INTERNATIONAL
STANDARD

ISO
3887

Third edition
2017-11

Steels — Determination of the depth of decarburization

Aciers — Détermination de la profondeur de décarburation

Reference number
ISO 3887:2017(E)

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Foreword

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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 voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document 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 3887:2003), which has been technically revised. The following changes have been made:

- the scope has been expanded from “non-alloy and low-alloy steels” to “steel products”;
- the definitions of “partial decarburization” and “complete decarburization” have been modified;
- the term “depth of ferrite decarburization” has been deleted;
- the terms “depth profile of carbon content” and “depth profile of hardness” have been added;
- more measurement details for the micro-indentation hardness method have been added;
- two new methods of measuring the carbon depth profile, by GDOES and EPMA, have been added;
- examples of typical decarburization microstructures have been added.

Steels — Determination of the depth of decarburization

1 Scope

This document defines the decarburization and specifies three methods of measuring the depth of decarburization of steel products.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 4545-1, *Metallic materials —Knoop hardness test — Part 1: Test method*

ISO 6507-1, *Metallic materials — Vickers hardness test — Part 1: Test method*

ISO 9556, *Steel and iron — Determination of total carbon content — Infrared absorption method after combustion in an induction furnace*

ISO 14594, *Microbeam analysis — Electron probe microanalysis — Guidelines for the determination of experimental parameters for wavelength dispersive spectroscopy*

ISO 14707, *Surface chemical analysis — Glow discharge optical emission spectrometry (GD-OES) — Introduction to use*

ISO 15349-2, *Unalloyed steel — Determination of low carbon content — Part 2: Infrared absorption method after combustion in an induction furnace (with preheating)*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at https://www.iso.org/obp

3.1
decarburization
reduction of carbon content from the surface zone of the steel

Note 1 to entry: A distinction is made between

a) partial decarburization, d_3, measured as the distance from the point at which the carbon content exceeds the solubility limit in ferrite, becoming visible as e.g. pearlite, to the point at which there is no more visible difference to the core carbon concentration, and

b) complete decarburization, also called ferrite decarburization, d_1, measured as the distance between the surface of the product and the point up to which the carbon content is below the solubility limit of carbon in ferrite so that only ferrite is present.

Note 2 to entry: The depth of complete decarburization as described in b) is determined by examination of the microstructure.

3.2
depth of functional decarburization
d_2
distance between the surface of the product and the point at which the carbon content or hardness is at the level where the performance of the product would be unaffected by a reduction in carbon content (i.e. at the minimum level specified in the product standard)

3.3
depth of total decarburization
d_4
distance between the surface of the product and the point at which the carbon content is that of the unaffected core, the sum of the partial and the complete decarburization $d_3 + d_1$being designated by the letters DD

EXAMPLE DD = 0,08 mm.

Note 1 to entry: Expressed in millimetres.

3.4
depth profile of carbon content
curve indicating the relationship between the perpendicular distance from the surface of steel material and the carbon content

3.5
depth profile of hardness
curve indicating the relationship between the perpendicular distance from the surface of steel material and the hardness

4 Sampling

Samples should be taken at locations that are representative of the bulk specimen. The location and number of samples taken depend on the nature of the material to be tested and are defined by agreement between the parties concerned.

Specimens for the metallographic methods or micro-indentation hardness tests or electron probe microanalysis should be cut from the bulk specimen perpendicular to the longitudinal axis of the product so that measurements are made on a transverse plane. This procedure permits the determination of the variation of decarburization around the periphery of the specimen. Unless otherwise specified, the corner parts which can generate abnormal decarburization shall not be included. For specimens up to about 2,5 cm diameter, the entire cross-section is polished and examined. For larger cross-sections, one or more specimens shall be prepared to assess variations in surface decarburization. The sampling scheme for large sections should be determined by agreement between the parties concerned.

Specimens for chemical analytical methods shall be of sufficient length so that the weight of incremental turnings is adequate for chemical analysis or the size of milled surfaces is large enough for sparking yet small enough to fit in the specimen holder.

5 Measuring methods

5.1 General

The choice of the method and its accuracy depend on the degree of decarburization, the microstructure, the carbon content of the product examined and the shape of the component.

The usual methods employed on finished products are as follows:

- metallographic method (see 5.2);

- method for measuring the micro-indentation hardness (Vickers or Knoop) for steels in the hardened or quenched and tempered condition (see 5.3);
- method for the determination of the carbon content by chemical analysis (see 5.4.2), spectrographic analysis (see 5.4.3), electron probe microanalysis (EPMA) (see 5.4.5), or glow discharge optical emission spectrometry (GDOES) (see 5.4.6).

The sample shall be examined in the as-delivered condition. Nevertheless if, by agreement between the parties concerned, a supplementary heat treatment is applied, every precaution shall be taken to prevent changes in mass percentage and/or in the distribution of carbon, e.g. a small sample, a short austenitization time, a neutral atmosphere.

The measuring method shall be agreed upon in writing by the parties concerned, unless it is defined in the product standard. In the absence of any agreement or requirement in a product standard, the metallographic method should be used.

5.2 Metallographic method

5.2.1 General

Unless otherwise specified, this method shall only be applied in situations where changes in the carbon content are reflected by resulting variations in microstructure.

This method is especially valid for steels showing an annealed, normalized, as-rolled or as-forged structure. It may apply, with reservations, for products showing a hardened or tempered structure where the interpretation of the structural variations becomes difficult.

5.2.2 Selection and preparation of the sample

The metallographic polishing, carried out by applying the usual methods, shall not round the edges. In order to achieve this, the sample may be mounted or held in a clamp, and the surface of the product may, if necessary, be protected by a metallic deposit obtained by electroless or electrolytic plating. Automatic/semi-automatic preparation techniques should be used, where possible.

Etching in a solution of 1,5 % to 4 % nitric acid in ethanol (nital) or 2 % to 5 % picric acid in ethanol(picral) will reveal the structure of the steel.

5.2.3 Measurement

As a rule, the reduction in the carbon content can be determined for the following:

a) ferrite and pearlite: from the decrease in the amount of pearlite;

b) pearlite and hypereutectoidally developed carbides: from the decrease in the amount of hypereutectoidally developed carbides and/or of pearlite;

c) ferrite matrix with dispersed carbides: from the decrease in the amount of carbides in the ferrite matrix.

This method can also be applied for assessing changes in the microstructure if the carbon content leads to clear changes in the microstructure, e.g. for hardened or quenched and tempered microstructures, but only if a distinct boundary which is decisive for the depth of decarburization exists within the characteristic structure. Examples of typical decarburization microstructure are given in Annex A.

The distance from the surface to the point at which the structure does not differ from that of the core shall be measured (total decarburization). The measurement shall be conducted using suitably calibrated equipment.

The choice of magnification depends on the depth of decarburization and shall be chosen by the assessor unless otherwise agreed between the parties concerned. The maximum magnification that allows the full extent of decarburization to be viewed should be adopted. A magnification of × 100 is recommended as a useful magnification for the majority of instances.

A preliminary examination of the whole surface at low magnification ensures that any great variation in the depth of decarburization along the periphery will be observed for further evaluation.

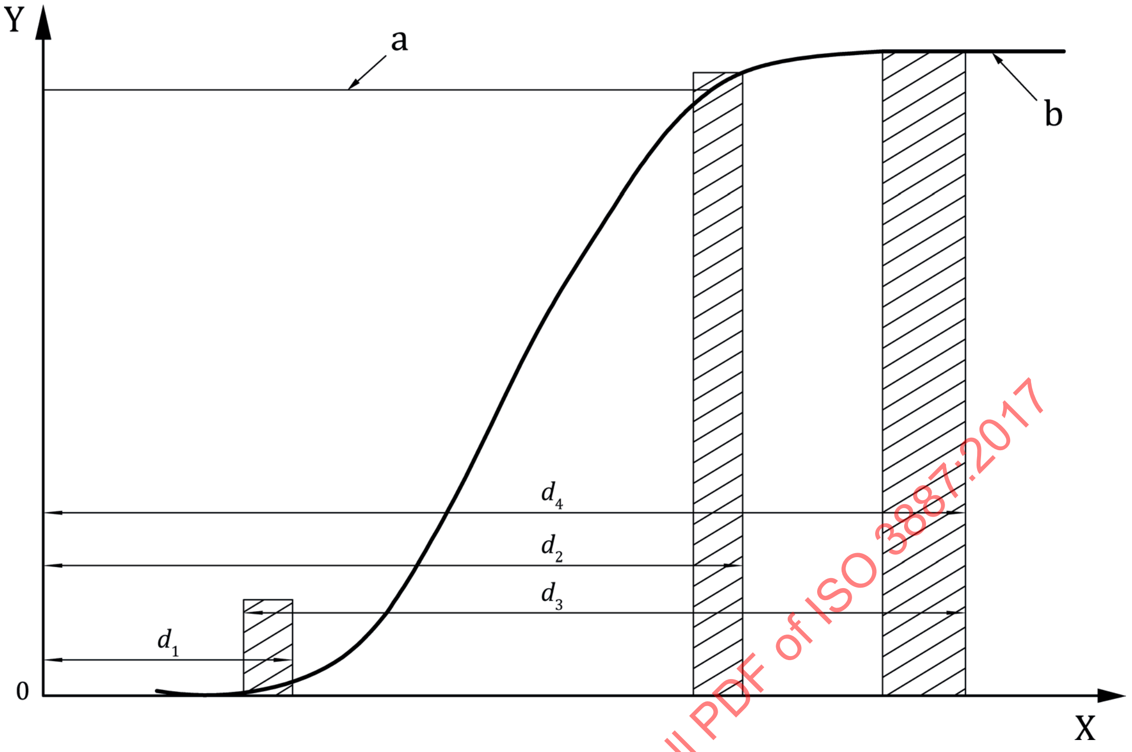
The deepest uniform decarburization zone is selected from the preliminary examination of the surface of the section. One of two methods of measurement should be used. The choice of the measurement method shall be in accordance with the agreement between the parties concerned.

- The worst field method is the simplest method and is adequate for many purposes. One measurement of the depth of decarburization is conducted and reported at the deepest uniform decarburization zone.
- The average method is the alternative method. Beginning at the deepest uniform decarburization zone, the first measurement point, the surface is divided into parts of equal size, at the ends of which the depth of decarburization is also measured. Unless otherwise agreed, four individual measured values are determined. The depth of total decarburization of the sample is defined as the average of these measurements. Measuring points that are affected by surface defects are not taken into account when determining the average.

The various bands of decarburization are shown schematically in Figure 1. The boundaries separating the various types of decarburization are shown as hatched bands with the width of the band illustrating the practical variability in measurements due to the uncertainty of interpretation.

If the product has undergone a process involving carburization, the definition of the "core" shall form the subject of an agreement between the parties concerned.

The permissible depth of decarburization shall be specified in the appropriate standard covering the product or shall be the subject of an agreement between the parties concerned.



Key

a	Minimum carbon content specified in the product standard.	d_1	complete decarburization
b	Core carbon content.	d_2	functional decarburization
X	distance from surface	d_3	partial decarburization
Y	carbon content	d_4	total decarburization

NOTE The depth of complete decarburization as described in d_1 is determined by examination of the microstructure.

Figure 1 — Depth profile of carbon content: schematic representation for a typical decarburized steel

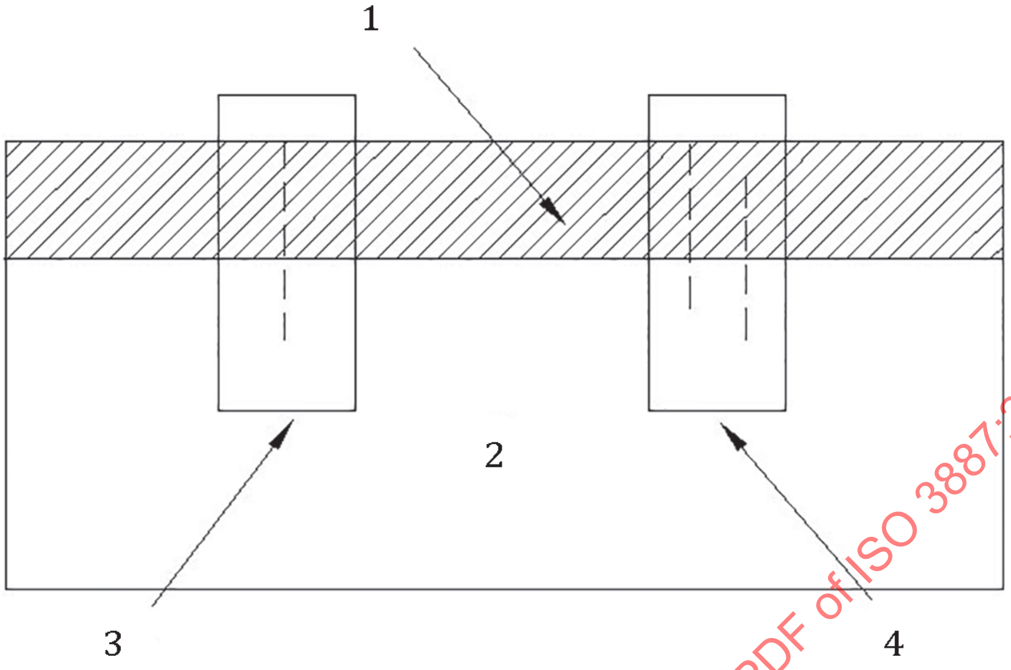
5.3 Methods for measuring the micro-indentation hardness

5.3.1 General

The methods under consideration are that of Vickers, in accordance with ISO 6507-1, and of Knoop, in accordance with ISO 4545-1.

Each method consists of determining the depth profile of the micro-indentation hardness on a cross-section of the product along a line perpendicular (see Figure 2) or oblique (see Figure 3) to the surface. The perpendicular line is convenient for the measurement of large and medium depths of decarburization and the oblique line for medium and small depths. In the case of the perpendicular line, finer measuring intervals can be obtained if a zigzag line is used.

This technique applies only to hypoeutectoid steels in the hardened, tempered or heat-treated condition, and to decarburized zones that are within a hardened zone, in order to avoid the occurrence of variations in hardness due to an imperfect penetration. The technique becomes inaccurate for low-carbon steels.

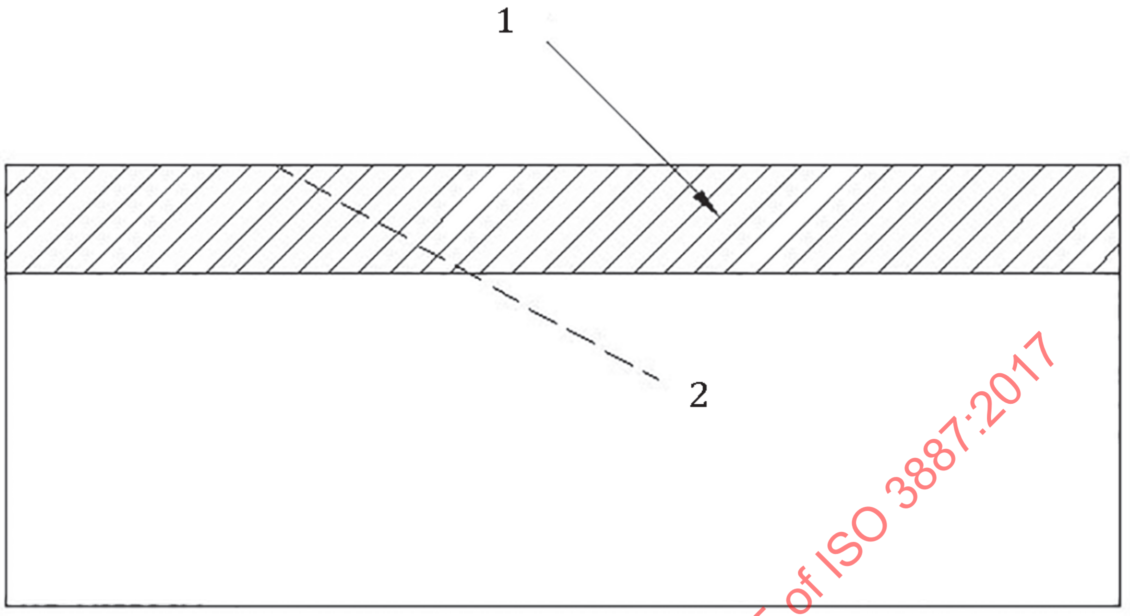


Key

1 decarburization
2 core material
3 one straight line
4 zigzag line

NOTE The dashed lines indicate the measurement lines.

Figure 2 — Schema of measurement on a cross-section of the product along a line perpendicular to the surface



Key

1 decarburization

2 core material

NOTE The dashed line indicates the measurement line.

Figure 3 — Schema of measurement on a cross-section of the product along a line oblique to the surface

5.3.2 Selection and preparation of the sample

The selection and preparation of the sample shall be identical to that used in the metallographic method (see 5.2.2) although, in general, the sample shall not be etched, in order to facilitate the measurement of the size of the impression.

5.3.3 Measurement

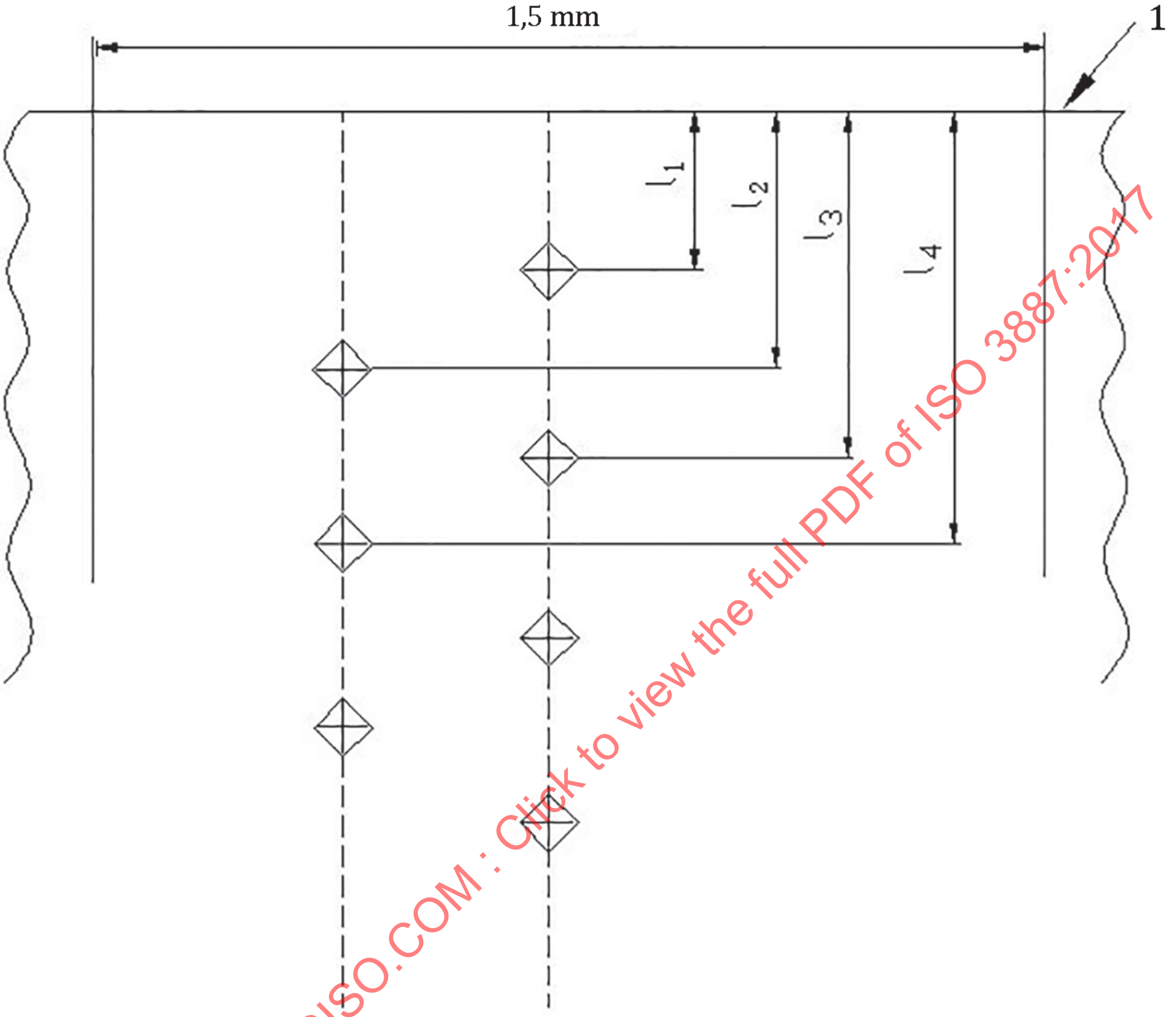
On the test plane, define the location of the line perpendicular or oblique to the surface, measure the Vickers hardness or Knoop hardness consecutively along the line perpendicular or oblique to the surface and draw the depth profile of hardness.

The load shall be as high as possible, in order to minimize the scatter of the measurements. The intervals between the measuring points at the time of making the depth profile of hardness by Vickers hardness test or by Knoop hardness test shall be 0,1 mm or smaller. The distance of the centres of two adjacent impressions shall meet the separation criteria in ISO 6507-1 or ISO 4545-1.

In the case of using a zigzag line, 2 to 5 points are chosen as starting points. These points shall lie within a band not wider than 1,5 mm while being sufficiently spaced to meet the separation criteria in ISO 6507-1 or ISO 4545-1. The hardness measurements are carried out along the lines drawn perpendicular to the surface through the starting points and the hardness values of all lines are combined into a single depth profile of hardness (see Figure 4).

The depth of total decarburization is defined by the distance from the surface to the point at which the core hardness is attained on one depth profile of hardness. In the case the core hardness is not obtainable, a previous agreement between the parties concerned is necessary.

The depth of functional decarburization is expressed with the distance from the surface to the point at which the required hardness is attained on one depth profile of hardness. However, it may depend on whether the required hardness is obtainable at the point of the specified depth. When judging the product to be acceptable or not by the depth of functional decarburization, a previous agreement between the parties concerned is necessary.



Key

1 surface

l_i measurement distance of indentation i, i = 1,2,3 and 4

NOTE The dashed lines indicate the measurement lines.

Figure 4 — Arrangement of hardness measuring points (zigzag method)

The depth of total decarburization and the depth of functional decarburization may be determined as averages of at least four depth profiles of hardness, in accordance with the agreement between the parties concerned. These depth profiles of hardness shall be recorded at locations as distant from each other as possible.

If the depth of decarburization cannot be determined by hardness test, a quench hardening treatment may be given, in accordance with an agreement between the parties concerned, under conditions not varying the depth of decarburization.

5.4 Methods of determination of the carbon content

5.4.1 General

The methods described in 5.4.2, 5.4.3, 5.4.5 and 5.4.6 determine the gradient of the carbon content in a direction perpendicular to the surface. They are applicable whatever the structure of the steel.

5.4.2 Chemical analysis

5.4.2.1 General

Chemical analysis applies only to products with a simple geometry (round base cylinder or plain faced polyhedron) and of a size suitable for machining facilities, and when decarburization is over the complete surface.

5.4.2.2 Selection of samples and test

Successive layers 0,1 mm thick, parallel to the surface of the piece, shall be removed by dry machining, avoiding any contamination. Any oxide films shall be removed beforehand.

The metal collected at each level shall be submitted for carbon determination by chemical means, in accordance with ISO 9556 and ISO 15349-2.

5.4.3 Spectrographic analysis

5.4.3.1 General

Spectrographic analysis applies only to products with flat faces of adequate size.

5.4.3.2 Selection of samples and test

The flat sample shall be subjected to successive grinding operations to different levels 0,1 mm apart. A spectrographic determination of the carbon shall be carried out at each level in such a way that successive sparkings are not superimposed.

5.4.4 Interpretation of the results (chemical and spectrographic methods)

The methods described in 5.4.2 and 5.4.3 allow the determination of the depth of functional decarburization, by measuring the distance from the surface to the point where the carbon content reaches the minimum specified value. Alternatively, the depth of total decarburization can be determined by measuring the distance from the surface to the point where the carbon content values become consistent, i.e. the core carbon content.

5.4.5 Electron probe microanalysis (EPMA)

5.4.5.1 General

The method under consideration shall be in accordance with ISO 14594.

This method is especially valid for hardened or tempered steel products with a single phase structure. It may apply, with reservations, for products showing a structure with more than one phase, where the interpretation of the carbon content variations becomes difficult.

5.4.5.2 Preparation of the sample

The preparation of the sample shall be identical to that used in the metallographic method (see 5.2.2) although, in general, the sample shall not be etched, in order to facilitate the measurement of the carbon content.

5.4.5.3 Measurement

The carbon content is subjected to a linear analysis by means of the electron probe microanalysis (EPMA) applied perpendicularly to the test plane. The depth profile of carbon content is drawn from the surface of the decarburization to the point where the carbon content of the core material is obtained. The depth of decarburization is determined from this profile.

The depth of total decarburization can be determined by measuring the distance from the surface to the point where the carbon content values become consistent, i.e. the core carbon content.

The depth of total decarburization may be obtained as the average of the values obtained, in accordance with the agreement between the parties concerned, from at least four depth profiles of carbon content drawn at locations as distant as possible.

5.4.6 Glow discharge optical emission spectrometry (GDOES)

5.4.6.1 General

The method under consideration shall be in accordance with ISO 14707.

This applies only to products with flat faces of adequate size and with a depth of decarburization smaller than 100 µm. The size of the test samples should be suitable for the glow discharge source used. Typically, round or rectangular samples with sizes (diameter, width and/or length) of 20 mm to 100 mm are suitable.

5.4.6.2 Preparation of the samples

Rinse the surface of the sample with an appropriate solvent (high purity acetone or ethanol) to remove oils or any residues. Blow the surface dry with a stream of inert gas (argon or nitrogen) or clean, oil-free, compressed air being careful not to touch the surface with the gas delivery tube. The wetted surface may be lightly wiped with a wetted soft, lint free cloth or paper to facilitate the removal. After wiping, flush the surface with solvent and dry as described above.

5.4.6.3 Measurement

A stream of argon ions mills the material from the sample surface. The sputtered material is then excited in a low pressure plasma discharge and the resulting light emission is used to characterize and quantify the sample's composition. The depth profile of carbon content is drawn from the surface of the decarburization to the point where the carbon content of the core material is obtained. The depth of decarburization is determined from this profile.

The depth of total decarburization may be obtained by measuring the distance from the surface to the point where the carbon content values become consistent, i.e. the core carbon content.

The depth of total decarburization may be obtained as the average of the values obtained, in accordance with the agreement between the parties concerned, from at least two depth profiles of carbon content drawn at locations as distant as possible.

6 Test report

The test report shall contain the following information:

a) a reference to this document and its year of publication, i.e. ISO 3887:2017;