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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

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.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 643 was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This third edition cancels and replaces the second edition (ISO 643:2003), of which it constitutes a minor revision. A note was added after the first paragraph of 7.1.2.

Steels — Micrographic determination of the apparent grain size

1 Scope

This International Standard 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 referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3785, *Steel — Designation of test piece axes*

ISO 14250, *Steel — Metallographic characterization of duplex grain size and distributions*

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.

3.1
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

A distinction is made between:

3.1.1
austenitic grain
crystal with a face-centered cubic crystal structure which may, or may not, contain annealing twins

3.1.2
ferritic grain
crystal with a body-centered cubic crystal structure which never contains annealing twins[1)]

1) Ferritic grain size is generally estimated for non-alloy 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
index
positive, zero or possibly negative number G which is derived from the mean number m of grains counted in an area of 1 mm^2 of the section of the specimen

NOTE By definition, G = 1 where m = 16; the other indices are obtained by the formula

$$m = 8 \times 2^G$$

3.3
intercept
N
number of grains intercepted by a test line, either straight or curved

See Figure 1.

NOTE 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, $\overline{N}_L$.

3.4
intersection
P
number of intersection points between grain boundaries and a test line, either straight or curved

See Figure 1.

NOTE $\overline{P}$ 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 and abbreviated terms

The symbols used are given in Table 1.

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.

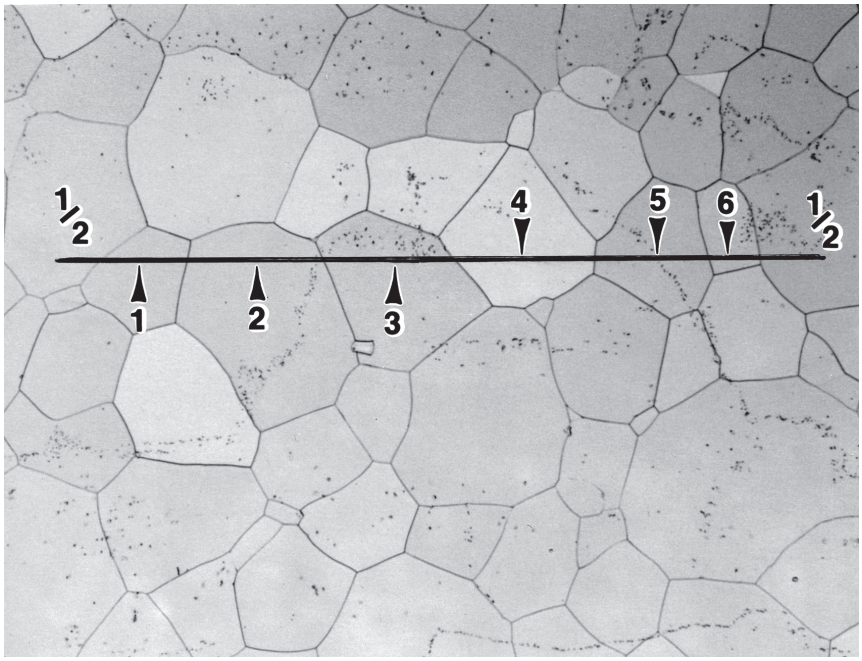
This average size is characterized either

a) by an index obtained

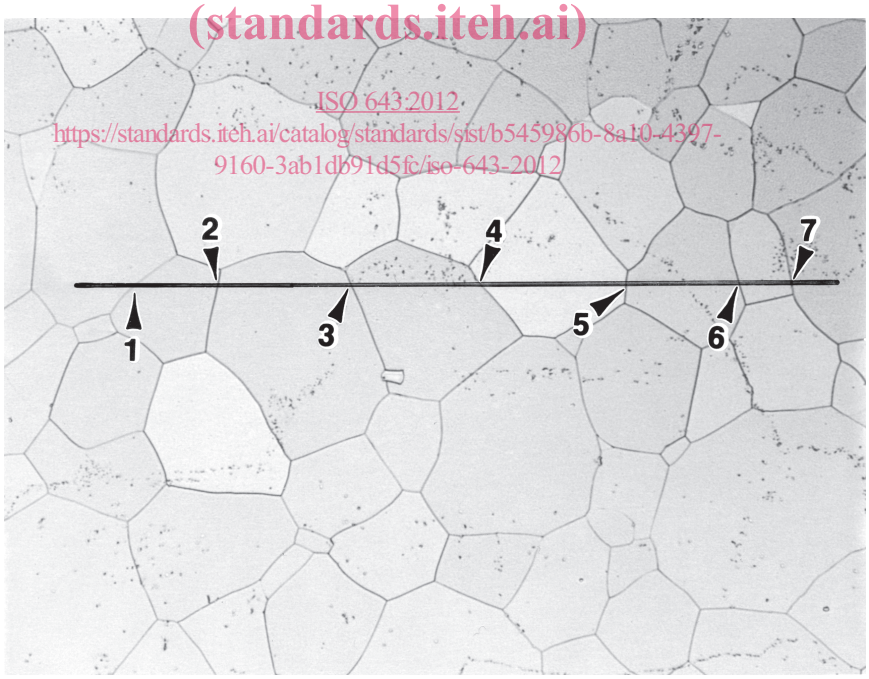
   — usually by comparison with standard charts for the measurement of grain size;

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

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



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 ($2 \times 1/2 = 1$ N) and $N = 7$



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 1 — Examples of intersection, P, and interception, N

Table 1 — Symbols

Symbols	Definition	Value
$\bar{a}$	Mean area of grain in square millimetres	$\bar{a} = \frac{1}{m}$
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 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	–
K	Conversion factor from linear magnification $\times g$ to linear magnification $\times 100$	$K = \frac{g}{100}$
l	Mean lineal intercept length, generally expressed in millimetres	$l = 1/\bar{N}_L = 1/\bar{P}_L$
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 $\times$ 100) $m = 2\ K^2 n_g$ (magnification $\times g$)
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 $\times g$)	–
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 $\times$ 100)	$n_{100} = n_1 + \frac{n_2}{2}$
$\bar{N}$	Mean number of grains intercepted per unit length L	–
$\bar{N}_L$	Mean number of grains intercepted per unit length of the line	$\bar{N}_L = \bar{N} / L_T$
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]	–
$\bar{P}$	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	$\bar{P}_L = \bar{P} / L_T$

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

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 carbon 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 may vary from a few minutes to more than one hour. Heating of the solution to 60 °C may 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 1 The oxidation method can be done without the inert atmosphere.

NOTE 2 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:

— picric acid 1 g

— hydrochloric acid 5 ml

— ethanol 100 ml

6.3.3.3 Result

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

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

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

6.3.4 "McQuaid-Ehn" method by carburization at 925 °C

6.3.4.1 Field of application

This is a method specifically for carburizing steels and shows up austenitic grain boundaries formed during carburization of these steels. It is not usually suitable for revealing grains actually formed during other heat treatments.

NOTE The "mock carburizing" procedure may 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 6.3.2.

6.3.4.2 Preparation

The specimens shall be free from any trace of decarburization or of surface oxidation. Any prior treatment, either cold, hot, mechanical, etc., may have an effect on the shape of the grain 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 must 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 (925 ± 10) °C for 6 h. This is generally done by keeping the carburizing chamber at (925 ± 10) °C for 8 h, including a pre-heating period. 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.

6.3.4.3 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 1 min, 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.

6.3.4.4 Result

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

6.3.5 Proeutectoid ferrite method

NOTE Guidelines for the use of this method depending on the microstructure of the steel product are given in Annex A.

6.3.5.1 Principle

This method is suitable for carbon 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.

6.3.5.2 Preparation

Use the austenizing conditions as given in the product standard. In the case of carbon or other low hardenability steel, either air cool, furnace cool or partially transform isothermally the test pieces 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 test pieces 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 test piece 12 mm × 6 mm × 3 mm is suitable to obtain uniform transformation during the isothermal treatment.

6.3.5.3 Polishing and etching

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

6.3.6 Bainite or gradient-quench method

NOTE Guidelines for the use of this method depending on the microstructure of the steel product are given in Annex A.

6.3.6.1 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.

6.3.6.2 Preparation

Heat the test piece 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.

6.3.7 Sensitization of austenitic stainless and manganese steels

The grain boundaries may be developed through precipitation of carbides by heating within the sensitizing temperature range, 482 °C to 704 °C (900 °F to 1 300 °F). Any suitable carbide-revealing etchant can be used.

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

6.3.8 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[2)] in these cases.

7 Characterization of grain size

7.1 Characterization by an index

7.1.1 Formulae

The index is defined in 3.2 by the formula

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

This formula may be stated as

2) Amongst these methods are the following:

— precipitation on the grain boundaries during cooling;

— gradient quenching method, etc.