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Steels — Micrographic determination of the apparent grain size

Aciers — Détermination micrographique de la grosseur de grain apparente

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Foreword

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

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For an explanation on 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 the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This fourth edition cancels and replaces the third edition (ISO 643:2012), which has been technically revised. The main changes compared to the previous edition are as follows:

— 7.1.2 has been modified;

— the original Annex B has been deleted and the original Annex C has been renumbered as Annex B.

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.

This corrected version of ISO 643:2019 incorporates the following corrections:

— minus sign replaced with plus sign between the values in Formula B.9.

Steels — Micrographic determination of the apparent grain size

1 Scope

This document specifies a micrographic method 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

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.

ASTM E112, *Standard Test Methods for Determining Average Grain Size*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

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

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

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

3.1 Grains

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

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

Note 1 to entry: Ferritic grain size is generally estimated for unalloyed steels with a carbon content of 0,25 % or less. If pearlite islands of identical dimensions to those of the ferrite grains are present, the islands are then counted as ferrite grains.

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).

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

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 interception. $\overline{N}$ is the average of a number of counts of the number of grains intercepted by the test line applied randomly at various locations. $\overline{N}$ 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, $\overline{N}_L$.

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: $\overline{P}$ is the average of a number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations. $\overline{P}$ 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, $\overline{P}_L$.

4 Symbols

The symbols used are given in Table 1.

Table 1 — Symbols

Symbols	Definition	Value
$\overline{a}$	Mean area of grain in square millimetres	$\overline{a} = \frac{1}{m}$
A_F	Apparent area of the test figure in square millimetres	—
$\overline{d}$	Mean grain diameter in millimetres	$\overline{d} = \frac{1}{\sqrt{m}}$
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 test piece	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	—
[a] The method for designating the direction conforms to ISO 3785.		

Table 1 *(continued)*

Symbols	Definition	Value
K	Conversion factor from linear magnification × g to linear magnification ×100	$K = \frac{g}{100}$
l	Mean lineal intercept length, generally expressed in millimetres	$l = 1/\overline{N}_L = 1/\overline{P}_L$
L_T	True length of the test line divided by the magnification, in millimetres	—
m	Number of grains per square millimetre of test piece surface in the area examined	$m = 2\ n_{100}$ (magnification × 100) $m = 2\ K^2 n_g$ (magnification × g)
M	Number of the closest standard chart picture where g is not 100.	—
n_g	Total equivalent number of grains examined on the image of diameter D (with a magnification × g)	—
n_1	Number of grains completely inside the circle of diameter D	—
n_2	Number of grains intersected by the circle of diameter D	—
n_{100}	Total equivalent number of grains examined on the image of diameter D (with magnification × 100)	$n_{100} = n_1 + \frac{n_2}{2}$
$\overline{N}$	Mean number of grains intercepted per unit length L	—
$\overline{N}_L$	Mean number of grains intercepted per unit length of the line	$\overline{N}_L = \overline{N} / L_T$
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]	—
$\overline{P}$	Mean number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations	—
$\overline{P}_L$	Mean number of grain boundary intersections per unit length of test line	$\overline{P}_L = \overline{P} / L_T$

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

5 Principle

The grain size is revealed by micrographic examination of a polished section of the specimen prepared by an appropriate method for the type of steel and for the information sought.

NOTE If the order or the International Standard defining the product does not stipulate the method of revealing the grain, the choice of this method is left to the manufacturer.

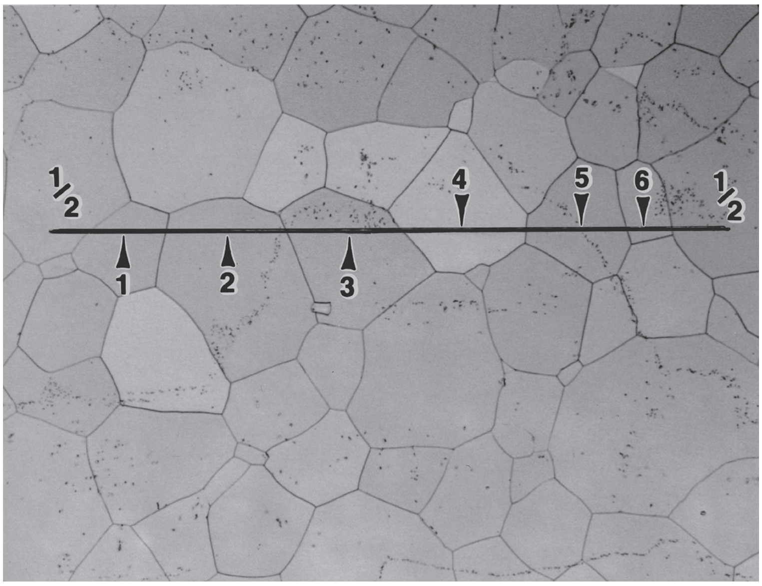
This average size is characterized either

a) by an index obtained

   1) usually by comparison with standard charts for the measurement of grain size;

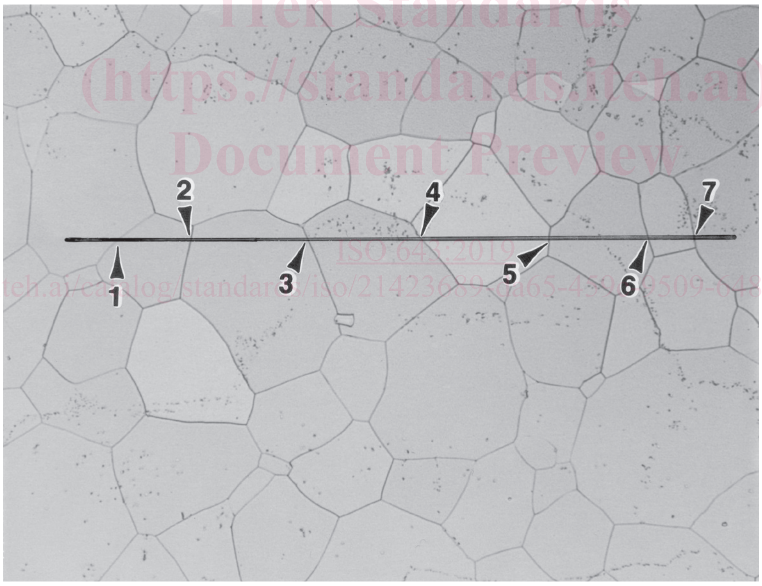
   2) or by counting to determine the average number of grains per unit area;

b) or by the mean value of the intercepted segment.



NOTE Interception, *N*, counts for a straight line on a single-phase grain structure where the arrows point to 6 intercepts and two line segments ending within grain (2 × 1/2 = 1 *N*) and *N* = 7.

Figure 1 — Example of interception, *N*



NOTE 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 International 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. Therefore, it is recommended that two or more sections be 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 face of the specimen shall be longitudinal, i.e. parallel to the principal axis of deformation in wrought products. 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 % nitric acid solution), or with an 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 two-phase austenitic structure (delta ferrite grains in an austenitic matrix) at ambient temperature, the grain 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 % HNO_3.

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 6.3.2).

— "Kohn" method by controlled oxidation (see 6.3.3).

— "McQuaid-Ehn" method by carburization (see 6.3.4).

— grain boundary sensitization method (see 6.3.7).

— 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.

6.3.2 "Bechet-Beaujard" method by etching with aqueous saturated picric acid solution

6.3.2.1 Field of application

This method reveals 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.

6.3.2.2 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 test piece are not provided for by the International Standard defining the product and there is no specification to the contrary, the following conditions shall be applied in the case of heat-treated structural unalloyed 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 test piece shall be quenched into water or oil.

6.3.2.3 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.

6.3.2.4 Result

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

6.3.3 “Kohn” method by controlled oxidation

6.3.3.1 Field of application

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

6.3.3.2 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 International 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.

