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Steels — Micrographic determination of the apparent grain size

Aciers — Détermination micrographique de la grosseur de grain apparente

FDIS stage

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Contents

Foreword

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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);~~A.4);

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

— ~~Annex B~~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)~~(subclause 7.3) and the remainder of the annex has been renumbered as ~~Annex C;~~Annex C;

— ~~—~~ ~~New Annexes D and E~~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~~.

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Steels — Micrographic determination of the apparent grain size

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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/obphttps://www.iso.org/obp

— ——IEC Electropedia: available at https://www.electropedia.org/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][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)~~(3.1.1) counted in an area of 1 mm² 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).~~Formula (1).

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

Note 1 to entry: See ~~Figure 1.~~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. ~~$\bar{N}$~~ $\tilde{N}$ is the average of a number of counts of the number of grains intercepted by the test line applied randomly at various locations. ~~$\bar{N}$~~ $\tilde{N}$ 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, ~~$\bar{N}_L$~~ $\tilde{N}_L$.

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

Note 1 to entry: See ~~Figure 2.~~Figure 2.

Note 2 to entry: ~~$\bar{P}$~~ $\tilde{P}$ is the average of a number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations. ~~$\bar{P}$~~ $\tilde{P}$ 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, ~~$\bar{P}_L$~~ $\tilde{P}_L$.

4 Symbols

The symbols used are given in ~~Table 1.~~Table 1.

Table 1 — Symbols

Symbols	Definition	Value
~~$\bar{a}$~~ $\tilde{a}$	Mean area of grain in square millimetres	~~$\bar{a} = \frac{1}{m}$~~ $\tilde{a} = \frac{1}{m}$
A_B	True area of the test box	mm²
A_C	True area of the test circle	mm²
A_F	Apparent area of the test figure in square millimetres	—
~~$\bar{d}$~~ $\tilde{d}$	Mean grain diameter in millimetres	~~$\bar{d} = \frac{1}{\sqrt{m}}$~~ $\tilde{d} = \frac{1}{\sqrt{m}}$

Symbols	Definition	Value
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 mm²)
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	~~$l = 1/\bar{N}_L = 1/\bar{P}_L$~~ $l = 1/\bar{N}_L = 1/\bar{P}_L$
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	—
m	Number of grains per square millimetre of specimen surface in the area examined	$m = n_t/A_C$ $m = n_t/A_B$
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	—
~~$\bar{N}$~~ $\bar{N}$	Mean number of grains intercepted per unit length L	—
~~$\bar{N}_L$~~ $\bar{N}_L$	Mean number of grains intercepted per unit length of the line	~~$\bar{N}_L = \bar{N}/L_T$~~ $\bar{N}_L = \bar{N}/L_T$
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]	—
~~$\bar{P}$~~ $\bar{P}$	Mean number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations	—
~~$\bar{P}_L$~~ $\bar{P}_L$	Mean number of grain boundary intersections per unit length of test line	~~$\bar{P}_L = \bar{P}/L_T$~~ $\bar{P}_L = \bar{P}/L_T$
Q	Correction factor for non-standard magnification	~~$Q = 2log_2\left(\frac{g}{100}\right)$~~ $Q = 2log_2(\frac{g}{100})$

[a] The method for designating the direction conforms to ISO 3785~~[2]~~.[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) ~~a)~~ comparison method using standard charts (see ~~7.2);~~7.2);

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

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

All methods give comparable results.

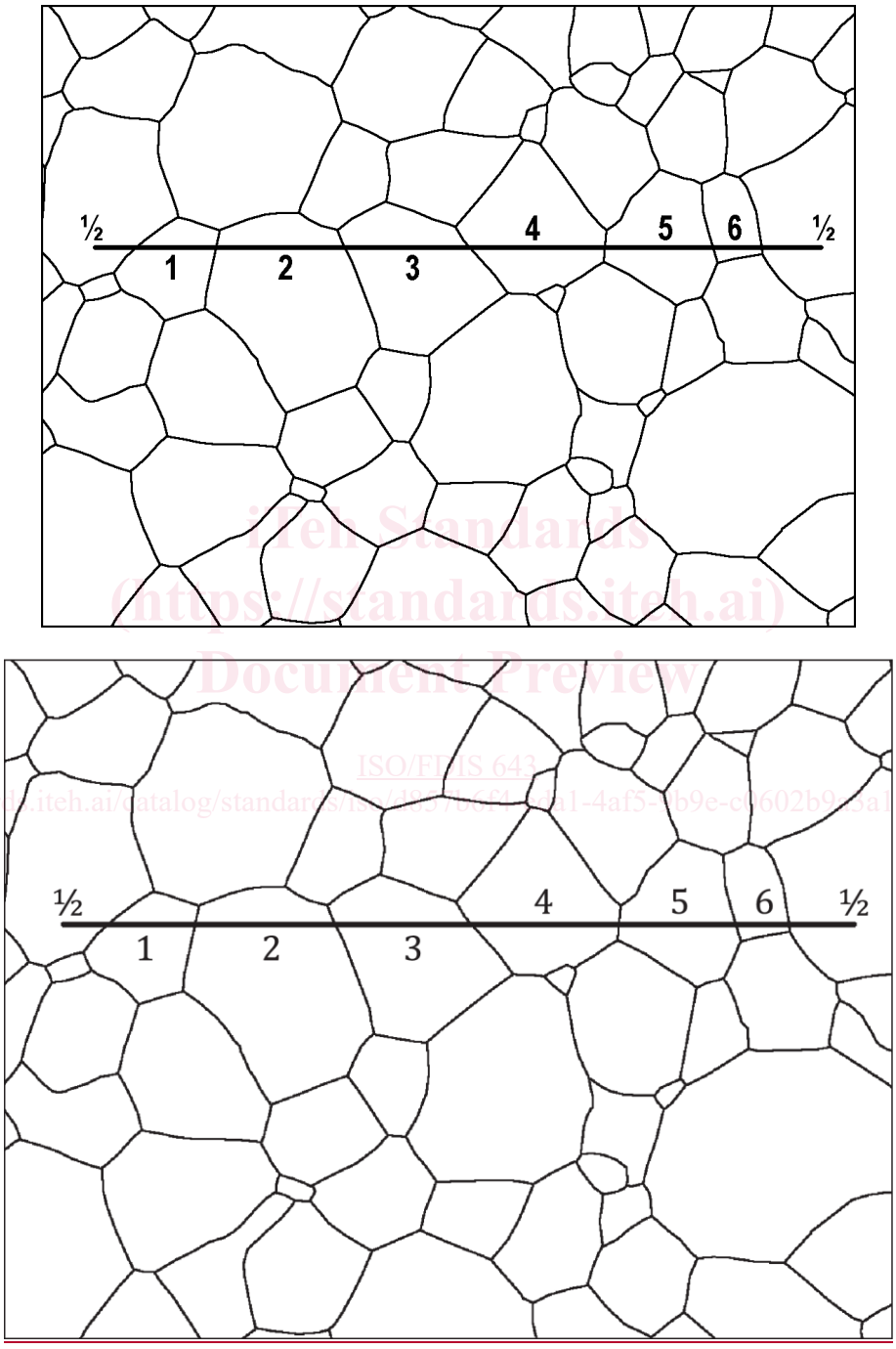
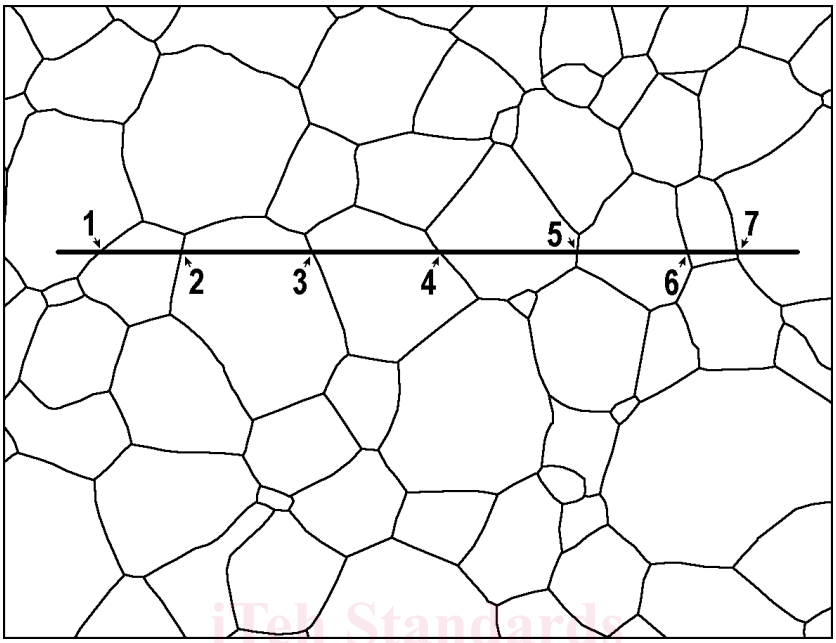


Figure 1 — Example of intercept, *N*

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 1 — Example of intercept, *N***~~



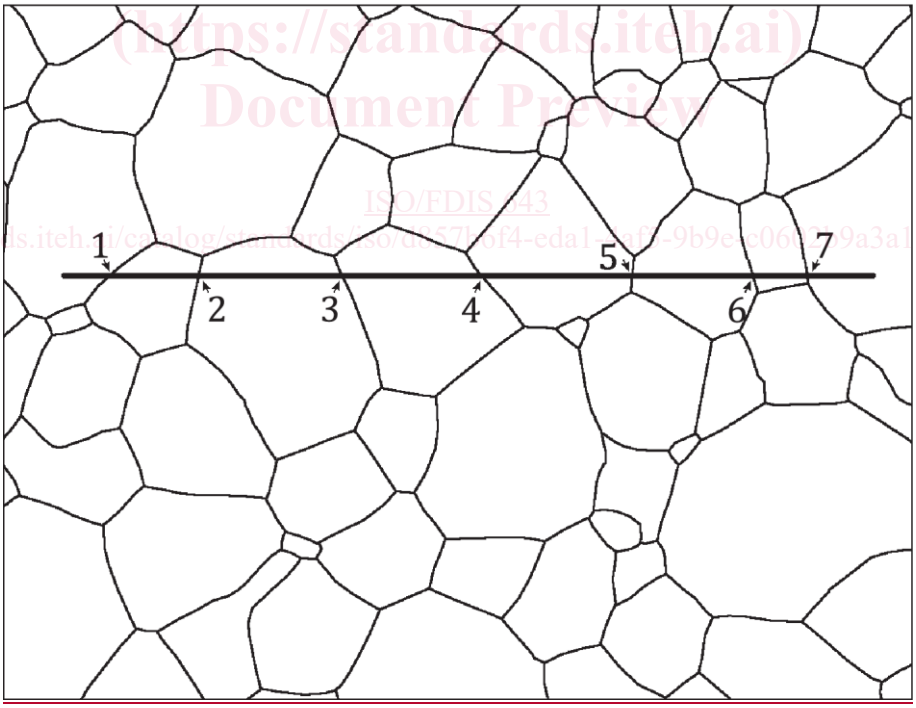


Figure 2 — Example of intersection, *P*

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.

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

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);~~A.2);

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

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

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