



Standard Test Methods for Estimating the Depth of Decarburization of Steel Specimens¹

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This standard has been approved for use by agencies of the U.S. Department of Defense.

INTRODUCTION

These test methods may be used to estimate the average or greatest depth of decarburization in hardened or non-hardened steel products. The test methods described range from simple screening tests to more statistically rigorous test methods depending upon the needs of the investigation.

1. Scope

1.1 These test methods cover procedures for estimating the depth of decarburization of steels irrespective of the composition, matrix microstructure, or section shape. The following basic procedures may be used:

- 1.1.1 Screening methods.
- 1.1.2 Microscopical methods.
- 1.1.3 Microindentation hardness methods.
- 1.1.4 Chemical analysis methods.

1.2 In case of a dispute, the rigorous quantitative or lineal analysis method (see 7.3.5 and 7.3.6) shall be the referee method. These methods can be employed with any cross-sectional shape. The chemical analytical methods generally reveal a greater depth of decarburization than the microscopical methods but are limited to certain simple shapes and by availability of equipment. These techniques are generally reserved for research studies. The microindentation hardness method is suitable for accurate measurements of hardened structures with relatively homogeneous microstructures.

1.3 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

¹ These test methods are under the jurisdiction of ASTM Committee E04 on Metallography and are the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

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2. Referenced Documents

2.1 ASTM Standards:²

- A941 Terminology Relating to Steel, Stainless Steel, Related Alloys, and Ferroalloys
- E3 Guide for Preparation of Metallographic Specimens
- E7 Terminology Relating to Metallography
- E340 Test Method for Macroetching Metals and Alloys
- E350 Test Methods for Chemical Analysis of Carbon Steel, Low-Alloy Steel, Silicon Electrical Steel, Ingot Iron, and Wrought Iron
- E384 Test Method for Knoop and Vickers Hardness of Materials
- E407 Practice for Microetching Metals and Alloys
- E415 Test Method for Analysis of Carbon and Low-Alloy Steel by Spark Atomic Emission Spectrometry
- E1951 Guide for Calibrating Reticles and Light Microscope Magnifications

3. Terminology

3.1 Definitions:

3.1.1 For definitions of terms used in these test methods, see Terminology E7 and Terminology A941.

3.2 Definitions of Terms Specific to This Standard:

3.2.1 *average depth of decarburization*—the mean value of five or more measurements of the total depth of decarburization.

3.2.2 *average free-ferrite depth*—the mean value of five or more measurements of the depth of complete decarburization.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

3.2.3 *complete decarburization*—loss of carbon content at the surface of a steel specimen to a level below the solubility limit of carbon in ferrite so that only ferrite is present.

3.2.4 *free-ferrite depth*—the perpendicular distance from the surface of the specimen to that location where the structure is no longer fully ferritic, that is, other transformation products are observed.

NOTE 1—The term *free ferrite* has also been used to describe globular, isolated grains of proeutectoid ferrite in the microstructure of medium-carbon hypoeutectoid steels.

3.2.5 *maximum depth of decarburization*—the largest measured value of the total depth of decarburization.

3.2.6 *partial decarburization*—loss of carbon content at the surface of a steel specimen to a level less than the bulk carbon content of the unaffected interior but greater than the room temperature solubility limit of carbon in ferrite.

3.2.7 *total depth of decarburization*—the perpendicular distance from the specimen surface to that location in the interior where the bulk carbon content is reached; that is, the sum of the depths of complete and partial decarburization.

4. Summary of Test Method

4.1 These test methods are designed to detect changes in the microstructure, hardness, or carbon content at the surface of steel sections due to decarburization. The depth of decarburization is determined as the depth where a uniform microstructure, hardness, or carbon content, typical of the interior of the specimen, is observed.

5. Significance and Use

5.1 These test methods are used to detect surface losses in carbon content due to heating at elevated temperatures, as in hot working or heat treatment.

5.2 Results of such tests may be used to qualify material for shipment according to agreed upon guidelines between purchaser and manufacturer, for guidance as to machining allowances, or to assess the influence of processing upon decarburization tendency.

5.3 Screening tests are simple, fast, low-cost tests designed to separate non-decarburized samples from those with appreciable decarburization. Based on the results of such tests, the other procedures may be utilized as applicable.

5.4 Microscopical tests require a metallographically polished cross section to permit reasonably accurate determination of the depth and nature of the decarburization present. Several methods may be employed for estimation of the depth of decarburization. The statistical accuracy of each varies with the amount of effort expended.

5.5 Microindentation hardness methods are employed on polished cross sections and are most suitable for hardened specimens with reasonably uniform microstructures. This procedure can be used to define the depth to a specific minimum hardness or the depth to a uniform hardness.

5.6 Chemical analytical methods are limited to specimens with simple, uniform shapes and are based on analysis of incremental turnings or after milling at fixed increments.

5.7 Microscopical tests are generally satisfactory for determining the suitability of material for intended use, specification acceptance, manufacturing control, development, or research.

6. Sampling

6.1 Samples should be taken at locations that are representative of the bulk specimen. The location and number of samples taken depends on the nature of the material to be tested and will be defined upon agreements between manufacturer and purchaser.

6.2 Specimens for screening tests using bulk hardness tests, such as the Rockwell test, should be small enough so that they can be properly supported on the anvil of the tester. The specimen surface should not be altered except for scale removal (if present) using a method that will not alter the subsurface metal.

6.3 Specimens for the microscopical methods or for micro-indentation hardness tests or for macroscopic screening methods 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 determination of the variation of decarburization around the periphery of the specimen.

6.3.1 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. Figs. 1-3 show examples of typical sampling schemes that may be used for larger sections; the sampling scheme for large sections should be determined upon mutual agreement between manufacturer and purchaser.

6.4 Specimens for chemical analytical methods must 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.

7. Procedure

7.1 Screening Methods:

7.1.1 *Bulk Surface Hardness*—For hardened specimens, particularly those in the as-quenched condition, a short section of the material to be heat treated is cut and heat treated in the same manner, or along with, the material of interest. The test specimen, however is not tempered. Any scale on the test piece is removed by wire brushing, glass-bead blasting, etc., and hardness tested, usually with the Rockwell C scale. The presence of decarburization is indicated by the difference between the surface hardness and the theoretical maximum hardness for the carbon content of the steel. This method is most suitable for those steels with bulk carbon contents below about 0.55 % carbon but will detect gross decarburization in steels with higher bulk carbon contents. The method is not suitable for steels that cannot be quench-hardened, for example, low-carbon steels.

7.1.2 *Macroscopical Etch Appearance*—The presence of decarburization is indicated by a difference in etching contrast between the surface and the interior of the specimen. A transverse section can be ground and macroetched or polished

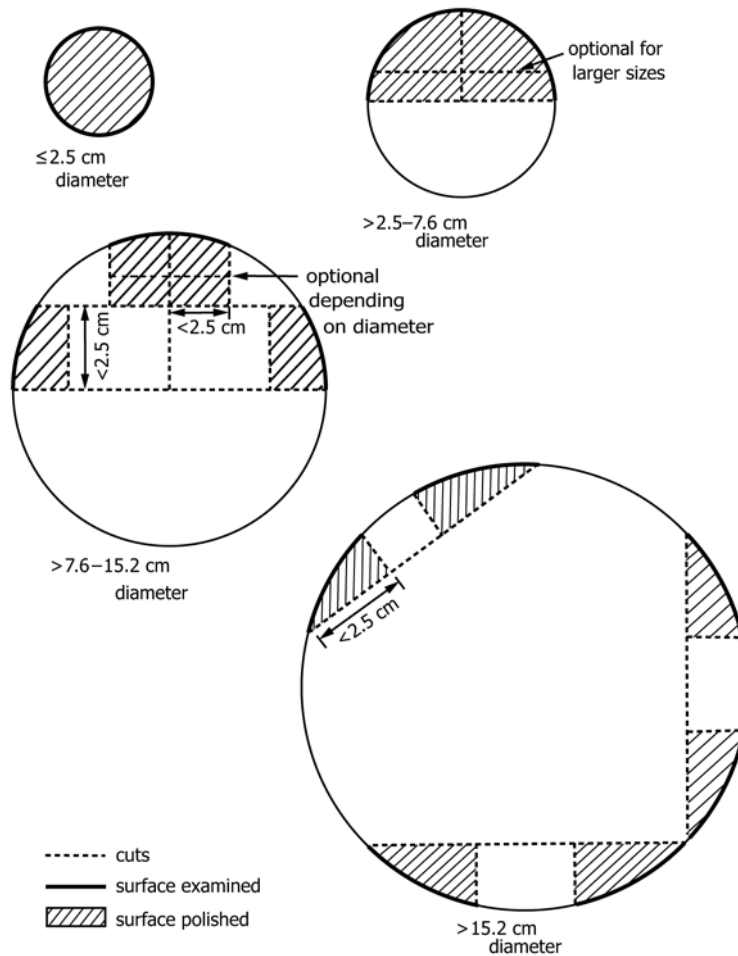


FIG. 1 Typical Sampling Schemes for Round Bars of Different Size

and microetched. The method is suitable for as-rolled, as-forged, annealed, normalized, or heat-treated specimens. The decarburized surface layer, if present, usually exhibits a light-etching appearance. Suitable macroetchants are listed in Test Method E340.

7.2 Microscopical Methods:

7.2.1 Microscopical methods are most suitable for measuring the depth of decarburization of as-hot rolled, as-forged, annealed, or normalized specimens. These methods can also be applied to heat-treated specimens, although with less certainty in determining the maximum affected depth. Spheroidize-annealed or cold-worked specimens can also be evaluated; but, detection of structural variations due to decarburization is more difficult than with hot-worked or fully annealed structures.

7.2.2 Measurement of the depth of decarburization is based on evaluation of the variation in microstructure at the surface due to the change in carbon content. The depth of complete decarburization is easiest to assess due to the excellent contrast between the free-ferrite layer, when present, and the interior structure. The depth of partial decarburization can best be assessed when this zone contains ferrite and pearlite. If the specimen has been spheroidized, the variation in carbide content in the partially decarburized zone is used to assess the total depth of decarburization. For heat-treated specimens, the

presence of non-martensitic structures in the partially decarburized zone is used to estimate the total depth of decarburization. Such measurements will generally underestimate the total depth of decarburization. For certain highalloy spheroidize-annealed tool steels, the depth of decarburization can be estimated by changes in the etch color. For austenitic manganese steels in the solution-annealed condition, depths corresponding to certain carbon contents can be defined by changes in the microstructure due to decarburization. Examples of decarburization for as-rolled, heat treated, and spheroidize-annealed steels are shown in Figs. 4-9, respectively.

7.2.3 Specimen polishing must be conducted in a manner that does not produce edge rounding. Unmounted, unprotected specimens can be satisfactorily prepared using certain automatic polishing devices. Low-nap cloths should be employed; polishing with abrasives finer than 1- μ m diamond is often unnecessary. When such devices are not available, or when specimens are small or of an inconvenient shape for such devices, specimens should be mounted in clamps or in various plastic media. With some mounting media, edge preservation may be inadequate. The compression mounting epoxy materials generally provide the best edge retention of the commonly available plastics. Electrolytic or electroless plating provides

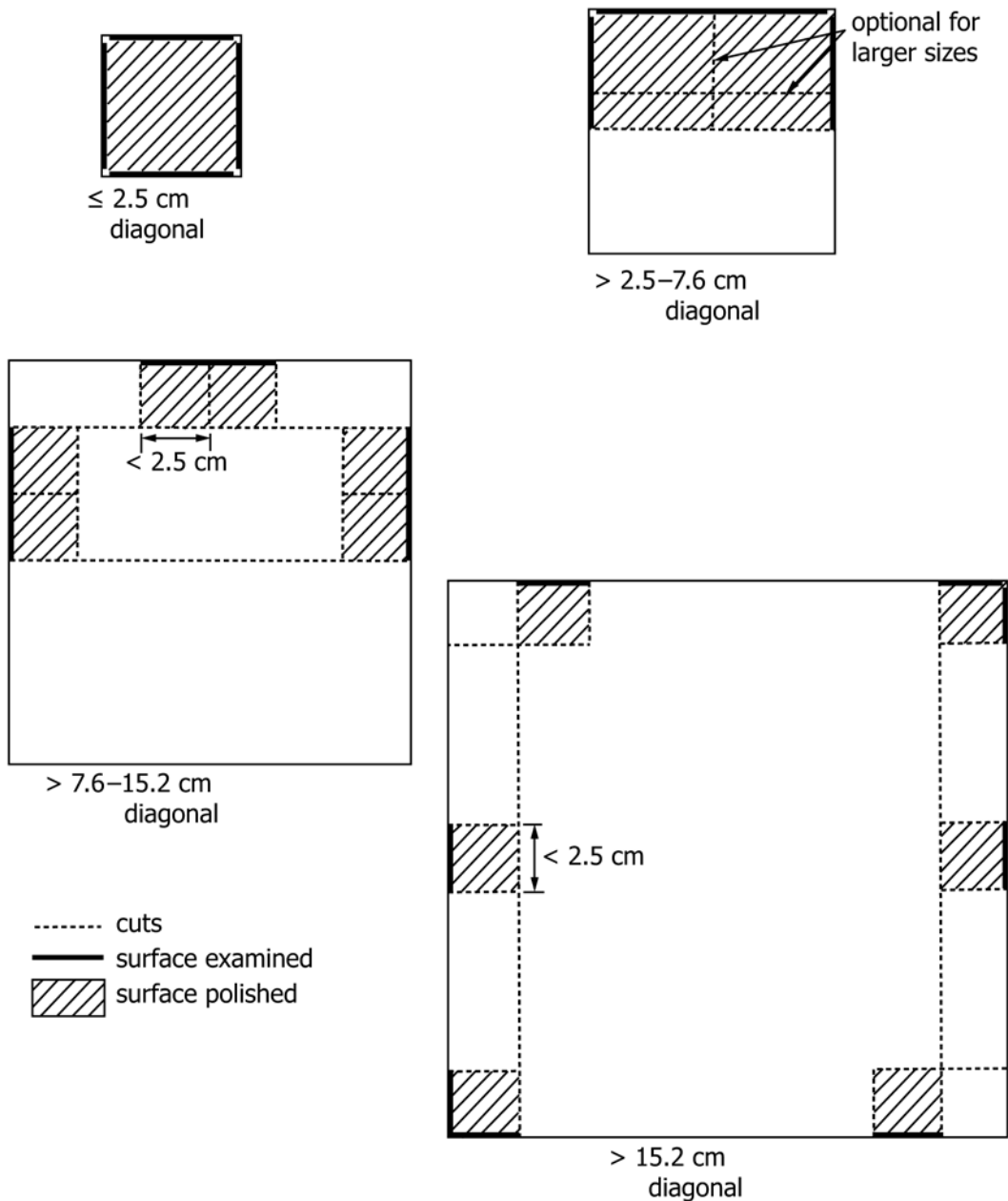


FIG. 2 Typical Sampling Schemes for Square Bars of Different Size

optimum edge retention and is recommended for critical work. Polishing must be practiced using techniques that produce a true representation of the surface microstructure, as described in Guide E3.

7.2.4 Etching should be conducted using standard etchants, (see Test Methods E407) such as nital or picral, based on the experience of the rater with the material being tested. Special etchants can be used if dictated by the situation encountered. In such cases, agreement should be obtained between manufacturer and purchaser.

7.2.5 For solution-annealed austenitic manganese steels, epsilon martensite will be present in the surface region where the carbon content is below about 0.5 % carbon. This structure is best revealed by etching first with 2 % nital for 5 s and then with 20 % aqueous sodium metabisulphite for about 20 s. After

measurement of the depth of this layer, the specimen can be aged at about 560°C for 1 h to precipitate pearlite at the grain boundaries in the core region where the carbon content is above 1.16 %. Etching with nital or picral will reveal the pearlite.^{3, 4} Figs. 10 and 11 illustrates these conditions.

7.3 Measurement:

7.3.1 The depth of complete or partial decarburization, or both, can be assessed in a variety of ways depending on the

³ Sedriks, A. J., and Mulhearn, T. O., "Austenitic Manganese Steel: Structure and Properties of Decarburized Layer," *Journal of the Iron and Steel Institute*, Vol 202, November 1964, pp. 907-911.

⁴ Sedriks, A. J., "Austenitic Manganese Steel: A Technique for Measuring the Extent of Decarburization," *Journal of the Iron and Steel Institute*, Vol 204, February 1966, pp. 142-145.

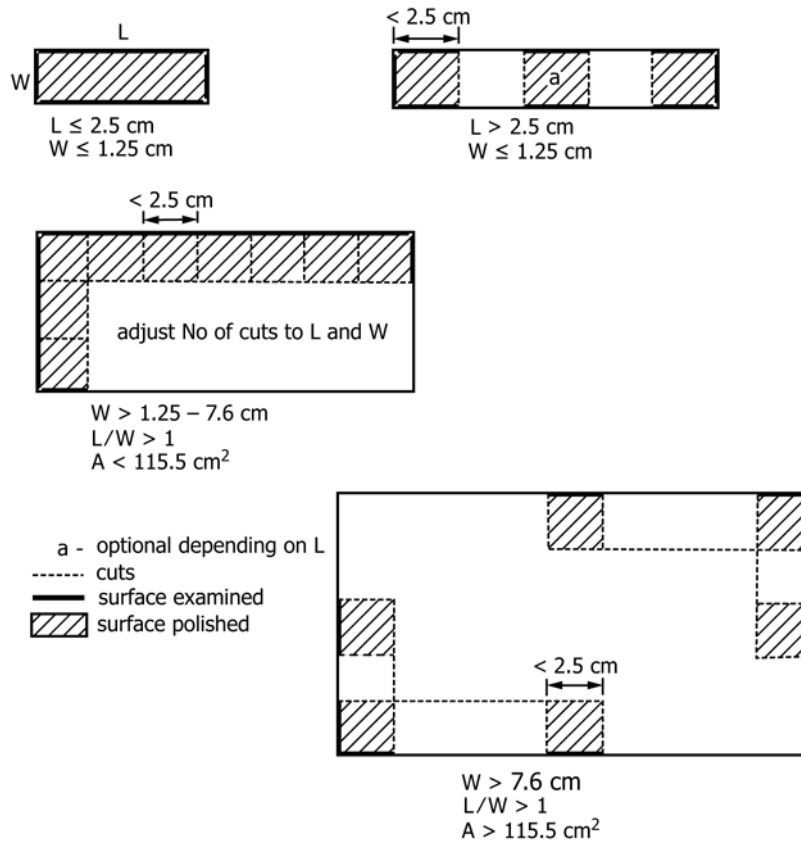


FIG. 3 Typical Sampling Schemes for Flat and Rectangular Bars of Different Size

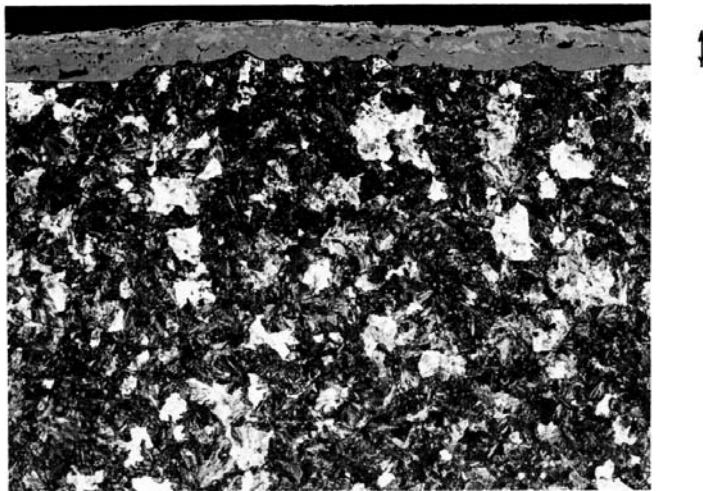


FIG. 4 Example of an As-Rolled, Fully Pearlitic Alloy Steel Microstructure With No Apparent Decarburization. Dark Layer at Surface Is Iron Oxide (Mill Scale) (200x, 2 % Nital Etch)

desired accuracy. Measurements can be made using an ocular micrometer reticle, a screw (Filar) micrometer ocular, or with a scale placed against a ground-glass projection screen. Measurements can be made on the image or on photographs. Measurements can also be made using image analysis techniques. The accuracy of the measurement device should be established using a stage micrometer. Light microscope calibration procedures can be found in Guide E1951.

7.3.2 The optimum magnification for measurement must be chosen based on the observed structure. It may be helpful to scan the specimen at low magnification for the measurement. The magnification used should be high enough to permit adequate resolution of the structure and achieve the desired measurement precision and avoid bias.

7.3.3 Before performing the measurement, scan the entire specimen surface at a suitable magnification(s) to ensure that

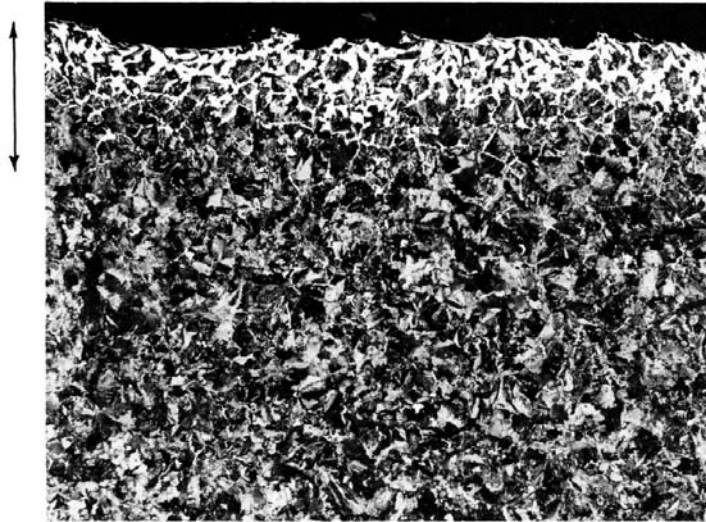


FIG. 5 Example of Partial Decarburization (Note Ferrite At and Near the Surface) in an As-Rolled, Fully Pearlitic Alloy Steel Microstructure (200x, 2 % Nital Etch)

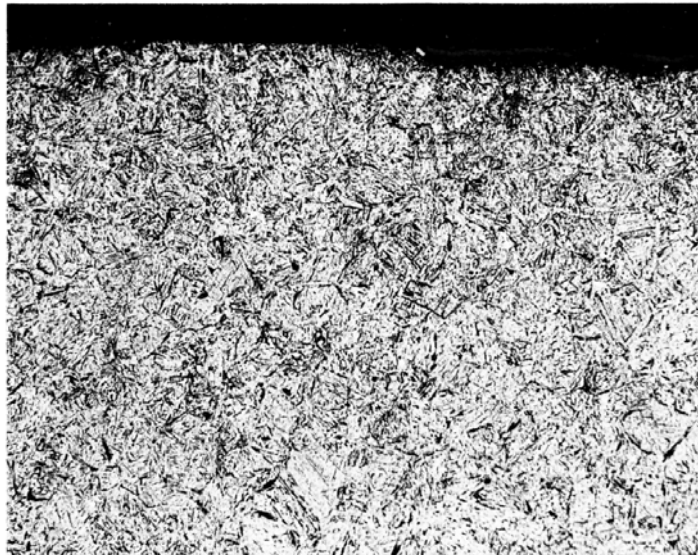


FIG. 6 Example of a Heat-Treated Alloy Steel Microstructure (Fully Martensitic) With No Apparent Decarburization (200x, 2 % Nital Etch)

the specimen has been properly prepared, to evaluate the uniformity of the decarburization present, and to assess the nature of the decarburization present, that is, complete and partial or only partial. If complete decarburization (free ferrite) is present, note the continuity of this condition.

7.3.4 The simplest measurement procedure, adequate for many purposes, is to select for measurement those locations on the specimen that appear to represent average and worst conditions of complete and partial decarburization. Measurement of these conditions is made using the devices in 7.3.1. For section shapes that contain corners, decarburization at such locations is not measured unless required. In such cases, it is reported as a separate measurement.

7.3.5 For quality control or research studies, the average depth of decarburization, either complete, partial or total, may be desired but with greater statistical precision. For such cases, the decarburization condition can be measured with greater statistical definition by making a number of measurements around the specimen periphery at randomly selected locations. The mean, standard deviation, and 95 % confidence limits of such measurements can be calculated. The number of measurements required depends on the size of the specimen and the desired accuracy.

7.3.6 For research studies, the lineal analysis method may be employed to determine the depth at any location where the microstructure becomes uniform and representative of the

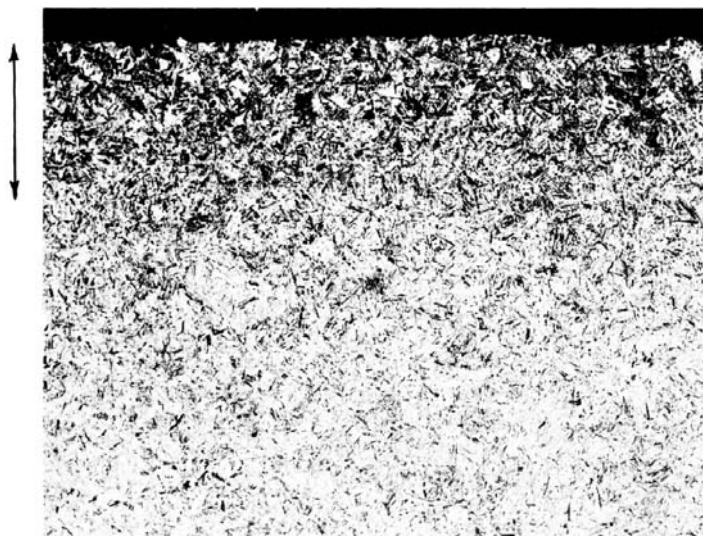


FIG. 7 Example of Partial Decarburization at the Surface of a Heat-Treated Alloy Steel Martensitic Specimen (200x, 2 % Nital Etch)

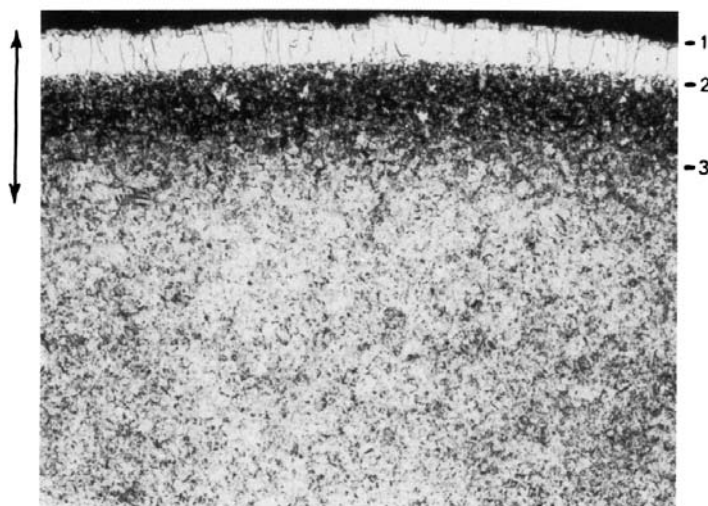


FIG. 8 Example of Complete Decarburization (Free-Ferrite Zone 1 to 2) and Partial Decarburization (Dark Zone 2 to 3) in a Heat-Treated Martensitic Alloy Steel. The Total Depth of Decarburization is the Sum of the Depths of Complete and Partial Decarburization (1 to 3) (200x, 2 % Nital Etch)

interior microstructure. Lineal traverses are made at known increments parallel to the surface and the percentage of each constituent present is determined.⁵

7.3.7 If any unusually deep localized penetration of decarburization is observed, such as might be associated with a seam or lap, the depth of decarburization at this imperfection should be measured and reported separately with an explanation of the nature of the condition observed.

7.3.8 For the determination of the depth of decarburization of spheroidized high-alloy tool steels, for example, high-speed steels, the variation in etch color has been utilized. The polished specimen is etched about 60 s with 4 % nital (using methanol) until the surface color is blue-green. The total depth

of decarburization is measured as the depth at 100x where the color changes from blue-green to green-tan.⁶

7.4 Microindentation Hardness Method:

7.4.1 The microindentation hardness method is most suitable for measuring decarburization depth in heat-treated specimens. It is not recommended for specimens containing two constituents of significantly different hardness.

7.4.2 Assessment of the total depth of decarburization is based on the variation in microindentation hardness with respect to the depth below the surface. The total depth of decarburization is at that depth where the hardness becomes constant and equal to that of the interior. Alternatively, the

⁵ Underwood, E. E., *Quantitative Stereology*, Addison-Wesley Publishing Co., Reading, MA, 1970.

⁶ Powell, J. R., "Measurement of a Decarburized Layer on High-Speed Steel," *Metallurgia*, Vol 70, No. 422, December 1964, pp. 295-297.

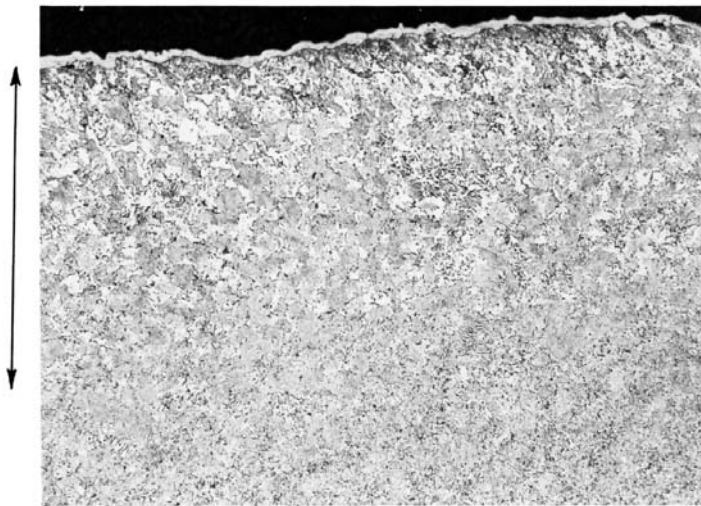


FIG. 9 Example of Partial Decarburization in a Spheroidize-Annealed Carbon Tool Steel. The Partially Decarburized Region Contains Less Carbide than the Matrix and Much of the Carbide is Pearlitic Rather than Spheroidal. The White Layer at the Surface is Electroless Nickel Used for Edge Preservation (100x, 4 % Picral Etch)



FIG. 10 Example of Epsilon Martensite Formation in the Decarburized Surface Zone of a Solution-Annealed Austenitic Manganese Steel Where the Carbon Content is Less than 0.5 % (100x, Etched With 2 % Nital for 5 s, then 20 % Aqueous Sodium Metabisulphite for 20 s)

depth to some specified hardness, based on an agreement between manufacturer and purchaser, can be determined to estimate an “effective depth of decarburization.” This method is best suited for steels with carbon contents below about 0.55 %. Above that carbon content, the hardness will not change unless there is a variation in residual carbide content or retained austenite.

7.4.3 Specimen preparation is conducted in the same manner as defined in 7.2.3. Care must be taken to ensure that the preparation does not alter the hardness.

7.4.4 Specimens should be etched and examined before hardness testing to select typical areas or specific areas of interest for hardness testing. Caution should be taken whenever completing hardness determination (see 7.12 in Test Method E384)

7.4.5 A series of microindentation hardness impressions are made at known increments from the surface inward using either Knoop or Vickers test impressions until a constant hardness is obtained within the normal scatter of test values. These test methods are described in Test Method E384.

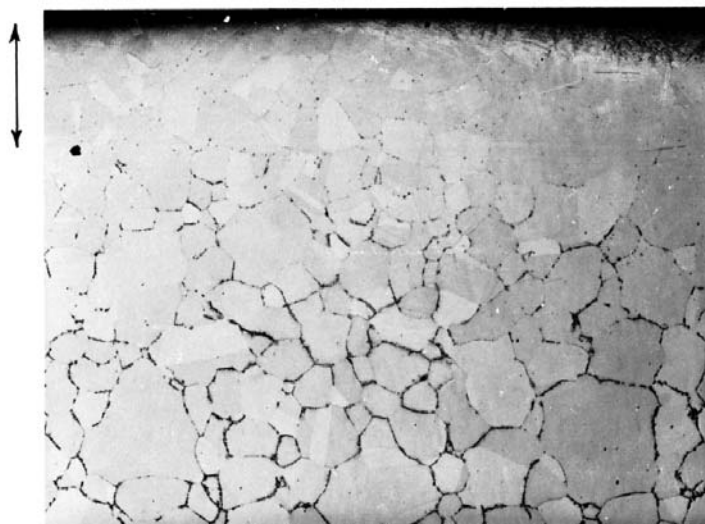


FIG. 11 Example of a Solution Annealed Austenitic Manganese Steel Aged 1 h at 560°C to Precipitate Pearlite at the Grain Boundaries in Regions Where the Carbon Content is Above 1.16 % (Nominal Carbon Content about 1.3 %) (50 \times , 2 % Nital Etch)

7.4.6 The Knoop indentation is more suitable for such tests because the hardness gradient will distort the shape of the Vicker's indentation along the diagonal perpendicular to the surface. When using the Knoop indenter, the long axis of the indenter should be parallel to the specimen surface. Knoop indentations can be spaced more closely than Vickers indentations without danger of altering the hardness value by the strain field from an adjacent impression because of the smaller strain field around a Knoop indentation.

7.4.7 The applied test load should be as high as possible to minimize diagonal measurement inaccuracy and to avoid low-load measurement problems. Loads under 25 grams force should be avoided, if possible.

7.4.8 Impressions should be spaced apart a distance at least 2.5 \times the diagonal length in the direction perpendicular to the surface, that is, along the transverse direction. If intermediate measurements are required, they can be made on a closely spaced parallel traverse.

7.4.9 To determine the total depth of decarburization or the effective depth, it is not necessary to make a complete hardness traverse from the surface inward. A partial traverse can be made based on the observed microstructural condition to minimize the required effort.

7.4.10 A single hardness traverse will define the total depth or effective depth of decarburization at only one location. To obtain greater statistical accuracy in defining these depths, several partial traverses can be made at locations selected based on the observed microstructure and an average can then be computed.

7.5 Chemical Analysis Methods:

7.5.1 Chemical analysis methods provide a direct measure of the carbon concentration as a function of depth. These methods are applicable to any steel composition and microstructure but they are restricted by the specimen shape. These methods are generally used only in research studies. Measurements by such methods will usually produce greater estimates of the total depth of decarburization than the other methods.

7.5.2 Combustion Method:

7.5.3 In this method, the specimen is turned or milled incrementally at known depths and the chips are analyzed using standard analytical methods for carbon. This method is restricted to simple shapes, for example, rounds or flats. High-hardness specimens must be back-tempered (but not annealed) at 600–650°C to permit machining.

7.5.4 Round bars must be accurately centered before turning. Dry machining is recommended. Remove any surface scale before machining. When milling flat specimens, avoid the corners. The weight of the chips must be sufficient to permit accurate analysis, and a recheck, if necessary.

7.5.5 The total depth of decarburization is defined as that depth where the bulk carbon content is reached within the accuracy of the analytical method. Test Method E350 covers the analytical details.

7.5.6 Spectrometric Method:

7.5.7 In this method, the specimen is ground to known depths and the surface is sparked to determine the carbon content using an optical emission vacuum spectrometer. The method is restricted to flat surfaces.

7.5.8 Before the initial grind, remove any surface scale. The specimen is ground incrementally at known depths. Depth measurements are made using a micrometer to measure the specimen thickness after each grind.

7.5.9 After each grind, the surface is sparked and the carbon content is determined. Successive sparkings should not be superimposed and corners should be avoided. Test Method E415 describes the analytical technique.

7.5.10 The total depth of decarburization is at that depth where the carbon content becomes constant and equal to the bulk carbon content within the accuracy of the method.

8. Reporting Test Results

8.1 The test report shall include the following information:

- 8.1.1 Identification of the specimen, heat, lot, etc.
- 8.1.2 Number and location of the test specimens.

8.1.3 Method used to measure decarburization and relevant test variables, for example, magnification, etchant, indenter type, and test load.

8.1.4 For microscopical methods, list the depth of complete or total decarburization, or both, for average and worst conditions. List the depth observed at any imperfection and the nature of the condition. If information on decarburization at corners is required by the purchaser, list the depth of complete or total decarburization, or both. If only a portion of the surface is decarburized, this should be noted.

8.1.5 If the microindentation hardness method was used, list the depth of total or effective (and the hardness criterion) decarburization.

8.1.6 If the chemical analysis method was used, list the depth of total decarburization and the method used.

8.1.7 If the lineal analysis, microindentation hardness, or chemical analysis methods are employed, a plot of the data as a function of depth can be prepared as desired or required based on agreements.

8.1.8 Any manufacturer-purchaser agreements regarding the tests should be documented.

9. Precision and Bias

9.1 Screening Methods:

9.1.1 Screening methods are “go, no-go” tests designed to save time. Under highly controlled conditions, the bulk surface hardness test can be developed to give a reasonably accurate estimate of the total depth of decarburization. The macroscopical etch method cannot be used to give a reliable estimate of the depth of decarburization.

9.2 Microscopical Methods:

9.2.1 The simple scanning method to pick out areas that appear to be typical or worst is subject to some error. Specimen preparation, for example, poor edge retention, can reduce the measurement accuracy. In general, the depth of complete

decarburization can be estimated more accurately than the depth of partial or total decarburization.

9.2.2 For microscopical measurements based on the average of a number of measurements made at randomly selected areas, the accuracy and reproducibility increases as the number of measurements increases. For 20 or more measurements, decarburization depths can be estimated to within 0.025 mm with a relative accuracy of 10 to 20 %. Estimates of the depth of complete decarburization (that is, the free-ferrite depth) are more accurate and more reproducible than estimates of the depth of partial decarburization or the total depth of decarburization.

9.2.3 For any of the measurements, somewhat different values will be obtained on specimens from different locations, even if they are adjacent.

9.2.4 Estimates of total or effective decarburization by microindentation hardness at a given location are generally accurate within 0.025 mm. However, because only one or a few areas are tested, these measurements may not be typical of the overall specimen.

9.2.5 For the analysis of incremental turnings or millings, results are representative of the specimen because each increment samples a substantial surface area. Accuracy is decreased if the turnings are not concentric with the bar diameter, or if millings are not made parallel to the surface.

9.2.6 For the incremental sparking method, an area about 5 mm in diameter and about 1 mm deep is sampled. This gives reasonable accuracy but is not as good as the incremental turning-milling method.

10. Keywords

10.1 carbon content; complete decarburization; decarburization; ferrite; microindentation hardness; partial decarburization; steels; total decarburization

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