
Determination of percentage of resolvable pearlite in high carbon steel wire rod

*Détermination du pourcentage de perlite résolvable dans les fils
machine en acier à haut carbone*

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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.

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 meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: [Foreword - Supplementary information](#)

The committee responsible for this document is ISO/TC 17, Steel, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

Introduction

This test method produces an estimate of the percentage of pearlite resolvable at 500x magnification in wire rod as a function of the cooling method after hot rolling to rod. The drawability of high-carbon pearlitic rod is influenced by the amount of resolvable pearlite present. As the percentage of resolvable pearlite increases, drawability decreases. The methods are used to check if the content of resolvable pearlite can fulfil the requirement of the wire rod with improved characteristics intended for drawing.

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Determination of percentage of resolvable pearlite in high carbon steel wire rod

1 Scope

This International Standard defines resolvable pearlite and specifies two methods of determining the percentage of resolvable pearlite.

The methods are applicable for wire rod made from control cooling steel with carbon content greater than 0,65 % C.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 9042, *Steels — Manual point counting method for statistically estimating the volume fraction of a constituent with a point grid*

ISO 16120-1, *Non-alloy steel wire rod for conversion to wire — Part 1: General requirements*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 9042 and ISO 16120-1 apply.

4 Symbols and abbreviated terms

The symbols and corresponding designations are given in [Table 1](#).

Table 1 — Symbols and designations

Symbol	Designation
o_1	the 1st measuring location
o_2	the 2nd measuring location
o_3	the 3rd measuring location
o_4	the 4th measuring location
o_i	the i^{th} measuring location, $i = 1, 2, 3$ and 4
Si_1	percentage of resolvable pearlite in the first field of view of the o_i measuring location
Si_2	percentage of resolvable pearlite in the second field of view of the o_i measuring location
Si_3	percentage of resolvable pearlite in the third field of view of the o_i measuring location
$\bar{S}$	average percentage of resolvable pearlite in the cross-section of wire rod

5 Principle

5.1 This International Standard defines resolvable pearlite as pearlite where the ferrite and iron carbide lamellae can be observed at 500x magnification under optical microscopy with 0,8 or higher numerical aperture. The light source shall typically be white light, but another light source can be used.

5.2 Resolvable pearlite is revealed by chemical etching of a polished section of the wire rod using an appropriate etchant. The choice of etchant shall refer to [6.3](#).

5.3 This International Standard specifies two methods of measuring the percentage of resolvable pearlite: manual point count method and automatic image analysis method.

5.3.1 The manual point count method: a grid with a number of regularly arrayed points, which can be a clear plastic test grid or eyepiece reticule, is superimposed over the image, or a projection of the image, produced by a light microscope, and the number of points falling within the constituent of interest are counted and divided by the total number of grid points yielding a point fraction, usually expressed as a percentage, for that field.

5.3.2 Automatic image analysis method: constituents with different microstructure are separated by grey-level intensity differences; for example, pearlites with smaller width between ferrite and iron carbide lamellae are darker than pearlites with bigger width. Thus, the measurement of the content of different constituents can be made on each field by the image analyser on the nature of the discriminated picture point elements in the image.

5.3.3 In case of a dispute, manual point count method shall be the referee method.

6 Selection and preparation of samples

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. 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.

Unless otherwise stated by the product standard or by agreement with the customer, the polished face of the specimen shall be transversal.

6.2 Grinding and polishing

The specimens should be prepared metallographically using a well-conceived method, starting with sectioning with a device that imparts minimal damage, mounting with a good resin with either a hot compression mounting press or cast resins, followed by an appropriate sequence of grinding and polishing steps, finishing with an abrasive of at least 1 μm , to yield a flat surface with minimal preparation-induced damage. After polishing, the specimen should be carefully washed with water, cleaned with alcohol and finally dried.

6.3 Etching

The following two etchants are recommended:

- a) a picral etchant, which is a solution of 4 g of picric acid in 100 ml of ethanol;
- b) a nital etchant, which is a solution of a 2 ml of nitric acid ($\rho_{20} = 1,33 \text{ g/ml}$) in 100 ml of ethanol.

NOTE Nital is orientation sensitive and can reveal the pearlite in different colonies in different contrast. Picral can reveal pearlite uniformly and can be superior to nital for most high carbon wire rod.

The polished surface is etched at ambient temperature in the etching solution for a minimum of 10 s or until the surface is etched clearly.

After etching, wash the specimen under a stream of lukewarm water to stop the etching reaction and to remove the etchant from the surface. Then, squirt ethanol onto the surface to displace the water and dry the specimen using either clean compressed air or a flow of hot air from a device similar to a hair dryer or a hand dryer. If bleed out occurs during drying, it may be necessary to clean the mounted specimen in an ultrasonic cleaner.

7 Evaluation of the percentage of resolvable pearlite

7.1 Parameters of optical microscope

The etched surface shall be observed with an optical microscope using the following conditions:

- a) magnification: 500x;
- b) numerical aperture: 0,8 or higher;
- c) light source: white or another light source.

7.2 Determination of measuring field of view

7.2.1 Determination of measuring locations

The measuring locations of the cross-section of wire rod are defined as the following. Four measuring locations, which are designated as 01, 02, 03 and 04, are equally spaced in the circle being $D/4$ away from the centre of wire rod (mid-radius, as shown in [Figure 1](#)).

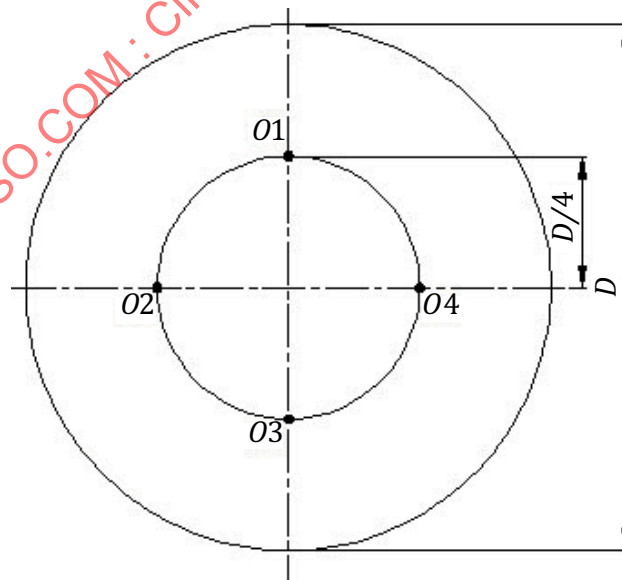


Figure 1 — The schematic diagram of measuring locations

7.2.2 Determination of measuring fields of view

On each measuring locations O_i ($i = 1, 2, 3$ and 4), three continuous fields of view are taken to measure the percentage of resolvable pearlite and recorded as $Si1, Si2, Si3$.