

ISO

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION

ISO RECOMMENDATION

R 643

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.

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 mm^2 of the section of the sample. By definition, $G = 1$ where $m = 16$; the other indices are obtained by the formula

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

* 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.