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**Steels — Micrographic determination of  
the apparent grain size**

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## Foreword

This Japanese Industrial Standard has been revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by The Japan Iron and Steel Federation (JISF) with a draft being attached, based on the provision of Article 12, paragraph (1) of the Industrial Standardization Act applied mutatis mutandis pursuant to the provision of Article 16 of the said Act. This edition replaces the previous edition (**JIS G 0551:2013**), which has been technically revised.

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# Steels — Micrographic determination of the apparent grain size

## Introduction

This Japanese Industrial Standard has been prepared based on **ISO 643** : 2012, Edition 3, with some modifications of the technical contents.

The vertical lines on both sides and dotted underlines indicate changes from the corresponding International Standard. A list of modifications with the explanations is given in Annex JE. Annexes JA to JD are unique to **JIS** and not given in the corresponding International Standard.

## 1 Scope

This 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.

**NOTE 1** 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.

**NOTE 2** The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 643 : 2012 *Steels — Micrographic determination of the apparent grain size* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standards and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

**WARNING** Persons carrying out tests based on this Standard should be familiar with normal laboratory practice. This Standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this Standard to establish appropriate safety and health practices.

## 2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS G 0561 *Method of hardenability test for steel (End quenching method)*

ISO 3785 *Metallic materials — Designation of test specimen axes in relation to*

*product texture*

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

### 3 Terms and definitions

For the purpose of this Standard, 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

Grains may be austenitic or ferritic, each defined as follows.

##### 3.1.1

##### **austenitic grain**

crystal with a face-centred cubic crystal structure which may, or may not, contain an annealing twin

##### 3.1.2

##### **ferritic grain**

crystal with a body-centred cubic crystal structure which never contains annealing twins

NOTE Ferritic grain size is generally estimated for unalloyed steels with a carbon content of 0.25 % (by mass) or less or ferritic stainless steels. 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<sup>2</sup> of the section of the specimen

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

NOTE 1 By definition,  $G = 1$  where  $m = 16$ .

NOTE 2 The mean index may be expressed in units of 0.5 in the assessment by comparison with the standard grain size charts (comparison method) or expressed to one decimal place for the integrated determination.

#### 3.3

##### **intercept, $N$**

number of grains [see Figure 1 a)] intercepted by a test line, either straight or curved

NOTE Straight test lines will normally end within a grain. These end segments are counted as 1/2 an interception.  $\bar{N}$  is the average of a number of counts of the number of grains intercepted or passed 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,  $\overline{N_L}$ .

### 3.4

#### intersection, $P$

number of intersection points between grain [see Figure 1 b)] boundaries and a test line, either straight or curved

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,  $\overline{P_L}$ .

### 3.5

#### fine grain steel and coarse grain steel

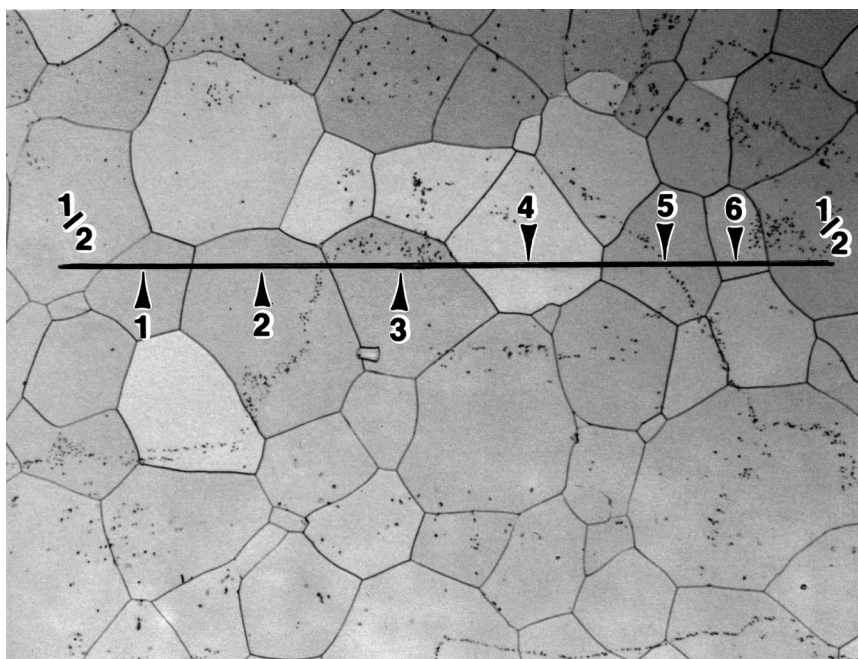
steel with an index of 5 or greater and less than 5, respectively

This distinction is made based on the test results obtained according to 6.3.2.

### 3.6

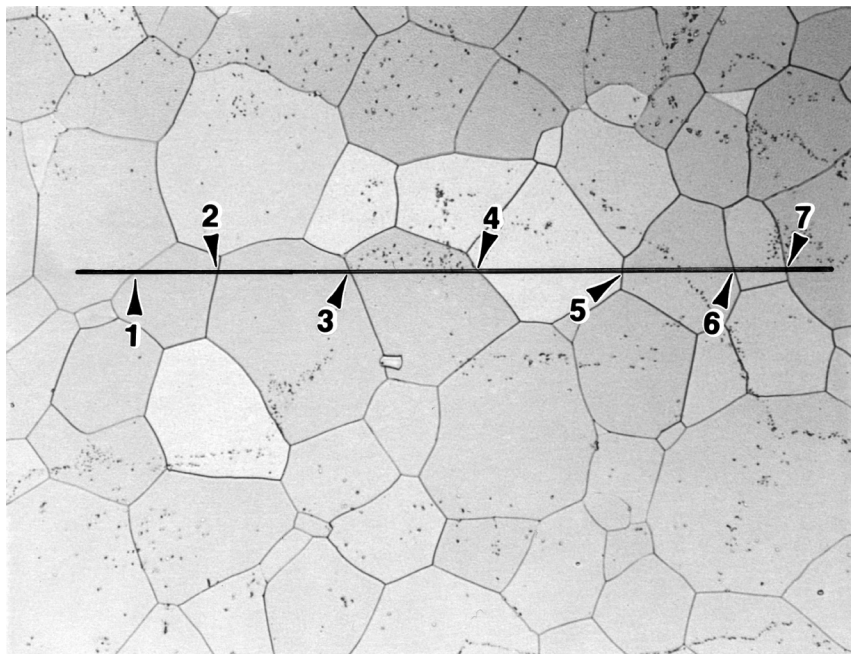
#### mixed grain

state in which grains whose index deviates by 3 or more from that of the maximum-count grains are unevenly distributed within one visual field and occupy approximately 20 % or more of the area, or state in which the index differs by 3 or more between any two different visual fields



The arrows point to 6 intercepts and two line segments ending within grain ( $2 \times 1/2 = 1$ ) and  $N = 7$ .

a) Counting of intercept,  $N$ , for straight test line over single-phase grain structure



Arrows point to 7 intersection points and  $P=7$ .

- b) Counting of intersection,  $P$ , for straight test line over single-phase grain structure

Figure 1 Examples of intercept,  $N$ , and intersection,  $P$

#### 4 Symbols

The symbols used in this Standard are given in Table 1.

Table 1 Symbols

| Symbol                                                                                             | Definition                                                                                                                                                 | Value                                                                                      |
|----------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------------------|
| $\bar{a}$                                                                                          | Mean area of grain (unit : mm <sup>2</sup> )                                                                                                               | $\bar{a} = \frac{1}{m}$                                                                    |
| $A_F$                                                                                              | Apparent area of the visual field (image on the projection screen or photomicrograph) (unit : mm <sup>2</sup> )                                            | —                                                                                          |
| $\bar{d}$                                                                                          | Mean grain diameter (unit : mm)                                                                                                                            | $\bar{d} = \frac{1}{\sqrt{m}}$                                                             |
| $D$                                                                                                | 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<br>(area = 5 000 mm <sup>2</sup> )                                                 |
| $g$                                                                                                | Linear magnification (image on the projection screen or photomicrograph)                                                                                   | In principle 100                                                                           |
| $G$                                                                                                | Equivalent index of grain size                                                                                                                             | —                                                                                          |
| $K$                                                                                                | Conversion factor from linear magnification $g \times$ to linear magnification 100 $\times$                                                                | $K = \frac{g}{100}$                                                                        |
| $l$                                                                                                | Mean lineal intercept length (unit : mm)                                                                                                                   | $l = 1/\bar{N}_L = 1/\bar{P}_L$                                                            |
| $L_T$                                                                                              | True length of the test line divided by the magnification (unit: mm)                                                                                       | —                                                                                          |
| $m$                                                                                                | Number of grains per square millimetre of test piece surface in the area examined                                                                          | $m = 2n_{100}$ (magnification 100 $\times$ )<br>$m = 2K^2 n_g$ (magnification $g \times$ ) |
| $M$                                                                                                | 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 $g \times$ )                                                 | —                                                                                          |
| $n_1$                                                                                              | 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 $\times$ )                                                 | $n_{100} = n_1 + \frac{n_2}{2}$                                                            |
| $\bar{N}$                                                                                          | Mean number of grains intercepted per unit length $L$                                                                                                      | —                                                                                          |
| $\bar{N}_L$                                                                                        | Mean number of grains intercepted per unit length of the line                                                                                              | $\bar{N}_L = \frac{\bar{N}}{L_T}$                                                          |
| $N_x$                                                                                              | Number of intercepts per millimetre in the longitudinal direction                                                                                          | —                                                                                          |
| $N_y$                                                                                              | Number of intercepts per millimetre in the transverse direction                                                                                            | —                                                                                          |
| $N_z$                                                                                              | Number of intercepts per millimetre in the perpendicular direction                                                                                         | —                                                                                          |
| $\bar{P}$                                                                                          | 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                                                                                   | $\bar{P}_L = \frac{\bar{P}}{L_T}$                                                          |
| The method for designating the directions of $N_x$ , $N_y$ and $N_z$ conforms to <b>ISO 3785</b> . |                                                                                                                                                            |                                                                                            |

NOTE **ISO 3785** was established to provide a method for designating the loca-

tion of specimen for ductility and toughness measurements in relation to the product grain flow. It designates the specimen axes making use of an X-Y-Z coordinate system for metallic products shown as follows :

- a) X axis : coincident with the main direction of grain flow (longitudinal direction)
- b) Z axis : coincident with the direction of main working force (perpendicular direction)
- c) Y axis : normal to X- and Z-axis (transverse direction)

## 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.

If the steel product standard or the agreement between the purchaser and the manufacturer does not stipulate the method of revealing the grain, the choice of this method is left to the manufacturer.

The mean aggregate size, unless otherwise specified, is characterized either by the following a) or b) at the discretion of the manufacturer. Where necessary, the method described in A.3 may be used.

- a) An index obtained by
  - 1) comparison with standard charts for the measurement of grain size (see 7.2);
  - 2) counting to determine the average number of grains per unit area (planimetric method) (see A.1).
- b) Mean value of the intercepted segment (intercept method) (see A.2)

The index is determined from this value with reference to Table A.1.

Annex JB may be used for the measurement of ferritic grain size by the intercept method.

## 6 Selection and preparation of the specimen

### 6.1 Test location

If the steel product standard or the agreement between the purchaser and the manufacturer 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.

It has been shown that precision of grain size determination increases as the higher the number of specimens are 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.).

Unless otherwise stated by the steel product standard or by agreement between the

purchaser and the manufacturer, 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 may be on a transverse plane if the grain shape is not equiaxial.

## 6.2 Revealing ferritic grain boundaries

The ferritic grains shall be revealed by etching with nital <sup>1)</sup> 2 % to 5 % (by volume), or with an appropriate reagent.

Note <sup>1)</sup> Ethanol solution containing nitric acid of specified volume fraction [(60 % to 62 % by mass), hereafter referred to as nitric acid].

## 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 glyceresia, Kalling's reagent (No. 2) and Marble's reagent.

The best electrolytic etch for single or two-phase stainless steels is 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.

NOTE Aqueous 10 % (by mass) oxalic acid, 6 V d.c., up to 60 s, is commonly used but is less effective than electrolytic nitric acid.

For other steels, one or other of the methods specified in 6.3.2 or 6.3.3 below shall be used depending on the information required.

### 6.3.2 Carburized grain size test method (McQuaid-Ehn method by carburization at 925 °C)

#### 6.3.2.1 Field of application

This method reveals austenitic grains during carburization of the steels at 925 °C for a predetermined period. It is not usually suitable for revealing grains actually formed during other heat treatments.

#### 6.3.2.2 Heat treatment

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 by agreement between the purchaser and the manufacturer shall state the treatments to be carried out before determination in cases where it is advisable to take into account these considerations.

The specimen is buried and sealed in a vessel filled with carburizing agent, and heated in an electric furnace or other suitable furnace. The furnace is heated to 925 °C in about 2 h, and the specimen is maintained at this temperature for 6 h. The specimen is then cooled at a rate slow enough to precipitate cementite at the grain boundaries in the hypereutectoid surface region of the carburized specimen. After carburizing, the

specimen should be gradually cooled to 600 °C at a rate of 30 °C/h to 150 °C/h. In most cases, a carburized layer of approximately 1 mm is obtained.

The carburizing compound shall be the mixture of dried pellet charcoal of 60 % to 80 % (by mass) and barium carbonate of 40 % to 20 % (by mass). Other mixture ratio may be used if suitable for the specific steel grade. The amount of carburizing compound used should be 30 times or more of the volume of specimen. Fresh carburizing compound shall be used each time.

### 6.3.2.3 Preparation and etching

The carburized specimen shall be sectioned normally to its surface. One of the sections shall be prepared for micrographic examination and etched using one of the following.

- a) Alkaline sodium picrate. Use electrolytic etching 6 V d.c. for 60 s where necessary.
- b) Nital, 2 % to 5 % (by volume)
- c) Picric acid-ethanol solution or other reagents may be used as long as the same results as **a)** and **b)** are obtained.

NOTE An alkaline sodium pictrate may be “le Chatelier and Igewski” reagent (picric acid 2 g, sodium hydroxide 25 g, and water 100 ml).

### 6.3.3 Heat treated grain size test method

The heat treated grain size test methods are as given in Table 2. These methods apply to the grain size measurement at highest heating temperature on heat treatment such as annealing, normalizing, quenching, solution heat treatment of steels. The details of each test method are given in Annex JA.

**Table 2 Heat treated grain size test methods**

| Test method                                                                                                                                            | Applicable steel                                                                                                                                                              | Annex            |
|--------------------------------------------------------------------------------------------------------------------------------------------------------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|------------------|
| “Bechet-Beaujard” method by etching with aqueous saturated picric acid solution                                                                        | Mainly martensite containing phosphorus of 0.005 % or more, tempered martensite or bainite steels                                                                             | See <b>JA.2.</b> |
| Pro-eutectoid ferrite method                                                                                                                           | Mainly coarse-grained carbon steels of 0.25 % to 0.6 % in carbon content and low alloy steel such as Mn-Mo steel, 1 % Cr-Mo steel, and 1.5 % Ni-Cr steel                      | See <b>JA.3.</b> |
| Sensitization heat treatment method of austenitic stainless steel and austenitic manganese steel <sup>a)</sup>                                         | Mainly unstabilized austenitic or two-phase stainless steels with a carbon content over 0.025 %.                                                                              | See <b>JA.4.</b> |
| <u>Slow cooling method</u>                                                                                                                             | Mainly hypoeutectoid steels of carbon contents more than middle class. For hypereutectoid steel, however, it is only applicable for measuring grain size over $A_{cm}$ point. | See <b>JA.5.</b> |
| <u>Quenching and tempering method</u>                                                                                                                  | Mainly carbon steels for machine structural use and alloy steels for machine structure use.                                                                                   | See <b>JA.6.</b> |
| <u>End quenching method</u>                                                                                                                            | Mainly low hardenability steels, i.e., hypoeutectoid steels and eutectoid steels of carbon contents more than middle level                                                    | See <b>JA.7.</b> |
| Oxidizing method                                                                                                                                       | Mainly carbon steels for machine structural use and alloy steels for machine structure use.                                                                                   | See <b>JA.8.</b> |
| <u>Quenching method</u>                                                                                                                                | Mainly high speed tool steels and alloy tool steels.                                                                                                                          | See <b>JA.9.</b> |
| Note <sup>a)</sup> Austenitic manganese steels will precipitate fine carbide aggregates at grain boundaries when sensitized between 482 °C and 704 °C. |                                                                                                                                                                               |                  |

## 7 Characterization of grain size

### 7.1 General

The assessment of grain size is made by : comparison with the standard grain size chart <sup>2)</sup> (see **7.2**); grain size obtained by counting the number of grains per unit surface area (see **A.1**); and the intercept method by obtaining the intercept,  $N$ , intersection,  $P$ , per a test line of 1 mm (see **A.2**).

Note <sup>2)</sup> As shown in Plate I and Plate IV given in **ASTM E112**.

The index can be obtained using Table A.1 after obtaining the mean lineal intercept length,  $l$ , obtained from the mean number of grains intercepted per millimetre,  $\overline{N_L}$ , or the mean number of grain boundary intersections per millimetre,  $\overline{P_L}$ , according to the intercept method.

The index can be defined by Formula (1) according to **3.2** :

$$m = 8 \times 2^G \quad \dots\dots\dots (1)$$

The index shall be obtained using Formula (2a) or Formula (2b) obtained from Formula (1) :

$$G = \frac{\log m}{\log 2} - 3 \quad \dots\dots\dots (2a)$$

$$G = \frac{\log m}{0.301} - 3 \quad \dots\dots\dots (2b)$$

where,  $m$  : mean number of grains per square millimetre of specimen cross-section observed

$G$  : index

## 7.2 Assessment by comparison with standard grain size charts (Comparison method)

The image examined on the screen (or on a photomicrograph) shall be compared with a series of standard charts defined in **ASTM E112** or overlays (eye-piece graticules designed for grain size measurement can be used providing these are traceable to **ASTM E112**). The standard charts at a magnification of  $100\times$  are numbered from  $-1(00)$  to 10 so that their number is equal to the index  $G$ .

**NOTE** Plate IV is normally used for carburized grain size test method (McQuaid-Ehn method by carburization at 925 °C).

The same plate of standard grain size chart should be consistently used all through measurement.

The standard grain size chart having the grain size closest to that of the examined visual fields of the specimen shall be determined. In the case of Plate IV, 0.5 shall be added to the lower index if the grain size is recognized to be the middle of indices.

A minimum of three (preferably 5 to 10) randomly-selected visual fields shall be evaluated on each specimen.

Where the magnification  $g$  of the image on the screen or photomicrograph is not  $100\times$ , 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) :

$$G = M + 6.64 \log \frac{g}{100} \quad \dots\dots\dots (3)$$

Table 3 gives the relationship between the indices for the usual magnifications.