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ISO RECOMMENDATION

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MICROGRAPHIC DETERMINATION OF THE AUSTENITIC GRAIN SIZE OF STEELS

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BRIEF HISTORY

The ISO Recommendation R 643, *Micrographic determination of the austenitic grain size of steels*, was drawn up by Technical Committee ISO/TC 17, *Steel*, the Secretariat of which is held by the British Standards Institution (BSI).

Work on this question by the Technical Committee began in 1958 and led, in 1965, to the adoption of a Draft ISO Recommendation.

In January 1966, this Draft ISO Recommendation (No. 920) was circulated to all the ISO Member Bodies for enquiry. It was approved, subject to a few modifications of an editorial nature, by the following Member Bodies:

Argentina	Germany	Poland
Australia	Hungary	Romania
Austria	India	Sweden
Belgium	Israel	Switzerland
Brazil	Italy	Turkey
Canada	Japan	U.A.R.
Chile	Korea, Rep. of	United Kingdom
Czechoslovakia	Netherlands	U.S.A.
Denmark	New Zealand	Yugoslavia
France	Norway	

No Member Body opposed the approval of the Draft.

The Draft ISO Recommendation was then submitted by correspondence to the ISO Council, which decided, in December 1967, to accept it as an ISO RECOMMENDATION.

ISO Recommendation R 643 December 1967

MICROGRAPHIC DETERMINATION OF THE AUSTENITIC GRAIN SIZE OF STEELS

1. SCOPE

1.1 This ISO Recommendation defines the methods of obtaining an estimate of the austenitic grain size of unalloyed and low alloyed * steels and gives the rules which will characterize the grain size observed.

NOTE. — The position at which the samples are taken from the product should be agreed between the purchaser and the supplier.

1.2 The average grain size is shown by micrographic examination of a polished cross-section of the sample, prepared by a method appropriate to the information required.

1.3 The index is obtained

— usually, by comparison with the standard grain size charts, or

— by count.

2. DEFINITIONS

2.1 Grain. A closed polygonal shape, with more or less curved sides, which can be revealed within the network of the flat cross-section of the sample, polished and prepared for micrographic examination.

2.2 Index. The whole positive, zero or possibly negative number G which is estimated from the *mean number* m of grains defined above, counted in an area of 1 mm² of the section of the sample. By definition, $G = 1$ where $m = 16$; the other indices are obtained by the formula

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

* This refers only to steels in which any alloying element does not exceed 5 % (*m*/*m*).

3. REVEALING OF THE AUSTENITIC GRAIN

Depending on the information required, one of the appropriate methods described below should be used:

— McQuaid Ehn Method by carburization, see clause 3.1,

— Kohn Method by light oxidation, see clause 3.2,

— Béchet Beaujard Method by concentrated picric acid etching, see clause 3.3,

— or, if need be, other methods stated either in the product specification or in the order for the specification, see clause 3.4.

3.1 McQuaid Ehn Method by carburization

This method, generally applied to carburizing steels, shows the austenitic grain boundaries formed during the carburization of the steel. In general, it is not suitable for the determination of grains actually formed during other heat treatments.

3.1.1 *Preparation.* The samples should be free from all traces of decarburization and surface oxidation. All previous treatments, whether cold, hot, mechanical or any other, can have an influence on the shape of the grain obtained; the product specification should state the treatments to be carried out before making the determination.

3.1.1.1 In general, the samples should be conveniently spaced in a carburizing chamber having a lid and filled with dry and new carburizing compound.* Usually the carburizing compound should consist principally of 60 % of charcoal in grains and 40 % (*m*/*m*) of barium carbonate ($BaCO_3$). The volume of carburizing compound used should be at least 30 times the volume of the samples to be carburized.

3.1.1.2 Carburization is achieved by maintaining the carburizing chamber at 925 °C for 8 hours. The carburizing chamber is then cooled to 600 °C at a rate depending on the type of samples, but in every case between 30 and 100 °C per hour, the rate of subsequent cooling below 600 °C having no influence on the test result.

3.1.2 *Polishing and etching.* The carburized sample should be sectioned. One of the section faces should be polished for micrographic examination. It should be etched

— either by means of a boiling alkaline solution of sodium picrate:

picric acid	2 g
caustic soda	25 g
water	100 ml

— or by nitric acid (nital):

HNO_3	3 to 5 ml
ethanol	sufficient quantity per 100 ml

* The re-use of a carburizing compound already used should be forbidden.

The use of other reagents is permitted, provided the same results are obtained.

3.1.3 *Result.* The grain boundaries of the carburized layer, which is about 1 mm thick, should consist of proeutectoid cementite.

3.2 Kohn Method by light oxidation

This method reveals the austenitic grain formed during austenitization, at the temperature of the desired heat treatment.

The application of this method on a number of samples from the same source and in various conditions makes possible the study of the grain size variation as a function of temperature and of time of austenitization.

3.2.1 *Preparation.* One face of the sample should be polished. The rest of its surface should not have traces of oxide. The sample should be placed in a laboratory tube furnace in which inert gas is circulating. It should be austenitized under the conditions of temperature and time agreed or, in the absence of such, under the conditions stated in the product specification for the treatment of test pieces for mechanical testing.

At the end of the heating time stated, air is introduced into the tube for 30 to 60 seconds.

The sample is then quenched as stated above.

3.2.2 *Polishing and etching.* The oxide which has formed and is adhering to the already polished face should be removed by light polishing with a fine abrasive, and the polishing completed in the usual manner, by etching,

— either by Villela's reagent:

picric acid	1 g
hydrochloric acid	5 ml
ethanol	100 ml

— or by Benedicks reagent:

metanitrobenzene sulphonic acid	5 ml
ethanol	100 ml

3.2.3 *Result.* The preferential oxidation of the boundaries will reveal the grain size.

Correct preparation should eliminate the appearance of oxide globules at the grain boundaries.

It may be necessary, in certain cases, for improved grain boundary delineation by the effect of relief, to use oblique illumination.

3.3 Béchet Beaujard Method by concentrated picric acid etching

This method is applicable to samples taken either from sample product treated so as to produce martensitic or bainitic structures or on portions of test pieces already used to determine the mechanical properties. It shows up the austenitic grain formed during heat treatment of this sample product or this test piece.

3.3.1 *Preparation.* No special preparations or treatments are required. If a new austenitization is carried out, this treatment should be carried out under the conditions of temperature and time stated on the order, or, where no such requirement exists, in accordance with the requirements of the product specification for the test pieces used to determine the mechanical properties (see clause 3.2.1).

3.3.2 *Polishing and etching.* One flat face of the sample should be polished for micrographic examination. It should be etched for a sufficient period by a saturated aqueous solution of picric acid with 0.5 % of sodium alkylsulphonate.

NOTE. — The time of oxidation can vary from a few minutes to more than an hour. If necessary, the procedure given in the Note to clause 3.3.3. should be followed.

3.3.3 *Result.* The boundaries of prior austenite grains appear directly in the micrographic examination.

NOTE. — Successive polishings and etching are sometimes necessary to ensure a sufficient contrast between the grain boundaries and the general form of the sample.

3.4 Other methods of preparation applicable to certain steels

For certain steels, after a simple heat treatment (annealing or normalizing, hardening, hardening and tempering, etc.), the pattern of the austenite grains may appear under micrographic examination in the following forms: a network of proeutectoid ferrite surrounding pearlite grains, a network of very fine troostite surrounding martensite grains, etc. The product specification should, in these cases, mention these simplified methods.*

4. CHARACTERIZATION OF GRAIN SIZE

4.1 Re-affirmation of definition

The index is defined in clause 2.2 by the formula

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

This formula can be stated as

$$G = \frac{\log_{10} m}{\log_{10} 2} - 3 \qquad (2)\ **$$

4.1.1 The surface image of the sample, prepared as stated above in section 3, should be examined on the ground glass screen of a microscope or a photomicrograph.

4.2 Count

4.2.1 The magnification g of the image examined should be such that at least approximately 50 grains can be counted in an area enclosed by a 79.8 mm diameter circle (area 5000 mm²) traced on the ground glass screen of a microscope (or on a photomicrograph). The number of grains completely enclosed by the circle n_1 and the number of grains intersected by the circumference n_2 should be counted.*** The number of equivalent whole grains n within the circle at the magnification used is

$$n = n_1 + \tfrac{1}{2} n_2$$

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* Of these methods, the following may be mentioned:
— martensitic hardening followed by tempering, etching by picric acid or hydrochloric acid,
— precipitation of the grain boundaries during cooling,
— vacuum heat treatment,
— gradient quench method, etc.

** In this formula $\log_{10} 2 = 0.301\ 03$.

*** The deduction can also be made on a surface limited to a square whose sides are 70.7 mm (area 5000 mm²).

The number m of grains per square millimetre of the surface of the sample is equal to

$$m = 2\left(\frac{g}{100}\right)^2 n = \gamma n \qquad (3)$$

where

g = magnification used,

γ = factor for the calculation of the number of grains per square millimetre.

The values of γ for the various magnifications are given in Table 1 below:

TABLE 1

Magnification g	Factor γ for the calculation of the number of grains per square millimetre
10	0.02
25	0.125
50	0.5
etc.	etc.

NOTES

1. If $g = 25\ D$, then $m = 12.5\ n$

2. The mean diameter of a grain is $d = \frac{1}{\sqrt{m}}$ mm (4)

   The mean area of a grain is $a = \frac{1}{m}$ mm² (5)

4.2.2 The index G is taken as equal to that which, in Table 2, page 10, corresponds to the number of grains nearest to the derived number m.*

4.3 Standard grain size charts (see pages 12, 13 and 14)

4.3.1 Standard grain size charts, Nos. I to VIII, are such that their number (in Roman figures) is equal to the index G when the magnification g is equal to 100.

The image of the sample examined on the ground glass screen of a microscope (or on a photomicrograph) of magnification g should be enclosed by a 79.8 mm diameter circle. By comparing with the charts, that chart whose grain size is nearest to that of the sample can be determined. Let M be its number. The index G is given by the following relationship:

$$G = M + \frac{2}{\log_{10} 2} \times \log_{10} \frac{g}{100} = M + 6.64 \times \log_{10} \frac{g}{100}$$

where

$$G = M + K \qquad (6)$$

The attached graph (see Annex B) shows the relationship between K and the various values of magnification g used (from $\times 10$ to $\times 1600$).

NOTES

1. *Grains of different size indices.* In certain cases, the surface examined can include grains belonging to two or more systems of indices of different sizes. This can be recognized, for example, by the presence of several grains of widely different dimensions from those of the whole. One should then make the count by dimensions and state that two or more indices occur at a certain frequency.
2. *Estimate of the index.* When the estimate is carried out by comparison or by count, the accuracy obtained is rarely greater than a half-unit. The index shown therefore is rounded off in the test report.

5. TEST REPORT

The test report should state

— the type of steel examined and the heat-treatment conditions (temperature, time, method of hardening, etc.),

— the particular method of examination,

— the index number of the grain size observed.

* This Table can be obtained by applying formulae (1), (3) and (5) above (see clauses 4.1 and 4.2.1).

ANNEX A

A.1 Certain properties of structural steels are influenced by the size of austenite grains formed by austenitization, during heat treatment.

Initially, it is often useful to know the austenite grain size of the steel:

(*a*) at a given temperature and the tendency to grain growth,

(*b*) as a function of austenitizing temperature, or

(*c*) resulting from hot working to which it is subjected.

In consequence of the transformations occurring during cooling, it is not possible in genera to observe directly on the sample examined cold the austenite grain network which existed when it was hot.

A.2 The metal grains have an irregular polyhedral form but the measurement of their mean size can be carried out in a simple manner. The established practice is to designate as " grain " the geometric figures outlining the sections of these polyhedrons on the flat surface of the section. It should be recognized that, under these conditions, the estimation of the mean grain size of the steel cannot be accurate. In fact, if the steel consists of equal grains, the areas of the observed polygons on any flat surface can take in all the values between a maximum value and zero (the statistical distribution of values of those areas can be calculated, if the form of polyhedral structure is known).

A.3 The determination of the mean austenitic grain size of a structural steel necessitates therefore two successive operations:

(1) the network of austenite grains which existed in the metal during the course of earlier austenitization on a polished section of the sample, examined cold, should be revealed. This will necessitate the employment of heating or chemical procedures;

(2) an estimation of the mean value of the area of the more or less regular polygons thus revealed.

A.3.1 This examination makes it possible to characterize the mean size of the observed grain by a conventional index.

A.3.2 This index can also be used to define the grain observable on the polished section of a sample of any steel.

A.4 Two principal methods of characterization of grain size by an index exist. The index is related to

(1) the number of grains counted in units on the surface of the section examined, or

(2) the predominating size of grain (among all the size of grain) which can be seen on the surface examined (index JKM).

The method stated above in this ISO Recommendation is based on the first of these principles.*

* It is that which is used, among others, in the French Standard A 04-102, the USA ASTM Standard E 112-63, the USSR Standard GOST 5639-51, etc.

TABLE 2

Value of the grain size indices	Nominal number of grains per square millimetre	Average area of 1 grain in square millimetres
0	8	0.125
0.5	12	0.083 3
1	16	0.062 5
1.5	24	0.041 7
2	32	0.031 2
2.5	48	0.020 8
3	64	0.015 6
3.5	96	0.010 4
4	128	0.007 81
4.5	192	0.005 2
5	256	0.003 90
5.5	384	0.002 6
6	512	0.001 95
6.5	768	0.001 3
7	1024	0.000 98
7.5	1536	0.000 65
8	2048	0.000 49
8.5	3072	0.000 325
9	4096	0.000 244

A.5 *As regards the numerical relation between various indices of grain size*, in the case of regular structures, one can establish empirically that the sum: number of index JKM + number of index ISO is between 16 and 18.

The ASTM index (E 112-63) is defined by the number of the grains p_1 counted on an area of 1 in² with a magnification of 100.

The index defined by this ISO Recommendation is related to the number of grains p_2 on an actual area per square millimetre. The ratio of numbers p_1 and p_2 is thus

$$\frac{p_1}{p_2} = \frac{(25.4)^2}{(100)^2} \qquad (7)$$

whence

$$\log_{10} p_1 = \log_{10} p_2 - 4 + 2.8096 = \log_{10} p_2 - 1.1904 \qquad (8)$$

but, the ASTM index has the value $N = \dfrac{\log_{10} p_1}{\log_{10} 2} + 1$ (9)

The ISO index is (formula (2), clause 4.1)

$$G = \frac{\log_{10} p_2}{\log_{10} 2} - 3 \qquad (10)$$

Taking $\left.\begin{aligned} \log_{10} p_1 &= (N - 1) \log_{10} 2 \\ \log_{10} p_2 &= (G + 3) \log_{10} 2 \end{aligned}\right\}$ (11)

into formula (8), one obtains

$$\log_{10} p_1 = (N - 1) \log_{10} 2 = (G + 3) \log_{10} 2 - 1.1904 \qquad (12)$$

and $$N = G + 0.0456 \qquad (13)$$

The ASTM index gives a grain slightly larger than that defined by this ISO Recommendation, but the difference is not 1/20th of an index unit. This is negligible because the estimation of grain size cannot generally be closer than ½ a unit under the most favourable conditions.

ANNEX B

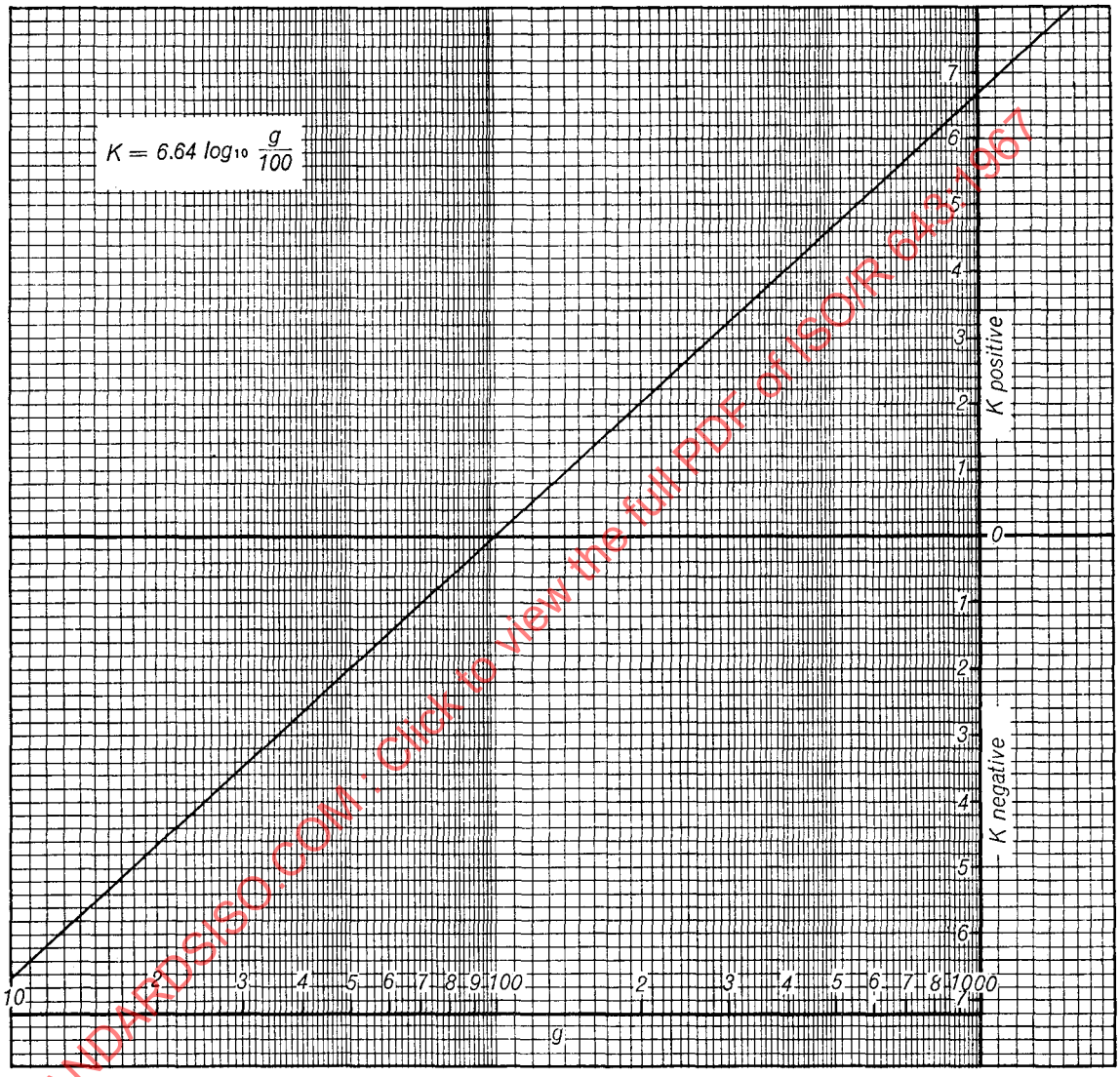

FIGURE — **Representative graph of** $K = 6.64 \log_{10} \frac{g}{100}$