INTERNATIONAL STANDARD

ISO 18203

First edition
2016-12-01

Steel — Determination of the thickness of surface-hardened layers

Acier — Détermination de l'épaisseur des couches durcies superficielles

Reference number
ISO 18203:2016(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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The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This first edition of ISO 18203 cancels and replaces ISO 2639:2002, ISO 3754:1976 and ISO 4970:1979, which have been technically revised.

Introduction

In the past, there are three ISO standards for measuring surface-hardened layer. Because those standards employed almost the same principle of measuring, it is intended to make it easy for maintenance of the standards and application of test by integrating these three standards.

The method of estimating uncertainty of measurement is not included in this document. In future revision, uncertainty of measurement may be reflected based on real applications to this test.

Steel — Determination of the thickness of surface-hardened layers

1 Scope

This document specifies a method of measuring the case hardening depth, surface hardening depth, nitriding hardness depth and total thickness of surface hardening depth obtained, e.g. thermal (flame and induction hardening, electron beam hardening, laser beam hardening, etc.) or thermochemical (carbonitriding, carburizing and hardening, hardening and nitriding, etc.) treatment.

NOTE Surface-hardened layer can be produced by mechanical method (shot blasting, shot peening, etc.). The depth of these layers is generally shallow. Measuring a profile of hardened depth may require lower test force of hardness test.

2 Normative reference

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 4545-2, *Metallic materials — Knoop hardness test — Part 2: Verification and calibration of testing machines*

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

ISO 6507-2, *Metallic materials — Vickers hardness test — Part 2: Verification and calibration of testing machines*

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 http://www.iso.org/obp

3.1
case hardening depth
CHD
perpendicular distance between the surface and the layer having a hardness of 550 HV in accordance with ISO 6507-1 or equivalent Knoop hardness in accordance with ISO 4545-1

Note 1 to entry: For steels which present a hardness greater than 450 HV at a distance of three times the case hardening depth (determined with a limiting hardness value of 550 HV) from the surface, a limiting hardness value greater than 550 HV, in steps of 25 units, can be selected for the determination of the case hardening depth by agreement between interested parties.

Note 2 to entry: In general, case hardening consists of carburizing or carbonitriding followed by quench hardening (see ISO 4885).

3.2
surface hardening depth
SHD
distance between the surface and the layer where HV is equal to the value specified by the term "hardness limit"

Note 1 to entry: Hardness limit is a function of the minimum surface hardness required for the part, given by the following formula:

hardness limit (HV) = A × minimum surface hardness (HV)

Note 2 to entry: 0,80 is usually used for A. Other values can be applied.

Note 3 to entry: SHD measurement should be applied for parts which, in the surface hardened condition, have a hardness less than (hardness limit – 100 HV) at a distance 3 × SHD from the surface. Where these conditions are not satisfied, the definition of SHD should be agreed between interested parties.

Note 4 to entry: Recommended hardness limit values are listed in Table 1.

Table 1 — Recommended hardness limit values of SHD

Minimum surface hardness HV	**Hardness limit** HV
300 to 330	250
335 to 355	275
360 to 385	300
390 to 420	325
425 to 455	350
460 to 480	375
485 to 515	400
520 to 545	425
550 to 575	450
580 to 605	475
610 to 635	500
640 to 665	525
670 to 705	550
710 to 730	575
735 to 765	600
770 to 795	625
800 to 835	650
840 to 865	675

3.3
compound layer thickness
CLT
thickness of the surface layer formed during the thermochemical treatment and made up of the chemical compounds formed by the elements introduced during the treatment and certain elements from the base metal

3.4
nitriding hardness depth
NHD
distance from the surface of nitride layer to the point where the material hardness limit is 50 HV above core hardness

Note 1 to entry: The core hardness is determined by at least three hardness measurements and rounded to the nearest multiple of 10 HV.

3.5
total thickness of surface hardening depth
THD
distance from the examined surface to the limit that shows the same hardness as the matrix hardness

Note 1 to entry: The distance is often measured by the microstructural method, which is the distance from the surface examined to the limit beyond which no visible structure variation is detected, as compared with the structure of the unaffected metal. The microstructural method does not measure as deep as the hardness method.

4 Symbols, abbreviations and designations

The symbols and abbreviations used in this document and their corresponding designations are given in Table 2.

Table 2 — Symbols, abbreviations and designations

Symbol/ abbreviation	Unit	Designation
di	mm	distance of the centre of each impression from the surface
Δd	mm	distance of the centre of two adjacent impression
CHD	mm	case hardening depth
SHD	mm	surface hardening depth
CLT	µm	compound layer thickness
NHD	mm	nitriding hardness depth
THD	mm	total thickness of surface hardening depth
H_{limit}	HV	hardness limit

5 Principle

The case hardening depth, surface hardening depth and nitriding hardness depth are determined from the gradient of hardness on a cross-section normal to the surface.

They are derived graphically from a curve representing the variation in hardness as a function of the distance from the surface of the part.

The total thickness of surface hardening depth is determined from the structure variation observed by micrographic method or by measuring hardness variation.

Compound layer thickness is determined from the chemical compound layer observed by microscopic method.

6 Apparatus

The hardness testing machine for determining Vickers hardness shall be verified and calibrated in accordance with ISO 6507-2.

By agreement between the parties concerned, measurement of the Knoop hardness may be used. The hardness testing machine for determining Knoop hardness shall be verified and calibrated in accordance with ISO 4545-2.

For measuring the total thickness of surface hardening depth, it is recommended that a microscope that can display the hardened layer as 1/3 to 2/3 of the view field should be used (see ISO 1463).

7 Test specimen

7.1 Selection and preparation of samples

The measurement shall be made, unless otherwise specially agreed, on a cross-section of the part in the following specified condition:

— a section perpendicular to the longitudinal axis of the product, or, if the product has no longitudinal axis, a section perpendicular to the surface at a location to be agreed between the parties concerned;

For thin hardened layers, the following sample can be applied by agreement between interested parties.

NOTE Because stepped test piece and oblique test piece may result in a different outcome from cross-section measurements, using lower test force including microhardness test to cross-section measurement is a better alternative (see ISO 6507-1, ISO 4545-1 and ISO 14577-1).

— a test piece with steps: These steps are precision-ground from the surface of the product to the point where the structure is that of the basis metal, and are 0,05 mm or 0,10 mm thick. The stepped test piece is to be used when a limiting value for the thickness of the surface layer is specified (see Figure 1 and Figure A.2);

— an oblique section (see Figure 2).

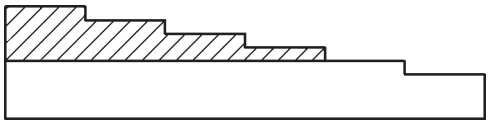
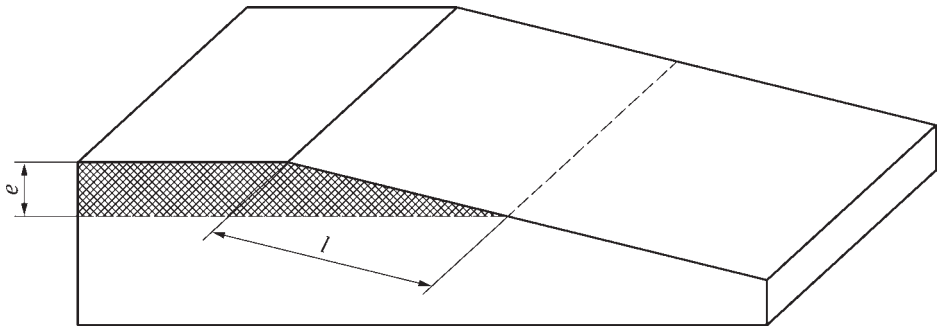


Figure 1 — Stepped test piece



Key

l distance measured

e actual depth

NOTE The values of the distance measured should be adjusted by the slope (*e*/*l*).

Figure 2 — Oblique section

7.2 Preparation of the surface to be examined

Cut specimens for preparation with the gentlest possible technique, using only laboratory equipment. Mount specimens with a high-quality resin designed to provide excellent edge retention. Grind and polish using a proper sequence of abrasives to remove the damage from sectioning and grinding while maintaining edge retention. Exercise care to ensure that the region of interest is not altered by the sample preparation. The lower the test load in hardness testing, the higher the preparation quality must be. After polishing, etch the specimen with an appropriate solution to determine if the preparation was adequate and to see if there is a modification to the surface. Repeat one or more of the final preparation steps, if the quality is inadequate. Carefully clean off any films or residue on the surface. Do not touch the surface with fingers. For microindentation hardness testing, it is recommended to test an as-polished, non-etched surface. For the micrographic method, use the appropriate etch to reveal and discriminate between the different constituents present.

8 Method of measurement

8.1 Hardness testing method

Make hardness impressions along one or more parallel lines normal to the surface and within a band of width, *W*, of 1,5 mm (see Figure 3). The distance between these lines shall meet the requirement of ISO 6507-1.

The distance, Δd, separating two adjacent impressions shall be not less than three times their diagonal (see Figure 3). The difference between the successive distances of each impression from the surface (e.g. $d_2 - d_1$) shall not exceed 0,1 mm and the cumulative distances from the surface shall be measured to an accuracy of ±25 µm. The diagonals of the impression shall be measured with the accuracy specified in ISO 6507-2.

The centre of the first impression shall be at a distance, d_1, from the surface of at least 2,5 times its diagonal.

Tests using Vickers or Knoop indents are performed at forces from 0,980 7 N to 9,807 N. Measurements of the impressions shall be made with a high quality light optical microscope and proper illumination, with or without a camera system (and possible by image analysis), at a high enough magnification so that the diagonals are between 25 % and 75 % of the eyepiece or screen width or height and the indent tips can be focused without distortion.

NOTE Due to the spacing of indents shown in Figure 3, test forces of 0,980 7 N to 2,942 N are normally used for this work.

Make the measurements on the prepared surface in two or more bands, the location of which shall be agreed between the parties concerned, and for each band plot the results in order to obtain the curve representing the variations in hardness as a function of distance from the surface.

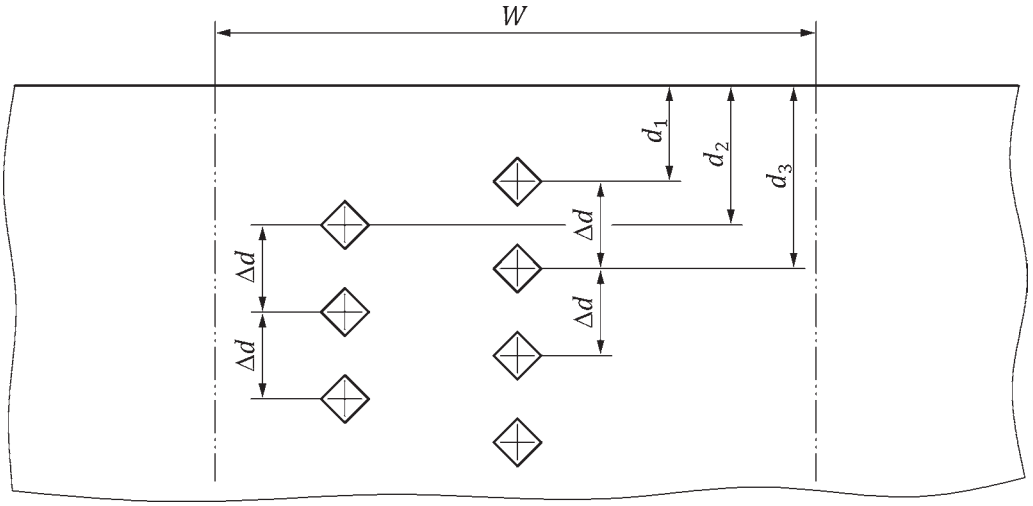


Figure 3 — Position of hardness impressions

8.2 Microscopic methods

8.2.1 Total thickness of surface hardening depth (THD)

Examine the surface of the hardened part with the specimen etched with the best reagent for discrimination between the constituents present. Scan parallel to the surface at low magnification to get an overall impression of the uniformity of the total thickness of surface hardening layer. If it is non-uniform, measure the depths to what appears to be the minimum and maximum total thickness of surface layer. If the total thickness of surface hardening layer appears to be reasonably uniform, make at least five measurements of the total thickness of surface hardening layer (spaced apart from each other) and determine the average depth.

NOTE 1 For some specimens, it may be possible to visually estimate the location where the microstructure is 50 % martensite and 50 % other constituents, such as ferrite, pearlite and/or bainite. If so, measure the depth to 50 % martensite in at least five locations (spaced apart from each other) and determine the average hardened depth.

NOTE 2 Microstructural measurement of the surface hardening depth, that is, the depth to some % of martensite (such as 80 %), may be quite difficult to do without making quantitative measurements of the microstructure by lineal analysis on lines parallel to the surface at known depths.

8.2.2 Compound layer thickness (CLT)

The compound layer thickness shall be determined from a sample cut perpendicular to the surface layer. When preparing the sample for the metallographic evaluation, special caution is needed to avoid damaging the layer. The layer can be protected during grinding and polishing, for example with a mechanical screen or with an electrolytic deposit.

The determination should be carried out at a magnification that displays the layer as 1/3 to 2/3 of the view field.

It is recommended to make 10 measurements within 5 mm band and average (see Annex B).[5]

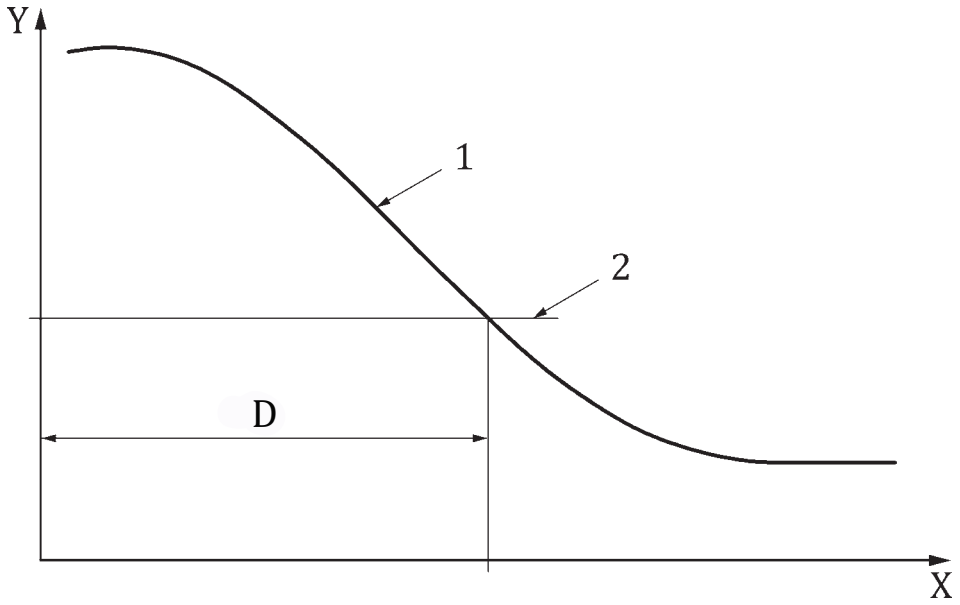
9 Evaluation of the results

9.1 Case hardening depth (CHD), surface hardening depth (SHD) and nitriding hardness depth (NHD)

In accordance with the measuring method defined in 8.1, the distance from the surface of the test piece to the layer/point which the hardness is equal to the limit hardness as defined in Clause 3. This distance corresponds to the case hardening depth (CHD), surface hardening depth (SHD) or nitriding hardness depth (NHD), respectively (see Figure 4).

If the thickness of the carburized case is specified, the case hardening depth can be determined using the method specified in Annex A.

NOTE Measurement uncertainty analysis is useful for identifying major sources of measuring results. A paper for estimation of the uncertainty of nitriding hardness depth determination is available.[6]



Key

1	hardness profile	X	distance from the surface
2	hardness limit defined in Clause 3	Y	hardness
D	depth of hardened layer defined in Clause 3		

Figure 4 — Hardness profile for determining the hardening depth

9.2 Total thickness of surface hardening depth (THD)

The average depth of the total thickness of surface hardening layer shall be calculated from at least five measurements.

9.3 Compound layer thickness (CLT)

The average depth of CLT shall be calculated from individual measurements. The number of measurements shall be agreed between interested parties.

10 Test report

The test report shall contain at least the following information unless otherwise agreed by the parties concerned:

a) a reference to this document, i.e. ISO 18203:2016;

b) method of surface hardening;

c) method of measuring (hardness test method, test force, if micrographic method is applied, the etchant, magnification, spacing between parallel lines, etc.);

d) result of case hardening depth (CHD), surface hardening depth (SHD), nitriding hardness depth (NHD), total thickness of surface hardening depth (THD) or compound layer thickness (CLT), for example:

   1) CHD,

   CHD = … mm (limiting hardness value is 550 HV)

   CHD 600 HV1 = … mm (limiting hardness value is 600 HV, measured by HV1)

   CHD HK0,3 = … mm (limiting hardness value is 550 HV, measured by HK0,3)

   2) SHD,

   SHD 450 = … mm (hardness limit is 450 HV)

   SHD 450 HV1 = … mm (hardness limit is 450 HV, measured by HV1)

   3) NHD,

   NHD 400 HV0,3 = … mm (core hardness is 350 HV, measured by HV0,3)

   NHD 300 HK = … mm (core hardness is 250 HK, measured by HK)

   4) THD,

   THD = … mm

   5) CLT,

   CLT = … µm

   NOTE In certain cases, CLT can be zero.

e) sample name, identification number, tested position, etc.;

f) any incidents occurring during measurement.

Annex A
(normative)

Interpolation method for determining the case hardening depth

A.1 General

If the thickness of the carburized case is specified, the following interpolation method may be used to determine the case hardening depth. This is possible because the hardness gradient may be approximately represented by a straight line in that transitional area where the case hardening depth, as defined in this document, would end.

A.2 Interpolation method for determining the case hardening depth

Make at least five impressions on a normal cross-section of the part under test, at each of the distances d_1 and d_2 from the surface, distance d_1 and d_2 being respectively below and above the value for the specified case hardening depth (see Figure A.1). The value of $d_2 - d_1$ shall not exceed 0,3 mm.

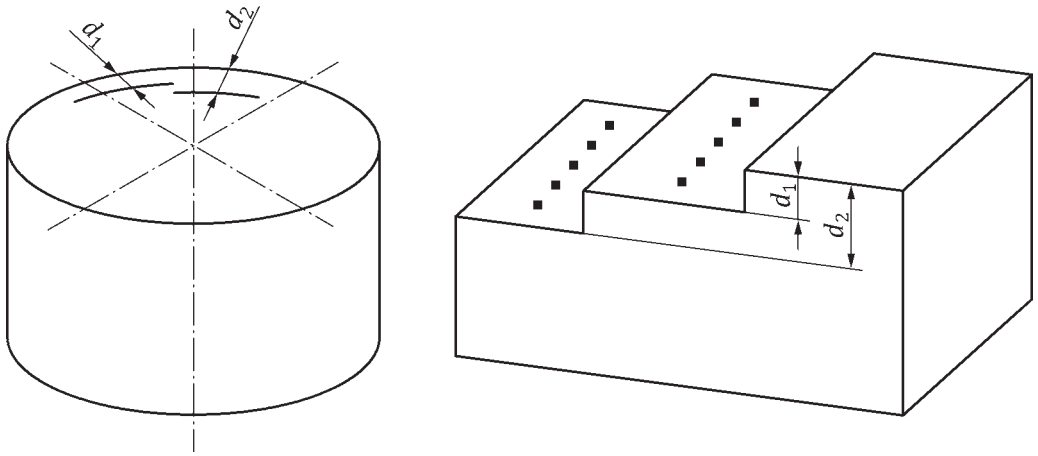
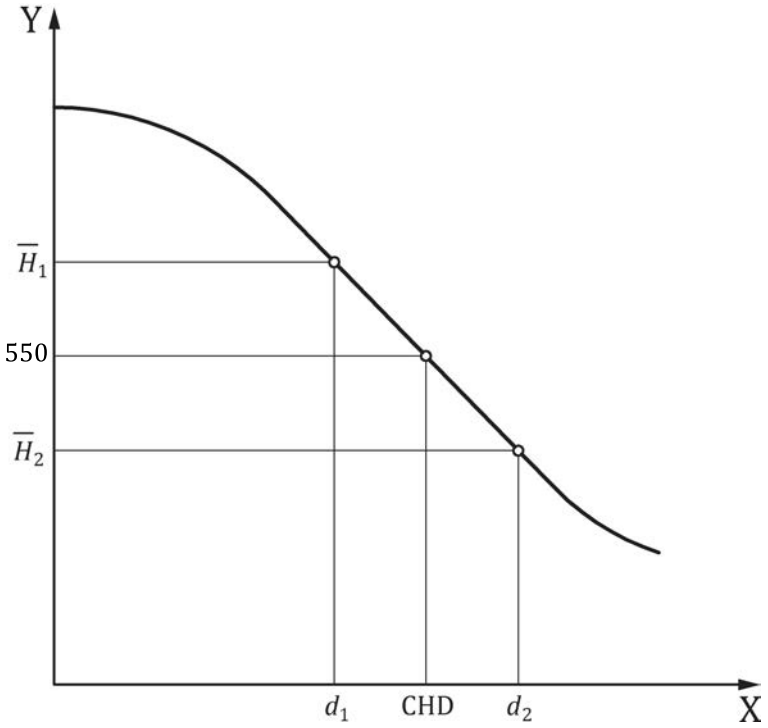


Figure A.1 — Position of hardness measurement points

The case hardening depth is given by Formula (A.1):

$$\mathrm{CHD} = d_1 + \frac{\left(d_2 - d_1\right)\left(\overline{H_1} - 550\right)}{\overline{H_1} - \overline{H_2}} \tag{A.1}$$

where $\overline{H_1}$, $\overline{H_2}$ are the arithmetic means of the hardness values measured at distances d_1 and d_2 (see Figure A.2).



Key

$\overline{H_1}$ arithmetic mean of the hardness values at distance d_1

$\overline{H_2}$ arithmetic mean of the hardness values at distance d_2

X distance from the surface, expressed in mm

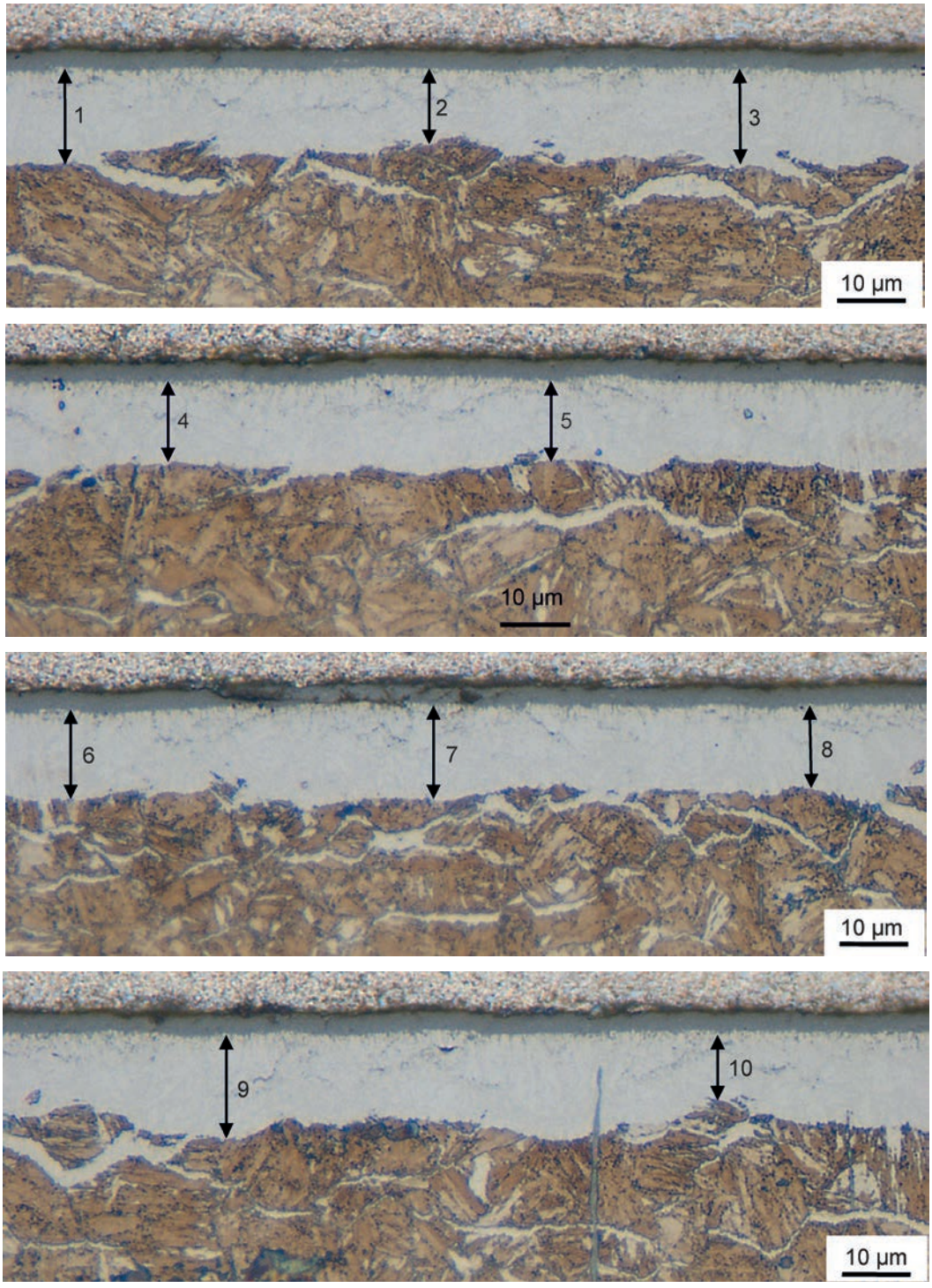
Y Vickers hardness or equivalent Knoop hardness, HV or HK

Figure A.2 — Interpolation method for determining the case hardening depth

NOTE When using the interpolation method, it is good practice to check the hardness immediately below the surface. If there is excessive retained austenite in the subsurface case, the hardness in this area may be below the critical level of 550 HV.

Annex B
(informative)

Examples of CLT measurements



NOTE See Reference [5].

Figure B.1 — Examples of CLT measurements

Bibliography

[1] ISO 1463, *Metallic and oxide coatings — Measurement of coating thickness — Microscopical method*

[2] ISO 4885, *Ferrous products — Heat treatments — Vocabulary*

[3] ISO 14577-1, *Metallic materials — Instrumented indentation test for hardness and materials parameters — Part 1: Test method*

[4] ISO 15787, *Technical product documentation — Heat-treated ferrous parts — Presentation and indications*

[5] DIN 30902, *Light-microscopical determination of the depth and porosity of the compound layer of nitrided and nitro-carburized ferrous parts*

[6] St. Stücklin, "Schätzung der Messunsicherheit bei Härtetiefenbestimmungen", Prakt. Met. Sonderband 46 (2014)

ICS 77.040.99

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