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Microbeam analysis — Electron backscatter diffraction — Quantitative determination of austenite in steel

Analyse par microfaisceaux — Diffraction d'électrons rétrodiffusés — Détermination quantitative de l'austénite dans l'acier

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Foreword

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This document was prepared by Technical Committee 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

This test method produces a total quantification result of austenite, separate quantification results of austenite with different aspect ratio, as well as morphology and distribution of austenite. The retained austenite in steel, such as transformation induced plasticity (TRIP), twinning induced plasticity (TWIP), quenching and partitioning (Q&P) steel, can give steel good plasticity due to its TRIP effect, which is that high plasticity can be induced during deformation by the transformation of austenite into martensite. The TRIP effect is greatly affected by the content and stability of austenite. The stability of austenite can be divided into chemical stability and mechanical stability. The chemical stability is mainly influenced by the carbon content of the austenite. The mechanical stability is primarily affected by the morphology, aspect ratio, distribution in the matrix of austenite. It is important to understand the effect of austenite on the mechanical properties through the quantitative results of electron backscatter diffraction (EBSD).

In a conventional scanning electron microscope (SEM) with a tungsten filament, a spatial resolution of about 0,25 µm for EBSD can be achieved; however, with a field-emission gun SEM (FEG-SEM), the resolution limit is 10 nm to 50 nm for EBSD, although the value is strongly dependent both on the instrument and the instrument operating parameters. See ISO 24173.

Microbeam analysis — Electron backscatter diffraction — Quantitative determination of austenite in steel

1 Scope

This document specifies procedures for quantitative analysis of austenite in steel using electron backscatter diffraction (EBSD). This document is mainly applied in low and medium carbon steels, low and medium carbon alloy steels.

This document is used to analyse austenite with grain size larger than 50 nm. This method is not used to quantify austenite with grain size smaller than 50 nm, which can significantly affect the accuracy of the analysis results.

NOTE 1   The size limit is strongly dependent both on the instrument and the instrument operating parameters.

NOTE 2   The size limit is the minimum grain size of the detectable austenite.

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 13067, *Microbeam analysis — Electron backscatter diffraction — Measurement of average grain size*

ISO 22309, *Microbeam analysis — Quantitative analysis using energy-dispersive spectrometry (EDS) for elements with an atomic number of 11 (Na) or above*

ISO 22493, *Microbeam analysis — Scanning electron microscopy — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13067, ISO 22493, ISO 24173 and the following 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
equivalent circle diameter
ECD
diameter of the circle with an area equivalent to the grain section area

3.2
aspect ratio
ratio of the length of the minor axis to the length of the major axis of an ellipse fitted round a grain

Note 1 to entry: It is sometimes referred to as grain elongation.

Note 2 to entry: The value lies in the range 0 to 1.

Note 3 to entry: There are several ways of fitting the ellipse round the grain. Different fitting methods can result in small difference in the measured aspect ratio.

3.3
pattern quality
sharpness of a diffraction band or the contrast range of a diffraction pattern

Note 1 to entry: Different terms, such as band contrast, band slope, image quality, and so on, are used in different commercial software packages.

3.4
indexing reliability
numerical value that indicates the confidence/reliability that the indexing software places in an automatic analysis

Note 1 to entry: This parameter varies between EBSD manufacturers, but can include:

a)   the average difference between the bands experimentally determined and the bands calculated from the orientations 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.

3.5
non-indexing
non-assignment of an orientation, e.g. due to insufficient *pattern quality* (3.3) of the diffraction pattern

Note 1 to entry: 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 the pattern is from an unanticipated phase.

3.6
misindexing
assigning an incorrect orientation or phase to the measured EBSD pattern, or dissatisfying the required *indexing reliability* (3.4)

Note 1 to entry: 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.

3.7
hit rate
percentage of electron backscatter diffraction patterns (EBSPs) that have been reliably indexed

Note 1 to entry: Both the *misindexing* (3.6) and *non-indexing* (3.5) data are considered to be unreliably indexed.

3.8
data cleaning
process chosen to accommodate *non-indexing* (3.5) and *misindexing* (3.6) data within the map, using a given set of parameters, typically based on the characteristics (orientation, phase) of a certain number of nearest neighbours

Note 1 to entry: 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 Specimen preparation

4.1 Sampling

If the order, or the International Standard defining the product, does not specify the number of specimens and the location at which they are to be taken from the product, these are left to the manufacturer. It is recommended that two or more sections are assessed. Care shall be taken to ensure that the specimens are representative of the bulk of the product.

4.2 Preparation

In order to achieve a high degree of indexing of individual pixels, it is necessary to produce a surface polish 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. 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. The surface of specimens used for EBSD test should be free from deformation due to specimen preparation and flat. Poor deformation can leave at, or just below, the surface, or can leave contaminants, oxides, reaction product layer on the specimen surface. Due to the high tilt of the specimen surface (typically 70°) with respect to the electron beam, minimizing surface relief is also important part of good specimen preparation. Guideline on specimen preparation for EBSD should refer to standard texts on polishing and etching and ISO 24173. In general, electro- polishing is recommended. Ion milling is not recommended for the preparation of austenite specimen because austenite is easy to transform into martensite during this process.

5 Procedure

5.1 Stage positioning and calibration

The procedures for stage positioning and calibration 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 polished surface of the specimen should be tilted 70° towards the EBSD detector. The good conductivity of the specimen shall be ensured in accordance with ISO 22309. Carbon coating is not suitable for this application.

5.2 Setting of test parameters

5.2.1 The setting of acceleration voltage, beam current, spot size, measurement time and EBSP signal background shall be in accordance with ISO 24173. However, with increasing the acceleration voltage and beam current, the pattern quality increases, meanwhile the diameter of the electron beam increases and the resolution decreases. Therefore, on the premise of satisfying the quality of EBSP, the lower acceleration voltage and beam current are recommended. Generally, the acceleration voltage for iron and steel specimens should be not less than 10 kV.

5.2.2 The scanning step size shall not exceed 1/5 of the average grain size of austenite. The average grain size of austenite can be roughly estimated by using EBSD, which is to conduct a quick scan on one field to estimate the average grain size according to ISO 13067.

5.2.3 A minimum of 6 fields should be scanned under 1 000× magnification. More fields are needed to ensure the statistics when the magnification is increased. The total scanning area should be more than 0,065 mm^2.

NOTE 1 More fields might be needed to decrease the %RA value if the %RA is higher than 30 %. The calculation of the %RA can be seen in 6.3.3.

NOTE 2   The derivative process of the minimum request of the total scanning area is in Annex A.

5.3   EBSD data acquisition

5.3.1   Select body-centered cubic (BCC) and face-centered cubic (FCC) iron phases for indexing and start scanning, the EBSPs can be stored for later re-analysis if necessary.

5.3.2   Define the critical value of the indexing reliability. The critical value should be not more than 1 degree (≤1°) in the system using mean angle difference (MAD) as the critical value of the indexing reliability. The critical value should be more than 0,1 (>0,1) in the system using confidence index (CI) as the critical value of the indexing reliability. Then find the misindexing data and set the data non-indexing. The critical value shall be specified in the report.

5.3.3   The hit rate shall be not less than 85 % for martensite-containing microstructure, and 90 % for martensite-free microstructure, respectively. The higher the hit rate is, the higher the probability of correct austenite quantification is. In order to improve the measurement precision, the appropriate specimen preparation method should be chosen to obtain as high as possible of the hit rate.

6   Data calculation

6.1   Data processing

6.1.1   Define grain boundaries. The misorientation angle that is used to define the grain boundary shall be 15°.

6.1.2   Clear small grains. The grains, in which the number of data points is less than a critical value, shall be removed and set the data non-indexing. The critical value shall be defined by user (typically 3 to 5 for a square grid, and 3 to 4 for a hexagonal grid), or be determined by consultation with the customer.

6.1.3   Data cleaning. Assign every non-indexing with the phase and average orientation of its certain number of neighbours. The number of neighbours shall be defined by user (typically 5 to 6 for a square grid, and 3 to 4 for a hexagonal grid), or be determined by consultation with the customer.

NOTE   In a square grid, if a group of non-indexing data contains 5 pixels, the minimal number of neighbours used for data cleaning with an iterative operation is 5, and for 3 pixels the minimal number is 6. In a hexagonal grid, if a group of non-indexing data contains 4 pixels, the minimal number of neighbours used for data cleaning with an iterative operation is 3, and for 3 pixels the minimal number is 4. If the noise reduction level is lower than those minimal number, the artificial errors will increase.

6.2   Calculation of the content of austenite

For each field the content of austenite is the data percentage of FCC phase, V_{A_i} (i = 1,~N). The average value of austenite in this sample can be obtained through Formula (1).

$$\overline{V_A} = \frac{1}{N}\sum_1^N V_{A_i} \tag{1}$$

where

$\overline{V_A}$ is the average value of the austenite content measurements for N fields, %;

V_{A_i} is the individual field measurements of austenite content, %;

N is the number of measurement fields.

6.3 Calculation of 95 % confidence interval and relative accuracy

6.3.1 General

In this document, the standard deviation, 95 % confidence interval (95 % *CI*) and relative accuracy (*%RA*) are employed to characterize the measurement error of austenite content, if the number of measurement field is more than 1.

6.3.2 Calculation of standard deviation

Calculate the standard deviations S of the austenite content measurements for N fields, according to Formula (2):

$$S = \left[\frac{1}{N-1} \sum_{i=1}^{N} \left(V_{A_i} - \overline{V_A} \right)^2 \right]^{\frac{1}{2}} \tag{2}$$

where

S is the standard deviation, %;

$\overline{V_A}$ is the average value of the austenite content measurements for N fields, %;

V_{A_i} is the individual field measurements of austenite content, %;

N is the number of measurement fields.

6.3.3 Calculation of 95 % confidence interval

Calculate the 95 % confidence interval for each measurement according to Formula (3):

$$95\,\%CI = \pm \frac{t \times S}{\sqrt{N}} \tag{3}$$

where

95 %*CI*   is 95 % confidence interval, %;

S is the standard deviation, %;

t is a coefficient related to the number of measurement fields and used in conjunction with the standard deviation of the measurements to determine the 95 % *CI*;

          t varies with the number of fields N (see Table 1);

N is the number of measurement fields.

Table 1 — t values for calculating 95 % confidence interval

N	t	N	t	N	t
1	—	11	2,228	21	2,086
2	12,706	12	2,201	22	2,080
3	4,303	13	2,179	23	2,074
4	3,182	14	2,160	24	2,069
5	2,776	15	2,145	25	2,064

Table 1 *(continued)*

N	*t*	*N*	*t*	*N*	*t*
6	2,571	16	2,131	26	2,060
7	2,447	17	2,120	27	2,056
8	2,365	18	2,110	28	2,052
9	2,306	19	2,101	29	2,048
10	2,262	20	2,093	30	2,045

6.3.4   Calculation of relative accuracy

Calculate the relative accuracy of each measurement according to Formula (4):

$$\%RA = \frac{95\,\%CI}{\overline{V_A}} \times 100\,\% \tag{4}$$

where:

$\%RA$ relative accuracy, %;

$95\,\%CI$ 95 % confidence interval, %;

$\overline{V_A}$ the average value of the austenite content measurements for *N* fields.

The relative accuracy is an estimate of the error of each measurement as influenced by the field-to-field variability of the values. A relative accuracy of 30 % or less is generally adequate. If the $\%RA$ is substantially higher, additional fields may be made to improve the $\%RA$ value.

6.3.5   Expression of austenite content

The content of austenite in the specimen is expressed as shown in Formula (5):

$$V_A = \overline{V_A} \pm 95\,\%CI \tag{5}$$

where:

V_A is the austenite content in the specimen, %;

$95\,\%CI$ is 95 % confidence interval, %;

$\overline{V_A}$ is the average value of the austenite content measurements for *N* fields, %.

6.4   Analysis of grain size and shape of austenite

6.4.1   Analyse the grain size of austenite according to the area and the diameter of an equal area circle.

6.4.2   Divide the aspect ratio (*r*) into several intervals and analyse the content and distribution of austenite with different aspect ratio.

6.5   Example

An example of quantitative analysis of austenite in TRIP 590 steel is given in Annex B.

7 Precision and bias

There are no standards that can be used to rigorously define the precision of austenite content measurements and detect bias. The uncertainty of analysing the austenite content is associated with the typical factors as specimen preparation, calibration of the instrument, test conditions, hit rate, the number of scanning fields, data processing parameters, etc.

8 Test report

The test report shall contain the following information:

a) the International Standard used (including its year of publication);

b) identification of the specimen, heat, lot, etc.; the number and the location of the samples taken from the test piece;

c) the test and data processing parameters, such as the number of fields, area of each field, total scanning area, hit rate (or the percentage of non-indexing), step size, data cleaning parameter, small grain criteria and so on. If necessary, the typical morphology of grain orientation can be provided;

d) the result(s), such as the austenite content, 95 % confidence interval, relative accuracy. If necessary, the content and distribution of the austenite with different aspect ratio, r, can be provided;

e) any deviations from the procedure;

f) any unusual features observed;

g) the date of the test.

Annex A
(informative)

Determination of the total scanning area in TRIP 590

A.1   Randomly select three separate fields (Magnification: 500×) from the testing surface. The relative positions of the three fields on the testing surface are shown in Figure A.1. The scanning area of each field is 450 00 μm^2. The austenite content of each field was determined, which are 3,99 %, 3,69 % and 3,55 %, respectively. The average content is 3,74 %. The total scanning area is 0,135 mm^2, which is large enough to take the average content 3,74 % as the true value of the testing surface. The phase distribution maps of three fields is shown in Figure A.2.
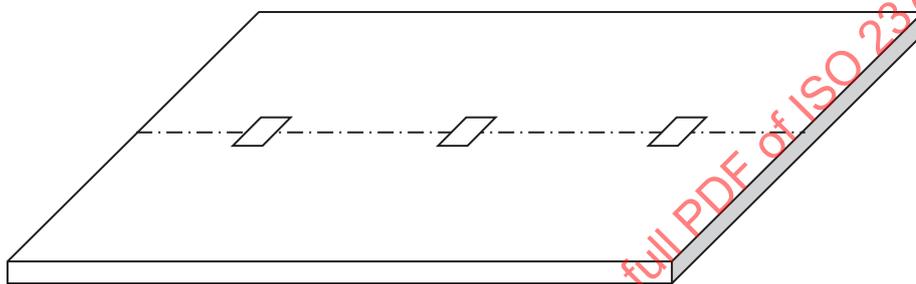

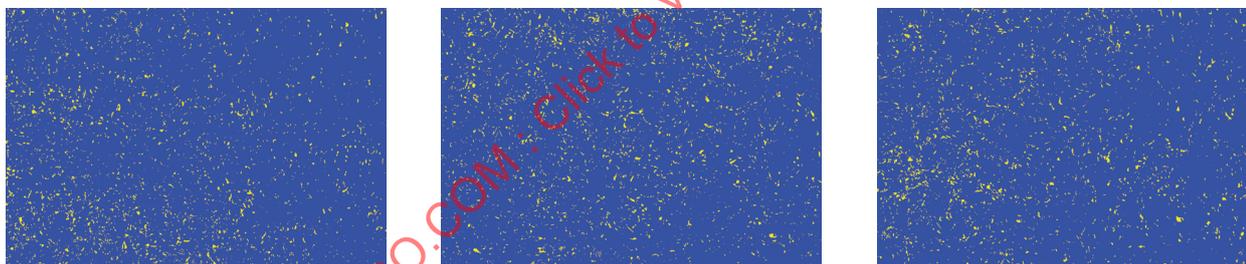**Figure A.1 — Schematic diagram of three fields on the test surface**

Figure A.2 — EBSD phase distribution maps of retained austenite in TRIP steel (three 500× fields)

A.2 Each 500× field can be divided into 16 fields in 2 000× magnification, 9 fields in 1 500× magnification, or 4 fields in 1 000× magnification, as shown in Figure A.3. The austenite content of every divided field can be obtained from the testing results of the first three 500× fields.

Key

a   1 000×(4 fields of view)

b   1 500×(9 fields of view)

c   2 000×(16 fields of view)

Figure A.3 — Schematic diagram of division of 500× field

A.3 Then randomly select two or three 1 000× fields from each 500× field to group into six or nine 1 000× fields in total. As well we can randomly select two, three, four or more 1 500× or 2 000× fields from each 500× field to group into six, nine, twelve or more 1 500× or 2 000× fields in total. The total scanning area, the average austenite content, 95 %CI, and the %RA can be calculated from the above each group of testing data, as can be seen in Table A.1. The relationship of total scanning area and %RA is drawn in Figure A.4. As can be seen from Figure A.4, when the total scanning area reaches around 650 00 μm^2, %RA tends to reach a stable value.

Table A.1 — Testing results of different number of fields

Magnifi-cation	Number of fields n	Total scanning area μm^2	Average V_{Ar}	95 %CI	%RA
1 000×	6	676 50	3,7	1,1	29,3
	9	101 475	3,8	0,7	19,1
1 500×	6	300 66	2,5	0,8	31,5
	9	450 99	3,5	1,0	28,9
	12	601 32	3,8	0,7	17,6
	15	751 65	3,8	0,7	19,2
	18	901 98	3,8	0,7	19,1
	21	105 231	3,9	0,6	16,1
	24	120 264	3,6	0,5	13,7

Table A.1 *(continued)*

Magnifi-cation	Number of fields	Total scanning area	Average	95 %CI	%RA
	n	μm²	V_{Ar}		
	6	169 14	4,1	2,4	59,5
	9	253 71	3,3	1,2	37,1
	12	338 28	3,4	0,8	24,6
2 000×	15	422 85	4,1	0,9	20,5
	18	507 42	3,7	0,4	10,5
	21	591 99	3,5	0,6	15,8
	24	676 56	3,7	0,7	18,4
	27	761 13	3,8	0,7	17,65
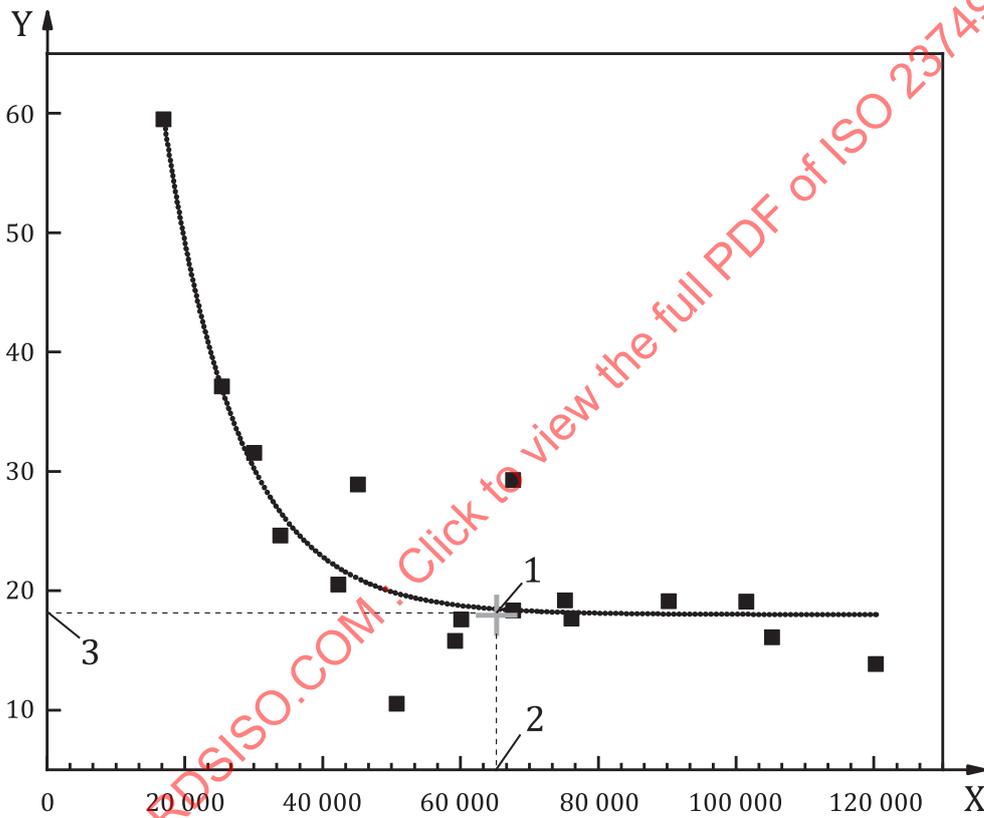
Key

X total scanning area, μm²

Y %RA

1 turning point

2 corresponding X value of the turning point, 651 48 μm²

3 corresponding Y value of the turning point, 18

Figure A.4 — Relationship between the total scanning area and %RA

Annex B
(informative)

Example of quantitative analysis of austenite in TRIP 590 steel

B.1 Estimation of the austenite grain size

Set the acquiring parameters as follows: accelerating voltage 15 kV, step size 0,15 μm, analysis area 64×44 μm² (Magnification: 2 000×), and then acquire an EBSD map. The morphology and distribution of austenite is shown in Figure B.1. In Figure B.1 the blue phase is austenite and grey phase is ferrite. From Figure B.1, it can be obtained that the average grain size of austenite is 0,693 μm.

= 20 μm; BC+Pblue; Step=0.15 μm; Grid432x297
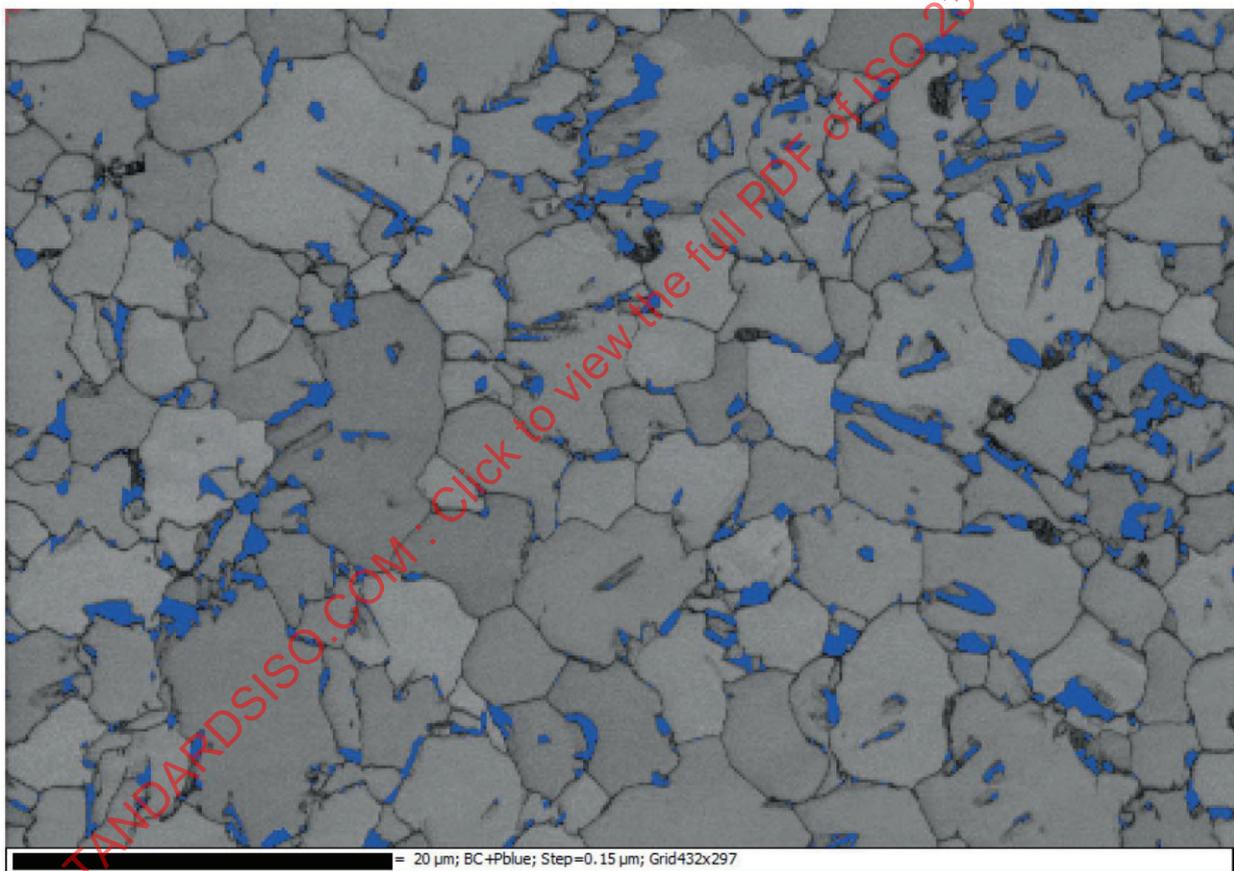
NOTE   The TRIP 590 sample was finally electrolytically polished using 10 % to 20 % perchloric acid ethanol electrolyte.

Figure B.1 — Morphology and distribution of austenite in TRIP 590 steel under step size 0,15 μm and Magnification: 2 000×

B.2 Selection of step size

According to 5.2.2, the suitable step size should not exceed 1/5 of the average grain size of austenite (0,693 μm ÷ 5 = 0,139 μm). Therefore, a step size of 0,1 μm is selected for the following austenite analysis in TRIP 590 steel.