International Standard

ISO 643

Fifth edition
2024-08

# Steels — Micrographic determination of the apparent grain size

*Aciers — Détermination micrographique de la grosseur de grain apparente*



Reference number
ISO 643:2024(en)



ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: 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.

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 document 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).

ISO draws attention to the possibility that the implementation of this document may involve the use of (a) patent(s). ISO takes no position concerning the evidence, validity or applicability of any claimed patent rights in respect thereof. As of the date of publication of this document, ISO had not received notice of (a) patent(s) which may be required to implement this document. However, implementers are cautioned that this may not represent the latest information, which may be obtained from the patent database available at www.iso.org/patents. ISO shall not be held responsible for identifying any or all such patent rights.

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*, in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459, *ECISS - European Committee for Iron and Steel Standardization*, in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 643:2019), which has been technically revised.

The main changes are as follows:

— the test temperature of McQuaid-Ehn method has been modified for case hardening steels to 950 °C (see A.4);

— subclause 7.2 has been modified with reference to new Annex B and amended Table 2;

— Annex B from the third edition (ISO 643:2012) has been reinstated, now with new ISO grain size charts instead of ASTM charts;

— parts of the old Annex B (evaluation method) have been revised and moved to the main body of the standard (subclause 7.3) and the remainder of the annex has been renumbered as Annex C;

— new Annexes D and E have been added.

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.

# Steels — Micrographic determination of the apparent grain size

**WARNING — This document calls for the use of substances and/or procedures that may be injurious to health if adequate safety measures are not taken. This document does not address any health hazards, safety or environmental matters associated with its use. It is the responsibility of the user of this document to establish appropriate health, safety and environmentally acceptable practices.**

## 1 Scope

This document specifies micrographic methods of determining apparent ferritic or austenitic grain size in steels. It describes the methods of revealing grain boundaries and of estimating the mean grain size of specimens with unimodal size distribution. Although grains are three-dimensional in shape, the metallographic sectioning plane can cut through a grain at any point from a grain corner, to the maximum diameter of the grain, thus producing a range of apparent grain sizes on the two-dimensional plane, even in a sample with a perfectly consistent grain size.

## 2 Normative references

There are no normative references in this document.

## 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 Grains

**3.1.1**
**grain**
closed polygonal shape with more or less curved sides, which can be revealed on a flat section through the sample, polished and prepared for micrographic examination

Note 1 to entry: In ISO 4885[1] grain is defined as “space lattice formed by atoms with regular interstices".

Note 2 to entry: If any other constituent (e.g. pearlite) of similar dimensions to the grains of interest is present, that constituent can be counted as grains of interest.

**3.1.2**
**austenitic grain**
crystal with a face-centred cubic crystal structure which may, or may not, contain annealing twins

**3.1.3**
**ferritic grain**
crystal with a body-centred cubic crystal structure which never contains annealing twins

## 3.2 General

**3.2.1**
**index**
positive, zero or possibly negative number $G$ which is derived from the mean number $m$ of *grains* (3.1.1) counted in an area of 1 mm$^2$ of the section of the specimen

Note 1 to entry: By definition, $G = 1$ where $m = 16$; the other indices are obtained by Formula (1).

**3.2.2**
**intercept**
$N$
number of *grains* (3.1.1) intercepted by a test line, either straight or curved

Note 1 to entry: See Figure 1.

Note 2 to entry: Straight test lines will normally end within a grain. These end segments are counted as 1/2 an intercept. $\bar{N}$ is the average of a number of counts of the number of grains intercepted by the test line applied randomly at various locations. $\bar{N}$ is divided by the true line length, $L_T$ usually measured in millimetres, in order to obtain the number of grains intercepted per unit length, $\bar{N}_L$.

**3.2.3**
**intersection**
$P$
number of intersection points between *grain* (3.1.1) boundaries and a test line, either straight or curved

Note 1 to entry: See Figure 2.

Note 2 to entry: $\bar{P}$ is the average of a number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations. $\bar{P}$ is divided by the true line length, $L_T$ usually measured in millimetres, in order to obtain the number of grain boundary intersections per unit length, $\bar{P}_L$.

## 4 Symbols

The symbols used are given in Table 1.

**Table 1 — Symbols**

| Symbols | Definition | Value |
|---|---|---|
| $\bar{a}$ | Mean area of grain in square millimetres | $\bar{a} = \frac{1}{m}$ |
| $A_B$ | True area of the test box | mm$^2$ |
| $A_C$ | True area of the test circle | mm$^2$ |
| $A_F$ | Apparent area of the test figure in square millimetres | — |
| $\bar{d}$ | Mean grain diameter in millimetres | $\bar{d} = \frac{1}{\sqrt{m}}$ |
| $D$ | Diameter of the circle on the ground glass screen of the microscope or on a photomicrograph enclosing the image of the reference surface of the specimen | 79,8 mm (area = 5 000 mm$^2$) |
| $g$ | Linear magnification (to be noted as a reference) of the microscopic image | In principle 100 |
| $G$ | Equivalent index of grain size | $G = \log_2 m - 3$ |
| $l$ | Mean lineal intercept length, generally expressed in millimetres | $l = 1 / \bar{N}_L = 1 / \bar{P}_L$ |
| $l_0$ | Mean lineal intercept length for $G = 0$, in millimetres | 0,32 |
| $L_T$ | True length of the test line divided by the magnification, in millimetres | — |
| [a] The method for designating the direction conforms to ISO 3785[2]. | | |

**Table 1** *(continued)*

| Symbols | Definition | Value |
|---|---|---|
| $m$ | Number of grains per square millimetre of specimen surface in the area examined | $m = n_t/A_C$<br>$m = n_t/A_B$ |
| $M$ | Number of the closest standard chart picture where $g$ is not 100 | — |
| $n_e$ | Number of grains completely inside the circle of diameter $D$ | — |
| $n_i$ | Number of grains intersected by the circle of diameter $D$ | — |
| $n_t$ | Total equivalent number of grains examined on the image of diameter $D$ | — |
| $\bar{N}$ | Mean number of grains intercepted per unit length $L$ | — |
| $\bar{N}_L$ | Mean number of grains intercepted per unit length of the line | $\overline{N_L} = \bar{N} / L_T$ |
| $N_x$ | Number of intercepts per millimetre in the longitudinal direction[a] | — |
| $N_y$ | Number of intercepts per millimetre in the transverse direction[a] | — |
| $N_z$ | Number of intercepts per millimetre in the perpendicular direction[a] | |
| $\bar{P}$ | Mean number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations | — |
| $\bar{P}_L$ | Mean number of grain boundary intersections per unit length of test line | $\overline{P_L} = \bar{P} / L_T$ |
| $Q$ | Correction factor for non-standard magnification | $Q = 2log_2\left(\frac{g}{100}\right)$ |

[a] The method for designating the direction conforms to ISO 3785[2].

## 5 Principle

This document is applicable to grain structures that have a unimodal size distribution. The apparent grain size is determined by micrographic examination of appropriately prepared sections of the specimen.

The following principal methods are available to obtain an index representing the mean value of the grain size:

a) comparison method using standard charts (see 7.2);

b) planimetric method counting grains to determine the mean number of grains per unit area, (see 7.3);

c) intercept method counting the number of grains or grain boundaries along a line of a known length (see 7.4).

All methods give comparable results.

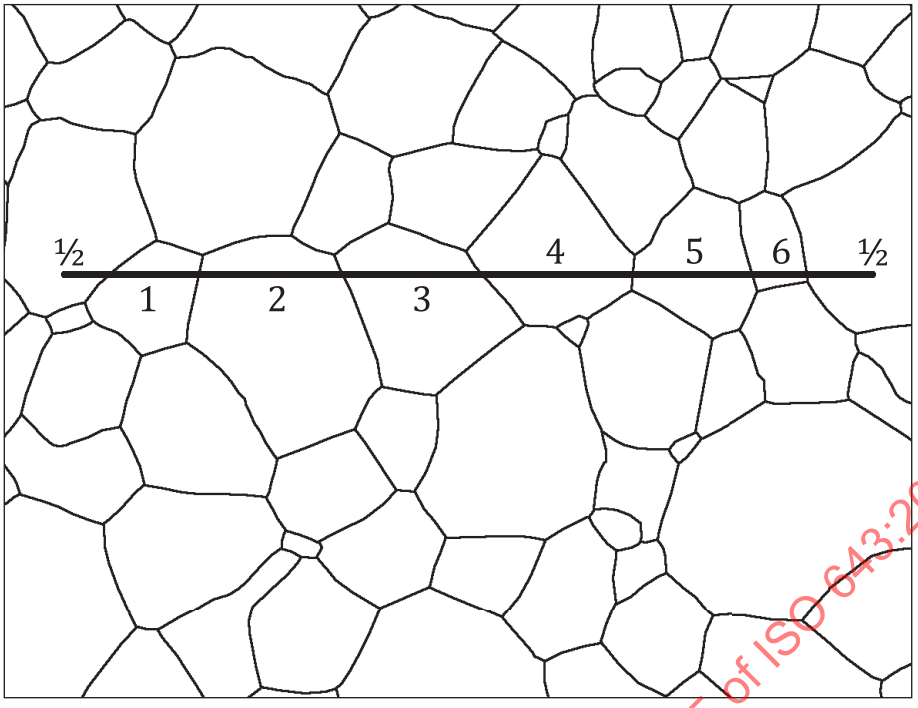


**Figure 1 — Example of intercept, *N***

Intercept, *N*, grain counts for a straight line on a single-phase grain structure. Six intercepts and two line segments ending within a grain equals 2 × 1/2 + 6 = 7.

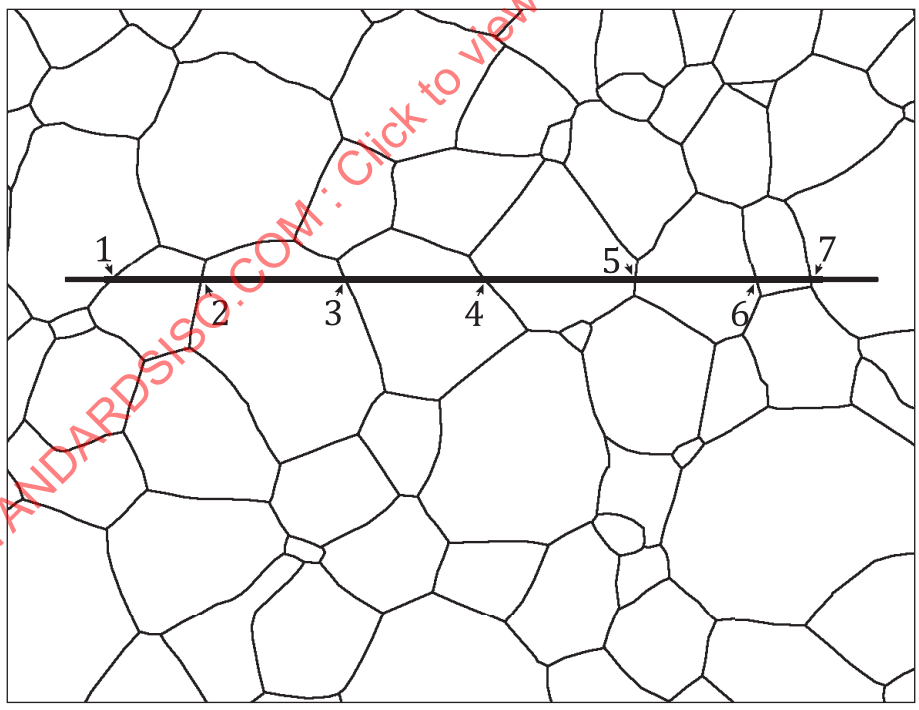


**Figure 2 — Example of intersection, *P***

Intersection, *P*, counts for a straight test line placed over a single-phase grain structure where the arrows point to 7 intersection points and *P* = 7.

# 6 Selection and preparation of the specimen

## 6.1 Test location

If the order, or the standard defining the product, does not specify the number of specimens and the point at which they are to be taken from the product, these are left to the manufacturer, although it has been shown that precision of grain size determination increases the higher the number of specimens assessed. Care shall be taken to ensure that the specimens are representative of the bulk of the product (i.e. avoid heavily deformed material such as that found at the extreme end of certain products or where shearing has been used to remove the specimen, etc.). The specimens shall be polished in accordance with the usual methods.

Unless otherwise stated by the product standard or by agreement with the customer, the polished surface can be randomly selected for the specimens with equiaxial grains. The polished surface shall be parallel to the principal axis of deformation in wrought products, for the specimens with deformed grains.

NOTE Measurements of the grain size on a transverse plane will be biased if the grain shape is not equiaxial.

## 6.2 Revealing ferritic grain boundaries

The ferritic grains shall be revealed by etching with nital [ethanolic 2 % to 3 % (by volume) nitric acid solution], or with another appropriate reagent.

## 6.3 Revealing austenitic and prior-austenitic grain boundaries

### 6.3.1 General

In the case of steels having a single-phase or dual-phase mainly austenitic structure (delta ferrite grains in an austenitic matrix) at ambient temperature, the grains shall be revealed by an etching solution. For single phase austenitic stainless steels, the most commonly used chemical etchants are glyceregia, Kalling's reagent (No. 2) and Marble's reagent. The best electrolytic etch for single or two-phase stainless steels is aqueous 60 % nitric acid at 1,4 V d.c. for 60 s to 120 s, as it reveals the grain boundaries but not the twin boundaries. Aqueous 10 % oxalic acid, 6 V d.c., up to 60 s, is commonly used but is less effective than electrolytic 60 % nitric acid.

For other steels, one or other of the methods specified below shall be used depending on the information required:

— "Bechet-Beaujard" method by etching with aqueous saturated picric acid solution (see A.2);

— "Kohn" method by controlled oxidation (see A.3);

— "McQuaid-Ehn" method by carburization (see A.4);

— grain boundary sensitization method (see A.7);

— other methods specially agreed upon when ordering.

NOTE The first three methods are for prior-austenitic grain boundaries while the others are for austenitic Mn or austenitic stainless, see Annex A.

If comparative tests are carried out for the different methods, it is essential to use the same heat treatment conditions. Results may vary considerably from one method to the other.

# 7 Characterization of grain size

## 7.1 General

### 7.1.1 Characterization methods

The apparent grain size can be determined by three micrographic methods: comparison method, planimetric method and intercept method.

### 7.1.2 Formulae

The index is defined by Formula (1):

$$m = 8 \times 2^{G} \tag{1}$$

This formula may be stated as Formula (2):

$$G = \log_2 m - 3 \tag{2}$$

NOTE An alternative system of grain size definition is known as the ASTM grain size (see C.2).

### 7.1.3 Accuracy of the methods

In general, the comparison method allows for an accuracy of 0,5; the planimetric and intercept segment methods allow for an accuracy of 0,1, see Reference [3]. For comparison between methods, the indexes obtained are usually rounded to multiples of 0,5.

Due to the randomness of the spatial position in which each grain is cut through by the sectioning plane and due to the measurement error, no determination of apparent grain size can be an exact measurement. Therefore, for planimetric and intercept methods it can be of interest to calculate the 95 % confidence interval of the grain size measurement result and adjust the number of fields inspected according to the percentage relative accuracy, % RA, of counting corresponding to the uncertainty of ±0,25 grain size units, taking into account that for a symmetric error of $G$ the % RA of the measured quantity is not symmetric, see Annex D.

The methods described in this document yield representative results for specimens with a unimodal grain size distribution. Applying them to specimens with bimodal (or more complex) size distributions will yield an average value that likely has no meaningful relationship with the various grain populations but may still represent the specimen on average. ISO 14250[4] may be the more appropriate standard for characterizing these specimens, see Annex E.

## 7.2 Comparison method

**7.2.1** The image examined on the screen (or on a photomicrograph) shall be compared with a series of standard charts presented in Annex B or overlays (using eye-piece graticules designed for grain size measurement can be used provided these are traceable to national or international standards). The standard charts at a magnification of 100:1 are numbered from -1 to 10 so that their number is equal to the index $G$. Images for grain sizes -1 to 3 are included in the chart but when determining grain sizes in this range it is recommended for reasons of accuracy to reduce the operating magnification of the microscope, in combination with index conversion according to Table 2.

Using ASTM E112 charts gives substantially the same results as using the comparison charts of Annex B, see C.2.4.

**7.2.2** The standard chart with the grain size closest to that of the examined fields of the specimen can then be determined. A minimum of three randomly selected fields shall be assessed on each specimen.

**7.2.3** Where the magnification $g$ of the image on the screen or photomicrograph is not 100:1, the index $G$ shall be equal to the number $M$ of the closest standard chart, modified as a function of the ratio of the magnifications, as given by Formula (3):

$$G = M + 2log_2 \frac{g}{100} = M + 6,64lg \frac{g}{100} \quad (3)$$

Table 2 gives the relationship between the indices for the usual magnifications.

**Table 2 — Relationship between indices for the usual magnifications**

| Standard chart no. $M$ | | 1 | 1,5 | 2 | 2,5 | 3 | 3,5 | 4 | 4,5 | 5 | 5,5 | 6 | 6,5 | 7 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Magnification of the image $g$ | Q [a] | Grain size [b] | | | | | | | | | | | | | | | | | | |
| 25 | −4 | −3 | −2,5 | −2 | −1,5 | −1 | −0,5 | 0 | 0,5 | 1 | 1,5 | 2 | 2,5 | 3 | 3,5 | 4 | 4,5 | 5 | 5,5 | 6 |
| 50 | −2 | −1 | −0,5 | 0 | 0,5 | 1 | 1,5 | 2 | 2,5 | 3 | 3,5 | 4 | 4,5 | 5 | 5,5 | 6 | 6,5 | 7 | 7,5 | 8 |
| 100 | 0 | 1 | 1,5 | 2 | 2,5 | 3 | 3,5 | 4 | 4,5 | 5 | 5,5 | 6 | 6,5 | 7 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 |
| 200 | +2 | 3 | 3,5 | 4 | 4,5 | 5 | 5,5 | 6 | 6,5 | 7 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 | 10,5 | 11 | 11,5 | 12 |
| 400 | +4 | 5 | 5,5 | 6 | 6,5 | 7 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 | 10,5 | 11 | 11,5 | 12 | 12,5 | 13 | 13,5 | 14 |
| 500 | +4,5 | 5,5 | 6 | 6,5 | 7 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 | 10,5 | 11 | 11,5 | 12 | 12,5 | 13 | 13,5 | 14 | 14,5 |
| 800 | +6 | 7 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 | 10,5 | 11 | 11,5 | 12 | 12,5 | 13 | 13,5 | 14 | 14,5 | 15 | 15,5 | 16 |
| 1 000 | +6,5 | 7,5 | 8 | 8,5 | 9 | 9,5 | 10 | 10,5 | 11 | 11,5 | 12 | 12,5 | 13 | 13,5 | 14 | 14,5 | 15 | 15,5 | 16 | 16,5 |

[a] The values for $g = 500$ and $g = 1\,000$ are rounded to the nearest multiple of 0,5.

[b] EXAMPLE: At a magnification $g = 50$ the standard chart number $M = 3$ corresponds to a grain size of $G = 1$.

**7.2.5** For the comparison method, if the difference between the maximum index $G_{max}$ and the minimum index $G_{min}$ determined is less than three (e. g. range from $G$ = 6 to 8,5), compute the test result as the arithmetic mean of the found indices. If the indices are calculated using Formula (3), the arithmetic mean is to be calculated after the modification for non-standard magnification. If this condition is not fulfilled, the operator may perform an additional series of at least six determinations of the grain size. If the difference between the maximum index and the minimum index determined in this new series is less than three, then compute the test result as the arithmetic mean of all determinations of both the first and second series. If this latter condition is not fulfilled, note the spread and a comment on the findings in the final report. Alternatively, ISO 14250 should be considered. For a more elaborate discussion on specimens of non-unimodal distribution, see Annex E.

The calculated arithmetic mean of indices shall be rounded to the nearest multiple of 0,5.

## 7.3 Planimetric method

**7.3.1** Historically a circle measuring 79,8 mm in diameter ($A_F$ = 5 000 mm$^2$), see Figure 3, was drawn or superimposed over a micrograph or a live image on a ground glass projection screen. The magnification was then adjusted so that the circular area contained at least 50 grains in order to minimize the counting error associated with a circular test pattern. The following procedure and formulae are magnification neutral.

NOTE The circle referenced an apparent size specifically at 100:1 magnification as perceived by an operator at a microscope using ×10 oculars and ×10 objective. This reference also was, and still is, used in other applications such as inclusion assessments. It is moreover a component of the recommended concentric circle grid used in the intercept method, as well as one of the reference circles used in the comparison method.

However, more recent tools like image software allow for optimizing the combination of circle diameter and magnification to facilitate the count and to make sure that the number of grains within the circle reach at least 50. Therefore, it is no longer always relevant to reference a specific circle size at a specific magnification.

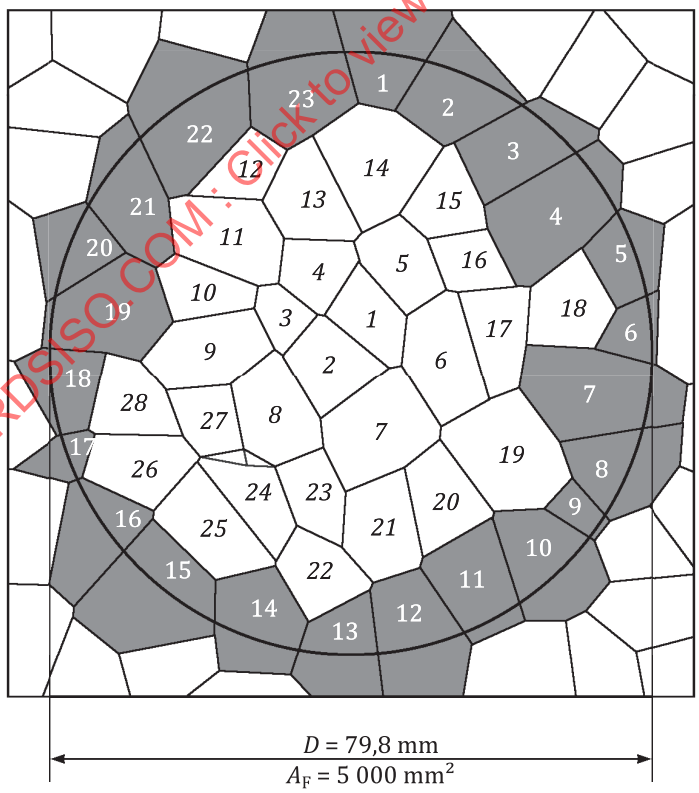


**Figure 3 — Evaluation of the number of grains in an area enclosed by a circle**

**7.3.2** Two counts are made; the number of grains completely enclosed within the test circle of any given size, $n_e$, and the number of grains intercepted by the test circle, $n_i$.

The total number of equivalent grains, $n_t$, is calculated using Formula (4):

$$n_t = n_e + \frac{n_i}{2} \tag{4}$$

The number of grains per mm$^2$, $m$, is calculated using Formula (5):

$$m = \frac{n_t}{A_C} \tag{5}$$

where $A_C$ is the true area of the circle.

**7.3.3** The planimetric approach assumes that on average, half of the grains on the test circle are within and half are outside test circle. This assumption is only fully valid along a straight line through a grain structure, but not for a curved line, including a circle. The bias created by this assumption increases as the number of grains inside the test circle decreases. If the number of grains within the test circle is at least 50, the bias becomes about 2 %.

**7.3.4** A simple way to avoid this bias, irrespective of the number of grains within the test figure, is to use a square or a rectangle (see Figure 4). However, the counting procedure then shall be modified.

First, it is assumed that the grains on each of the four corners are, on average, one fourth within the figure and three-fourths outside. These four corner grains together equal one grain within the test box. Ignoring the four corner grains, a count is made of the grains completely enclosed by the test box, $n_e$, and the grains intercepted by the four sides of the box, $n_i$, (see Figure 4). The total number of grains is calculated as in Formula (6):

$$n_t = n_e + \frac{n_i}{2} + 1 \tag{6}$$

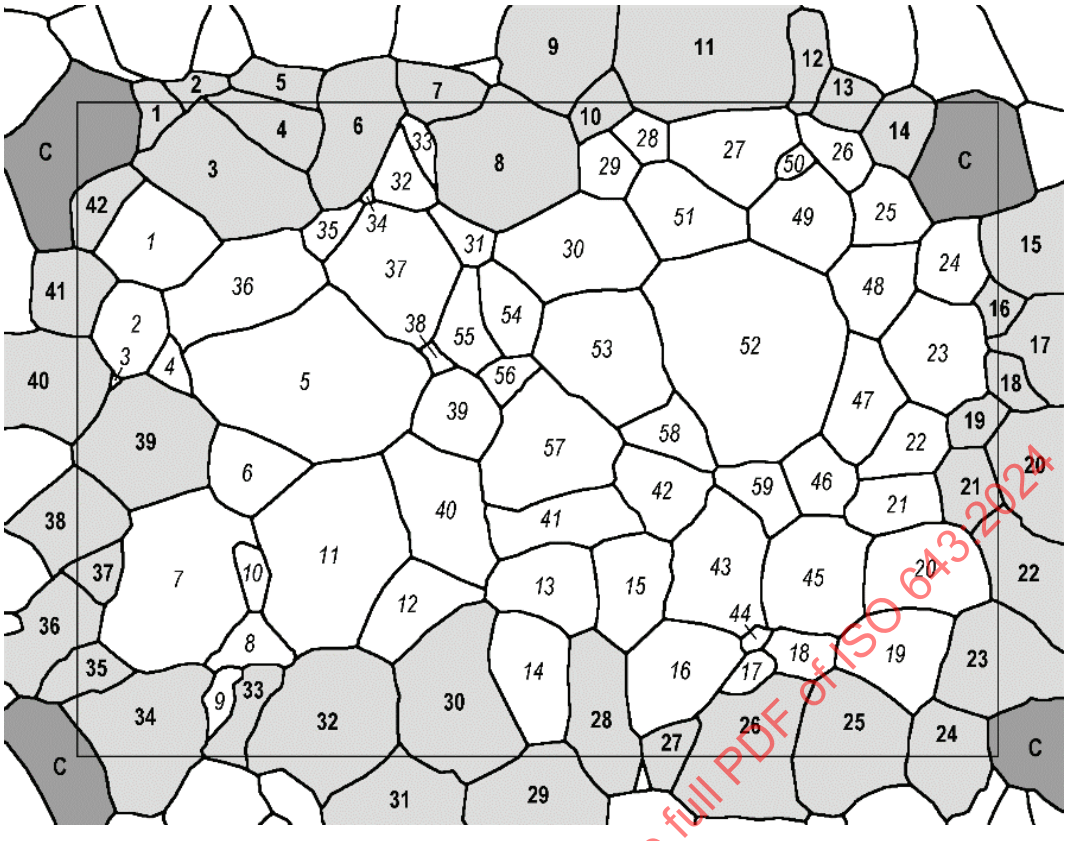

**Figure 4 — Evaluation of the number of grains in an area enclosed by a square or rectangle**

**7.3.5** The number of grains per mm$^2$, $m$, on the surface of the specimen is given by Formula (7):

$$m = \frac{n_t}{A_B} \tag{7}$$

where $A_B$ is the true area of the test box used for counting.

**7.3.6** Regardless of using a circle or a box, the mean grain area, $\bar{a}$, in mm$^2$ is calculated from Formula (8):

$$\bar{a} = \frac{1}{m} \tag{8}$$

**7.3.7** By assuming a uniform geometrical grain shape, there are several ways to calculate a mean grain "diameter". Assuming that all grains are square-shaped, the mean diameter, $\bar{d}$, can be calculated with Formula (9):

$$\bar{d} = \sqrt{\bar{a}} \tag{9}$$

This is the diameter listed in Table 3.

A mean equivalent circle diameter, $d_{EC}$, can be calculated with Formula (10):

$$d_{EC} = \sqrt{\frac{4\bar{a}}{\pi}} \tag{10}$$

NOTE The mean equivalent circle diameter is often referred to in literature as $\overline{ECD}$.

Assuming that all grains are hexagonal in a perfect honeycomb pattern, the mean vertex-to-vertex distance, $\overline{d_{\text{v-v}}}$, can be calculated with Formula (11):

$$\overline{d_{\text{v-v}}} = \sqrt{\frac{8 \cdot \bar{a}}{3\sqrt{3}}} \tag{11}$$

and the mean distance between parallel sides, $\overline{d_{\text{s-s}}}$, can be calculated with Formula (12):

$$\overline{d_{\text{s-s}}} = \sqrt{\frac{2 \cdot \bar{a}}{\sqrt{3}}} \tag{12}$$

**7.3.8** A nominal value of $m$ corresponds to each value of $G$. The values of $m$ calculated by Formula (5) or Formula (7) within the limits given in Table 3 are given in 0,5 steps of $G$.

**Table 3 — Evaluation of number of grains as a function of various parameters**

| Grain size indices $G$ | Number of grains, per square millimetre $m$ | | | Mean diameter of grain $\bar{d}$ | Mean area of grain $\bar{a}$ | Mean lineal intercept length $l$ or $\frac{1}{\bar{P}_{\text{L}}}$ | | | Mean number of intercepts on the measuring line, per millimetre or $\bar{N}_{\text{L}}$ |
|---|---|---|---|---|---|---|---|---|---|
| | Nominal value | Limit values | | | | Nominal value | Limit values | | |
| | | from (excl.) | to (incl.) | mm | $mm^2$ | mm | from (excl.) | to (incl.) | |
| -1 | 4 | 3,4 | 4,8 | 0,5 | 0,25 | 0,453 | 0,494 | 0,415 | 2,21 |
| -0,5 | 5,7 | 4,8 | 6,7 | 0,42 | 0,18 | 0,381 | 0,415 | 0,349 | 2,63 |
| 0 | 8 | 6,7 | 9,5 | 0,35 | 0,125 | 0,320 | 0,349 | 0,293 | 3,13 |
| 0,5 | 11,3 | 9,5 | 13,5 | 0,30 | 0,088 4 | 0,269 | 0,293 | 0,247 | 3,72 |
| 1 | 16 | 13,5 | 19,0 | 0,25 | 0,062 5 | 0,226 | 0,247 | 0,207 | 4,42 |
| 1,5 | 22,6 | 19,0 | 26,9 | 0,21 | 0,044 2 | 0,190 | 0,207 | 0,174 | 5,26 |
| 2 | 32 | 26,9 | 38,1 | 0,177 | 0,031 3 | 0,160 | 0,174 | 0,147 | 6,25 |
| 2,5 | 45,3 | 38,1 | 53,8 | 0,149 | 0,022 1 | 0,135 | 0,147 | 0,123 | 7,43 |
| 3 | 64 | 53,8 | 76,1 | 0,125 | 0,015 6 | 0,113 | 0,123 | 0,104 | 8,84 |
| 3,5 | 90,5 | 76,1 | 108 | 0,105 | 0,011 0 | 0,095 1 | 0,104 | 0,087 2 | 10,5 |
| 4 | 128 | 108 | 152 | 0,088 4 | 0,007 81 | 0,080 0 | 0,087 2 | 0,073 4 | 12,5 |
| 4,5 | 181 | 152 | 215 | 0,074 3 | 0,005 52 | 0,067 3 | 0,073 4 | 0,061 7 | 14,9 |
| 5 | 256 | 215 | 304 | 0,062 5 | 0,003 91 | 0,056 6 | 0,061 7 | 0,051 9 | 17,7 |
| 5,5 | 362 | 304 | 431 | 0,052 6 | 0,002 76 | 0,047 6 | 0,051 9 | 0,043 6 | 21,0 |
| 6 | 512 | 431 | 609 | 0,044 2 | 0,001 95 | 0,040 0 | 0,043 6 | 0,036 7 | 25,0 |
| 6,5 | 724 | 609 | 861 | 0,037 2 | 0,001 38 | 0,033 6 | 0,036 7 | 0,030 8 | 29,7 |
| 7 | 1 024 | 861 | 1 218 | 0,031 3 | 0,000 977 | 0,028 3 | 0,030 8 | 0,025 9 | 35,42 |
| 7,5 | 1 448 | 1 218 | 1 722 | 0,026 3 | 0,000 691 | 0,023 8 | 0,025 9 | 0,021 8 | 42,0 |
| 8 | 2 048 | 1 722 | 2 435 | 0,022 1 | 0,000 488 | 0,020 0 | 0,021 8 | 0,018 3 | 50,0 |
| 8,5 | 2 896 | 2 435 | 3 444 | 0,018 6 | 0,000 345 | 0,016 8 | 0,018 3 | 0,015 4 | 59,5 |
| 9 | 4 096 | 3 444 | 4 871 | 0,015 6 | 0,000 244 | 0,014 1 | 0,015 4 | 0,013 0 | 70,7 |
| 9,5 | 5 793 | 4 871 | 6 889 | 0,013 1 | 0,000 173 | 0,011 9 | 0,013 0 | 0,010 9 | 84,1 |
| 10 | 8 192 | 6 889 | 9 742 | 0,011 0 | 0,000 122 | 0,010 0 | 0,010 9 | 0,009 17 | 100 |
| 10,5 | 11 585 | 9 742 | 13 777 | 0,009 29 | 0,000 086 3 | 0,008 2 | 0,009 17 | 0,007 71 | 119 |
| 11 | 16 384 | 13 777 | 19 484 | 0,007 81 | 0,000 061 0 | 0,007 07 | 0,007 71 | 0,006 48 | 141 |
| 11,5 | 23 170 | 19 484 | 27 554 | 0,006 57 | 0,000 043 2 | 0,005 95 | 0,006 48 | 0,005 45 | 168 |
| 12 | 32 768 | 27 554 | 38 968 | 0,005 52 | 0,000 030 5 | 0,005 00 | 0,005 45 | 0,004 59 | 200 |
| 12,5 | 46 341 | 38 968 | 55 109 | 0,004 65 | 0,000 021 6 | 0,004 20 | 0,004 59 | 0,003 86 | 238 |

NOTE This table gives the values between the different parameters for equiaxed grains.

**Table 3** *(continued)*

| Grain size indices $G$ | Number of grains, per square millimetre $m$ | | | Mean diameter of grain $\bar{d}$ | Mean area of grain $\bar{a}$ | Mean lineal intercept length $l$ or $\frac{1}{\bar{P}_L}$ | | | Mean number of intercepts on the measuring line, per millimetre or $\bar{N}_L$ |
|---|---|---|---|---|---|---|---|---|---|
| | Nominal value | Limit values | | | | Nominal value | Limit values | | |
| | | from (excl.) | to (incl.) | mm | $mm^2$ | mm | from (excl.) | to (incl.) | |
| 13 | 65 536 | 55 109 | 77 936 | 0,003 91 | 0,000 015 3 | 0,003 54 | 0,003 86 | 0,003 24 | 283 |
| 13,5 | 92 682 | 77 936 | 110 218 | 0,003 28 | 0,000 010 8 | 0,002 97 | 0,003 24 | 0,002 73 | 336 |
| 14 | 131 072 | 110 218 | 155 872 | 0,002 76 | 0,000 007 63 | 0,002 50 | 0,002 73 | 0,002 29 | 400 |
| 14,5 | 185 364 | 155 872 | 220 436 | 0,002 32 | 0,000 005 39 | 0,002 10 | 0,002 29 | 0,001 93 | 476 |
| 15 | 262 144 | 220 436 | 311 744 | 0,001 95 | 0,000 003 81 | 0,001 77 | 0,001 93 | 0,001 62 | 566 |
| 15,5 | 370 728 | 311 744 | 440 872 | 0,001 64 | 0,000 002 70 | 0,001 49 | 0,001 62 | 0,001 36 | 673 |
| 16 | 524 288 | 440 872 | 623 487 | 0,001 38 | 0,000 001 91 | 0,001 25 | 0,001 36 | 0,001 15 | 800 |
| 16,5 | 741 455 | 623 487 | 881 744 | 0,001 16 | 0,000 001 35 | 0,001 05 | 0,001 15 | 0,000 964 | 951 |
| 17 | 1 048 576 | 881 744 | 1 246 974 | 0,000 977 | 0,000 000 954 | 0,000 884 | 0,000 964 | 0,000 811 | 1 131 |
| NOTE This table gives the values between the different parameters for equiaxed grains. | | | | | | | | | |

## 7.4 Intercept method

### 7.4.1 General

**7.4.1.1** Count the number of grains intercepted, $N$, or the number of grain boundary intersections, $P$, with a test line or a grid of test lines of known true length $L$. The count may be performed using a projection screen, a reticle, a video monitor or a photomicrograph of the specimen.

**7.4.1.2** The test line may be straight or circular. The test grid in Figure 5 shows the types of recommended test lines.

**7.4.1.3** The line or grid of lines shall be applied only once to the field examined. It is applied at random to an adequate number of fields to obtain a valid count for $N$ or $P$.

Figure 5 shows a test pattern that can be used to measure grain size by the intercept method, and which is scaled to be convenient for $g$ = 100. The three concentric circles have a total line length of 500 mm; their diameters are 79,58 mm, 53,05 mm and 26,53 mm. A circular test grid averages out variations in the shape of equiaxed grains and avoids the problem of lines ending within grains. Figure 5 also has four straight lines: two oriented diagonally, one vertically and one horizontally. Each diagonal line has a length of 150 mm while the horizontal and vertical lines are each 100 mm long. The straight lines will also average out variations in the shape of equiaxed grains. Alternatively, if the degree of grain elongation is of interest, grain counts can be made using only the vertical and horizontal lines (separately) when they are aligned so that one line is parallel to the deformation axis (and the other line is then perpendicular to the deformation axis) on a longitudinally-oriented polished plane [see 7.5, c)].

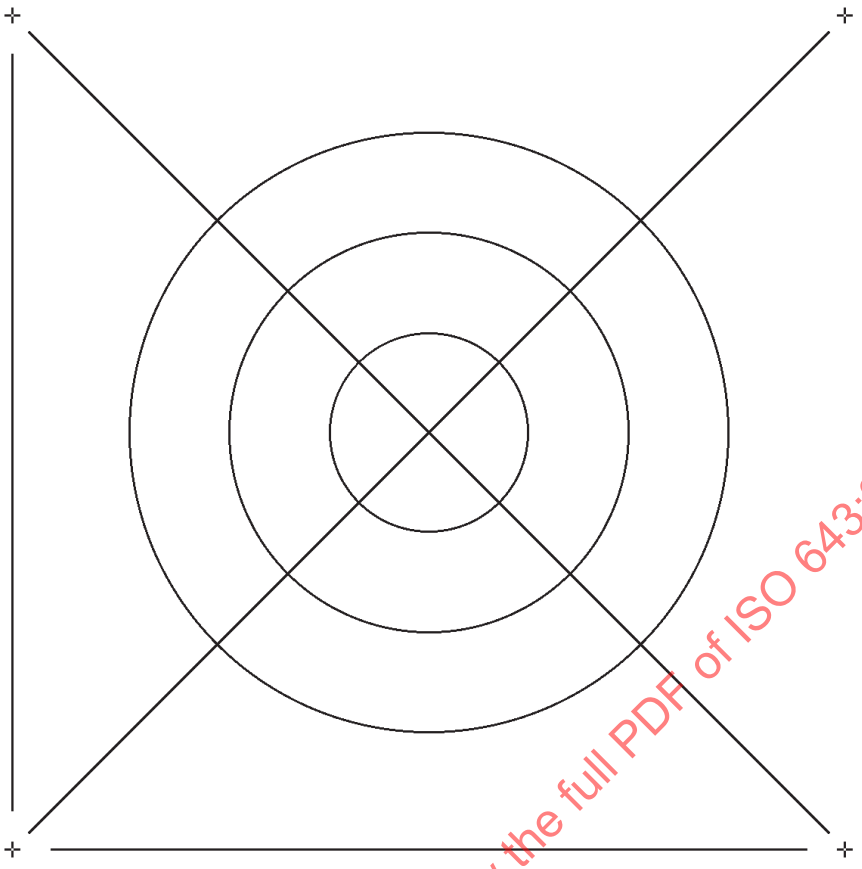

**Figure 5 — Recommended measurement grid for the intercept segment methods**



Straight lines indicate the linear intercept method (see 7.4.2) and circles indicate the circular intercept method (see 7.4.3). These methods should be used separately.

**7.4.1.5** The true total length of the grid of lines $L_T$ is the measured length divided by the magnification $g$. $L_T$ can also be calculated using a scale bar or a software calibration factor.

### 7.4.2 Linear intercept method

**7.4.2.1** The pattern of straight lines shown in Figure 5 is recommended. The magnification $g$ should be selected so that at least 50 intercepts are obtained in any one field. At least five randomly selected fields shall be assessed with a total number of intercepts of at least 250.

NOTE If the grain size of the specimen requires the magnification to be changed in order to achieve the required number of intercepts, the length of the measuring lines can also be varied providing that the orientation of the measuring lines is arranged to take account of the effects of anisotropy.

**7.4.2.2** The following rules apply to intercept and intersection counts of single-phase grain structures using straight test lines:

a) when the number of intercepted grains, $N$, is counted:

— if a test line goes through a grain, $N$ is 1;

— if a test line terminates within a grain, $N$ is 0,5;

— if a test line is tangential to a grain boundary, $N$ is 0,5.

b) when the number of grain boundary intersections, *P*, is counted:

— if a test line passes through a grain boundary, *P* is 1;

— if a test line is tangential to a grain boundary, *P* is 1;

— if a test line intersects a triple point, *P* is 1,5.

NOTE The "Snyder-Graff" method, described in C.1, Annex C, represents a linear intercept method for tool steel (high-speed steels).

### 7.4.3 Circular intercept method

**7.4.3.1** The pattern of circles shown in Figure 5 is recommended.

**7.4.3.2** The measuring line consists either of a set of three concentric circles as shown in Figure 5 or of one single circle.

**7.4.3.3** The magnification or diameter of the circle should be selected so that there are 40 to 50 intercepts when the measurement grid is superposed on the field to be examined. At least five randomly selected fields shall be assessed with a total number of intercepts of at least 250.

**7.4.3.4** In the case of a single circle, the largest circle is used. In this case, the magnification to be used should enable at least 25 intercepts to be counted. At least five randomly selected fields shall be assessed with a total number of intercepts of at least 250.

**7.4.3.5** The circular intercepted segment method tends to slightly overestimate intercepted values and thus slightly underestimate the number of intersections. In order to compensate for this, the intersections caused by a triple point shall be counted as 2 intersections instead of 1,5 as is the case with the linear intercept method.

### 7.4.4 Assessment of results

**7.4.4.1** Counts of the number of intercepts, *N*, or intersections, *P*, are made on a number of fields selected at random. The mean value of the number of intercepts, $\bar{N}$, or intersections, $\bar{P}$, is calculated. From this value, the mean number of intercepts per millimetre, $\bar{N}_L$, or the mean number of intersections per millimetre, $\bar{P}_L$, is calculated using Formulae (13) and (14):

$$\bar{N}_L = \bar{N} / L_T \tag{13}$$

$$\bar{P}_L = \bar{P} / L_T \tag{14}$$

**7.4.4.2** For the case of non-equiaxed grain structures**,** counts can be made of the number of intercepts, *N*, or intersections, *P*, with straight test lines oriented parallel to the three principal directions. These three directions can be found on any two of the three principal test planes (longitudinal, transverse and planar).

**7.4.4.3** The mean number of intercepts per millimetre, $\bar{N}_L$, or the mean number of intersections per millimetre, $\bar{P}_L$, is determined from the cube root of the product of the three measurements, according to Formulae (15) and (16):

$$\bar{N}_L = \left(\bar{N}_{Lx} \times \bar{N}_{Ly} \times \bar{N}_{Lz}\right)^{1/3} \tag{15}$$

$$\bar{P}_L = \left(\bar{P}_{Lx} \times \bar{P}_{Ly} \times \bar{P}_{Lz}\right)^{1/3} \quad (16)$$

where the bars above the quantities indicate that they are the arithmetic means of a number of measurements and x, y, and z indicate the principal directions (longitudinal, transverse and planar).

**7.4.4.4** Calculate the mean lineal intercept length $l$ from $\bar{N}_L$ or $\bar{P}_L$ as defined in Table 1.

**7.4.4.5** Calculate $G$ using Formula (17) or determine $G$ according to Table 3.

**7.4.4.6** Because there is no exact relation between $G$ and the mean lineal intercept length $l$ in millimetres, the relationship is defined to be as stated in Formula (17):

$$G = 2\log_2\left(\frac{l_0}{l}\right) \approx -3{,}288 - 2\log_2 l \quad (17)$$

**7.4.4.7** The relationship between the mean grain diameter shown in Table 3 and the mean lineal intercept length can be derived from Formula (17) using Formulae (8), (9), and (1) and is shown in Formula (18):

$$l = \bar{d}\sqrt{8}l_0 \quad (18)$$

## 7.5 Other methods

In special cases, other methods can be used, for example:

a) **Grains of different size indices:** in certain cases, the specimen examined may include grains belonging to two or more different systems of size indices. This can be recognized by the presence of several grains of greatly differing dimensions from those of the whole, e.g. see Annex E and ISO 14250.

b) **Twinned grains**: unless otherwise specified, these are counted as a single grain, that is, twin boundaries are ignored (see Figure 6).

c) **Non-equiaxed grains**: the grain shape can be expressed by dividing the mean lineal intercept length in the deformation direction by the mean lineal intercept length perpendicular to the deformation direction using a longitudinally oriented test specimen. This is referred to as the grain elongation ratio, or the anisotropy index.

d) **Modern methods of grain size measurement**: such as ultrasonic methods, automatic image analysis, electron backscatter diffraction (see ISO 13067[5]) etc., can be used to measure grain size of applicable materials providing that the accuracy of the methods has previously been proven by an extensive cross correlation.

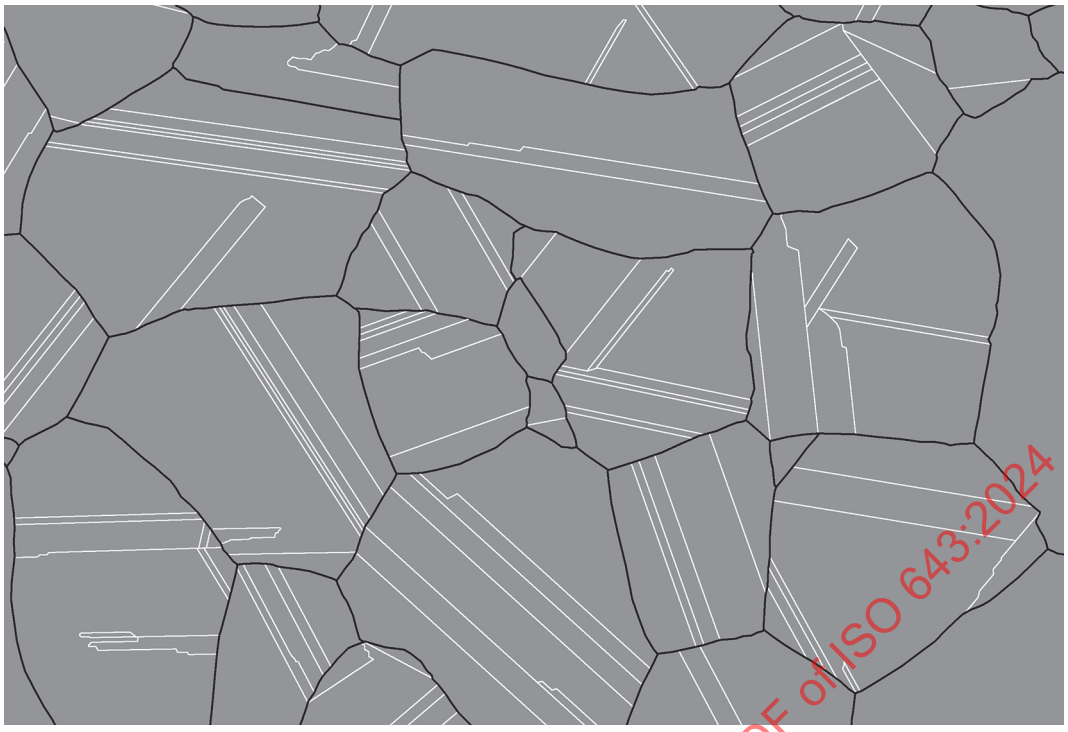

**Figure 6 — Evaluation of number of grains (twin grains)**

## 8 Test report

The test report shall contain the following information:

a) a reference to this document, i.e. ISO 643:2024;

b) specifics for identifying and tracking the sample (e.g. heat, cast, lot, unknown);

c) type of grain determined;

d) method used, standard charts used (if applicable), operating conditions, method of evaluation (i.e. manual or automatic image analysis);

e) grain size index, or, where explicitly specified, the average number of grains per $mm^2$ or the value of the mean segment in mm;

f) any deviations from the procedure;

g) any unusual features observed;

h) the date of the test.

# Annex A
(informative)

# Methods for revealing austenitic or prior-austenitic grain boundaries in steels

## A.1 Overview

Table A.1 gives a summary of methods for revealing ferritic, austenitic or prior-austenitic grain boundaries in steels.

**Table A.1 — Summary of methods for revealing ferritic, austenitic or prior-austenitic grain boundaries in steels**

| Method | Applicable steels |
|---|---|
| The "Bechet-Beaujard" etch method (see A.2) | Steels with martensitic, tempered martensitic or bainitic structures that contain ≥ 0,005 % phosphorus |
| The "Kohn" oxidation method (see A.3) | Non-alloyed and low-alloy steels |
| The "McQuaid-Ehn" carburizing method (see A.4) | Case-hardening steels |
| The mock carburizing method (see A.4) | |
| The proeutectoid ferrite delineation method (see A.5) | Coarse-grained non-alloyed steels with between 0,26 % and 0,6 % carbon; also low-alloy steels such as Mn-Mo, 1 % Cr, 1 % Cr-Mo and 1,5 % Cr-Ni |
| The bainite or gradient quench method (see A.6) | Coarse-grained steels of approximately eutectoid carbon content, i.e. 0,7 % to 0,8 % carbon |
| The grain boundary sensitization method (see A.7) | Unstabilized austenitic or duplex stainless steels with a carbon content > 0,025 %[a] |
| The quenching and tempering method (see A.8) | Non-alloyed steels |

[a] Austenitic Mn steels will precipitate a fine carbide aggregate at the grain boundaries when aged between 550 °C and 600 °C.

## A.2 "Bechet-Beaujard" method by etching with aqueous saturated picric acid solution

### A.2.1 Field of application

This method reveals prior austenitic grains formed during heat treatment of the specimen. It is applicable to specimens which have a martensitic or bainitic structure. For this etch to work, there shall be at least 0,005 % P.

### A.2.2 Preparation

The Bechet-Beaujard etchant is normally used on a heat-treated steel specimen. Normally, no subsequent heat treatment is necessary if the specimen has a martensitic or bainitic structure. If this is not the case, heat treatment is necessary.

If the conditions for treating the specimen are not provided for by the standard defining the product and there is no specification to the contrary, the following conditions can be applied in the case of heat-treated structural non-alloyed steels and low-alloy steels:

— 1,5 h at (850 ± 10) °C for steels whose carbon content is greater than 0,35 %;

— 1,5 h at (880 ± 10) °C for steels whose carbon content is less than or equal to 0,35 %.

After this treatment, the specimen shall be quenched into water or oil.

### A.2.3 Polishing and etching

A flat specimen surface shall be polished for micrographic examination. It shall be etched for an adequate period of time by means of an aqueous solution saturated with picric acid together with at least 0,5 % sodium alkylsulfonate or another appropriate wetting agent.

NOTE The period of etching can vary from a few minutes to more than one hour. Heating of the solution to 60 °C can improve the etching action and reduce etching time.

Several successive etching and polishing operations are sometimes necessary to ensure a sufficient contrast between the grain boundaries and the general base of the specimen. In the case of through-hardened steel, tempering may be carried out before selecting the specimen.

**WARNING — When heating solutions containing picric acid, caution shall be taken to avoid the solution boiling dry as picric acid can become explosive.**

### A.2.4 Result

The prior-austenite grain boundaries shall be immediately apparent on microscopic examination.

## A.3 "Kohn" method by controlled oxidation

### A.3.1 Field of application

This method shows up the prior austenitic grain pattern formed by preferential oxidation of the boundaries during austenization at the temperature of a given heat treatment.

### A.3.2 Preparation

One surface of the specimen shall be polished. The rest of its surface shall not show any traces of oxide. The specimen shall be placed in a laboratory furnace in which either a vacuum of 1 Pa is attained, or an inert gas is circulated (e.g. purified argon). Heat treat the specimen in accordance with the austenitizing procedure specified by the customer, or as defined by the standard governing the product.

At the end of this specified heating period, air shall be introduced into the furnace for a period of 10 s to 15 s.

The specimen shall then be water-quenched. The specimen can usually be directly examined using a microscope.

NOTE The oxidation method can be done without the inert atmosphere.

The oxide adhering to the previously polished surface should be removed by light polishing with a fine abrasive, taking care that the oxide network which has formed on the grain boundaries is retained; then the polishing should be completed by the usual methods. The specimen should then be etched using Vilella's reagent:

— 1 g of picric acid;

— 5 ml of hydrochloric acid;

— 100 ml of ethanol.

### A.3.3 Result

The preferential oxidation of the boundaries shows up the pattern of austenitic grains.

If the preparation is applied out correctly, no oxide globules should appear at the grain boundaries.

In certain cases, it may be necessary to use oblique illumination, or differential interference contrast (DIC) methods, to show up the boundaries in better relief.

## A.4 "McQuaid-Ehn" method by carburization

### A.4.1 Field of application

This is a method specifically developed for case-hardening steels and shows up prior austenitic grain boundaries formed during carburization of these steels at temperatures of 915 °C to 960 °C. It is not usually suitable for revealing grains actually formed during other heat treatments.

NOTE The "mock carburizing" procedure can also be used. The specimen is subjected to the same thermal treatment but without a carbon-rich atmosphere. It is then heat-treated as the product would be treated. The Bechet-Beaujard reagent is used to reveal the grain boundaries, see A.2.

### A.4.2 Preparation

The specimens shall be unaffected by decarburization or surface oxidation. Any prior treatment, either cold, hot, mechanical, etc., may have an effect on the shape of the grains obtained; the product specification shall state the treatments to be carried out before determination in cases where it is advisable to take into account these considerations.

After carburizing, the specimen shall be cooled at a rate slow enough to precipitate cementite at the grain boundaries in the hypereutectoid surface region of the carburized specimen.

Carburization shall be achieved by maintaining the specimen at 915 °C to 960 °C for 6 h. This is generally done by keeping the carburizing chamber at 915 °C to 960 °C for 8 h, including a pre-heating period. For case hardening steels the temperature shall be (950 ± 10) °C. For other steel grades the temperature can be between 915 °C to 960 °C depending on carbon and alloy content. In most cases, a carburized layer of approximately 1 mm is obtained. After carburizing, cool the specimen at a rate slow enough to ensure that the cementite is precipitated at the grain boundaries of the hypereutectoid zone of the carburized layer.

Fresh carburizing compound shall be used each time.

### A.4.3 Specimen preparation

The carburized specimen shall be sectioned normally to its surface. One of the sections shall be prepared for micrographic examination and etched using either a) or b).

a) "Le Chatelier and Igewski" reagent (alkaline sodium picrate):

| | | |
|---|---|---|
| — | picric acid | 2 g |
| — | sodium hydroxide | 25 g |
| — | water | 100 ml |

Use this reagent by immersion at 100 °C, for at least 60 s, or at room temperature by means of electrolytic etching 6 V d.c. for 60 s.

b) Nital:

| | | |
|---|---|---|
| — | nitric acid | 2 ml to 5 ml |
| — | ethanol | to make up to 100 ml |

Other reagents may be used as long as the same results are obtained.

### A.4.4 Result

The prior-austenite grain boundaries in the hypereutectoid carburized surface layer will be delineated by proeutectoid cementite.

## A.5 Proeutectoid ferrite method

### A.5.1 Principle

This method is suitable for non-alloyed steel with about 0,25 % to 0,6 % carbon and for low-alloy steels such as manganese-molybdenum, 1 % chromium, 1 % chromium-molybdenum and 1,5 % nickel-chromium. The prior-austenitic grain boundaries are revealed as a network of proeutectoid ferrite.

### A.5.2 Preparation

Use the austenizing conditions as given in the product standard. In the case of non-alloyed or other low hardenability steel, either air cool, furnace cool or partially transform isothermally the specimens in such a manner as to outline the austenitic grain boundaries with ferrite.

In the case of alloy steels, after austenitizing, partially transform isothermally the specimens at an appropriate temperature within the range 650 °C to 720 °C and then water quench.

NOTE 1 The time required for transformation will vary according to the steel, but usually sufficient ferrite has precipitated in 1 min to 5 min, although longer times, up to about 20 min, can sometimes be required.

NOTE 2 For alloy steels, a specimen 12 mm × 6 mm × 3 mm is suitable to obtain uniform transformation during the isothermal treatment.

### A.5.3 Polishing and etching

Section, polish and etch the specimens for micrographic examination. Etch the specimens with a suitable etchant such as hydrochloric acid and picric acid (Vilellas' reagent).

## A.6 Bainite or gradient-quench method

### A.6.1 Principle

This method is suitable for steels of approximately eutectoid composition, i.e. having a carbon content of 0,7 % by mass or higher. The boundaries of the prior-austenitic grains are revealed by a network of fine pearlite or bainite outlining the martensite grains.

### A.6.2 Preparation

Heat the specimen to a temperature not more than 30 °C above $A_{C3}$ (i.e. the temperature at which ferrite completes its transformation to austenite during heating) to ensure full austenitization.

Cool the specimen at a controlled rate to produce a partially hardened structure of fine pearlite or bainite outlining the martensite grains.

This structure may be produced in one of the following ways:

a) by completely quenching in water or oil, as appropriate, a bar of cross-sectional dimensions such that it will fully harden at the surface but only partially harden in the centre;

b) by gradient quenching a length of bar, 12 mm to 25 mm diameter or square, by immersing it in water for a part of the length only.

Then polish and etch.

## A.7 Sensitization of austenitic stainless and manganese steels

The grain boundaries may be developed through precipitation of carbides by heating within the sensitizing temperature range, about 480 °C to 700 °C (900 °F to 1 300 °F). Any suitable carbide-revealing etchant can be used.

This method should not be used in case of very low carbon contents in austenitic grades.

## A.8 Other methods for revealing prior-austenitic grain boundaries

For certain steels, after simple heat treatment (annealing or normalizing, quenching and tempering, etc.), the pattern of the austenitic grains may appear in the following forms under micrographic examination: a network of proeutectoid ferrite surrounding pearlite grains, a network of very fine pearlite surrounding martensite grains, etc. The austenitic grain may also be revealed by thermal etching under vacuum (not necessarily followed by oxidation). The product specification shall mention these simplified methods in these cases.

NOTE Amongst these methods are the following:

— precipitation on the grain boundaries during cooling;

— gradient quenching method, etc.

# Annex B
(normative)

# Determination of grain size with standard comparison charts

Figures B.1 to B.22 have been created using actual micrographs and subsequently checked using round robin comparisons and manual planimetric counting. Figures B.1 to B.22 were taken setting the optical magnification to 100:1. The reference circle corresponds to 0,5 $mm^2$ on the sample surface. Its diameter is thus 798 µm, but for practical purposes a diameter of 800 µm is acceptable. At 100:1 magnification, the printed circle measures 79,8 mm. When working with different magnifications grain size index conversion according to Table 2 applies. Grain size charts are given from 0 to 10 in 0,5 size steps and in addition for grain size -1.

NOTE For custom-scaled printouts, TIFF images of the standard comparison charts are available from https://standards.iso.org/iso/643/ed-5/en.

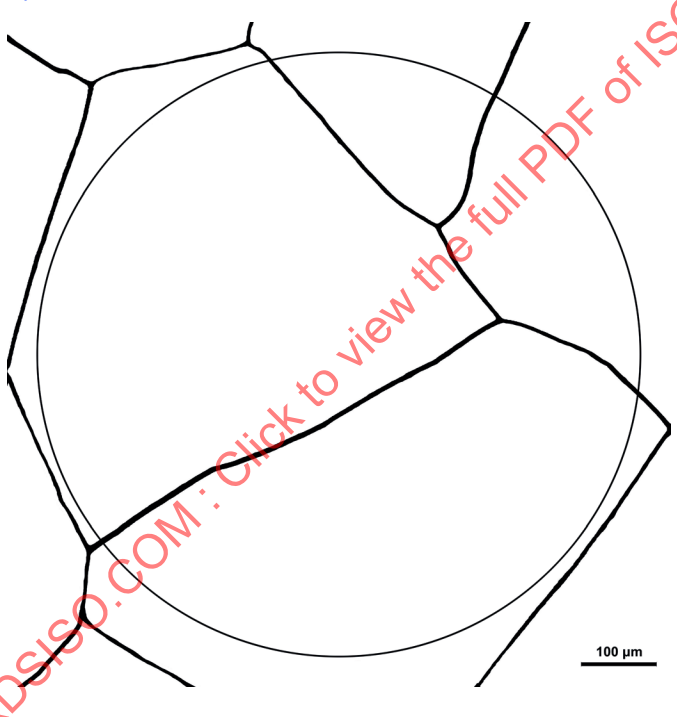


**Figure B.1 — Grain size -1**

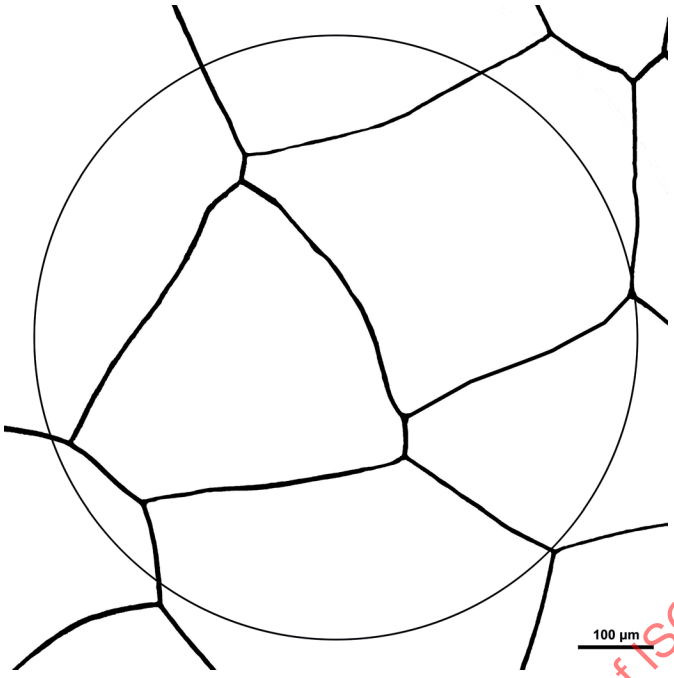


**Figure B.2 — Grain size 0**

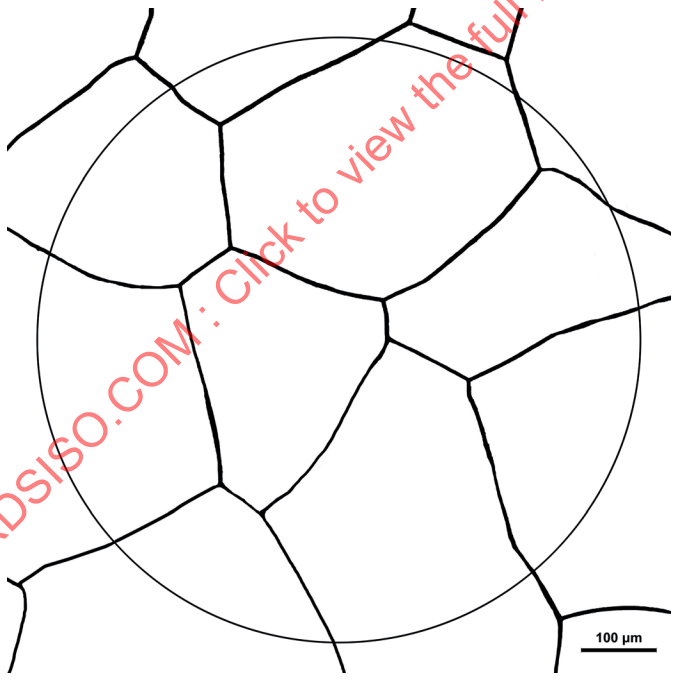


**Figure B.3 — Grain size 0,5**

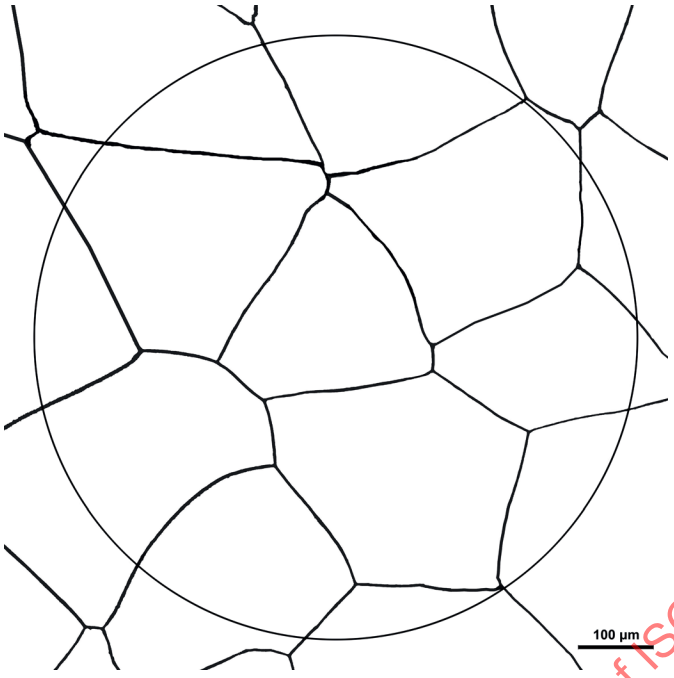


**Figure B.4 — Grain size 1**

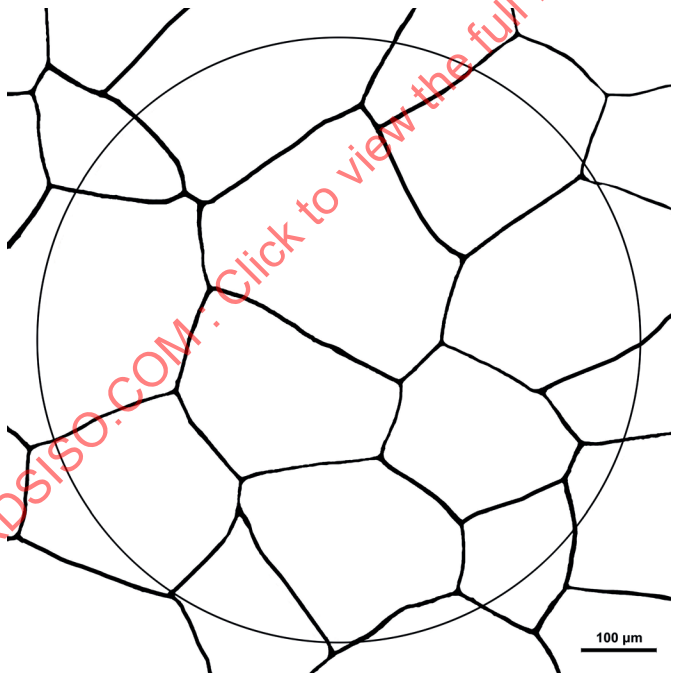


**Figure B.5 — Grain size 1,5**

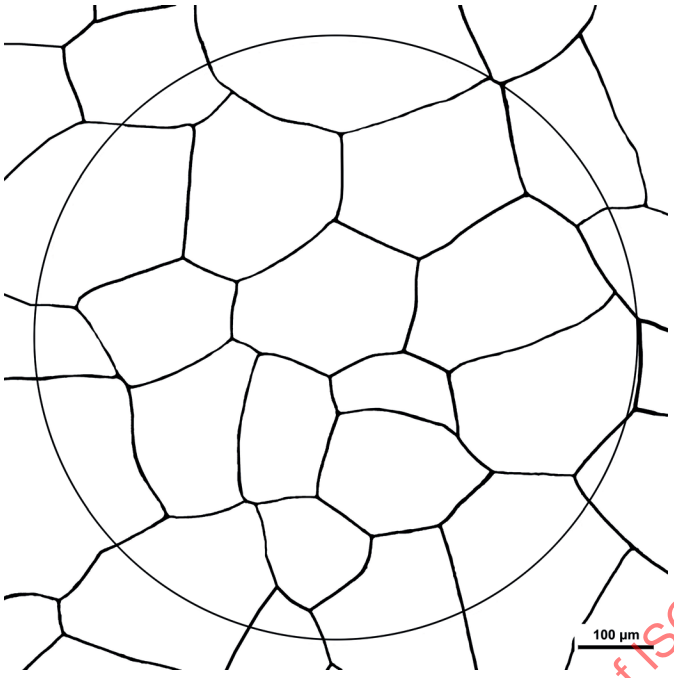


**Figure B.6 — Grain size 2**

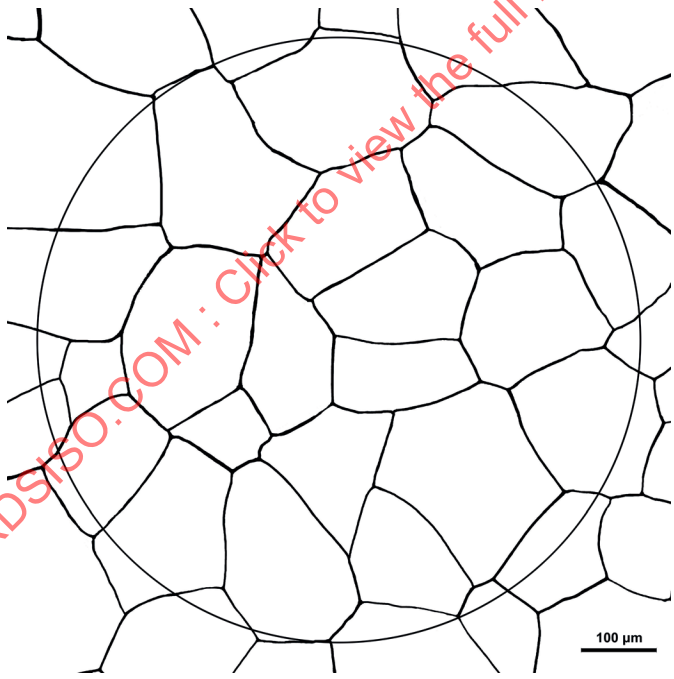


**Figure B.7 — Grain size 2,5**

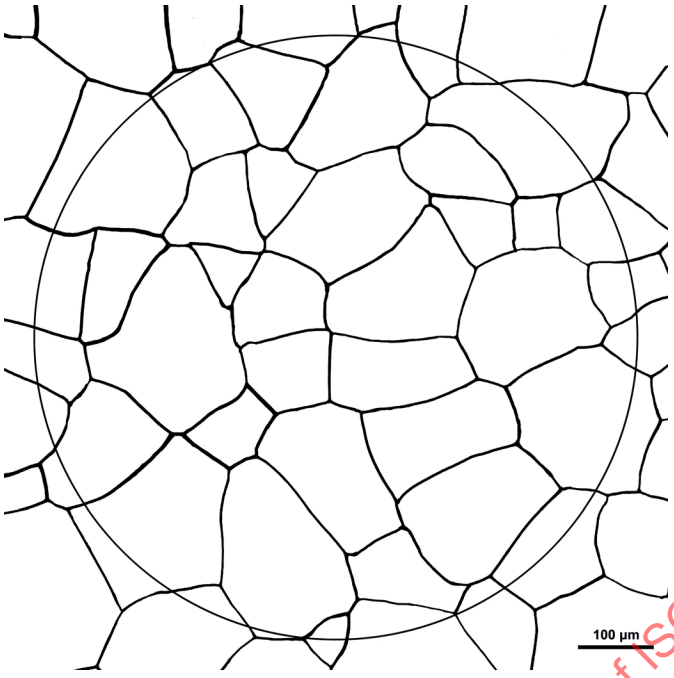


**Figure B.8 — Grain size 3**

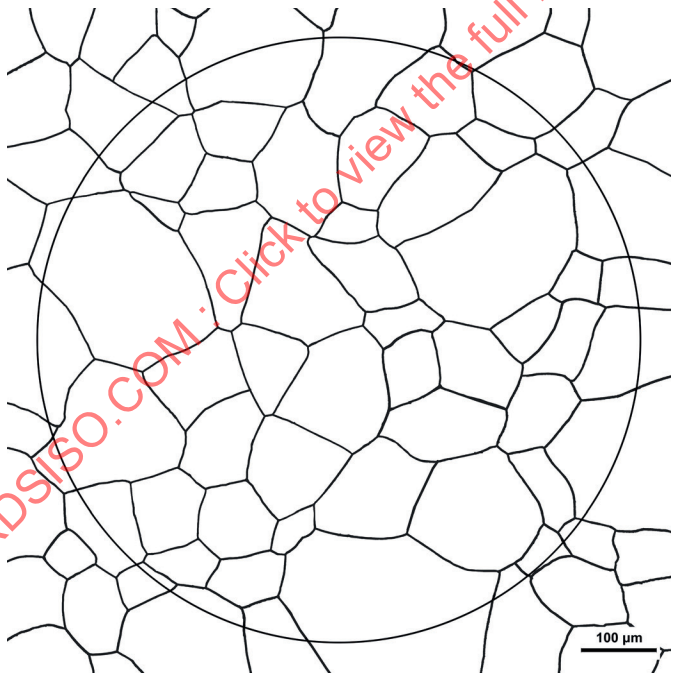


**Figure B.9 — Grain size 3,5**

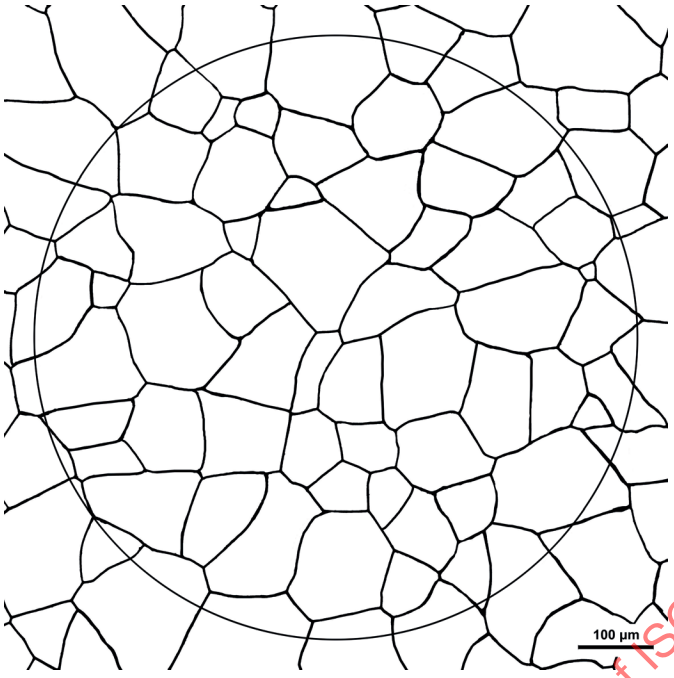


**Figure B.10 — Grain size 4**

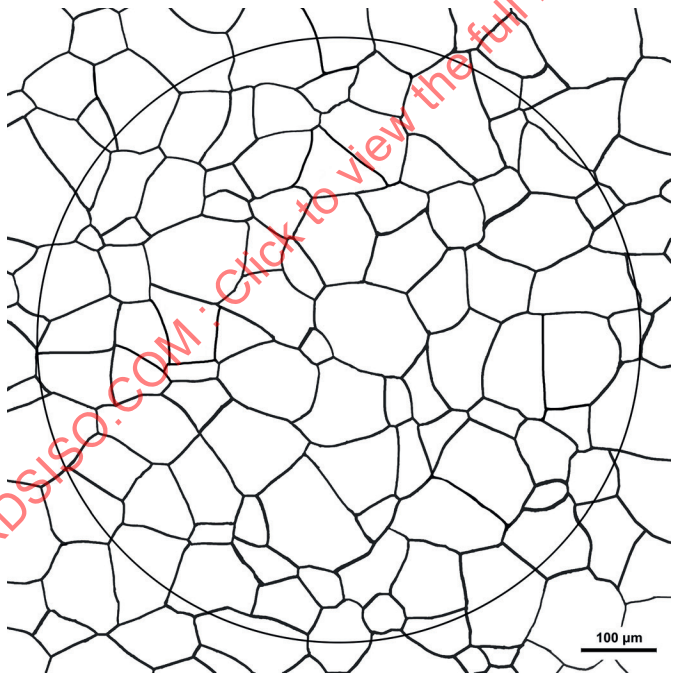


**Figure B.11 — Grain size 4,5**

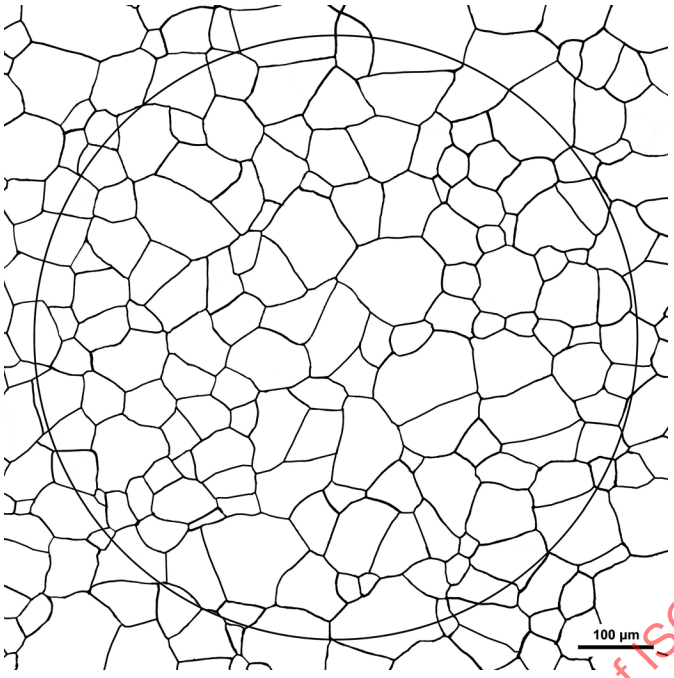


**Figure B.12 — Grain size 5**

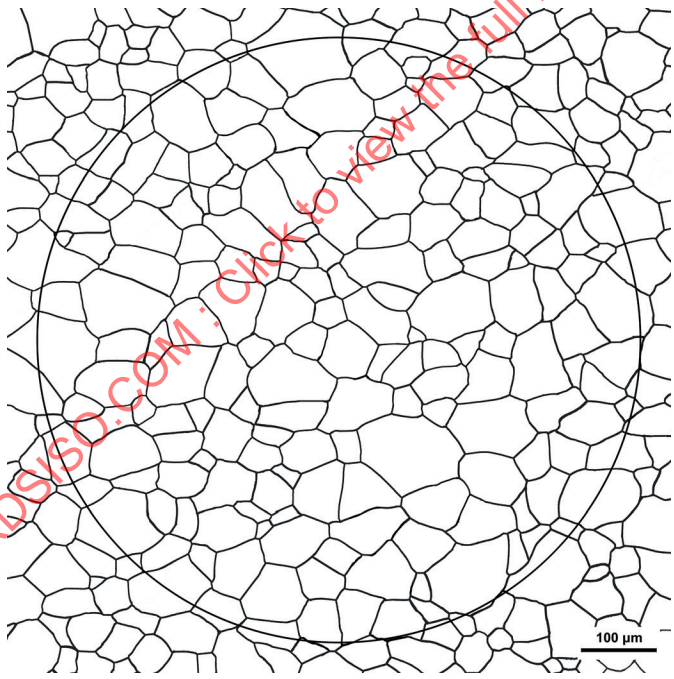


**Figure B.13 — Grain size 5,5**

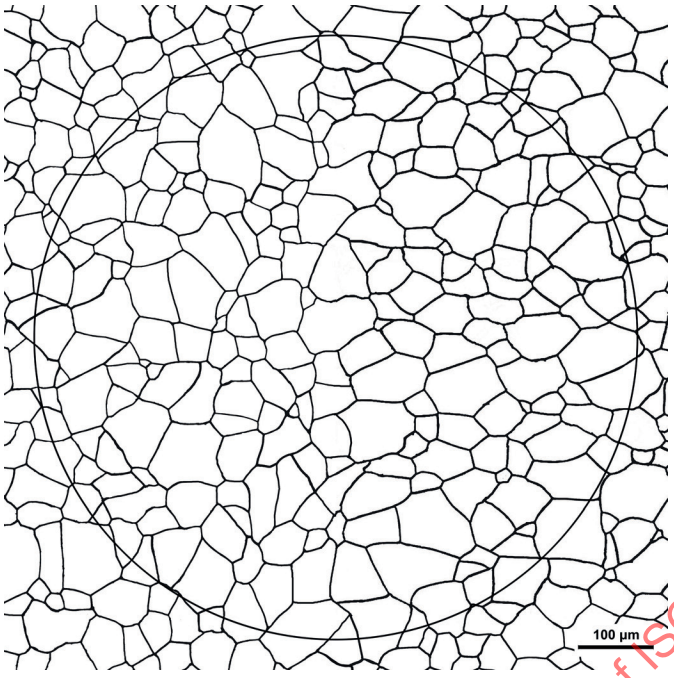


**Figure B.14 — Grain size 6**

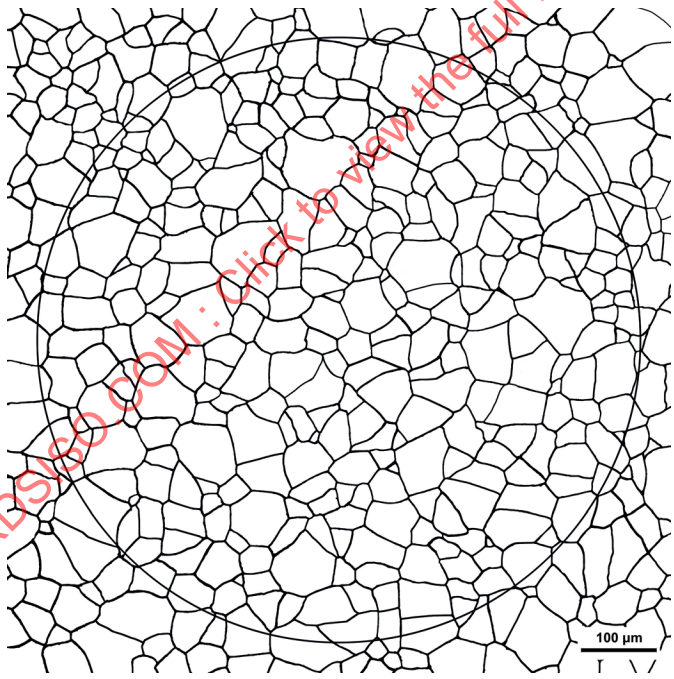


**Figure B.15 — Grain size 6,5**

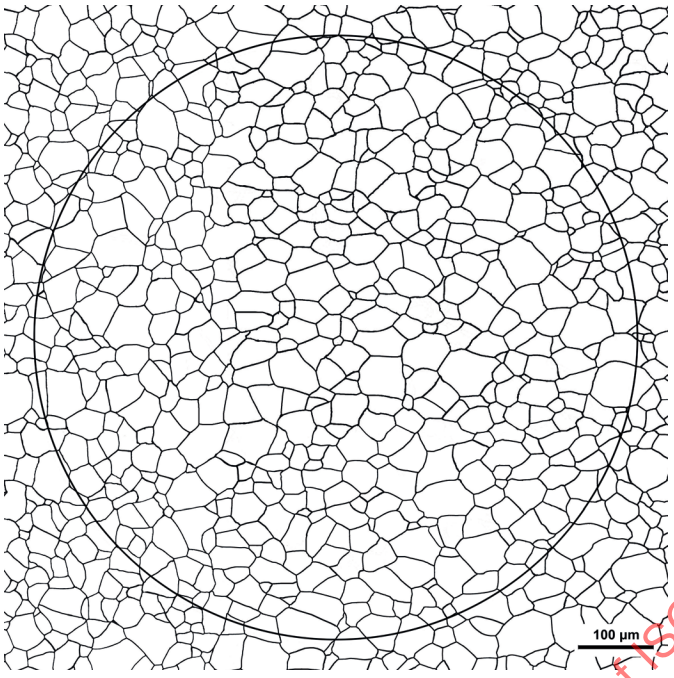


**Figure B.16 — Grain size 7**

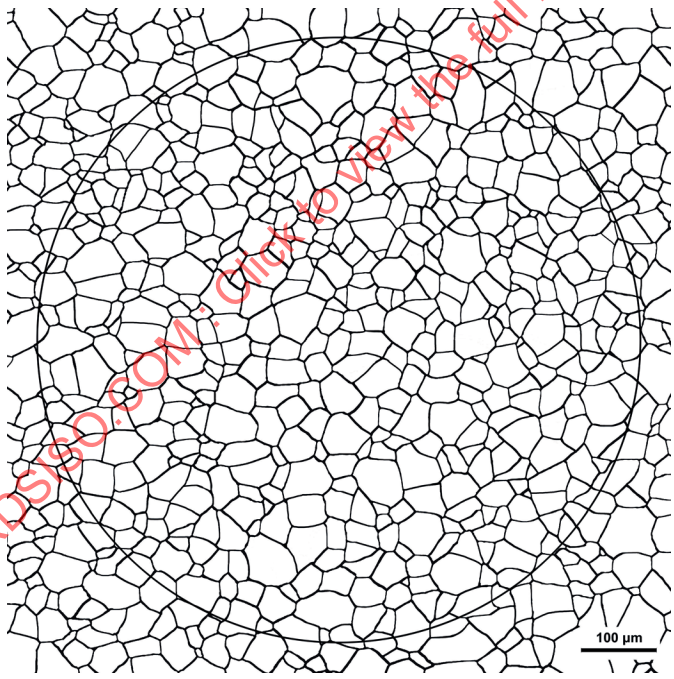


**Figure B.17 — Grain size 7,5**

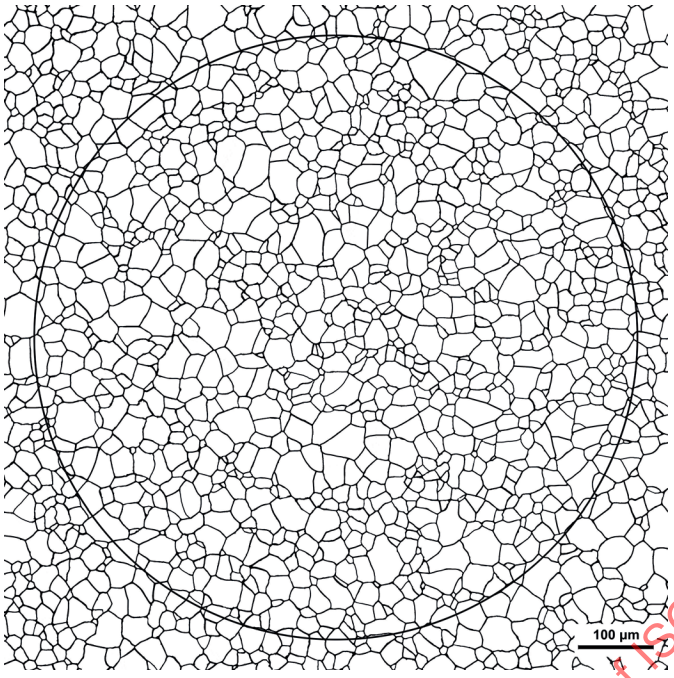


**Figure B.18 — Grain size 8**

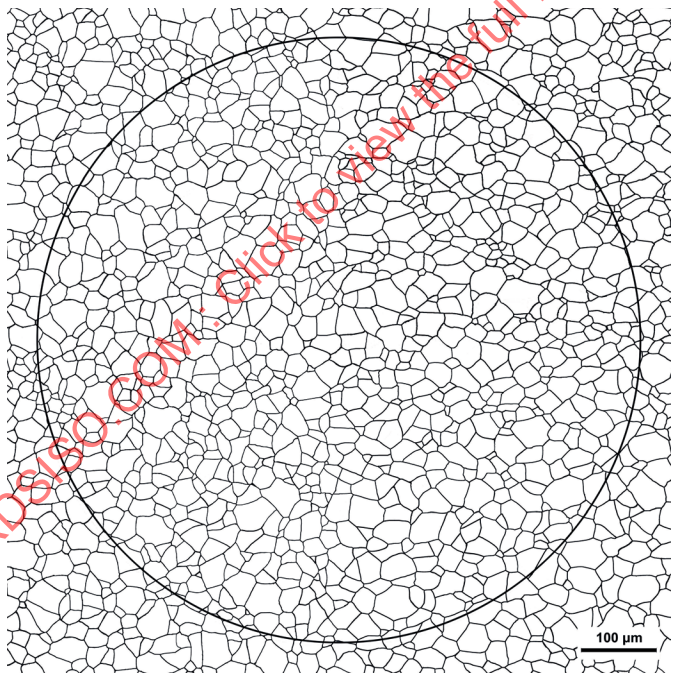


**Figure B.19 — Grain size 8,5**

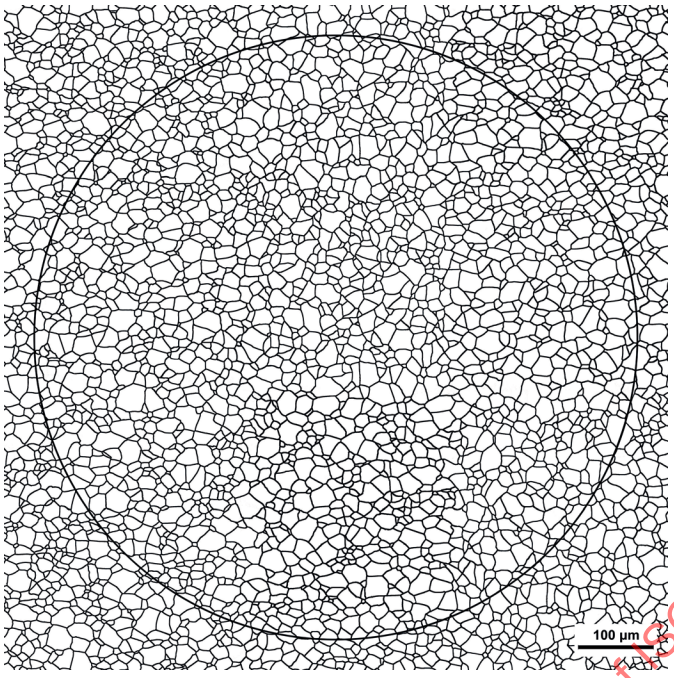


**Figure B.20 — Grain size 9**

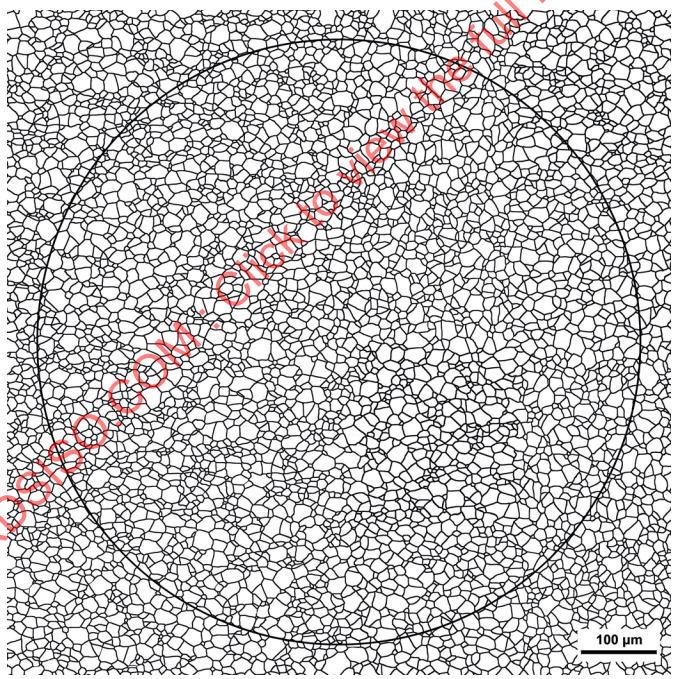


**Figure B.21 — Grain size 9,5**

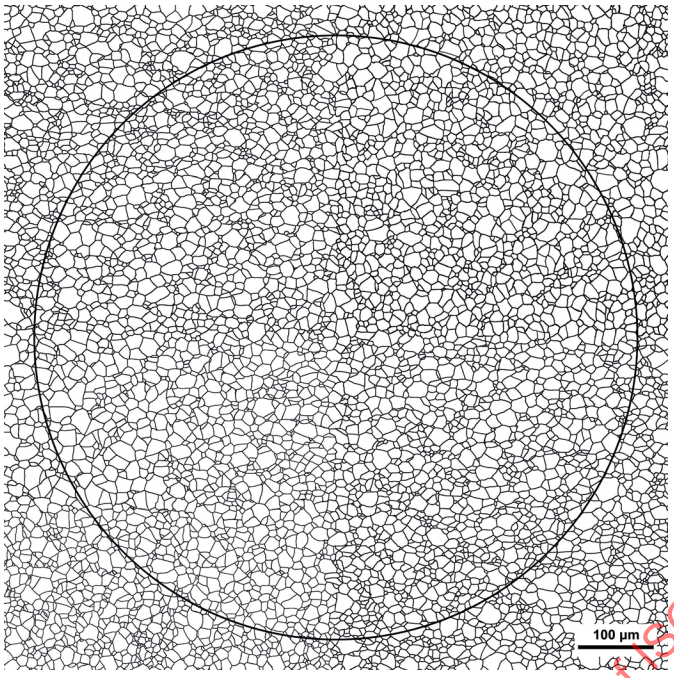


**Figure B.22 — Grain size 10**

# Annex C
(informative)

# Evaluation method

## C.1 "Snyder-Graff" method[6]

### C.1.1 Field of application

This method is used for determining the prior-austenitic grain size of hardened and tempered high-speed steels by means of the linear intercept method.

### C.1.2 Preparation

The specimen, taken from the product that is usually in the hardened and tempered condition, shall not receive any supplementary heat treatment.

After being polished, the specimen shall be etched using nital containing up to 10 % by volume of nitric acid in ethanol. Etch the specimen long enough to clearly reveal the prior-austenitic grain boundaries. Several successive polish/etch cycles may be necessary. The surface of the specimen is more or less coloured depending on the type of heat treatment undergone by the product.

### C.1.3 Measuring

Under a magnification of 1 000:1, the number of grains intercepted by a measuring line 125 mm long shall be counted. Five counts shall be carried out in different directions in fields selected at random.

### C.1.4 Result

Unless specified to the contrary, the arithmetic mean of the number of grains intercepted in five counts characterizes the grain size. This value multiplied by 1,6 corresponds to the mean number of grains intercepted per unit length of the line $\bar{N}_L$. From that value, the mean intercepted segment or $G$ may be determined.

## C.2 Alternative system of grain size definition

### C.2.1 General

In addition to the grain size definition system described in this document, there is one other system, as used in the USA.

This system (see ASTM E112[3]) defines the grain size by an index $G$, known as the ASTM grain size, as shown in C.2.2 and C.2.3.

### C.2.2 Mean intersected segment method

Index $G$ (ASTM) = 0, corresponds to a mean intersected segment of 32,0 mm measured at a magnification of 100:1.

The formula giving the other indices as a function of

— the mean intersected segment is given by Formula (C.1):

$$G(\text{ASTM}) = -3,287\,7 - 2\log_2 l \quad \text{(C.1)}$$

— the mean number of intercepts per unit length (mm) is given by Formula (C.2):

$$G(\text{ASTM}) = -3,287\,7 - 2\log_2 \bar{N}_L \quad \text{(C.2)}$$

### C.2.3 Count method

By definition, index $G$ (ASTM) = 1 corresponds to 15,5 grains per unit area (square millimetre).

The formula giving the other indices as a function of the number of grains per unit area (square millimetre) is given by Formula (C.3):

$$G(\text{ASTM}) = -2,954\,2 + \log_2 m \quad \text{(C.3)}$$

### C.2.4 Numerical ratios between the various grain size indices in the case of regular structures

The ASTM index gives a grain size slightly larger than the one defined by this document, but the difference does not reach one twentieth of an index unit. This is negligible, as the estimation of grain size cannot generally be accurate to more than one half a unit under even the most favourable conditions.

Formulae (2) and (3) may be written as Formula (C.4):

$$G = -3 + \log_2 m \quad \text{(C.4)}$$

Comparing this formula with Formula (C.3) shows that, as given by Formula (C.5):

$$G(\text{ASTM}) - G = 0,045\,8 \quad \text{(C.5)}$$

This is the computed difference between the planimetric methods as defined in this document and ASTM E112. It is well within the deviation that arises from permissible deviations in microscopic magnification as stated in ISO 8039[7].

Because the human eye is unable to detect such a small difference reliably, the comparison methods of the two standards yield comparable results. Furthermore, because such a small difference in grain size has a negligible influence on the material properties, the ASTM E112 comparison charts may also be used without losing compliance with this document.

For the intercept method, there is no difference between the standards, as both standards use $l_0$ = 0,32 mm. As mentioned in 7.4.4.6, there is no exact relation between $G$ and the mean lineal intercept length $l$.

NOTE An argument can be made for $l_0$ = 0,315 mm (consistent difference between the standards across the methods) as well as for $l_0$ = 0,313 mm (for $l$ defined as $l = d_{EC}$), but to make it easier for the users of both standards and to stay consistent with past editions of this document, $l_0$ = 0,32 mm was upheld in this document.

# Annex D
(informative)

# Calculation of grain size and confidence interval

## D.1 General

The calculation methods include the planimetric method, intercept method and intercept length method.

For the planimetric method, an uncertainty of ±0,25 grain size units corresponds to the relative error $E_{rel}$ = 17,2 %. For the intercept method and intercept length method, an uncertainty of ±0,25 grain size units corresponds to the relative error $E_{rel}$ = 8,6 %. Thus, it is stipulated as follows: for the planimetric method, if $E_{rel}$ > 17 %, more fields should be measured until $E_{rel}$ ≤ 17 %; for the intercept method and intercept length method, if $E_{rel}$ > 8,5 %, more fields should be measured until $E_{rel}$ ≤ 8,5 %.

The planimetric method and intercept method are applicable for the determination of grain size of specimens with unimodal and relatively uniform grain size distribution. Five representative fields are chosen for the determination of the initial values (the number of intercepts, the number of intersections or the number of grains), based on which the relative error, $E_{rel}$, is calculated. If $E_{rel}$ is higher than the above stipulated value, more fields will be measured until $E_{rel}$ is equal to or smaller than the above stipulated value, then calculate the average grain size number.

For the planimetric method and intercept method, the calculation has a confidence interval (range). The nonuniform grain size may result in a too high relative error or too big confidence limit of the grain size. In this case, observe the whole specimen comprehensively and calculate the nonuniform areas separately or use single-circle method or intercept length method to calculate different grain size numbers.

The intercept length method is applicable for determination of grain size of specimens with nonuniform size distribution, see Annex E.

## D.2 Calculation method

**D.2.1** For the intercept method and planimetric method, at least five representative fields are chosen for calculating the number of intersections or the number of grains.

Calculate the mean value of $N_A$ or $l$ from the individual field values according to Formula (D.1):

$$\bar{X} = \frac{\sum X_i}{n} \tag{D.1}$$

where $\bar{X}_i$ represents an individual value, $\bar{X}$ is the mean and $n$ is the number of measurements.

**D.2.2** Calculate the standard deviation according to Formula (D.2):

$$s = \sqrt{\frac{\sum (X_i - \bar{X})^2}{n-1}} \tag{D.2}$$

where $s$ is the standard deviation.