Leica Metallurgy Application Briefing

Application Solutions
for Metallurgy

Leica Metallurgy Application Briefing

Leica Microsystems Imaging Solutions Ltd
Clifton Road
Cambridge
CB1 3QH
United Kingdom
Tel +44 1223 411101
Fax +44 1223 412526
Email: imaging.support@leica-microsystems.com
www.leica-microsystems.com

Table of Contents

Introduction

This Application Briefing is a digest of various national and international standards, pertinent to the use of the Leica QMetals metallurgy application suite. Although these standards, their measurement methods and the results derived from them are referenced within the document, it is not a standard itself, nor intended to be used as such. We hope that the metallurgy application suite user who is unfamiliar with these standards will find the briefing useful to consult as a guide.

The property and rights of standards belong to the body responsible for them. Any consequential resemblance of text, tables or diagrams in this document to those standards does not affect those rights. For additional information about the Leica Q550MW Materials Workstation or any of the Leica QMetals Application solutions, please refer to the Product Catalogue.

Style Reference

The following conventions have been used to make this manual easier to read:

Menus	FILE (bold, capitals).
Menu Commands/ Buttons/ Dialogs	Open, OK (bold).
References	*Chapter 1* (italics).
Notes, Tips, Warnings, References and Instructions	Appear as follows and contain additional information

Note: Additional information that should be noted.

Refer to: References to other relevant material.

Tip: Tips to help you use the program more productively.

WARNING: Important information to which you should pay careful attention.

One Step instructions: Operations that can be performed in a single step.

Step-by-step instructions: numbered instructional steps.

Information that only applies in certain situations is enclosed in boxes.

Coating Thickness

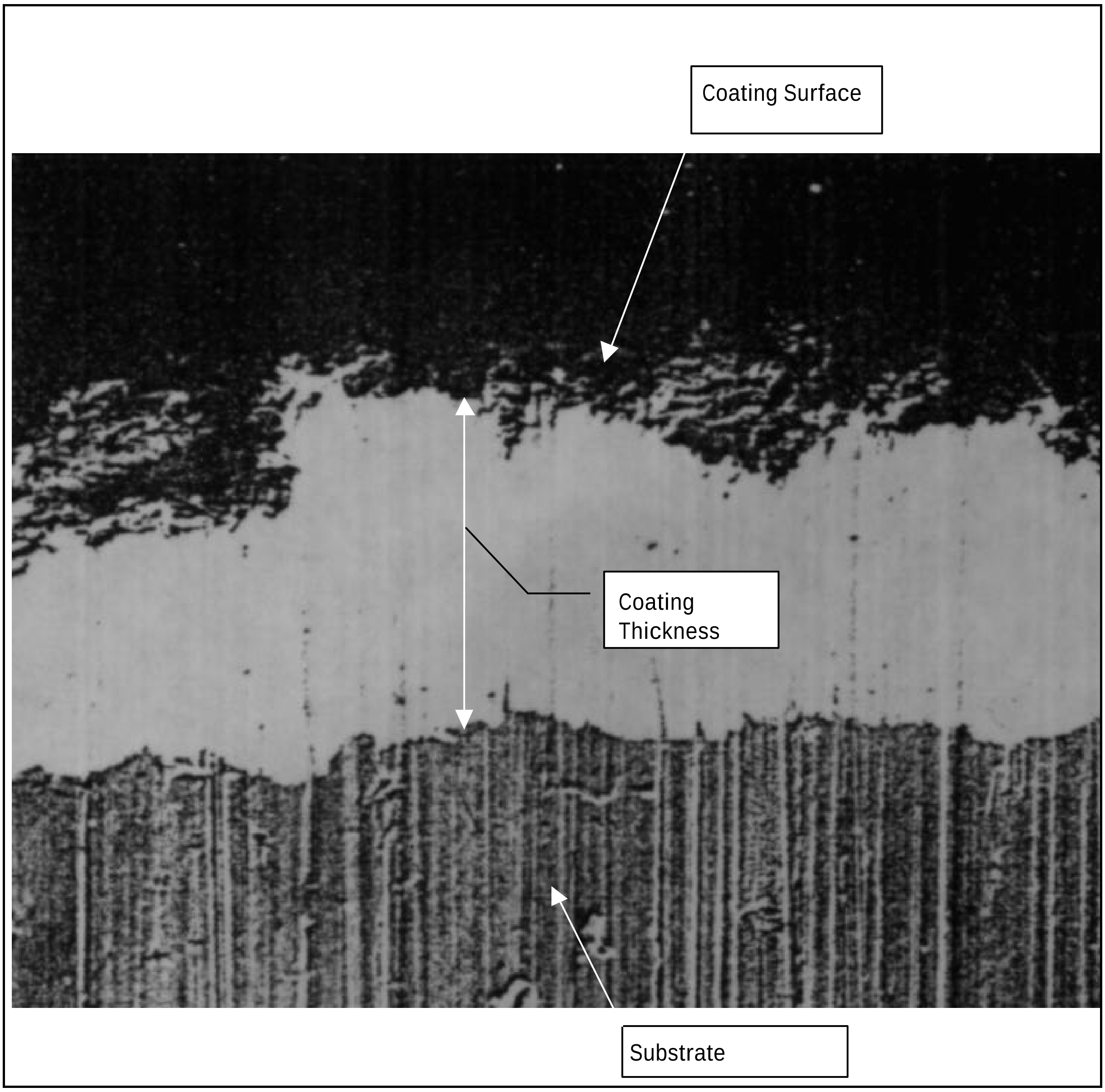
The standards relating to measurement of coating thickness are listed in the table below.

ASTM B487-85 (1990)	Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section.
ASTM B748-90	Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope.
BS 5411 Part 5(1984) ISO 1463	Metallic and Related Coatings Part 5 Measurement of Local Thickness of Metal and Oxide Coatings by Microscopical Examination.
BS 5411 Part 16 (1989) ISO 9220(1988)	Metallic and Related Coatings Part 16 Scanning Electron Microscope Method for the Measurement of Local Thickness of Coatings by Examination of Cross Section.

Note: The scanning electron microscope methods have not been implemented in phase one of the metallurgy applications.

Introduction to Coating Thickness.

Figure 1. An image of a coating on the surface of a substrate shown perpendicular to the coating surface.



There are many different applications for coatings of different types with in the field of materials production. Examples include paint, chrome plating, and plastic powder coatings. These are applied to the substrate surface with the intention of changing properties at the surface of the substrate material. Often this may be for protection from a corrosive environment, e.g. paint on a car, or perhaps, purely for decoration. Coating raw products e.g. steel can be carried out as an extension of the production process and as such requires quality control analysis. This could be performed by the application of the standards mentioned above and described briefly below.

ASTM B487 Measurement of Metal and Oxide Coating Thickness by Microscopical Examination of a Cross Section.

Method

Give attention to the following factors, which may introduce error.

Note: These factors apply to the application of manual methods of coating thickness measurement, and not to automated image analysis methods. See below for a further explanation.)

- Calibration: User dependent and should be made by the operator.
- Alignment errors: Can be introduced by backlash in the movement of the micrometer eyepiece.
- Uniformity of magnification: Magnification may not be uniform within the field of view and hence calibration should be carried out within the same part of the field of view as the measurements are made.
- Lens quality: Lack of sharpness of the image may contribute to uncertainty in the results. Using monochromatic light may improve sharpness.
- Orientation of eyepiece: The movement of the hairline of the eyepiece for alignment to the coating boundary has to be perpendicular. An angle of 10° produces an error of 1.5%.
- Tube length: The tube length may change when the eyepiece is repositioned this may occur whenever it is moved.

Calibrate the microscope and its measuring device using a certified stage micrometer. Measure the width of the image at no less than five points distributed along the micro section. Measure the length over which these measurements are made.

The use of automated image analysis and modern Leica microscopes can eliminate some of these errors. Using image analysis, the calibration becomes system dependent and does not change from user to user. The use of high quality optical components can help to eliminate the errors that are attributed to the equipment used. The affect of tube length is negated by the use of Leica infinite tube length microscopes.

Reporting Results

The following must be included.

- The date of the test.
- The number and title of this test method
- The identification of the test specimens.
- The location on the coated item at which the cross section was made.
- Thickness is measured in micrometers, unless it is greater than one millimetre at each point and the length of section over which the measurements where distributed, when it is in millimetres.
- The local thickness is by definition the arithmetic mean of the measured thickness.
- Any deviations from this test method.
- Any factors that might influence interpretation of the reported result.
- Name of the operator and testing laboratory.

ASTM B748-90, Measurement of Thickness of Metallic Coatings by Measurement of Cross Section with a Scanning Electron Microscope.

Method

Operate the SEM in accordance with the manufacturer's instructions. Take into account the following factors:

- Surface roughness: If the coat or substrate has a rough surface then it may not be possible to produce accurate measurements.
- Taper of cross section: If the plane of the cross section is not perpendicular to the plane of the coating the measure thickness will be greater. Than the true thickness.
- Specimen tilt: Errors will be introduced if the specimen surface is not perpendicular to the electron beam.
- Oblique measurement: If thickness measurement is not made perpendicular to the plane of the coating, the measured value will be greater than the true thickness.
- Deformation of coating: Excessive temperature or pressure during preparation of the sample can cause deformation of the coating; this is especially true of soft coatings and low temperature coatings.
- Rounding edge of coating: If the surface of the coating cross section is rounded then the true edge of the coating can not be measured.
- Over plating: The use of over plating to protect the surface coating may result in a low coating thickness measurement.
- Etching: Good etching should reveal the coating substrate boundary as a narrow well-defined line. Poor etching may produce a broader line resulting in a low coating thickness measurement.
- Smearing: Poor polishing may leave one metal smeared over the other, making an accurate measurement impossible. The preparation of multiple specimens should make it possible to detect poorly prepared specimens when wide variations in measurements occur.

Make a micrograph of the following under the same instrument settings as used to make the calibration and make an appropriate measurement of the micrograph image.

Conventional micrograph

With the boundaries of the coating clearly defined, make conventional micrographs of the test specimen and calibration micrometer scale. Measure the micrograph to at least 0.1mm using a diffraction plate reader or equivalent device.

Video wave form signal

Photograph the video wave form signal for a single scan across the specimen and across the stage micrometer scale. To measure the coating thickness, measure the horizontal distance between the inflection points of the vertical portions of the scan at the boundaries of coating. Make the measurements to the nearest 0.1mm using a diffraction plate reader or similar device.

Calculate the thickness according to the following expression.

Equation 1. Coating thickness calculated using linear distance and magnification

$$T = 1000 \times d / M$$

T= coating thickness microns, d =linear distance on micrograph in mm and M = magnification factor as defined in practice ASTM E766 Calculating the calibration of a scanning electron microscope.

Reporting Results

The following must be included.

- The date of the test.
- The number and title and year of the this test method
- The identification of the test specimens.
- The location on the coated item at which the cross section was made.
- Thickness is measured in micrometers, unless it is greater than one millimetre at each point and the length of section over which the measurements where distributed, when it is in millimetres.
- The measured values and the arithmetic mean of the measured thickness.
- The calibrated magnification as measured with a SEM micrometer scale.
- The measurement can be made using either conventional micrographs or video wave form signals.
- Any factors that might influence interpretation of the reported result.
- Name of the operator.

BS5411 Part 5:1984 - ISO 1463:1982, Metallic and Oxide Coatings - Measurement of Local Thickness of Metal and Oxide Coatings - Microscopical Method.

Method

Give appropriate attention to the following factors.

- Surface roughness: If the coating or substrate has a rough surface then it may not be possible to produce accurate measurements.
- Taper of cross section: If the plane of the cross section is not perpendicular to the plane of the coating, the measured thickness will be greater than the true thickness.
- Deformation of coating: Deformation of the coating can be caused by excessive temperature or pressure during preparation of the sample.
- Rounding edge of coating: If the surface of the coating cross section is rounded then the true edge of the coating cannot be measured.
- Over plating: The use of over plating to protect the surface coating may result in a low coating thickness measurement.
- Etching: Good etching should reveal the coating substrate boundary as a narrow well-defined line. Poor etching may produce a broader line resulting in a low coating thickness measurement.
- Smearing: Poor polishing may leave one metal smeared over the other making an accurate measurement impossible. The preparation of multiple specimen should make it possible to detect poorly prepared specimen when wide variation in measurements occur between specimens.
- Magnification: For any given coating thickness, the error associated with the measurement will increase at lower magnifications. The field of view is between 1.5 and 3 times the coating thickness.
- Calibration of stage micrometer: Any error in calibration of the stage micrometer will be reflected in the measurement of the specimen.
- Calibration of micrometer eyepiece: A filiar micrometer eyepiece is usually the most satisfactory means of making measurements.
- Alignment: Errors can be introduced due to backlash in the movement of the eyepiece.
- Uniformity of magnification: The magnification may not be the same over the entire field. Errors can occur if the calibration and measurements are made in different parts of the field.
- Lens quality: lack of sharpness can contribute to uncertainty in the measurement.
- Orientation of eyepiece: The movement of the hairline of the eyepiece should be perpendicular to the surface of the specimen.
- Tube length: The tube length may change when the eyepiece is repositioned. This may occur whenever it is moved.

Calibrate the microscope and it's measuring device using a certified stage micrometer.

Reporting Results

- The test report shall include.
- The location on the coated item at which the cross section was made.
- The measure thickness in micrometers (millimetres if greater than 1mm) at each point, and the length over which the measurements are made.
- The local thickness is the arithmetic mean of the measured thickness.

BS5411 Part 16:1989 - ISO 9220:1988, Metallic and Related Coatings Part16 -- Measurement of Local Thickness of Coatings by Examination of Cross Sections.

Method

Calibration of Instruments.

Measure the perpendicular centre to centre distance between the lines in the photographed image to the nearest 0.1 mm using a diffraction plate reader or similar device. Repeat the measurement in at least three different locations, which should be at least 3 mm apart.

Make a micrograph of the test specimen under the same condition and instrument settings as the calibration.

Conventional micrograph.

With the boundaries of the coatings clearly defined, make conventional micrographs of the stage micrometer scale and the test specimen. Measure the micrographs to at least 0.1 mm using a diffraction plate reader or other optical device for making accurate linear measurement on film or paper.

Video wave form signal.

Photograph the video waveform signal for a signal scan across the coating cross section and across the SEM stage micrometer scale. Calculate the thickness according to the following expression.

Equation 2. Thickness calculated using linear distance on micrograph and magnification factor

$$T = 1000 \times {}^{l_m}\!/_{n}$$

T= coating thickness in microns, l_m =linear distance on micrograph in mm and ν = magnification factor as defined in practice ASTM E766 Calculating the calibration of a scanning electron microscope.

Reporting results

The report must include.

- Reference to this international standard ISO 9220.
- The measured value.
- Identification of the test specimen.
- Location of the measurements on the test specimens.
- The magnification as measured before and after the test specimen measurements.
- Any unusual features of the measurements that may have affected the results.
- Date the measurements where made.
- Name of the individual responsible for the measurements.
- The measurement can be made using either conventional micrographs or video wave form signals.

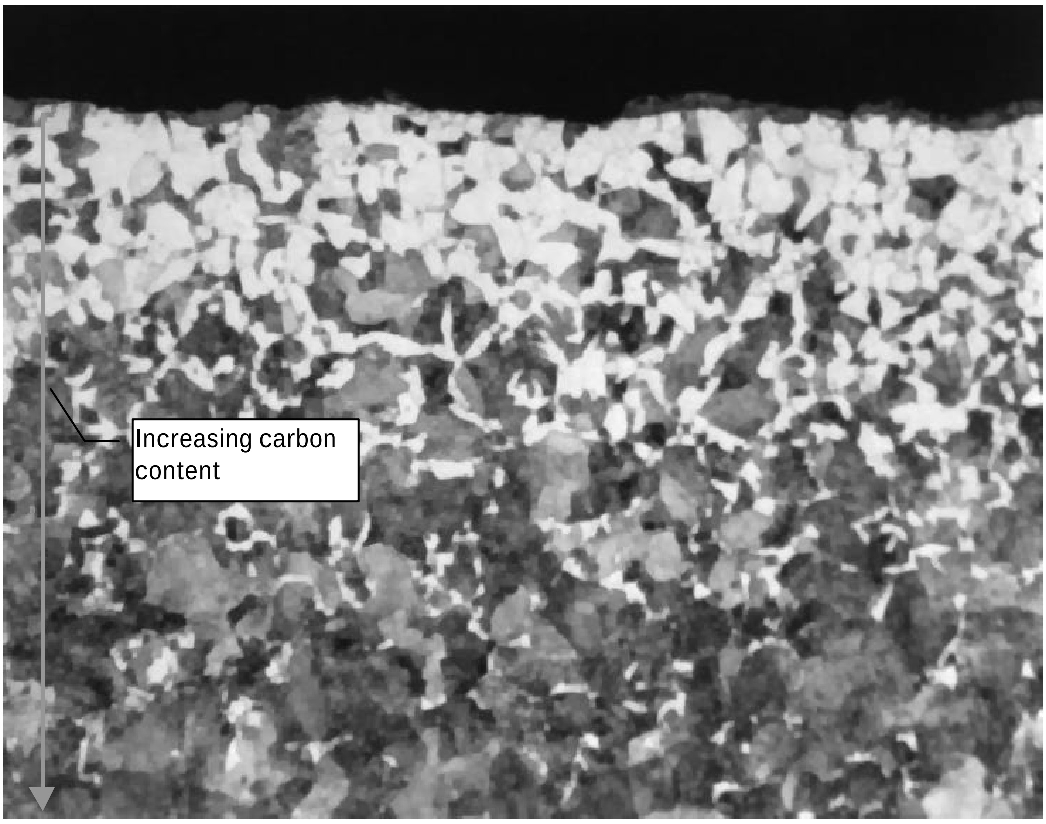
Decarburisation Depth Measurement.

The standards relating to measurement of decarburisation depth are listed in the table below.

DIN 50192 (1977-05)	Determination of the Depth of Decarburisation.
ISO 3887 -1976	Steel Non Alloy and Low Alloy Determination of Decarburisation Depth.
JIS G0558	Methods form Measuring Decarburised Depth of Steel.
ASTM E1077-91	Standard Test Method for Estimating the Depth of Decarburisation of Steel Specimens.

Introduction to Decarburisation Depth Measurement.

Figure 1. Section of decarburised surface layer of steel specimen perpendicular to specimen surface.



The measurement of decarburisation depth of steel products is important because of the effect that a reduction in carbon content can have on the surface of the product. A reduction in carbon content will result in softer metal at the surface of the product than in the interior. A softening in the metal will be disadvantageous where the surface in required to be hard e.g. bearing steels and other hardened products.

The depth of decarburisation is an indication of the deterioration in the metal at the surface. This may occur during processing or actually when in service, where the operating conditions are severe. Decarburisation is caused by the removal by oxidation of carbon from the surface of the product. The depth of decarburisation can be determined visually using image analysis, by determining the quantities of carbon bearing phases at the surface of the specimen and comparing the amount present to the unaffected interior. The extent of decarburisation is expected to decrease toward the centre of the specimen until the carbon content calculated by the frequency of features identified as carbon bearing phases decreases to a level where the content is a the same as the unaffected specimen core. The perpendicular distance from the surface to this point is the decarburisation depth.

DIN 50 192 Determination of Depth of Decarburisation.

This standard corresponds closely with ISO 3887

Definitions

Decarburisation: A reduction in the carbon content and is mostly restricted to the surface of a work piece. The term work piece refers not only to finished products, but also parts at any stage of manufacture, includes heat processing and machining.

Total decarburisation: Decarburisation with almost total carbon removal.

Partial decarburisation: Decarburisation where the carbon content is reduced but there is no total Decarburisation.

Depth of decarburisation: The vertical distance from the surface of a decarburised work piece to the point at which the carbon content is equivalent to an appropriate specified limit value, or limit characteristic.

The depth of decarburisation may also be characterised by other properties determined by carbon content, especially structural properties. The type and size of the limit value or limit characteristic are determined by the relevant conditions and requirements for test methods; e.g. the state of materials, etc.

Depth of total decarburisation: The depth of decarburisation of a totally decarburised work piece to the limit of the ferritic layer.

Depth of partial decarburisation: The difference between the depth of decarburisation and the depth of total decarburisation. It is equivalent to the depth of decarburisation provided there is no total decarburisation.

Methods

Microscopic determination.

A microscope is used to determine the depth of Decarburisation from the changes in structural quality. This method is predominately used for the following.

- Ferrite Pearlite microstructures
- Pearlite and hyper-eutectoid Carbides
- Ferrite matrix with Carbide inclusions

The depth of decarburisation is measured as the vertical distance though the specimen to the point at which the microstructure is no different than at the core of the specimen. At least four fields are required, depending on the accuracy required.

Determination by measuring hardness

With this test method, the depth of decarburisation is determined by means of low load hardness testing .This method is preferred for hyper–eutectoid steels which have been hardened, or hardened and tempered. In the case of parts which have not been fully hardened, the depth of decarburisation should be small compared with the depth of the hardened layer, so that the change in hardness between the hardened and decarburised layer is clearly discernible. The Vickers Hardness Method DIN50 133 low load range is usually used.

Chemical analysis

Wet chemical analysis and spectrographic analysis can be used to determine depth of Decarburisation.

Reporting Results

The report should include the following:

- The type of steel and ladle identification marking.
- The type and cross sectional dimensions of the work piece.
- The position of the points from which samples are taken from the workpiece (where this does not refer to the whole work piece).
- Treatment conditions of the work piece or sample.
- The test method including the limit value, or limit characteristic, for the depth of decarburisation is abbreviated as Ret M.
- The depth of decarburisation is taken as the mean of the measurements taken for each sample. Depending on the value, the figure should be rounded up to multiples of 0.02, 0.05, 0.1 as appropriate.
- Test house.
- Date of testing.

Abbreviations (designation)

"Ret" is used to denote depth of decarburisation the symbol for the test method used and if necessary the relevant limit value or limit characteristic.

For example:

- Microscopic determination Ret M =0.2mm
- Measurement of hardness Ret 600 HV 0.30.2mm
- Chemical determination Ret 0.5 C =0.2mm

If there is only partial Decarburisation or total Decarburisation, is to be specified separately then the details relating to each are to be separated by an oblique where the first abbreviation refers to Decarburisation, and the second to total Decarburisation. If only total Decarburisation is to be specified then the abbreviation shall be followed by the words Total Decarburisation.

ISO 3887 Steel Non-Alloy and Low Alloy Determination of Depth of Decarburisation.

Definitions

Decarburisation: The loss of carbon from the surface layer of metal. This loss may be either

- Partial decarburisation
- Complete (or almost) decarburisation

Total decarburisation is the sum of the two types of decarburisation namely both partial and complete.

Depth of total decarburisation is the distance between the surface of the specimen and the point at which the carbon content is that of the unaffected core.

Methods

Micrographic method

This method is best suited to annealed or Ferrite Pearlite microstructure. It is less well suited to hardened products where the interpretation of the microstructural features regarding carbon content is more complex.

The sample selected should be a section perpendicular to the longitudinal axes of the product. Small samples of less than $4cm^2$ should be examined. For large samples several sections should be taken. The number and specified position of the samples can be determined according to requirements.

The distance between the surface of the specimen and the point at which the microstructure is the same as that at the core, should be measured using a micrometric eyepiece, or directly on a projected image of the specimen. The recommended magnification is x100.

Method for measuring the micro hardness.

This method consists of determining the gradient of the micro hardness on a cross section of the product, along a line perpendicular to the edge of the specimen. The selection and preparation of the sample should be the same as for the micrographic method. The load should be as high as possible, in order to minimise scatter of measurements; 0.49N to 4.9N typically.

The depth of decarburisation is defined as the depth at which the hardness is the same as that at the core of the sample.

Chemical and spectrographic method

Chemical and spectrographic methods for carbon content analysis methods are also available.

Reporting Results

The report should include the following:

- The number and location of samples taken from the work piece
- The method used
- The results of the measurements, permitting the depth of Decarburisation to be defined.

ASTM E 1077-91 Standard Test Method for Estimating the Depth of Decarburisation of Steel Specimens.

Definitions

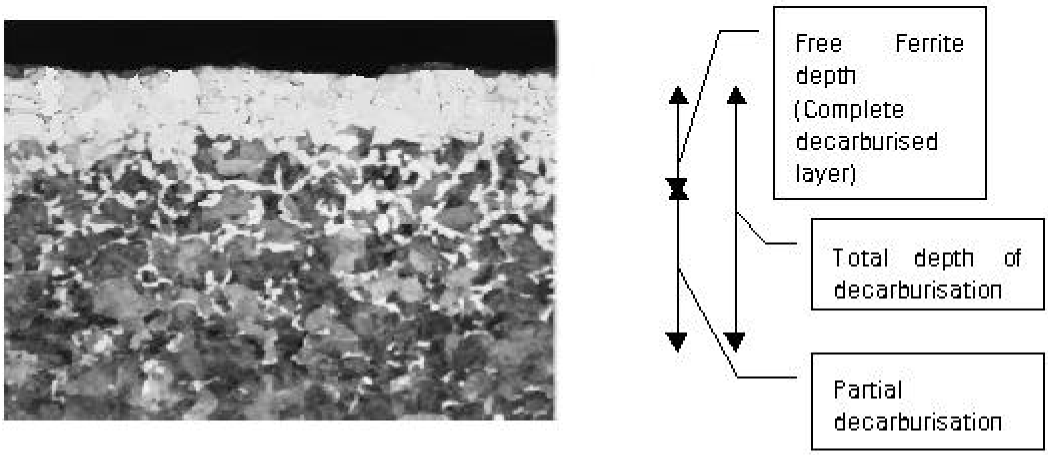
Average depth of decarburisation: The mean value of five or more measurements of the total depth of decarburisation.

Partial decarburisation; 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 then the room temperature solubility limit of carbon in Ferrite.

Complete Decarburisation: Loss of carbon content to a level below the solubility of carbon in Ferrite, so that only Ferrite is present.

Free Ferrite depth: The perpendicular distance to that point where the microstructure is no longer fully ferritic.

Figure 2. Total and partial decarburisation



Total depth of decarburisation: The perpendicular distance from the specimen surface to the point in the interior where the bulk carbon content is reached, i.e. the sum of the depths of complete and partial decarburisation.

Methods

Bulk surface hardness.

For hardened specimens, particularly in the as quenched condition, a short section of the material to be heat-treated is cut and heated treated in the same manner or along with the material of interest. The scale is removed from the sample using a wire brush, or by blasting with glass beads. The hardness of the surface of the sample is then tested using the Rockwell C Hardness Test. The presence of decarburisation is indicated by a lower hardness on the surface than the theoretical maximum hardness of the carbon content of the steel. This method is most suitable for steels with a carbon content below 5.5%.

Macroscopical etch appearance

Decarburisation is indicated by a difference in the etching contrast between the surface and the interior of the specimen.

Microscopical method

Measurement of the depth of decarburisation is based on the evaluation of the variation in the microstructure at the surface, due to the change in carbon content.

The depth of complete decarburisation is easiest to assess when there is an excellent contrast between the free Ferrite layer and the interior structure. The depth of partial decarburisation can best be assessed when this zone contains Ferrite and pearlite. The type and extent of decarburisation should be determined. The latter is determined using a graticule, or a screw micrometer ocular.

In heat-treated products, the presence of non-martensitic structures is used to determine the extent of decarburisation.

Micro-indentation hardness method

This method is most suitable for heat-treated specimens. It should not be used where two or more constituents are present which differ significantly in hardness. Specimens are prepared as for the microscopical method and should be scanned to find suitable sites for the test. A series of micro-hardness indentations should be made at specific increments from the surface inward.

Combustion method

In this method, the specimen is milled incrementally at known depths and the chips are analysed using standard analytical methods for carbon.

Spectrographic method

In this method, the specimen is ground at known depths, the surface is sparked and the carbon content is determined, using an optical emission vacuum spectrometer.

Reporting Results

The report should include the following:

- Identification of the specimen, heat, lot, etc.
- Number and location of the test specimens.
- Method used to measure decarburisation and relevant test variables; magnification, etchant, indenter type, test load.
- For micro-structural methods, list the depth of complete, or total decarburisation, or both, for average and worst conditions. List the depth observed for any imperfection and the nature of the condition. If only a portion of the surface is affected, this should be noted.
- If the micro indentation method is used, list the depth of total, or effective decarburisation (and the hardness criterion).
- If the chemical analysis method is used, list the depth of total decarburisation and the method used.
- If lineal analysis micro-indentation hardness or chemical analysis are employed, plot the data as a function of depth .

JIS G0558 Methods for Measuring the Decarburised Depth of Steel.

Definitions.

Decarburised layer: The surface layer of steel, where the carbon content is reduced by hot working or heat treatment.

Methods

Measuring method by microscope

A specimen is prepared which is cut from the product, revealing a section perpendicular to the surface of the sample. The decarburisation depth of a test plane, perpendicular to the specimen surface, is determined by the measurement of the decarburised state. This is observed as the area ratio of Ferrite, Pearlite and Carbide. The depth is measured using an eye piece graticule. Magnification should be in the range x100 x500.

Measuring method by hardness test

The Vickers Hardness Test is applied to a section of the product perpendicular to the surface. Starting at the surface and incrementally toward the core of the specimen, until the point where the hardness is the same as at the core of the specimen.

It is preferable to draw the hardness transition curve to illustrate the change in hardness across the specimen surface.

Reporting Results

The decarburisation should be expressed in millimetres, down to a second decimal place for the microscope observation, and down to the first decimal place for the hardness test. The symbols for the indication of decarburisation are shown in the following table.

Table 1. Designation of decarburisation test methods

	Measuring method by microscope.	Measuring method by hardness test.
Total decarburised depth	DM-T	DH-T
Ferrite decarburised depth	DM-F	
Decarburised depth by residual carbon ratio.	DM-S	
Practical decarburised depth		DH-P

Examples

- DM-T 0.28 means that the total decarburised depth is 0.28mm measured by microscope.
- DH (0.3)-T0.2 means that the total decarburised depth is 0.2 mm measured by Vickers Hardness Testing machine with a load of 0.3Kg{2.9N}
- DM-F 0.05 means that the Ferrite decarburised depth is 0.05 mm measured by microscope.
- DM-S (70) 0.01 means that the decarburised depth specified as residual carbon ratio 70% is 0.10 mm measured by microscope.
- DM-F0.05-S (50) 0.15-T 0.28 means that the Ferrite decarburised depth is 0.05mm measured by microscope.
- DH (0.3)-P (450) 0.2 means that the practical decarburised depth of HV is 450 0.2mm measured by a Vickers Hardness Testing machine with test load of 0.3kgf{2.90N}
- DHC-P (0.2) 45 means that HRC45 was obtained at a specified depth of 0.2mm measured on the C scale, using Rockwell Hardness Testing machine.

Units in {} brackets are based on the international system of units and are included for informative reference.

Grain Sizing

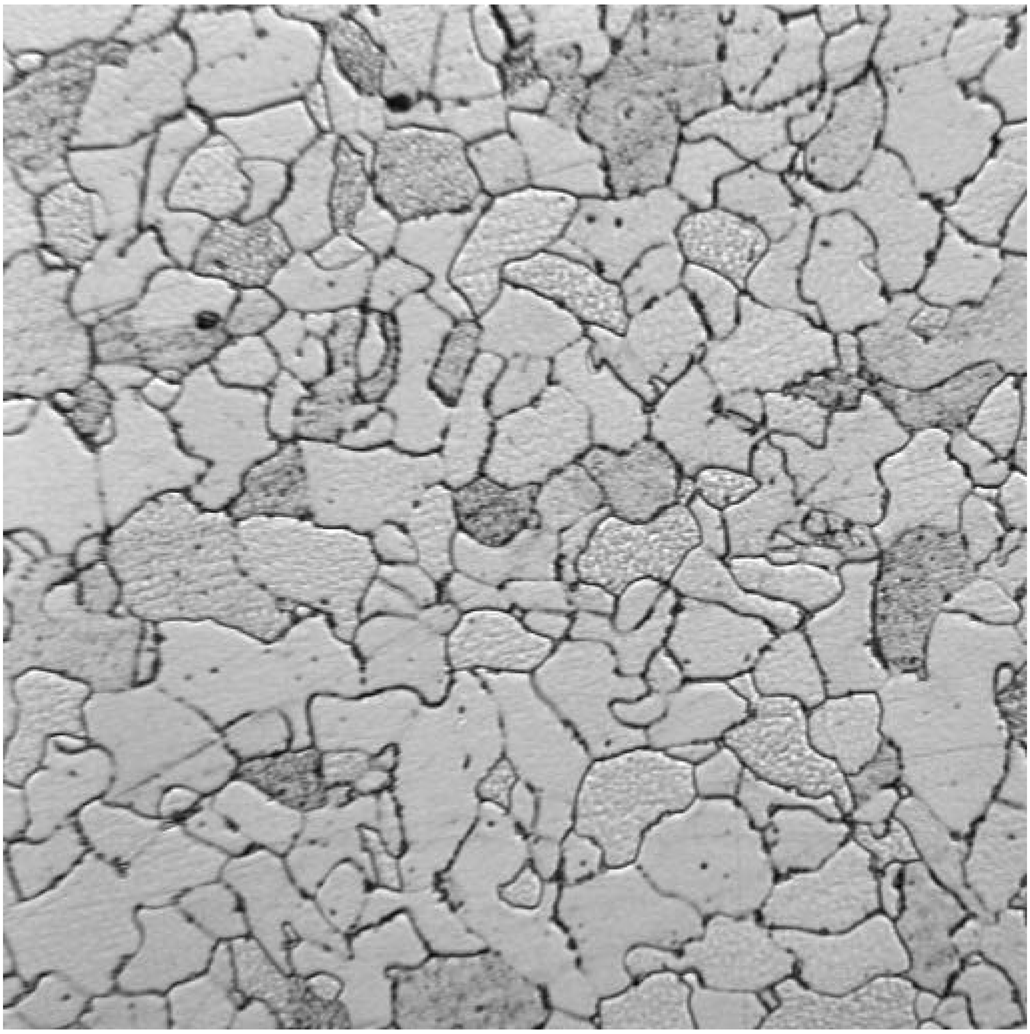
The standards relating to measurement of coating thickness are listed in the table below.

ASTM E930-92	Standard Test Methods for Estimating.
ASTM E1181-87	Standard Test Methods for Characterising Duplex Grain Sizes.
ASTM E1382-91	Standard test Methods for Determining Average Grain Size Using Semi-automatic and Automatic Image Analysis.
ASTM E 112	Standard Test Methods for Determining Average Grain Size.
DIN 50 601	Determination of Ferritic or Austenitic Grain Size of Steel and Ferrous Materials.
ISO 643- 1983	Steels – Micrographic Determination of the Ferritic or Austenitic Grain Size.
BS EN ISO 2624 –1995	Copper and Copper Alloys Estimation of Average Grain Size.
BS 4490	Micrographic Determination of the Grain Size of Steel.
SAE J418 DEC83	Grain Size Determination of Steels.
JIS G 551-1977	Method of Austenite Grain Size Test for Steel.
JIS G 552-1977	Method of Ferrite Grain Size Test for Steel.

Introduction to Grain Sizing.

The grain size in a metallic product is a factor which determines the hardness and ultimate tensile strength of that product. There are certain processing techniques that can be used to determine the grain size in a product, at the bulk product stage of manufacturing. As such, it is an important factor to record and use in the quality control of products. Grain size measurement is not restricted to metals and may be used in ceramics, or other situations involving grain like structures.

Figure 3. An Image of some grains in a metallic specimen.



The grain size may be expressed in terms of a grain size number: This is usually related to the number of grains per unit area by a logarithmic arithmetic relationship. The following standards describe the nature of these relationships. The grains in a metallic specimen are revealed when the specimen is polished and then etched, using a suitable etching reagent. This is due to differences in the way in which the grain is eroded by the reagent at the grain boundaries.

ASTM E112 Standard Method for Determining Average Grain Size.

Introduction

ASTM E112 is a basic manual method, standard issued by ASTM. It details manual procedures for use in grain sizing. The methods rate grain size in terms of ASTM grain size number.

It is important in using these methods to recognise that the estimation of average grain size is not a precise measurement. A metal structure is an aggregate of three-dimensional crystals of varying sizes and shapes. Even if all these crystals were identical in size and shape, the grain cross sections produced by a random plane through such a structure would have a distribution of areas varying from a maximum value to zero, depending on the microstructure in the plane of view.

Definitions

Grain: A grain is considered to be all that area within the confines of the original (primary) boundary. In materials having twinned grain structures a crystal and its twinned bands shall be considered as one grain.

Grain size: In materials consisting of two or more constituents, the grain size refers to the matrix. This is true, except in those materials where the second phase is of sufficient amount or size or continuity to be significant to the grain size. This may be estimated and recorded separately. Minor constituent phase's inclusions and additives are not normally considered in the estimation of grain size.

Sub Grains: The sizes of sub-grains may be estimated by the same methods applicable to the grains themselves.

Limitations of Use.

This standard should be used for grain sizing of metals consisting of a single phase. No attempt should be made to estimate the grain size of heavily cold worked or partially re-crystallized wrought alloys.

Comparison methods cannot be applied to the sizing of individual grains. The distribution of grains is random. No random process of positioning can improve this randomness. In order to select areas of a specimen to be measured, without introducing bias, the specimen should be divided into regions and a random starting point for the test pattern selected in each region by positioning the field of view, without looking at its content.

Methods

All the methods within ASTM E112 are intended to be manual methods. They are not restricted to use for measuring grain size of metals but can be used for measuring the mean grain, crystal or cell size of non metallic materials.

This standard contains instructions for the use of the following methods.

- Comparison Procedure
- Planimetric (Jeffries) Procedure
- Lineal Intercept (Heyn) Procedure
- Circular Intercept Procedure

Comparison Procedure

This method can be applied to completely re-crystallized or cast products.

Equation 3. Relation between grains per square inch and ASTM grain size number

$$N_{AE} = 2^{G-1}$$

N_{AE} is the number of grains per square inch at x100. G is ASTM grain size.

Refer to: ASTM E112 Table 6. for other relations with G, reproduced as Table 2 below.

Table 2 ASTM E112 calculating grain size number.

Equation	Units
G=(3.321928 $\log_{10} N_A$)-2.954	N_A number of grains per mm^2 in mm^{-2}
G=(6.643856 $\log_{10} N_L$)-3.288	N_L number of intercepts per unit length of test line in mm^{-1}
G=(6.643856 $\log_{10} P_L$)-3.288	P_L number of grain intersection boundaries per unit length of test line in mm^{-1}
G=(-6.643856 $\log_{10} l$) -3.288	mean l lineal intercept length in mm

The comparison procedure is a visual estimation, for which the results are generally reproducible within plus or minus a whole grain size number.

When the specimen is equiaxed this method is most convenient of the three grain sizing methods, and is sufficiently accurate.

Note: Experience has shown that unless the appearance of the standard reasonably well approaches that of the sample, errors may occur. To minimise such errors the comparison charts are presented in four categories as follows.

- Plate I: Un-twinned grains (flat etch) includes grain sizes 00, 0, ½, 1, 1½, 2, 2½, 3, 3 ½, 4, 4½, 5, 5½, 6, 6½, 7, 7½, 8, 8½, 9, 9½, 10 at 100x
- Plate II: Twinned grains (flat etch) includes grain size numbers 1, 2, 3, 4, 5,
- Plate II: Twinned grains (flat etch) includes grain size numbers 1, 2, 3, 4, 5, 6, 7, 8, 9 at 100x
- Plate III: Twinned grains (Contrast etch) includes the nominal grain diameters 0.200, 0.150, 0.120, 0.090, 0.070, 0.060, 0.050, 0.045, 0.035, 0.025, 0.020, 0.015, 0.001, 0.005 at 75x
- Plate IV: Austenite grains in steel (McQuaid-Ehn) Includes grain size numbers 1,2,3,4,5,6,7,8 at 100x

Note: If the grain size is reported in ASTM grain size number, it is convenient to use the following relationship.

Equation 4. Calculation of correction factor for magnification other than x100

$$Q = 2Log_2(M / M_b)$$

Where Q is the correction factor that is added to the apparent micro grain size of the specimen at magnification M, instead of the base magnification M_b x75, or x100 to calculate the true ASTM grain size number.

Grain size estimations on three areas should be made, on three or more representative areas of each sample section.

The small number of grains per field at the coarse end of the chart series, and the large number of grains at the fine end, may lead to errors. A more meaningful comparison can be made by changing the magnification such that the apparent grain size lies nearer the centre of the range.

Placing the standard and measured image side by side is the traditional method, although superimposition is probably more appropriate for IA.

Results of inter-laboratory grain size determinations show that there is a general bias: Ratings are generally claimed to be coarser than the actual grain size by ½ to 1 G lower than

Table 3. Suggested comparison charts for metallic materials. (ASTM E112 Table 1.)

Material	Plate Number	Basic Magnification
Aluminium	I	100x
Copper and copper base alloys	III	75x
Iron and Steel:		
Austenitic	II or IV	100x
Ferritic	I	100x
Carburized	IV	100x
Stainless	II	100x
Magnesium or Magnesium base alloys	I or II	100x
Nickel and Nickel base alloys	II	100x
Super strength alloys.	I or II	100x
Zinc or Zinc base alloys.	I or II	100x

Planimetric Procedure

The planimetric procedure should be treated as an estimation procedure that is only accurate to plus or minus half a grain size number, when no statistical control is applied. When sufficient measurements are made and statistically analysed to comply with the requirement of ASTM E112 section 13, the grain size can be stated to plus or minus one quarter of a grain size number. This procedure is used where a higher degree of accuracy is required over the comparison method.

Intercept Procedure

The intercept procedure should be treated as an estimation procedure that is only accurate to plus or minus half a grain size number, when no statistical control is applied.

When sufficient measurements are made and statistically analysed to comply with the requirement of ASTM Section 15. Then the grain size can be stated to the accuracy indicated but not normally lesser than plus or minus one tenth of a grain size number.

There is no direct mathematical relationship between the ASTM grain size number (G), and the mean lineal intercept, unlike the exact relation ship between G and N_{AE}. The relationship between the ASTM grain size number (G) and the mean lineal intercept is defined such that ASTM No.0 has a mean lineal intercept size of precisely 32.00 mm. for macroscopically determined grain size and of 32.00 mm on a field of view at 100x magnification, for the microscopically determined grain size scale.

Hence:

Equation 5. Relationship between mean intercept length and grain size number.

$$G = 10.00 + 2\log_2 \overline{N_L}$$

Where N_L is the number of intercepts per mm at 1x for macro grain size number, or the number per mm in a field at 100x for the microscopically determined grain size numbers.

Note: Using this scale of grain size number, the measured grain size number is within 0.01 G units of the grain size number of grain size determined using the planimetric method. This is well within the precision of the test methods.

Non equiaxed grain structures.

In order to measure the grain size of non-equiaxed grain structures it is necessary to make measurements on the three principle axes though the specimen and combine the results. Ref. ASTM E112 16.3. In cases of dispute, the intercept method should be the referee procedure in all cases.

Section 13 of the ASTM E112 defines the Heyn lineal intercept method: This method uses straight line intercept counting. 50 intercepts are required in one visual field and the magnification should be adjusted to permit this. The precision of the grain size method is a function of the number of grain interceptions counted. Between three and five widely separated fields should be selected to make the measurement. Provided the specimen is equiaxed the measurement will be valid however if the specimen is not equiaxed then more information can be gathered by making separate measurements along the three principle axes of the specimen. See ASTM E112 section 16, Specimens with non-equiaxed grain shapes.

Statistical Analysis

Ref. ASTM E112 Section 15.

No determination of average grain size can be an exact measurement. Thus, no measurement is complete without also calculating the precision within which the determined size may, with normal confidence, be considered to represent the actual average grain size of the specimen measured.

The statistical analysis applied to the results of grain size analysis is used to determine the validity of the result, it’s accuracy and precision. Also it can be used to determine the number of fields required to be measured in order to reach the required confidence limit.

The equations in Table 2 can be applied to the parameters N_A and l and the mean values calculated. The confidence interval and relative accuracy of these means can be used to assess their accuracy.

To calculate the 95% confidence interval, use the following equation:

Equation 6. Calculation of confidence interval

$$95\%CI = \frac{t \times s}{\sqrt{n}}$$

Where t is the confidence interval multiplier, s is the standard deviation; n is the number of measurements.

Table 4. Confidence interval multipier (t) as a function of the number of fields measured (n)

Number of fields (n)	t	Number of fields (n)	t
5	2.776	13	2.179
6	2.571	14	2.160
7	2.447	15	2.145
8	2.365	16	2.131
9	2.306	17	2.12
10	2.262	18	2.11
11	2.228	19	2.101
12	2.201	20	2.093

Equation 7. To calculate the relative accuracy of measured results.

$$RA\% = \frac{95\%CI}{\overline{X}} \times 100$$

As a rule a 10% relative accuracy or less is acceptable, where RA is the relative accuracy.

CI is the calculated 95% confidence interval for the parameter. X is the mean value of the parameter under consideration.

Specimens Containing Two or More Phases.

Minor amounts of second phase particles may be ignored. The identity and percentage of each phase should be determined by ASTM E 562. Determine the mean intercept length of the matrix phase according to the following equation:

Equation 8. Calculation for mean intercept length for matrix phase

$$\overline{l_a} = \frac{(V_{Va})(L/M)}{N_a}$$

Where the volume fraction of the of the α matrix, $V_{V\alpha,}$ is expressed as a fraction, L is the test line length M is the magnification

See Table 2 from ASTM E112 Table 6.

Equation 9. Calculation for grain size number from mean intercept length

$$G = (-6.643856\log_{10} l) - 3.288$$

Where l is the intercept length is in mm.

The grain size of the α grains can be calculated using. (ASTM E112 Table 4) or the equation in (ASTM E112 table 6). The mean lineal intercept length can be calculated for each field and the statistical procedure described in Section 15 can be applied. This describes the method for calculating the confidence interval and relative accuracy.

Reporting Results

See ASTM E112 Fig 10: This format of worksheet should be adopted for the intercept and planimetric methods. The report should document all of the pertinent identifying information, including:

- Composition.
- Specification or trade name.
- Customer or data requester.
- Date.
- Heat treatment or processing history.
- Specimen location and orientation.
- Preparation.
- Grain sizing method.
- List the number of measured fields.
- Magnification.
- Field area.
- The number of grains counted.
- A photomicrograph which is representative of the microstructure may be provided if required or desired.
- List the mean measurement value, its standard deviation, 95% confidence interval, percent relative accuracy and ASTM grain size number.

For the comparison method list only the grain size number.

For non-equiaxed grain structure, list the method of analysis, planes examined, directions evaluated and if applicable, the grain size estimate per plane or direction, the grain mean of planer measurements and the computed, or estimated, grain size number.

For two-phase structure, list the method of analysis, the amount of the matrix phase if determined, the grain size number of the matrix phase, the standard deviation, 95% confidence interval, percent relative accuracy and ASTM grain size number.

To express the average grain size of a group of specimens from a lot, do not simply average the grain size number, instead, compute the arithmetic average of the actual measurements, such as mean grains per mm^2 at x1 or lineal intercept length.

Notes

Equation 10. Relationship between grain size number and grains per square inch at x100

$$N_{AE} = 2^{G-1}$$

Where G is the ASTM grain size and N is the number of grains per square inch at x100 magnification. This is the basis of ASTM grain size number

Equation 11. Conversion for grains per mm2, at x1 to grains per inch2 at x100

$$G = 15.5000 \times n_a \, (per 1 in^2 at 100 \times)$$

Note: 1 inch is approximately 25.4mm; this approximation is sufficiently precise for the purposes of this standard.

ASTM E930-92 Standard Test Method for Estimating the Largest Grain Observed in Metallographic Section (ALA Grain Size).

This standard describes the test procedure for estimating the size of the largest grain observed in a metallographically prepared section.

Definitions

ALA grain: The largest grain observed in a random scatter of individual coarse grains, comprising 5% or less of the specimen area, where the apparent grain size of these grains differ by 3 or more ASTM grain size numbers from the balance of the microstructure.

Outlier grain: A grain substantially different in size from the predominant grain size in a microstructure; for example, an ALA grain.

The presence of large grains has been correlated with anomalous behaviour in, for example, crack initiation, crack propagation and fatigue.

Methods

Comparison Procedure

The comparison method requires the whole micro-section to be scanned and the larger grains to be noted.

The larger grains are then compared to the ALA grain size visual aid. This depicts a series, A to E, of sets of circles and ellipses with equivalent area to each other. The areas shown on the visual aid can be related with the ALA micro grain size. See

Table 5. below.

Table 5. Grain size number from ASTM E930

Area mm^2	Size
2.06	-4.0
1.46	-3.5
1.03	-3.0
0.703	-2.5
0.516	-2.0
0.365	-1.5
0.258	-1.0
0.182	-0.5
0.129	0.0
0.0912	0.5
0.0645	1.0
0.0456	1.5
0.0323	2.0
0.0228	2.5
0.0161	3.0
0.0114	3.5
0.00807	4.0
0.0057	4.5
0.00403	5.0
0.00285	5.5
0.00202	6.0
0.00143	6.5
0.00101	7.0

The comparison procedure requires the operator to locate the larger grain and make a comparison with the grain size visual aid in ASTM E930 A2. The comparison is made using the equivalent area of the individual grains, estimated using the visual aid. This method is accurate to plus or minus one grain size number.

Note: Each visual aid is only valid at the magnification shown.

Table 6. ASTM 930 A3 dimensions for ASTM 930 fig A2.1

Letter	Area mm^2	Diameters of figures mm (rounded to nearest 0.1 mm)				
		Circle	1:2 Ellipse		1:4 Ellipse	
			Major	Minor	Major	Minor
A	645	28.7	40.5	20.3	57.3	14.3
B	456	24.1	34.1	17.0	48.2	12.0
C	323	20.3	28.7	14.3	40.6	10.1
D	228	17.0	24.1	12.0	34.1	8.5
E	161	14.3	14.3	10.1	28.6	7.2

Measuring Procedure

For greater accuracy, the measurement method can be applied. Locate the position of the largest grain in a microscope image or in a photomicrograph. Using a measuring eyepiece, or graticule, measure the maximum calliper diameter and the calliper diameter in the direction perpendicular to the maximum calliper diameter.

Multiply the product of the two measurements by 0.785 to obtain the area of an ellipse with axes equal to the calliper diameters at the magnification used. Divide this area by the square of the magnification so that the area is that at 1x. Use

Table 5. to obtain the grain size rating by selecting the area nearest the area measured unless other wise specified.

The area as found by image analysis techniques could be used here instead. Any automatic or semi-automatic measuring device, which provides the area of a grain section, can also be used within the framework of this manual method.

Referee procedure.

The largest grain should be displayed using the largest magnification that shows the entire grain in the image area. A transparent grid can be applied to the image in order to determine the estimated area of the grain. Re-apply the grid three or four times at different angles. Take the average of the number of points falling within the grid and Use

Table 5. to calculate the ALA grain size number

Retain the photomicrograph. Record the average number of grid points counted for each grid placement, the total grid points counted, the average number of grid points counted, inter point spacing in a grid, magnification used and the ALA grain size number.

ASTM E1181-87 Standard Test Method for Characterising Duplex Grain Sizes.

This standard provides simple guidelines for determining whether or not duplex grain size exists. The test methods separate the duplex grain sizes into one of two distinct classes, and provide systems for grain size characterisation of each type. The two classes of duplex grain sizes are randomly varying, and topologically varying, and define specific types of grain sizes within these classes.

Recognising and Classifying Duplex Grain Size.

A Random Duplex grain size is defined the following.

- The presence of randomly distributed individual coarse grains, differing in size by three or more ASTM grain size numbers from the average size of the balance of the grains (henceforth referred to as the ALA). These individual coarse grains should comprise 5% or less of the area of the specimen.

A Topological Duplex grain size is defined as any of the following.

- The presence of systematic variation of grain size across the product, such that the grain size differs from one area to another by three or more ASTM grain sizes; or, the presence of different grain sizes in specific areas of the product, such that the grain size in these specific areas differs by three or more ASTM grain sizes. Both conditions henceforth known as the cross section condition.
- The presence of coarse grains, each surrounded by rings of finer grains, where the coarse and finer grains differ by three or more ASTM grain sizes is commonly referred to as a necklace condition.
- The presence of bands of distinct grain sizes, such that the sizes differ by three or more ASTM grain size number, is referred to as a banding condition

Methods

There are four procedures for estimating Area Fractions. For random class of duplex grain sizes, apply the chosen test method to 5 randomly selected areas of the specimen. For the topological class of duplex grain sizes, apply the selected test method to all of the specimen area if possible.

Comparison Procedure.

This procedure requires the use of a comparison chart to improve the accuracy of the visual estimates of area fractions occupied by distinct grain sizes. This comparison chart is shown in ASTM E1181 figure 1. Other comparison procedures that might be applicable are:

- Comparison method for largest grain size observed, E 930.
- Comparison procedure for estimating average grain size, E 112.

Point Count Procedure.

Point counting procedure, for estimating area fraction using a grid, as defined in ASTM E 562, can be used to estimate the area fractions occupied.

Outline the distinct grain size regions in each specimen. Mark the outline of the total field of view. Apply a regular two-dimensional grid to the image. The grid spacing should be matched to the image magnification in order to satisfy the conditions of E 562. Count the number of grid points falling within the predetermined grain size regions. Count the points falling on the boundary of a region as one half. Estimate the area fraction as the number of points falling within a certain area divided by the total number of points visible within the image outline. Note: The grid must be large enough to completely cover the image.

Planimetric Procedure.

A planimeter is a device which can be used to measure areas of irregular polygon and as such, may be used to measure areas occupied by distinct grain sizes on specific images of a specimen.

Outline all areas consisting of grains of a distinct size. Mark the outline of the total field of view. Use the planimeter to determine the area with each region of distinct grain size. Also, use the planimeter to calculate the total area. Calculate the area fraction occupied by the regions of each distinct grain size.

Direct Measurement Procedure.

This procedure may be applied to topological duplex specimens that show surface layers of differing grain sizes. Make at least 10 measurements at different locations of the depth of the given surface layer. Use the average depth, and overall product dimensions to calculate the estimated area fraction of that surface layer.

Determining Grain Sizes

Different applications of ASTM E 112 and E 930 can be applied. The comparison method is simple to apply, but is the least precise of all the E 112 methods. The planimetric method does not lend itself to measurement of small areas. The different intercept methods can be used on regions of discrete grain size; these methods may be more difficult to apply but provide better precision.

Statistical Determination of Grain Size Distribution.

Ref. ASTM E1181 Section 8.7 and Appendix X2

This method requires the use of a test grid of parallel lines with 5mm spacing. The test is conducted on four angular positions upon the specimen 0,45,90,135 degrees. Measurements from all grid positions should be treated as one data set. Classify the intercept lengths according to pre-selected classes intervals. This data may be used to determine the nature of the observed grain size distribution. Also, it can be used to determine mean intercept lengths and area fractions for distinct segments of a total distribution.

An example of the application of this procedure is provided in Section X1 of E1181: Formulas for determining mean intercept length and area fractions are described and the presentation of the data as a histogram or a frequency plot illustrated.

Estimates of the area fraction arising from this procedure are only valid if the conditions of ASTM E1181 Section 8.4.2 are met. For a random duplex grain distribution, at least five different randomly selected areas of the specimen should be sampled.

Reporting Results

Characterising and reporting duplex grain size.

Ref. ASTM 1181 Section 8.6

1. Begin a duplex grain size report, with the orientation used for the evaluation.
2. Use the abbreviation “Long” for longitudinal and “Trans” for transverse.
3. Use the abbreviation “AGS” for average grain size and the abbreviation “OCC ALA” for the phrase occasionally as large as.
4. For random ALA conditions, use ASTM E 930 to estimate the size of the largest individual specimen on the sample. Use E 112 to determine the average grain size of the balance of grains in the specimen. Report the results using the following designation:

   “Duplex, ALA, AGS, ASTM No. ________, OCC ALA ASTM No. ________”, with the proper values inserted in the blanks. For example:

   “Long. Duplex, ALA, AGS ASTM No. 6 OCC ALA ASTM No.1”
5. For the random, wide range condition use method E 112 to determine the average grain size of the specimen. Use test method E 112 to determine the smallest grain size in the specimen and method E 930 to estimate the largest grain size in the specimen. Report these results as:

   Duplex Wide Range AGS ASTM No. ____, range ASTM No. ____to ASTM No. ____ with the proper values filled in the blanks. For example:

   “Long Duplex Wide Range AGS ASTM No.4 range ASTM No.8 to ASTM No.1”
6. For random bimodal conditions, use test method E 112 to determine the average grain sizes of the two distinct populations of grain sizes. Use one of the procedures from E1181 to estimate the area fractions occupied by the two grain sizes. Report results as:
   Duplex Bimodal ____% AGS ASTM No. ___ ___% AGS ASTM No. ___ inserting the corresponding area fractions and average grain sizes in the blanks. For example:

   Long Duplex Bimodal 30% AGS ASTM No.7.70 AGS ASTM
7. For the topological cross section conditions, use test methods E 112 to determine the average size of the grains for at least two extremes of the variation pattern. Note the locations associated with the two extremes. Report these results as:

   Duplex Cross Section AGS ASTM No.____ at ____ to AGS ASTM No. ____ at ____, inserting the corresponding average size values and location s in the blank spaces. For example:

   Long duplex cross section AGS ASTM No.5 at centre to AGS ASTM No.0.5 at surface.
8. For the topological necklace condition use test method E 930 to estimate the typical size of the individual coarse grains and test method E 112 to determine the average grain size of the fine grains. Use one the procedures from E1181 Section 8.4 to estimate the area fractions occupied by the coarse grain and the fine grains. Report the results as:

   Duplex Necklace condition ____%ASTM No.____ ____%AGS ASTM No.____ , inserting the first two blanks, the area fraction and typical size of the coarse grains respectively and then the area fraction, the area fraction and the average size of the fine grains. For example:

   Long duplex Necklace 95% ASTM No.0, 5% AGS ASTM No.8

Examples of reporting results are illustrated in ASTM 1181 Section 8.6.

ASTM E1382-91 Standard Test Method for Determining Average Grain Size Using Semiautomatic and Automatic Image Analysis.

This standard defines test methods for determining ASTM grain size using the following methods: Measurement of grain intercept lengths, intercept counts intersection counts, grain boundary length and grain areas.

Limits of use.

These methods are applicable provided that the grain boundaries can be segregated. This is dependant on the composition of the sample and the preparation, which requires good polishing to remove scratches and etching to expose the grain boundaries to view.

Test Procedures

This standard makes provision of both semi-automatic and automatic image analysis.

Semiautomatic Image Analysis - Digitising Tablet

Ref. ASTM 1382. Section 12

This part of the standard defines a method by which a digitising tablet can be used to measure the grain size.

Methods

Intercept length method.

The intercept method is carried out using a digitising tablet; a template consisting of five parallel lines that are separated by a gap larger than the apparent mean grain diameter. Measure only the cord distances between successively intercepted grain boundaries. Generally each line will begin and end in a grain and the partial cords will not be measured. The template should be presented at two or more orientations in order to average any anisotropy. If anisotropy is clearly present, measurements at 0,45,90,135 should be carried out; see appendix ASTM 1181. This procedure should be repeated on at least five different micrographs, until at least 500 intercept cords have been measured.

If the grain elongation is of interest, the intercepts should be measured parallel and perpendicular to the direction of elongation. The extent of elongation is then the ratio of the average intercept length, in the direction parallel to the direction of elongation, to the intercept length perpendicular to the direction of elongation.

The average intercept length is calculated by summing the length of the intercepts and dividing by the number of intercepts. A histogram of intercept length can be constructed. This can be used to check the uniformity of grain intercept length and detect any duplex grain size conditions. The standard deviation s is calculated

Equation 12. Calculation of standard deviation

$$s = \left[\frac{1}{N-1} \sum (l_i - \bar{l})^2 \right]^{1/2}$$

N is the number of intercepts measured, l bar is the average intercept length and l_i is the individual measurements. If the histogram reveals a duplex condition in the structure, the standard deviation should be determined for each distribution.

Intercept and Intersection Count Methods.

A digitising tablet can be used to count the number of grain boundary intersections (P), or the number of grains intercepted (N); the former is preferred. A circular test line is used in the same manner as described in ASTM E 112. The test grid that consists of three circles, as described in test method E 112. Any size circle can be used provided it is larger than the largest grain in the field. The same recommendations as in E 112 standard apply; i.e. use 3 concentric circles, with total 500 mm in line length.

The average number of intersections should be 100, with a minimum of 70 and a maximum of 150 unless grain size is too coarse. This ideal may not always be achievable, depending on the magnification steps. Values outside these ranges may be used, but the number of fields counted should be changed to achieve counting totals of 500 grain boundary intercepts and a minimum of five randomly selected fields. A minimum of 500 intersections can be used as a defining requirement for the number of fields to be scanned. A similar method using straight lines can be used, however, the requirement to count the ends of the lines, as per the rules in E 112, requires a tally of half counts to be kept.

With a circular test grid, the line end counting problem is eliminated. The counting rules are as follows:

- A tangential intersection with a grain boundary is counted as one.
- Each grain boundary cut by the test line is counted as one intersection.
- Count the intersection with the junction of three grain boundaries (triple point) as 1 ½ Intersections.
- At the junction of four grains count the intersection as 2.

Grain count (Planimetric) method

This method, the Jeffries Planimetric Method,can be carried out using a digitising tablet to mark off the grains to get an accurate count.

5 fields are required. The number of whole grains in each field are counted and the area which they occupy is calculated. The number of grains per unit area from the count and the area are also calculated (number per mm^2 is preferred). The average number of grains per unit area for the number of fields measured is then determined.

Grain area method

Grain areas can also be measured by image analysis using a digitablet, this may be tedious and therefore is not recommended for general use. However, it may be useful for rating ALA grains as described in test ASTM E 930. The boundary of large grains in question is marked out using the digitablet and the area is measured. The area can be converted to a grain size number rating.

Methods for 2 phase structures

The grain size of a particular phase within the microstructure can be determined using a digitablet. The simplest method is to measure intercept lengths of straight lines in the phase of interest; only intercept lengths over grains of interest are recorded. The average intercept length and standard deviation should be calculated.

An alternative procedure, more difficult to implement, is to determine the area fraction of the phase of interest using area fraction, or point fraction procedure e.g. E 562.

Apply a circular test pattern and count the number of grains of the phase of interest intercepted by the test lines of known length. The mean lineal intercept length is then calculated.

Equation 13. Calculation of mean lineal intercept length

$$\overline{l_a} = \frac{(\overline{A_{Aa}})(L_{ti})}{N_a} = \frac{(\overline{P_{Pa}})(L_{ti})}{N_a}$$

Where A and P bar are the area fraction, α is the point fraction of the phase of interest, L is the true test line length of the grid, and N bar is the average value of the number of α grains intercepted by the test circle, or circles for fields measured (n), determine the standard deviation of $l_{\alpha i}$

Automatic Image Analysis

Ref. ASTM 1382 Section 13.

The accuracy of grain size measurements is highly dependant on the delineation of the grain boundaries on the specimen, since this has a direct effect on the ability of the automated image analysis system to segregate grain boundaries.

Grain boundary length/area method.

The simplest procedure for determining grain size is to detect the grain boundaries using grey level thresholding. The boundaries that intersect with the image boundaries are not removed. The total length of boundaries in the field is measured. Divide this length by the field area to obtain the grain boundary length per unit area in mm/mm^2 L_{iA} . Divide this value by $\pi/2$ to give P_{Li}, the number of grain boundary intersections per unit length of test line for each field and calculate the mean lineal intercept length for l all measured fields. The standard deviation should also be calculated.

Equation 14. Grain boundary intersections per unit length

$$P_{Li} = \frac{L_{Ai}}{p/2}$$

Equation 15. Mean intercept length is the reciprocal of the number of intesections per unit length

$$\bar{l} = \frac{1}{\overline{P_L}}$$

Intersection counts method.

Grain boundaries are detected and those intersecting the field boundary should be removed. The number of intersections of the grain boundaries and the scan lines are counted.

The number of grain boundary intersections is divided by the true total scan line length to give the number of intersections per unit length. This can be averaged over a number of fields. The image should be rotated and the measurement repeated to avoid the effect of any anisotropy that may be present. Horizontal and vertical scan lines can be used instead of rotating the image. The mean lineal intercept length and standard deviation can then be calculated.

Intercept cord length method.

Grain size can also be determined by field, or feature, specific, chord length measurements. Detect the grains interiors and remove those grains that intercept the field boundary to avoid measuring partial chords. The image should be rotated or measurements should be made in more than one direction so that the affect of anisotropy is avoided. If the extent of elongation is significant, the measurements should be made parallel and perpendicular to the direction of elongation. The ratio of average chord length parallel and perpendicular to the direction of elongation, defines the degree of grain elongation. The mean chord length for each field should be calculated. This measurement is repeated for at least five fields. The mean intercept length for n fields is calculated. If the grain size distribution is duplex then the grain size within each portion and the amount of each type is determined as described 12.3.5 and Appendix X2 of test method E1181.

Grain count method.

Grain size may also be determined by a count of the number of grains within a test area. Detect the grains interiors and remove those grains that intercept the field boundary to avoid measuring partial grains. The measurement area is the sum of the grain interiors and the grain boundaries between these grains. The grains completely within the measurement area are counted and divided by the test area to obtain the number of grain per unit area. This process is repeated for at least five fields. The average number of grains per unit area is calculated, as is the standard deviation.

Average grain area method.

The grain size can also be determined by measuring the total of all of the grains within a field and then dividing by the number of grains in the field to determine the average grain area. This should be repeated for at least five fields and the standard deviation should be calculated.

Individual grain area method.

The grain area of individual grains completely within the field boundary can be measured. This measurement should be repeated for as many fields as is required to measure at least 500 grains. Calculate the mean grain area for N grains, for true area units μm^2 and mm^2. A histogram of the frequency of grain areas can be constructed. Calculate the standard deviation. The area of the largest grain can be reported.

Methods for 2 phase structures.

The grain size of a particular phase can be determined using an automatic image analyser. The cord and individual area methods can be adapted so that the cords and areas of the phase of interest only are detected. The average cord length and grain area can be calculated as well as the standard deviation.

Calculating results.

In addition to the parameters measured above, the averages and standard deviation calculated the confidence limit can be reported.

Reporting Results.

The test report should document the identifying information regarding the specimen, including:

- Composition.
- Specification.
- Designation or trade name.
- Customer or data requestor.
- Heat treatment or processing history.
- Specimen location and orientation.
- Etchant etch method.
- Analysis method.
- List the number of fields measured.
- Magnification.
- Field area.
- Total measurement area.
- A photomicrograph (if desired).
- Image processing techniques applied to the grey image before analysis if applied.
- List the mean measurement value, its standard deviation, 95% confidence interval, and percent relative accuracy.
- List the computed or estimated ASTM grain size number.
- For a 2 phase microstructure, describe the nature of the phases or constituents present and the test results for the phase/ phases of interest.
- For a duplex microstructure, describe the nature of the duplex condition, and the test results for each portion of the distribution.
- For a specimen with a non-equiaxed structure, state the planes measured, the type of measurement method, random grain count, grain area, intercepts, direct test lines, the specific measurement for each test plane, or direction on the planes, the mean test measurement and the resulting mean grain size of the specimen. Report the pooled standard deviation of the mean values of the measured parameter.

ISO 643 Steels-Micrographic Determination of the Ferritic or Austenitic Grain Size.

Characterisation of ISO 643 grain size index.

Equation 16. Relation between ISO grain size number and grains per mm²

$$m = 8 \times 2^G$$

Where m is the number of grains per mm^2, G is the grain size number. This is the basis of the determination of ISO 643 grain size.

Methods

Preparation methods for samples are described in Section 5 of the standard.

Comparison Method

Table 7. Compensation for magnification using chart comparision

Magnification of the image.	Index of metal grain for an image identified on a standard chart No							
	I	II	III	IV	V	VI	VII	VIII
25	-3	-2	-1	0	1	2	3	4
50	-1	0	1	2	3	4	5	6
100	1	2	3	4	5	6	7	8
200	3	4	5	6	7	8	9	10
400	5	6	7	8	9	10	11	12
800	7	8	9	10	11	12	13	14

Equation 17. Calculation of grain size number at magnification other than x100

$$G = M + 6.64 \log \frac{g}{100}$$

When the magnification of the chart examined (g) is not 100, the index (G) should be equal to the number of the closest standard chart (M), modified as a function of the ratio of the magnifications (see above equation).

Area Method

By definition, index G (ASTM) = 1, corresponds to 15,500 grains per unit area (1 square mm) and the other indices can be calculated.

Equation 18. Calculation of ASTM grain size

$$G(ASTM) = -2.9542 + 3.3219 \log_{10} m$$

Where, m is the number of grains per mm^2

The number of grains in a circle of known area is counted. The grains per unit area value should be converted into a grain size index using the equation above.

Equation 19. Derivation of relation between ASTM grain size and ISO grain size

$$G_{(ASTM)} - G = 0.0458$$

This equation reveals that the ASTM grain size index gives a grain size slightly larger than the one defined by the ISO standard. The difference is less than one twentieth of an index unit and hence is negligible.

Intercept method

See section 6.2.1.2 for rule of intercept method.

The intercept method uses a measurement grid similar to that used in ASTM E112. The end of a measurement line is counted as only ½ when it exactly touches a grain boundary. When the intercept line is tangential to the grain boundary one intercept is also counted. When the intercept coincides with a triple point (junction of 3 grains), 1.5 intercepts are counted. In the case of irregular shape grains, where the line intercepts the same grain at two different points, both intercepts are counted.

A circular intercept method is described in Section 6.2.2

Count Method

The magnification is set so that at least 50 grains can be seen in a 5000 mm^2 circle, with diameter 79.8mm. The number of grains within the circle (n1) and the number of grains partially in the circle (n2) are counted. The total number of equivalent grains (n100) is calculated.

Equation 20. Calculation of nominal number of grains per mm^2 at any magnification

$$n_{100} = n1 + \frac{n2}{2}$$

The number of grains per square mm is shown below:

$$m = 2 \times n_{100}$$

Or in the case of any magnification g:

$$m = 2\left(\frac{g}{100}\right)^2 n_g$$

The value of m corresponds to a value of G, the ISO grain size number, according to the limits shown in

Table 8. below from ISO 643. This is only valid for equiaxed grains.

Table 8. ISO 643 Table 3 Grain size number relation to grains per square mm

Grain Size Indices G	Nominal Value grains per square mm	From (exclusive) grains per square mm	To (inclusive) grains per square mm
-7	0.0625	0.046	0.092
-6	0.125	0.092	0.185
-5	0.25	0.185	0.37
-4	0.5	0.37	0.75
-3	1	0.75	1.5
-2	2	1.5	3
-1	4	3	6
0	8	6	12
1	16	12	24
2	32	24	48
3	64	48	96
4	126	96	192
5	256	192	384
6	512	384	768
7	1024	768	1536
8	2048	1536	3072
9	4096	3072	6144
10	8192	6144	12288
11	16384	12288	24576
12	32768	24576	49152
13	65536	49152	98304
14	131072	98304	196608
15	262144	196608	393216
16	524288	393216	786432
17	1048576	786432	1572864

Assessment of Results

Calculate the mean number of intercepts over a number of fields, then calculate the mean intercept length. In the case of non-equiaxed structures the number of intercepts on each on the principle axes of the specimen should be calculated and the mean of these values reported.

Report Results

The following must be included:

- The grade of steel.
- The type of grain determined (ferritic or austenitic).
- The method used and the operating condition.
- The grain size index or the value of the mean segment.

DIN 50601 Determination of Ferritic or Austenitic Grain Size of Steel and Ferrous Materials

This standard is based on the ISO 643 and the EURONORM 103-71. From the explanatory notes it is understood that the only difference is that more details of the specimen treatment for austenitic steels are included.

Characterisation of grain size.

Equation 21. Grain per mm² relation to grain size number

$$m = 8 \times 2^{G}$$

Where m is the number of grains per mm², G is the grain size number. This is the basis of the determination of DIN grain size.

Pre-Treatment of Samples.

The pre-treatment of samples is used to make the grain boundaries visible for examination under the microscope. This applies particularly for steels in which the grain size of the austenite before the last γ-α phase transformation is measured.

Methods

Comparison method

Comparison is made with the eight standard chart images that are used. These images are included in the standard on page 5.

Intercept method using standard overlay

Determination of the grain size and the mean intersected segment length (Ls)can be achieved using the standard overlay (similar to ASTM E112 overlay). There is no relationship between intercept length (DIN) and grain size number. It is implied that intercept length is sufficient. The DIN grain size can be calculated from the ASTM grain size relationship with intercept length, and then correcting the grain size number. The DIN grain size number should be reported to whole figure accuracy only and so the 0.05 grain size number correction will have negligible effect.

Counting the grains intersected by a measurement line. (Snyder-Graff method)

This is a special application of the linear intersected segment method for determining the austenitic grain size of high speed steels and steel with particularly fine grain in the hardened and tempered condition.

Counting the grains within a circle and statement of the grain size index resulting from the mean grain area.

The number of grains per mm² is estimated by counting the number in a circle of known area and then using DIN 50 601 Table A2. converting to a grain size number (G).

Comparison with E112

Appendix A2 compares the DIN grain size with ASTM E 112.

The following definitions apply:

Using the intersected segment method.

ASTM E 112 defines the Ls as 32 at G (ASTM)=0

L_s is the mean length of intercepted segment in mm.

Equation 22. ASTM grain size number calculated from mean intercept segment

$$G_{(ASTM)} = 10 - 6.64 \log(100 \times L_S)$$

Using the counting method.

Equation 23. Relation between DIN grain size number and ASTM Grain size number

$$m = 8 \times 2^G$$

$$G_{(ASTM)} = \frac{\log m}{\log 2} - 2.95$$

$$G_{(ASTM)} - G = 0.05$$

$G_{(ASTM)}$ is the ASTM grain size, G is the DIN grain size and m is the number of grains per mm^2.

Reporting Results.

The following must be included:

Ref. Section 9 DIN50 601

- Steel grade and identification of cast.
- Material condition the thermal pre-treatment, and a description of the structure as appropriate.
- Type of grain size as described in sub clause 2.2 of the standard. For non-transformed steels, Austenite, Ferrite, and composites of both and steels with γ/α transformation Ferrite, Pearlite and composites of both, the grain size of austenite before the last γ/α transformation should be recorded.
- Method used as specified in the standard, giving the magnification, if this differs from the standard case.
- Grain size index to whole figure accuracy.
- Any deviation from this standard.

BS EN ISO 2624-1995 Copper and Copper Alloys-Estimation of Average Grain Size.

This standard is similar to the ASTM E 112, the standard chart images are those taken from the ASTM E112 1985.

Methods

Comparison Procedure

Compare a visual field with standard images at x100.

Intercept Procedure

The intercept method uses a measurement grid similar to that used in ASTM E 112. At least 50 intercepts are required, with at least 10 per line. 200 are required for referee purposes. For non-equiaxed specimens the mean intercept length on the three principle planes of the specimen should be determined. There are no instructions regarding the presentation of results. However, this standard has a strong resemblance to ASTM E112, so it does not seem unreasonable to do the same as the E112 does here.

Planimetric Procedure

A magnification which has at least 50 grains in a visual field should be used; alternatively, 200 grains for referee purposes and these figures should comprise the total number of complete grains, plus half the number of grain bisected by the field edge.

Reporting Results

Comparison method

The estimated grain size for each field should be reported. Where a single figure is required, the median of these results should be reported.

Planimetric and Intercept method.

In equiaxed material, the result for each of the three, or more required fields should be reported. If one figure is required, the median should be reported.

Mixed grain sizes.

When these are encountered, the estimated area fraction and size by the comparison method should be reported.

BS 4490 Micrographic Determination of the Grain Size of Steel.

This standard shares similarities with ISO 643 and ASTM E 112. The European grain size number (G_{E},) and American grain size number (G_{A}) are referred to. The standard chart images included are the AFNOR (EU103-71) images and also the ASTM E112 images.

The standard says in the “Foreword” that the main difference between this standard and the ISO 643 is the number of preparation methods supported.

Methods

Comparison procedure.

Comparison procedure is carried out using the AFNOR (EU103-71) images and also the ASTM E112 images. The image at x100 should be compared with the standard chart and the number (M) of the closest standard image. When using magnifications other than x100 a correction should be used. See Table 9.

Table 9. Relationship between indices for the usual magnifications.

Magnification of the image	I	II	III	IV	V	VI	VII	VIII
25	-3	-2	-1	0	1	2	3	4
50	-1	0	1	2	3	4	5	6
100	1	2	3	4	5	6	7	8
200	3	4	5	6	7	8	9	10
400	5	6	7	8	9	10	11	12
800	7	8	9	10	11	12	13	14

Intercept procedure (Lineal analysis).

The intercept procedure is similar to ISO 643, with some differences, and additions.

The procedure employs the familiar grid of straight lines, 500 mm in total length, and three circles, also with 500 mm total length, as used in ASTM E 112. It is superimposed upon the field of view at x100 magnification.

Linear intersected segment method.

Using the grid described above, count the intercepts of grain boundaries with the horizontal and vertical and diagonal lines. Apply the following rules to the counting of intercepts.

- If a measurement line terminates exactly on a grain boundary, count as plus 0.5
- If the measurement line is tangential to a grain, or phase boundary, count as plus 1.
- If the measurement line coincides with a triple point, count as plus 1.5.
- If the measurement line intersects the same grain or phase four times, count as plus 2: i.e. two segments within the grain or phase.

Calculate the mean intersected segment length L. The grain size index can then be calculated.

Equation 24. Calculation of grain index from mean intersected segment length

$$G_A or G_E = -3.2877 - 6.6439 \log_{10} \overline{L}$$

Circular intersected segment method.

Select the magnification, so that at least 50 intercepts are made with the measurement grid (three circles) which should be superimposed on the field to be measured. In cases where only the largest circle is used, 25 intercepts should be made. In order to offset the low number of intercepts, which is characteristic of the circular intercept method, the triple point intercept can be counted as 2 intercepts but a, b, d, still apply. Calculate the mean intersected segment length (L). The grain size index can then be calculated.

Equation 25. Count of number of grains per square millimetre

$$G_A or G_E = -3.2877 - 6.6439 \log_{10} \overline{L}$$

Count of number of grains per square millimetre method

Determine the number of grains per square millimetre by superimposing a circular reference grid on to the specimen image. Adjust the magnification so that at least 50 grains are enclosed in the reference grid. Count the number of grains enclosed by the grid and the number of grains intersected by the grid.

The number of grains per mm squared (m) can be calculated and the grain size number calculated.

Equation 26. Calculation of ASTM grain size index from grains per mm squared

$$G_A = -2.9542 + 3.3219 \log_{10} m$$

Where m is the number of grains per mm squared and G_A is the ASTM grain size index.

Alternatively:

Equation 27. Calculation of European grain size number

$$G_E = \frac{\log_{10} m}{\log_{10} 2} - 3$$

Reporting Results

The following must be reported.

- The grade of steel examined.
- The type of grain determined.(Ferrite, Pearlite, Austenite)
- The method used, reference standard and the operating conditions.
- The grain size index or the mean value of the intersected segments.
- The time, and temperature of any heat treatments that are used.

SAE Grain Size Determination of Steels SAE J418 DEC83

This standard is based on the ASTM E 112. Most of this standard is concerned with preparation of samples. The standard refers to the standard methods of grain size evaluation.

JIS G0552 Methods of Ferrite Grain Size Test for Steel

Limits of Use:

Used for steel with not more than 0.2 percent carbon.

Definitions:

i) Grain Size: Grain size of the Ferrite crystal expressed in grain size number.

ii) Mixed grain: The presence of 20 % of grains, or more, within one visual field, which differ in size by 3, or more, grain size numbers.

iii) Elongation rate: Ferrite crystal grains may be elongated due to the working that should be calculated by the following formula.

Where c is the elongation rate. n_1 is the number of grains intercepted by a line of a certain length in the orthogonal to the direction in which the grains have been elongated. n_2 is the number of grains intercepted by a line of a certain length in the direction in which the grains have been elongated.

Equation 28. Calculation of elongation rate

$$c = \frac{n_1}{n_2}$$

Testing Methods

There are only two methods of calculating grain size in this standard.

- Comparison Method
- Synthetic Determination.

Comparison Method

This method is usually used where grains are not elongated, and precise numerical accuracy is not required. Compare what you see down the microscope at 100x magnification with the standard images supplied. As a rule the magnification used should be x100. The actual visual field should have a diameter of 0.8mm. For a grain size falling between two consecutive grain sizes, the smaller grain size number shall be used, but the number 0. 5 shall be added to that number.

When it is difficult to make a comparison at 100x, 50x or 200x can be used. But, at 50x the grain size number obtained by the manual comparison method should be decreased by 2. When 200x is used then the grain size number should be increased by 2.

For mixed grains the proportion of the areas for each grain shall be determined by eye.

In the case where Pearlite and the like are mixed in great numbers, so far as then mixed state is banded or granular, the area proportion of mixed structure and Ferrite crystal should be determined by eye. Subsequently, determination of the corresponding grain size number for only the Ferrite crystal grain should be made. Compare with the standard figure Ref. JIS G0552 Fig 1.

Use table below when the measurement is made by the comparison method using JIS comparison images.

Table 10 Grain size number relationship with grains per unit area from JIS G0552

Grain size number	Number of crystal grain per mm^2 of sectional area	Mean sectional area of crystal grain mm^2	Mean number of crystal grain in 25 mm^2 at 100x Magnification (n)
-3	1	1	0.0625
-2	2	0.5	0.125
-1	4	0.25	0.25
0	8	0.125	0.5
1	16	0.0625	1
2	32	0.0312	2
3	64	0.0156	4
4	128	0.00781	8
5	256	0.0039	16
6	512	0.00195	32
7	1024	0.00098	64
8	2048	0.00049	128
9	4096	0.000244	256
10	8192	0.000122	512

Synthetic Determination.

Synthetic determination provides a method of combining the comparison method and grain size results for a number of fields on the same specimen, to produce a single weighted grain size number result.

Equation 29. Calculation of mean grain size number for synthetic determination

$$m = \frac{\sum a \cdot b}{\sum b}$$

Where m is mean grain size number, a is grain size number in each visual field and b is the number of visual fields indicating the identical grain size number.

Table 11 Calculation of grain size synthetic determination, example from JIS G0552.

Grain size number in each visual field (a)	Number of visual fields at each grain size number (b)	(a∗b)	Mean grain size number (m)	Grain size
6 6.5 7	2 6 2	12 39 14	65/10=6.5	6.5
Total	10	65		

See Reporting the Results below for details of how to report the results of this method

Intercept Method

The number of Ferrite grains intercepted by two segments of fixed length orthogonally crossing each other should be determined.

Two straight lines of fixed length oriented at right angle to each other are superimposed over the field of view. The intercept is the number of grains that the line passes though. When two grains are partially intercepted at each end of the line, they will be counted as one; hence the count is equal to the number of grain boundaries crossed by the line. In the event that the line partially intercepts only one grain, then no measurement should be made.

Magnification of the microscope should be adjusted so that the number of Ferrite crystal grain to be intercepted by one segment is not less than 10 in one visual field. The measurement should continue for several visual fields, until the number of grains intercepted amounts to not less than 50. Grain size number should be rounded of to the first decimal place. Elongation rate if necessary should be noted. Provision for a comment regarding the orientation of the elongation within the artefact should be made.

When Pearlite and the like are present and mixed in great numbers, the area proportion of the mixed structure and the Ferrite grain should be determined. Using an appropriate method e.g. point count method, gravermetric method or linear analysis method. The number of crystal grain in 25mm^2 at x100 should be measured by the intercept method. The results should be converted into the number of Ferrite crystal grain per 25mm^{2,} in order to obtain the grain size number determined by Equation 31. .

Note: The methods described in this standard are similar to those used in the ASTM E 112 standard. The methods are similar to the ASTM standard using SI units.

Comparison Method.

The grain sizes, appearing on the etched surface of the specimen, should be observed under a microscope and compared with a standard figure. The user should use grain images created using the current calibration. The relation between grain size number and grains per (25mm)^2 at x100 is as follows:

Equation 30. Relation between grains per 25mm^2 and JIS Grain size number

$$N = \frac{\log n}{0.301} + 1$$

Ref. G 0552 (1)

N = Grain size number, n = number of grains per (25mm)^2 at x100

The expression can be rearranged to provide a relation for the actual number of grains per mm^2 (g).

Equation 31. Relation between the actual number of grains per mm^2 and JIS grain size number

$$2^{N+3} = n_a$$

Where n_a is the actual number of grain per mm^2 at x1 magnification.

This is illustrated in Table 10 above, which is taken from the standard.

Hence by calculating the number of square mm in the image using the calibration value and the screen resolution dimension, an appropriate number of grains to be seeded within this area could be calculated. This method should provide a suitable comparison image, although the effect of grains on the edge of the image may make it difficult to verify the grain size displayed.

Testing of the comparison images should be carried out using the same methods as in the standard.

User input required for comparison method.

- Specimen ID.
- Plane orientation.
- Selection of method: i.e. comparison/intercept method.
- Is grain size mixed? If so, record how many different sizes are present.
- Number of visual fields is not necessarily needed; they can be counted in the process.
- Selection of matching comparison image.
- If the comparison falls between images, take lowest and add 0.5.
- Determination of fraction of mixed grains for each visual field.
- Location of test plane.
- General comment.

Reporting Results

1. Grains size number in the format as described in the following. Grain size number is limited to multiples of 0.5, in the range –3 to 10. In the first instance, some flexibility in these limits might be advantageous. Also, the ability to produce a warning when values in certain ranges are evaluated. These features would be useful in routine analysis. Users will be surprised by values which are well out side the accepted range or theoretically impossible. The standard notation should be used, see examples in "Reporting Results".
2. Comment on the nature of specimen; mixed grain sizes or Ferrite Pearlite structure details.
3. Comment on location of test plane.
4. Details of synthetic determination and mean grain size number and mean grain size proportion if mixed grain sizes are apparent.
5. Elongation rate is not appropriate for the comparison method.
6. The usual configuration data calibration value, date, time etc.

Intercept Method

Draw lines horizontally and vertically across the screen, then use the calculated screen width and height.

The intercepts should be counted for each of these lines and n is calculated. This value is used to calculate the grain size number N. Note: Ferrite crystal grains only should be counted. In microstructure consisting of Ferrite and Pearlite, the latter is ignored.

The grain size number N should be reported to one decimal place only.

Equation 32. Grains per 25 mm^2 at x100

$$n = 500\left(\frac{1}{100}\right)^2\left(\frac{I_1 \times I_2}{L_1 \times L_2}\right)$$

n is the grain per $(25mm)^2$, L total linear length (in mm) of the segments (1,2) crossing each other, I is the number of grain intercepted by the lines (1,2) and N is the grain size number.

Equation 33. Calculation of grain size number

$$N = \frac{\log n}{0.301} + 1$$

Elongation rate

Elongation rate should be reported when this method is used. It is important to determine the direction of elongation and the orthogonally orientated lines positioned, so that one of them is parallel to the direction of elongation.

Reporting Results

The reporting of the grain size should be formatted as follows:

Method

- FG_c : Comparison method
- FG_I : Intercept method

Orientation of section measured

- V : Vertical cross section
- P : Parallel section
- S : Surface

Examples of notation for reporting JIS Grain size

Not mixed grain

- A grain size of 3.5 in vertical cross section, derived by synthetic determination, obtained from 10 visual fields by comparison method: FG_c -V $3.5_{[10]}$

> Note: Grain size number should always be rounded to one decimal place.

- A grain size of 6.5, in a parallel section derived by intercept method: FG_I – P6.5
- A grain size of 3.5, in a parallel section, mixed with 20% banded Pearlite, derived by synthetic determination, obtained from 10 visual fields by comparison method as outlined in 5.1. FGc-P3.5[10](Mixed with 20% banded Pearlite)

For mixed grain

70% with grain size of 3, 30% with grain size of 6, in vertical cross section, derived by synthetic determination, obtained from 10 visual fields of mixed grains by comparison method as outlined in 5.1: FG_c-V$\{3(70\%)+6(30\%)\}_{[10]}$

Elongation rate

For elongated crystal growth, the elongation rate should also be noted as given below, where necessary:

e-P2.3

Location

If necessary the location of the tested planes shall also be noted.

Notes

$$N = \frac{\log n}{0.301} + 1$$

Equation 34. Derivation of relation between grain size number and grains per mm²

Ref. G 0552 (1)

Ref. JIS G0552 Fig. 3. This illustrates a relation ship within formula 1.

$$n_a = \frac{n}{(\frac{25}{100})^2} = 16n$$

Equation 35. Calculate the relation between the actual number of grain per mm2 and the number per 25mm² at 100x (n)

Substitute n grains per 25mm ^{2} at x100, with n_a the actual number of grain per mm^2.

$$N = \frac{\log n_a - \log 16}{0.301} + 1$$

0.301=log 2.

$$N = \frac{\log n_a}{0.301} - 4 + 1 = \log_2 n_a - 3$$

This relationship below can be clearly seen in Table 10.

$$2^{N+3} = n_a$$

Equation 36. Derivation expression for JIS grain size in terms of actual grain per mm²

Intercept Method

$$n = 500\left(\frac{M}{100}\right)^2\left(\frac{I_1 \times I_2}{L_1 \times L_2}\right)$$

Equation 37. Expression for grains per (25 mm)² at x100

Where N is the grain size number, n is the number of grain size in 25mm^2, under a microscope at 100x magnification, calculated from an image viewed at magnification M, the microscope magnification, L_1 (or L_2) total length (in mm) of one linear length of the segments orthogonally crossing each other and I_1 (or I_2) is total number of crystal grain intercepted by L_1 (or L_2).

Consider the case where M=1; the micrograph being analysed shows the grains at the same resolution as the specimen. Hence L is the real length of intercept in mm, I is the number of intercepts on the line length (L) and N is the number of grains per (25mm)^2 at x100. N the grain size number can then be deduced as before.

Equation 38. Derivation of relation between grain size number and grains per mm²

$$N = \frac{\log n}{0.301} + 1$$

JIS G0551 Method of Austenite Grain Size Test for Steel.

The JIS G0551grain size rating is the same as for JIS G0552. However, the difference is in the preparation of the specimen and its appearance. See standard chart images.

Limitation of Use

This Japanese standard specifies the testing method of measuring austenite grain size.

Definitions

Grain Size: The term grain size means the size of austenite grain, established by the heating temperature and holding time when steel has been heated to a temperature above its transformation point (Ac3,AC1,or Accm), or to a temperature for the solution treatment for purposes of annealing, normalising ,quenching, carburising or other heat treatment processes.

Grain Size Number: The grain size number refers to the number of the grain size measured by the method specified below, and classified in Table 10.

Ref. JIS G0551

Methods

Comparison procedure.

This method is the same as the JIS G0552.

Synthetic determination.

This method is the same as the JIS G0552.

Intercept Method.

There is no mention of the intercept method in this standard.

Table 12. Grain size number relation with grain per unit area, and mean grain area

Grain Size Number (N)	Number of grain size per 1 mm^2 (n)	Mean area of grain size mm^2
-3	1	1
-2	2	0.5
-1	4	0.25
0	8	0.125
1	16	0.0625
2	32	0.0325
3	64	0.0156
4	128	0.00781
5	256	0.00390
6	512	0.00195
7	1024	0.00098
8	2048	0.00049
9	4096	0.000244
10	8192	0.000122

Equation 39. Relation between grains per mm^2 and JIS grain size number

$$n = 2^{N+3}$$

Where N is the JIS grain size number and n is the number of grains per mm^2

Reporting Results

Indication of the test results of synthetic determination shall be made in order of classified symbol of testing method, grain size, number of visual fields, maximum heating temperature and holding time for heat treatment grain size, and proportion of area occupied by grains of different sizes for mixed grains.

The preparation method must be recorded. Classified symbols for test method are as follows.

- Gc: Testing method of Carburised grain size.
- Gf: Slow cooling method.
- Gd: Double quenching method.
- Gh: Quenching and tempering method.
- Gj: One end quenching.
- Go: Oxidising method.
- Gs: Solution treating method.
- Gq: Quenching method.

Examples for the result notation follow.

1. For grains other than mixed grains.

a) Fine grain steel.

Gc $8.5_{[10]}$

A grain size of 8.5 (fine grain steel), based on synthetic determination, obtained from 10 visual fields, by the method described in JIS G0551 Section 4 testing method of carburised steel.

Gf 6.5[10] (920°1 ½ hours)

A grain size of 6.5 (fine grain steel), based on synthetic determination, obtained from 10 visual fields, after being held for 1 ½ hours at 920° by the method described in JIS G0551 Section 5(1), slow cooling method.

b) Coarse grain steel.

Gc $3.6_{[10]}$

A grain size of 3.6 (coarse grain steel), based on synthetic determination, obtained from 10 visual fields by the method in Section 4, testing method of carburised grain sizes.

2. For mixed grains included partially in the visual fields.

Gf 6.3 [13] + {6.8(67%) + 2.5(33%)}[7]…(920°1 ½ hours)

A grain size number of 6.3, based on synthetic determination, obtained from 13 visual fields out of a total of 20 visual field. The balance of 7 visual fields consisted of mixed grains, with a grain size of 6.8, taking 67 percent with a grain size of 2.5 and taking 33 percent of the 7 fields total samples, heated to 920° for 1 ½ hours by the method of Section 5 (1), slow cooling method.

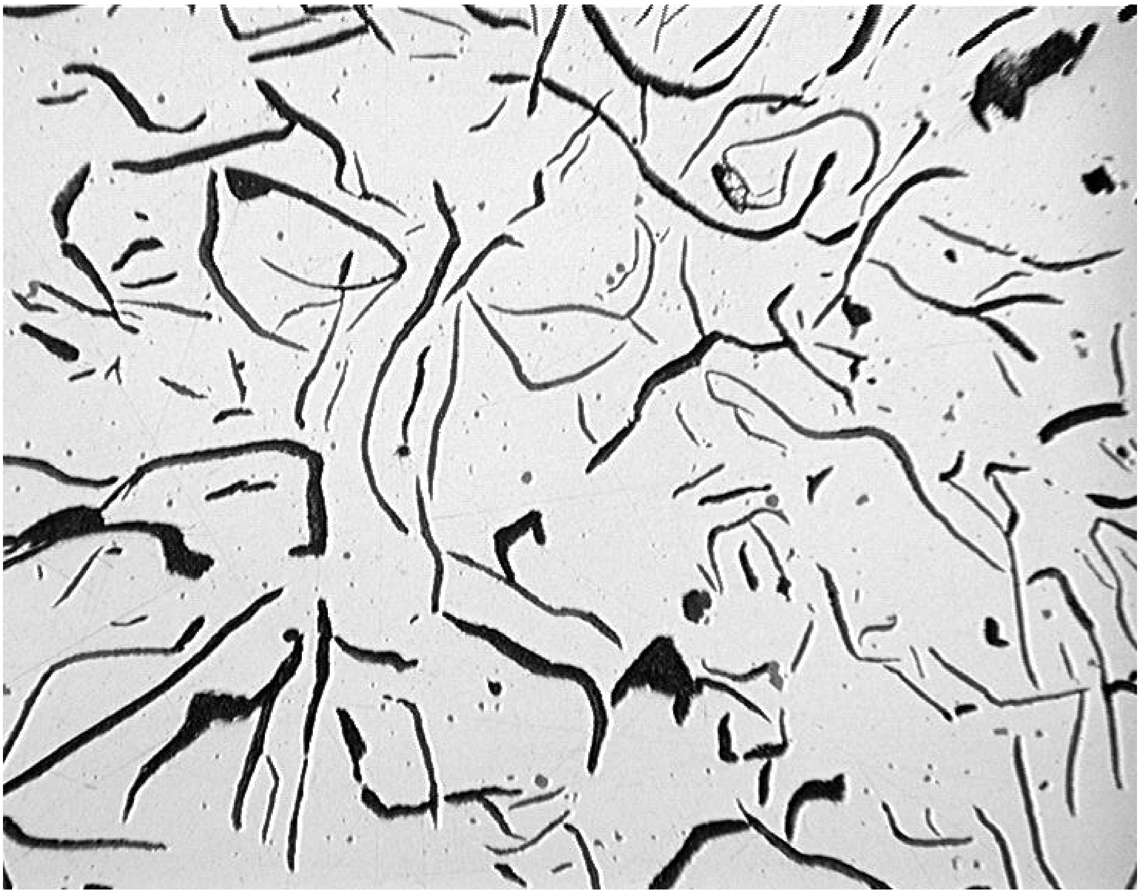
Graphite Nodules

Introduction to Graphite Nodule Analysis.

The standards relating to measurement of graphite nodules are listed in the table below.

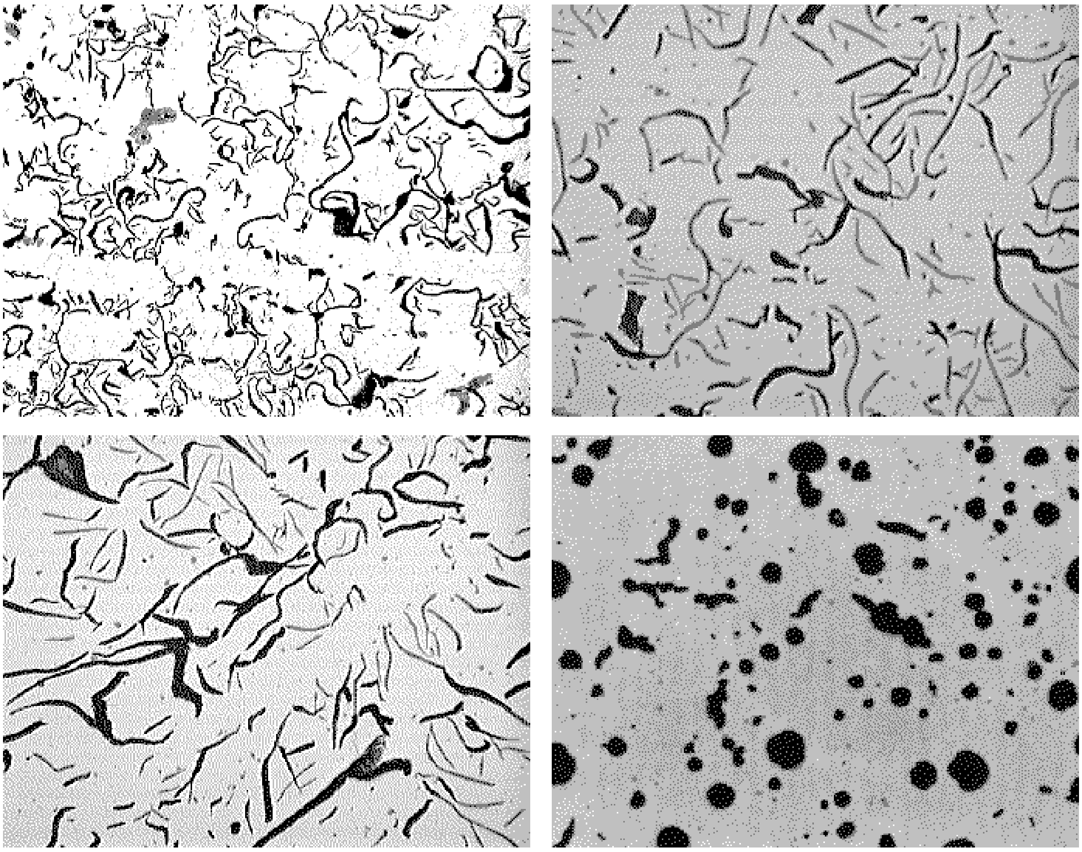
ASTM 247	Standard Method for Evaluating the Microstructure of Graphite in Iron Castings.
JIS G5501	Gray Iron Castings.
JIS G5502	Spheroidal Graphite Iron Castings.
JIS G5503	Austempered Spheroidal Graphite Iron Castings.
JIS G5504	Heavy Walled Ferritic Spheroidal Iron Castings for Low Temperature Service.
ISO 945	Cast Iron Designation of Graphite.
ISO 1083	Spheroidal Graphite Cast Iron Classification.

Figure 4 An image of graphite nodules flake graphite.



The analysis of graphite nodules in cast iron products is performed as part of the quality assurance procedure for these products. It is important to be able to control the composition and microstructure of the cast iron because this has a direct affect upon the properties which it displays. The graphite that is precipitated upon the cooling of the product will have characteristic size shape and distribution which is determined by its composition and heat treatment. The standards described below set out to measure and rate these properties.

Figure 5 Some of the different types and distributions of graphite nodules that may be encountered.



ASTM A247 Standard Method for Evaluating the Microstructure of Graphite in Iron Castings.

ASTM A 247 covers the classification of graphite in cast iron in terms of type distribution and size. ASTM Plates I, II and III describe the morphology of the different types and distributions of graphite nodules classified in this standard. The plates are not intended to be taken as a standard. These standard plates can be used to illustrate the different types, distributions and sizes, which the classifier should discriminate.

Classification by type.

Classification into seven types; see ASTM A 247 Plate I; designated I to VII. Graphite distribution is represented by capital letters A to E. Graphite size is referenced in Arabic numerals 1 to 8.

Classification by distribution.

The distribution of the graphite is then considered, as ASTM A247 Plate II. This is usually used to describe flake graphite and types A to E can be distinguished.

A: Uniform distribution random orientation.

B: Rosette groupings random orientation.

C: Superimposed flake sizes, random orientation.

D: Inter-dendritic segregation, random orientation.

E: Inter-dendritic segregation, preferred orientation.

Size Classification.

Plate III shows two series of size charts; one for spheroidal graphite and one for flake graphite. Usually visual comparison with the standard chart is adequate, but measurements may be made using ocular scales or an overlay may be applied.

Table 13. Size Classification from ASTM A247

Size Class	Maximum dimension at x 100 mm	Actual size mm
1	128	1.28
2	64	0.64
3	32	0.32
4	16	0.16
5	8	0.08
6	4	0.04
7	2	0.02
8	1	0.01

It is common practice in the analysis of malleable iron to use nodule count per unit area instead of the size chart. Where more than one size is present, the fraction of the total area covered by graphite represented by each size present may be reported.

Reporting Results.

The requirements for the reporting of results are defined in ASTM A247 Section 10.

The fraction of each type (I to VII) present should be reported as a percentage. Report headings will be Sample ID, Graphite Type, Graphite Distribution, and Graphite Size Class. Graphite type or types are represented by roman numerals I to VII and distribution by letters A to E. Graphite size is designated by arabic numerals, 1 through 8. Graphite distribution is always designated for flake graphite but may be omitted for malleable or/and ductile iron.

For example, a typical grey iron might be designated VII A4. If eutectiform graphite is present, the rating might be VIID7. Or, mixtures of the two might be described as

70% VII A4, 30%VII D7.

High quality ductile iron and graphite in malleable irons may be described as a type and size, with the distribution omitted.

IV5 etc.

JIS 5501 Gray Iron Castings.

No useful content.

JIS G 5502 Spheroidal Graphite Iron Castings.

This standard defines the tests required to characterise cast iron containing spheroidal graphite. This includes mechanical properties as well as a small reference to the calculation of spheroidal graphite rate using image analysis 10.7.5.2.

Measurements made at 100x and 5 visual fields should be covered.

Particles < 20 µm should not be measured.

Calculation Spheroidal Graphite Rate.

Section 10.7.4 describes the method for calculating spheroidal graphite rate.

Spheroidal graphite rate is calculated by determining the fraction of nodules in each of the form classifications, determined by the shape factor, or areal ratio of graphite to the circle of which diameter is of the maximum length of graphite.

Table 14. Classifcation of graphite nodules by form JIS G 5502

Form Number	1	2	3	4	5
Shape Factor	0	0.3	0.7	0.9	1.0
Informative reference areal ratio of graphite to the circle of which diameter is of the maximum length of graphite(%)	Up to 20	20 and over, up to 40	40 and over, up to 70	70 and over, up to 80	80 and over
Form type see G5502 figure 3	Long thin flake graphite				Spheroidal graphite

Equation 40. Calculation of spheroidal graphite rate

$$SperoidalGraphiteRate = \frac{0\times n_1 + 0.3\times n_2 + 0.7\times n_3 + 0.9\times n_4 + 1.0\times n_5}{n_1 + n_2 + n_3 + n_4 + n_5}\times 10$$

From JIS G5502 10.7.4

Where n_1 to n_5 are the number of graphite particles corresponding to form number of fig. 3.

JIS G5503 Austempered Spheroidal Graphite Iron Castings.

JIS G5503 as JIS G5502

JIS G5504 Heavy Walled Ferritic Spheroidal Iron Castings for Low Temperature Service.

JIS G5504 as JIS G5502

Equation 41. Calculation of Ferritic area rate

$$F(\%) = \frac{A - (A_G + A_C + A_P)}{A - A_G} \times 100$$

Also need to calculate the ferritic area rate as described in G5504 12.6.2.1

This standard requires the segregation of graphite, Pearlite and cementite from the Ferrite matrix of the sample in order to measure the area fraction of Ferrite visible on the surface of the sample.

Where:

- F is the ferritic area rate.
- A is the area of the visual field.
- A_G is the area occupied by graphite.
- A_C is the area occupied by cementite.
- A_P is the area occupied by Pearlite.
- JIS G5510 Austenitic Iron Castings

ISO 945 Cast Iron Designation of Graphite.

When iron carbon alloys are examined under a microscope, the graphite occurring in these alloys can be classified by form, distribution, and size.

Method

Specimen should be prepared and polished so that the whole surface of the specimen can be examined. The classification of graphite is made by comparison of specimens with a standard chart. First, form and distribution are classified and then comparison of size is to be made at x100.

Form

The reference diagram of graphite form as fig 1. in ISO 945, represented by the roman numerals I- VI are the typical forms of graphite, although other forms do occasionally occur.

Distribution

The reference diagrams in ISO 945 fig 2. show five examples of distributions that are referenced by the letters A to E.

Size

ISO 945 Figs. 3 - 6 and Table 15 are used to determine the graphite size. The range of sizes for each size designation numbered 1 to 8 is shown in the Table 15 The diagrams show examples of these sizes at x100 for comparison measurement.

Table 15 Size classification ISO 945

Reference number	Dimensions of the particle observed at x100 mm	True dimension mm
1	>100	>1
2	50 to 100	0.5 to 1
3	25 to 50	0.25 to 0.5
4	12 to 25	0.12 to 0.25
5	4 to12	0.06 to 0.12
6	3 to6	0.03 to 0.06
7	1.5 to 3	0.015 0.03
8	<1.5	<0.015

Reporting Results

The results should be written using the format form distribution size.

For example:. I A 4

This can be modified to provide designations to describe microstructures that are intermediate between rating values and combinations of microstructures. If the size of the graphite nodules falls between two rating values, both values can be used. In a given case the predominant size may be indicated by underlining the size

For example: 3/4

Mixed structures with different types of graphite may be defined by estimating the percentage proportion of the different types of graphite.

For example: 60% IA4 + 40%ID7.

ISO 1083 Spheroidal Graphite Cast Iron Classification.

Spheroidal graphite, or nodular graphite cast iron, is a casting alloy or iron and carbon, the latter being present mostly as graphite nodule of form VI ISO 945.

This international standard defines the classification of cast irons in accordance with the mechanical properties of the material.

The properties of spheroidal graphite depend on the form of the graphite and the structure of the matrix.

Samples of two types are used; separately cast test samples and test pieces from cast on test samples.

Methods

Microstructure

Examination of the microstructure should include verification that the microstructure contains minimum 80% of form V and VI graphite as defined in ISO 945.

Mechanical Properties

Tensile stress, proof stress, elongation and additional information.

Impact test

Brinell hardness

Reporting Results

The designation of the grade of iron is determined by the limits defined in Tables 1 and 2 of ISO1083. The classification is made using mechanical properties.

Hardness Testing

The standards appropriate for hardness testing are listed in the table below.

DIN 50 133	Vickers Hardness Test
ISO 4545	Metallic Materials Knoop test
ISO/ TR 10188	Steel Conversion of Hardness Values to Tensile Strength Values.
ISO 6507-1	Metallic Materials Hardness Test Vickers Test Part 1.
ISO 6507-2	Metallic Materials Hardness Test Vickers Test Part 2.
ISO 6507-3	Metallic Materials Hardness Test Vickers Test. Part3
ISO 4964	Steel Hardness Conversions.
BS EN 23878	Vickers Hardness Test
BS EN 10003-1	Metallic Materials Brinell Hardness Test
ISO 6506	Metallic Materials Hardness Brinell Test
ASTM E92-82	Standard Test, Method for Vickers Hardness of Metallic Materials.
ASTM E 10-93	Standard Test Method for Brinell Hardness of Metallic Materials
ASTM E 384	Standard Test Methods for Micro Hardness of Materials.
SAE J417 DEC 83	Hardness Tests and Hardness Number Conversions.
ISO 6507-1	Metallic materials Hardness test Vickers test Part 1.
ISO 6507-2	Metallic materials Hardness test Vickers test Part 2.
ISO 6507-3	Metallic materials Hardness test Vickers test. Part3
ISO 6507-1	Draft for Public Comment Metallic Materials Hardness Test Vickers Test Part 1.
ISO 6507-2	Draft for Public Comment Metallic Materials Hardness Test Vickers Test Part 2.
ISO 6507-3	Draft for public Comment Metallic Materials Hardness Test Vickers Test. Part3
JIS Z2251	Method of Knoop Hardness Test
JIS Z2243	Method of Brinell Hardness Test
JIS Z2244	Method of Vickers Hardness Test

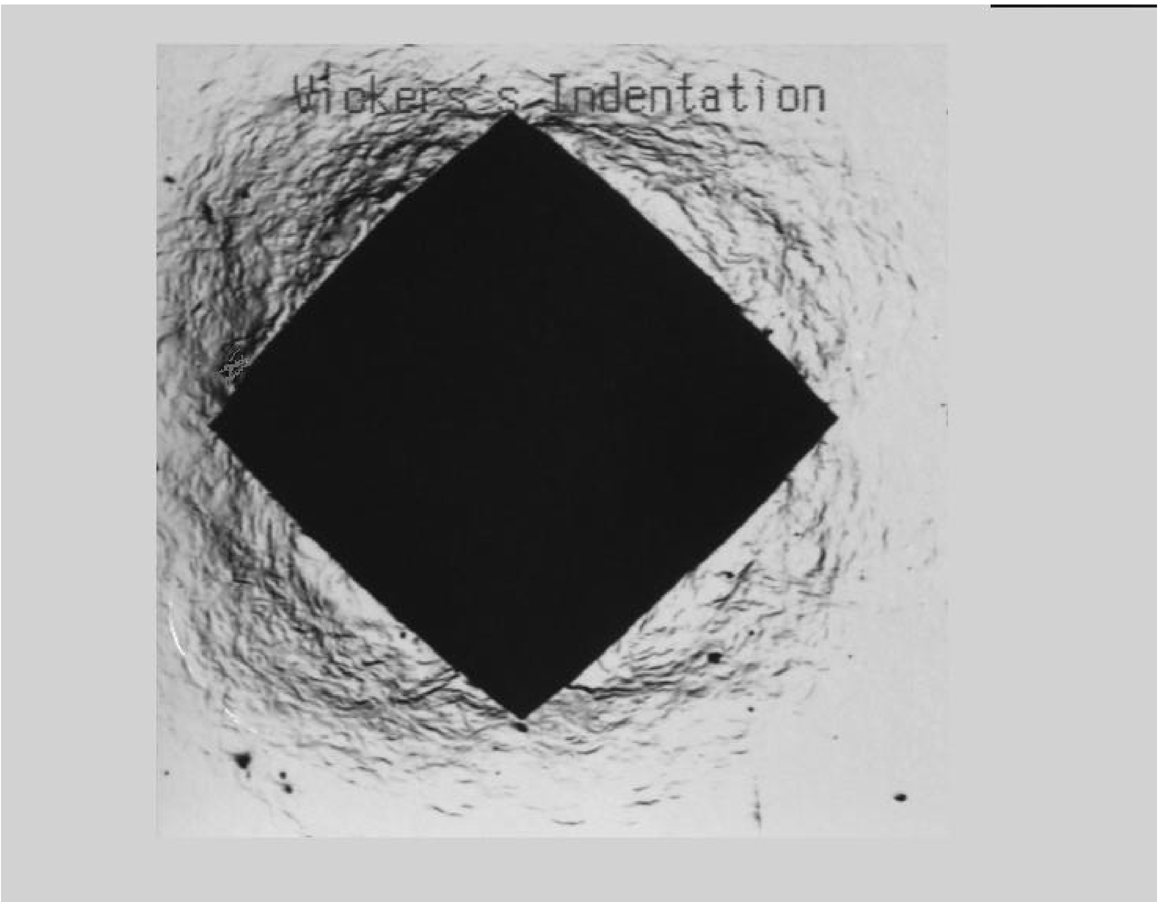
Introduction to Hardness Testing.

The hardness test uses mechanical test methods to measure the hardness. An indenter that should be harder than the material being tested is pushed into the surface of the specimen for a limited time using a known load. This is done using a hardness test machine to ensure the process can be automated and made to give reproducible results. Hardness machines are usually equipped with travelling microscopes with Vernier measurement devices.

The resulting indentation in the surface of the material can be measured and used to characterise the hardness of the material. This is done using a hardness number that is calculated using known parameters, including the size of the indenter and the test load. The exact formulas are provided below in each case. The three basic methods are Vickers, Brinell, and Knoop Tests. There are other test methods but these do not lend themselves to image analysis measurement techniques.

Hardness tests can be applied other materials besides metals, e.g. glass, plastic, ceramic.

Figure 6 A Vickers hardness indentation in the surface of a metal specimen.



DIN 50 133Vickers Hardness Test.

This standard defines the procedure for Vickers hardness testing. The standard is comparable to ISO 6507 Euro norm 5-79.

Table 16 Types of Vickers hardness test and associated parameters.

Type of Vickers hardness test	Test conditions	Test force F in N	Standard
Macro-hardness test	HV 5 to HV 100	49.03 to 980.7	DIN 50 133
Low load hardness test	HV 0.2 to < HV 5	1.961 to < 49.03	DIN 50 133
Micro-hardness test	< HV 0.2	<1961	In preparation

Test Procedure

The diagonal length of the square test indentation must be measured, for both diagonals. Using IA we can make comments on the square shape of the indentation. The indentation can be mis-shapen when the indentation is created and can produce a pin cushion shape, or another distortion, which may have an affect on the results.

Test method to calculate the arithmetic mean of the two diagonals is (d1+d2)/2=d

F is the force applied in units of Newtons (N).

Equation 42. Calculation of Vickers hardness load force divided by indentation surface area

$$HV = 0.1891\frac{F}{d^2}$$

F can be calculated using the gravitational constant g = 9.80665

F = Load Kg * g

Table 17 Ranges of hardness values (HV) for load.

Macro Range		Low load range	
Test Condition	Test Force F (N)	Test Condition	Test Force F (N)
HV 5	49.03	HV 0.2	1.961
HV10	98.07	HV 0.3	2.942
HV 20	196.1	HV 0.5	4.903
HV 30	294.2	HV 1	9.807
HV 50	490.3	HV 2	19.61
HV 100	980.7	HV 3	29.42

Micro hardness ranges for test condition will be <0.2

Correction for curved surfaces

Appendix B1 lists correction factors that must be applied in order to apply the standard to curved surfaces.

The diameter of the curved surface is required in order to calculate the ratio d/D

Designation

The standard designation is as follows.

Test DIN 50 133 – HV30

HV30 is the test load measured in Kg not the test force.

Reporting Results

The report should include the following information:

- Reference to this standard.
- All details necessary for the identification of the test piece.
- Hardness values obtained and the test conditions.
- Any deviations from the standard.

ISO 4545 Metallic Materials Hardness Test Knoop Test.

This standard specifies the method of Knoop hardness for metallic materials. It covers test forces up to and including 9807 N.

Note: A strict comparison of hardness values is only possible at identical test forces.

Test Procedure

The test piece is placed on a rigid support. The indenter is bought into contact with the sample perpendicular to the surface. The indentation is made by pushing the indenter into the polished surface of the test piece, using a known test force. Measure the length of the long diagonals of the Knoop indentation.

Table 18 Test Forces for HK Value ranges

Hardness Symbol	Nominal Test Force F (N)
HK 0.01	98.07×10^{-3}
HK 0.02	0.1961
HK 0.025	0.2452
HK 0.05	0.4903
HK 0.1	0.9807
HK 0.2	1.961
HK 0.3	2.942
HK 0.5	4.903
HK 1	9.807

Calculating HK

Equation 43. Calculating Knoop Hardness

$$HK = Constant \times \frac{TestForce}{ProjectedAreaofIndentation} = constant \times \frac{F}{d^2 \cdot c}$$

Where d is the length of the long diagonal of the Knoop indentation, F is the test force constant is reciprocal g, where g is the gravitational constant.

Equation 44. The constant used in the expression above evaluates:

$$Constant = \frac{1}{g_n} = \frac{1}{9.80665} \approx 0.102$$

Equation 45. The constant (c) that is a property of the indenter geometry is shown above

$$c = \frac{\tan\frac{b}{2}}{2\tan\frac{a}{2}}$$

For a Knoop indenter of dimensions defined in ISO 4546, then c=0.07028.

Designation

For example:

- 640 HK 0.1 = Knoop hardness of 640, determined with a test force of 0.9807 N, applied for 10 to 15 seconds.
- 640 HK 0.1/20 = Knoop hardness of 640, determined with a test force of 0.9807 N, applied for 20 seconds.

Test Report

The report should include the following information:

- Reference to this international standard ISO 4545
- All information necessary for identification of the test sample
- The result obtained in the standard designation format.
- All operations not specified by this international standard, or regarded as optional.
- Details of any circumstances that may affect the result.
- Temperature if it is out of the range of 23°C+- 5°C

ISO 6507-1 Metallic Hardness Test Vickers Test, HV 5 to HV 100.

Method

The test piece is placed on a rigid support. The indenter is bought into contact with the sample perpendicular to the surface.

Measure the length of the two perpendicular diagonals and calculate the arithmetic mean. This is used to calculate the Vickers hardness.

Equation 46. Calculation of Vickers hardness

$$HV = cons\tan t \times \frac{TestForce}{SurfaceAreaofIndentation}$$

Where d is the arithmetic mean diagonal length.

$$HV = 0.1891 \times \frac{F}{d^2}$$

Refer to the following table for the test force that should be used.

Table 19 Test forces for hardness value ranges.

Hardness Symbol	Test Force F Nominal Value
HV 5	49.03N
HV10	98.07N
HV 20	196.1N
HV 30	294.2N
HV 50	490.3N
HV100	980.7N

Corrections for curved surfaces.

Correction factors for curved surfaces can be applied to the measured Vickers hardness, by calculating d/D for each and using tables of conversion factors which are supplied in Appendix B of ISO 6507/1, for concave and convex surfaces.

The correction factor is multiplied into the measured HV.

Reporting Results

The following should be reported.:

- Reference to this part of ISO 6507.
- All details necessary for the identification of the test sample.
- Results obtained.
- All operations not specified in this part of ISO 6507, or regarded as option.
- Details of any occurrence that may have affected the result.

ISO 6507-2 Metallic Hardness Test Vickers Test, HV 0.2 to <HV 5.

Method

The test piece is placed on a rigid support. The indenter is bought into contact with the sample perpendicular to the surface.

Measure the length of the two perpendicular diagonals and calculate the arithmetic mean which can be used to calculate the Vickers hardness.

Equation 47. Calculation of Vickers hardness

$$HV = constant \times \frac{TestForce}{SurfaceAreaofIndentation}$$

d is the arithmetic mean diagonal length.

$$HV = 0.1891 \times \frac{F}{d^2}$$

Refer to the following table for the test force that should be used.

Table 20. Test forces for ranges of hardness values (HV)

Hardness Symbol	Test Force F Nominal Value
HV 0.2	1.961N
HV 0.3	2.942N
HV 0.5	4.903N
HV 1	9.807N
HV 2	19.61N
HV 2.5	24.52N
HV 3	29.42N

Corrections for curved surfaces

Correction factors for curved surfaces can be applied to the measured Vickers hardness, by calculating d/D for each and using tables of conversion factors for concave and convex surfaces, which are supplied in Appendix B of ISO 6507/2.

The correction factor is multiplied into the measured HV.

Reporting Results

The following should be reported.

- Reference to this part of ISO 6507.
- All details necessary for the identification of the test sample.
- Results obtained.
- All operations not specified in this part of ISO 6507, or regarded as option.
- Details of any occurrence that may have affected the result.

ISO 6507-3 Metallic Hardness Test Vickers Test Less Than HV 0.2.

Method

The test piece is placed on a rigid support. The indenter is bought into contact with the sample perpendicular to the surface. Make the indentation by pushing the pyramidal diamond indenter into the polished surface of the test piece, using a known test force which should be applied for the duration defined. Measure the length of the two perpendicular diagonals and calculate the arithmetic mean. This is used to calculate the Vickers hardness.

Equation 48. Calculation of Vickers hardness

$$HV = constant \times \frac{TestForce}{SurfaceAreaofIndentation}$$

d is the arithmetic mean diagonal length.

$$HV = 0.1891 \times \frac{F}{d^2}$$

Refer to the following table for the test force that should be used.

Table 21. Test forces recommended to be used, and the hardness symbols that are used to record the test force

Hardness Symbol	Test Force F Nominal Value (N)
HV 0.01	98.07×10^{-3}N
HV 0.02	0.1961N
HV 0.025	0.2452N
HV 0.05	0.4903N
HV 0.1	0.9807N

Reporting Results

The following should be reported:

- Reference to this part of ISO 6507.
- All details necessary for the identification of the test sample.
- Results obtained.
- All operations not specified in this part of ISO 6507, or regarded as optional.
- Details of any occurrence that may have affected the result.

BS EN 23878-23878 ISO 3878-1983 Hardmetals Vickers hardness Test.

Method.

The test force shall be in the range 9.807N (HV 1) to 490.3N (HV 50); the preferred force being 294.2(HV30). If possible, at least three hardness indentations should be made on the same piece. Measure the length of the two diagonals and determine the arithmetic mean for the calculation of the Vickers hardness.

Reporting Results

The following should reported:

- Reference to this part of ISO 3878.
- All details necessary for the identification of the test sample.
- Results obtained.
- All operations not specified in this part of ISO 6507, or regarded as option.
- Details of any occurrence that may have affected the result.

BS EN 10003/1-1995 Metallic Materials Brinell Hardness Test.

Method

This method uses a hardened steel ball, or hard metal ball, with diameter D, that is pressed into the surface of the test specimen. The steel ball is used for materials not exceeding HB 350. The hard metal ball is used for materials not exceeding HB 650.

The Brinell hardness is denoted by the following symbols.

- HBS in cases where the steel ball is used
- HBW in cases where a hard metal ball is used.

This symbol is preceded by the hardness value and supplemented by an index indicating the test conditions, diameter of the test ball in mm, a figure representing the test force and the duration of loading in seconds, if different from the specified time.

For example: 350 HB 5/750 = Brinell hardness of 350, determined using a steel ball of 5mm diameter and with a test force of 7355kN, applied for 10 to 15 seconds (the preferred time). Alternatively,; 600 HB 1/30/20 = Brinell hardness of 600, determined using a hard metal ball of 1mm diameter and with a test force of 294.2 N, applied for 20 seconds.

The test is usually carried out at ambient temperature within limits 10 to 35 °C. Tests carried out under controlled conditions should be carried out at 23 +- 5°C The test force should be chosen so that the test force lies between the values 0.24 D, and 0.6 D.

Table 22. Test forces for various combinations of ball size and Hardness value (HB)

Hardness Symbol	Ball diameter D mm	$0.102F/D^2$	Test Force F
			Nominal Value
HBS(HBW) 10/3/3000	10	30	29.42 kN
HBS(HBW) 10/1500	10	15	14.71 kN
HBS(HBW) 10/1000	10	10	9.807kN
HBS(HBW) 10/500	10	5	4.903kN
HBS(HBW) 10/250	10	2.5	2.452kN
HBS(HBW) 10/125	10	1.25	1.226kN
HBS(HBW) 10/100	10	1	980.7kN
HBS(HBW) 5/750	5	30	7.355kN
HBS(HBW) 5/250	5	10	2.452kN
HBS(HBW) 5/125	5	5	1.226kN
HBS(HBW) 5/62.5	5	2.5	612.9N
HBS(HBW) 5/31.25	5	1.25	306.5N
HBS(HBW) 5/25	5	1	245.2N
HBS(HBW) 2.5/187.5	2.5	30	1.838kN
HBS(HBW) 2.5/62.5	2.5	10	612.9N
HBS(HBW) 2.5/31.25	2.5	5	306.5N
HBS(HBW) 2.5/15.625	2.5	2.5	153.2N
HBS(HBW) 2.5/7.8125	2.5	1.25	76.61N
HBS(HBW) 2.5/6.25	2.5	1	61.29N
HBS(HBW) 2/120	2	30	1.177kN
HBS(HBW) 2/40	2	10	392.3N
HBS(HBW) 2/20	2	5	196.1N
HBS(HBW) 2/10	2	2.5	98.07N
HBS(HBW) 2/5	2	1.25	49.03N
HBS(HBW) 2/4	2	1	39.23N
HBS(HBW) 1/30	1	30	294.2N
HBS(HBW) 1/10	1	10	98.07N
HBS(HBW) 1/5	1	5	49.03N
HBS(HBW) 1/ 2.5	1	2.5	24.52N
HBS(HBW) 1/1.25	1	1.25	12.26N
HBS(HBW) 1/1	1	1	9.807N

The degree of loading ($0.102F/D^2$) should be chosen according to the material and the hardness test as indicated in Table 23. below.

Table 23. The degree of loading quoted for different materials.

Material	Brinell hardness HB	$0, 102F/D^2$
Steel - Nickel alloys Titanium alloys.	-	30
Cast iron	<140 >140	10 30
Copper and copper alloys	<35	5
	35 to200	10
	>200	30
Light metals and their alloys	<35	2.5
	35 to 80	5
		10
		15
	>80	10
		15
Lead tin		1
Sintered metal	see EN 24498-1	

Measure the diameter if the indentation in two directions at right angles. The arithmetic mean can be taken for the calculation of the Brinell hardness:

Equation 49. Calculation for Brinell hardness

$$HB = 0.102 \times \frac{2F}{pD(D - \sqrt{D^2 - d^2})}$$

HB is the Brinell hardness, F is the test force, D is the diameter of the ball in mm, d is the diameter of the indentation in mm. The minimum thickness of test piece permitted is as follows.

Table 24 Minimum thickness of test piece for differnt indenation sizes

Mean diameter of the indentation (mm)	Minimum thickness of the test piece (mm)				
	D=1	D=2	D=3	D=5	D=10
0.2	0.08				
0.3	0.18				
0.4	0.33				
0.5	0.54	0.25			
0.6	0.8	0.37	0.29		
0.7		0.51	0.40		
0.8		0.67	0.53		
0.9		0.86	0.67		
1.0		1.07	0.83		
1.1		1.32	1.02		
1.2		1.60	1.23	0.58	
1.3			1.46	0.69	
1.4			1.72	0.80	
1.5			2.00	0.92	
1.6				1.05	
1.7				1.19	
1.8				1.34	
1.9				1.50	
2.0				1.67	
2.2				2.04	
2.4				2.46	1.17
2.6				2.92	1.38
2.8				3.43	1.60
3.0				4.00	1.84
3.2					2.10
3.4					2.38
3.6					2.68
3.8					3.00
4.0					3.34
4.2					3.70
4.4					4.08

4.6					4.48
4.8					4.91
5.0					5.36
5.2					5.83
5.4					6.33
5.6					6.86
5.8					7.42
6.0					8.0

Reporting Results

The following should be reported:

- Reference to this part of EN10003
- All details necessary for the identification of the test sample.
- Results obtained.
- Additional requirements outside of the scope of this standard.
- Details of any occurrence that may have affected the result.
- The test temperature if not within the limits (23+-5 °C).

ISO 6506 Metallic Materials Hardness Test Brinell Test.

Method

A hard steel ball, or hard metal ball, with diameter D is forced in to the surface of the test piece with a force F. The size of the indentation (d) is measured in two directions and the arithmetic mean is used to calculate the Brinell hardness HBS or HBW.

Equation 50. Calculation of Brinell hardness

$$HB = 0.102 \times \frac{2F}{pD(D - \sqrt{D^2 - d^2})}$$

Refer to the following table for the test force that should be used.

Table 25. Test forces and degree of loading for various hardness values

Hardness Symbol	Ball diameter D mm	$0.102F/D^2$	Test Force F
			Nominal Value
HBS(HBW) 10/3/3000	10	30	29.42 kN
HBS(HBW) 10/1500	10	15	14.71 kN
HBS(HBW) 10/1000	10	10	9.807kN
HBS(HBW) 10/500	10	5	4.903kN
HBS(HBW) 10/250	10	2.5	2.452kN
HBS(HBW) 10/125	10	1.25	1.226kN
HBS(HBW) 10/100	10	1	980.7kN
HBS(HBW) 5/750	5	30	7.355kN
HBS(HBW) 5/250	5	10	2.452kN
HBS(HBW) 5/125	5	5	1.226kN
HBS(HBW) 5/62.5	5	2.5	612.9N
HBS(HBW) 5/31.25	5	1.25	306.5N
HBS(HBW) 5/25	5	1	245.2N
HBS(HBW) 2.5/187.5	2.5	30	1.838kN
HBS(HBW) 2.5/62.5	2.5	10	612.9N
HBS(HBW) 2.5/31.25	2.5	5	306.5N
HBS(HBW) 2.5/15.625	2.5	2.5	153.2N
HBS(HBW) 2.5/7.8125	2.5	1.25	76.61N
HBS(HBW) 2.5/6.25	2.5	1	61.29N
HBS(HBW) 2/120	2	30	1.177kN
HBS(HBW) 2/40	2	10	392.3N
HBS(HBW) 2/20	2	5	196.1N
HBS(HBW) 2/10	2	2.5	98.07N
HBS(HBW) 2/5	2	1.25	49.03N
HBS(HBW) 2/4	2	1	39.23N
HBS(HBW) 1/30	1	30	294.2N
HBS(HBW) 1/10	1	10	98.07N
HBS(HBW) 1/5	1	5	49.03N
HBS(HBW) 1/ 2.5	1	2.5	24.52N
HBS(HBW) 1/1.25	1	1.25	12.26N
HBS(HBW) 1/1	1	1	9.807N

The test force shall be chosen so that the diameter of the indentation lies between the value 0.24 D and 0.6 D. the ratio 0.102 F/D^2 shall be chosen according to the material and the hardness under test. When the thickness of the test piece permits, a ball diameter of 10 mm can be used. The minimum thickness of the test piece permitted is as follows.

Table 26. Minimum thickness of test piece for different indentation sizes and ball sizes

Mean diameter of the indentation (mm)	Minimum thickness of the test piece (mm)				
	D=1	D=2	D=3	D=5	D=10
0.2	0.08				
0.3	0.18				
0.4	0.33				
0.5	0.54	0.25			
0.6	0.8	0.37	0.29		
0.7		0.51	0.40		
0.8		0.67	0.53		
0.9		0.86	0.67		
1.0		1.07	0.83		
1.1		1.32	1.02		
1.2		1.60	1.23	0.58	
1.3			1.46	0.69	
1.4			1.72	0.80	
1.5			2.00	0.92	
1.6				1.05	
1.7				1.19	
1.8				1.34	
1.9				1.50	
2.0				1.67	
2.2				2.04	
2.4				2.46	1.17
2.6				2.92	1.38
2.8				3.43	1.60
3.0				4.00	1.84
3.2					2.10
3.4					2.38
3.6					2.68
3.8					3.00

4.0					3.34
4.2					3.70
4.4					4.08
4.6					4.48
4.8					4.91
5.0					5.36
5.2					5.83
5.4					6.33
5.6					6.86
5.8					7.42
6.0					8.0

Reporting Results

The following shall be reported:

- Reference to this part of ISO 6506.
- All details necessary for the identification of the test sample.
- Results obtained.
- All operations not specified in this part of ISO 6506 or regarded as optional.
- Details of any occurrence that may have affected the result.

ASTM E92-1992 Standard Test Method for Vickers Hardness of Metallic Materials.

Method

A testing machine equipped for Vickers harness testing is used. This consists of a rigid platform upon which the specimen can be placed. An indenter that is a square pyramidal diamond is pushed into the test specimen by a known force. The force can be varied by adding and removing weights of known mass to and from the indenter mechanism. The force is applied to the surface of the test piece for a duration of 10 to 15 seconds. (See ASTM)

Both diagonals of the indentation produced by the method described above are measured. The arithmetic mean is calculated and used to calculate the Vickers hardness.

Equation 51. Calculation of Vickers Hardness

$$HV = {2P\sin\left(\frac{a}{2}\right)} \Big/ {d^2} = 1.8544P \big/ d^2$$

α=136° the angle between opposite faces of the diamond, P is the load kgf, d is the diagonal of impression.

Reporting Results

The following shall be reported:

- The Vickers hardness number, e.g. 440 HV30 where 440 is the Vickers hardness under a 30kgf test applied for 10 to 15 second. Where the test duration varies from 10 to 15 seconds it should be reported e.g. a test producing the same HV value with the same load with a duration of 20 seconds is reported thus 440 HV30/20.
- The test load used.
- The loading time, if other than 10 to 15 seconds.

ASTM E10 -1993 Standard Test Method for Brinell Hardness of Metallic Materials.

Method

A hard steel ball, or hard metal ball diameter (D) is forced in to the surface of the test piece with a force F. The size of the indentation (d) is measured in two directions and the arithmetic mean used to calculate the Brinell hardness HBS or HBW. Tables for the estimation of HBS or HBW are provided, but the following equation can be used to calculate the HB value from the diameter of the indentation.

Equation 52 Calculation of Brinell hardness

$$HB = 0.102 \times \frac{2F}{pD(D - \sqrt{D^2 - d^2})}$$

Refer to the following table for the test force that should be used.

Table 27. Test force for hardness value ranges and ball sizes

Hardness Symbol	Ball diameter D mm	$0.102F/D^2$	Test Force F
			Nominal Value
HBS(HBW) 10/3/3000	10	30	29.42 kN
HBS(HBW) 10/1500	10	15	14.71 kN
HBS(HBW) 10/1000	10	10	9.807kN
HBS(HBW) 10/500	10	5	4.903kN
HBS(HBW) 10/250	10	2.5	2.452kN
HBS(HBW) 10/125	10	1.25	1.226kN
HBS(HBW) 10/100	10	1	980.7kN
HBS(HBW) 5/750	5	30	7.355kN
HBS(HBW) 5/250	5	10	2.452kN
HBS(HBW) 5/125	5	5	1.226kN
HBS(HBW) 5/62.5	5	2.5	612.9N
HBS(HBW) 5/31.25	5	1.25	306.5N
HBS(HBW) 5/25	5	1	245.2N
HBS(HBW) 2.5/187.5	2.5	30	1.838kN
HBS(HBW) 2.5/62.5	2.5	10	612.9N
HBS(HBW) 2.5/31.25	2.5	5	306.5N
HBS(HBW) 2.5/15.625	2.5	2.5	153.2N
HBS(HBW) 2.5/7.8125	2.5	1.25	76.61N
HBS(HBW) 2.5/6.25	2.5	1	61.29N
HBS(HBW) 2/120	2	30	1.177kN
HBS(HBW) 2/40	2	10	392.3N
HBS(HBW) 2/20	2	5	196.1N
HBS(HBW) 2/10	2	2.5	98.07N
HBS(HBW) 2/5	2	1.25	49.03N
HBS(HBW) 2/4	2	1	39.23N
HBS(HBW) 1/30	1	30	294.2N
HBS(HBW) 1/10	1	10	98.07N
HBS(HBW) 1/5	1	5	49.03N
HBS(HBW) 1/ 2.5	1	2.5	24.52N
HBS(HBW) 1/1.25	1	1.25	12.26N
HBS(HBW) 1/1	1	1	9.807N

The test force should be chosen so that the diameter of the indentation lies between the value 0.24 D and 0.6 D. The ratio 0.102 F/D^2 should be chosen according to the material and the hardness under test. A ball dameter of 10.000 mm can be used, with deviations of not more than 0.005 mm in any diameter

Usually a test force of 2.9kN(300kgf), 14.7kN(1500kgf), or 4.9kN (500kgf) is used. It is recommended that the indentation be between 25 and 60 % of the ball diameter.

The following forces are recommended for the ranges of hardness values shown below. It should be realised that the hardness number may vary if a different force is used to measure hardness of the same material and that force should be kept constant. This means that it may be prudent to select an appropriate force which can be kept constant when measuring a material which is expected to have hardness values in a certain range and hence produce comparable results in a continuous range of hardness measurements.

Table 28. Test forces recommended for use on material in a certain range of hardness values

Ball diameter mm	Force	Recommended range
10	2.94kN(300kgf)	96 to 600
10	14.7kN(1500kgf)	48 to 300
10	4.90kN (500kgf)	16 to 100

Smaller indenter balls may be used to test thin specimens. These tests are not regarded as standard tests.

Two diameters of the indentation should be measured at right angles to each other. The arithmetic mean of these two values, d, is used to calculation the Brinell hardness.

Reporting Results

The report should contain the following:

- The Brinell hardness number, HBS or HBW, in the following format:
- The ball size and force should be reported, if other than 10mm and 29.42kN 3000kgf and duration, if other than 10 to 15 seconds e.g. 330HBS5/4.90kN (500kgf)/20
- The test conditions, when the Brinell hardness number is determined from force other than 29.42kN (3000 kgf), ball diameters other than 10mm and test force applications other than 10 to 15 seconds.

ASTM E384-89 Standard Test Method for Microhardness of Materials.

Method

This procedure covers tests made using Vickers and Knoop indenter test loads in the range 1 to 1000 gf. Calculation of the HV value is made in the same way that it is calculated in ASTM E 92, the equation may be rearranged to permit units other than mm and kgf.

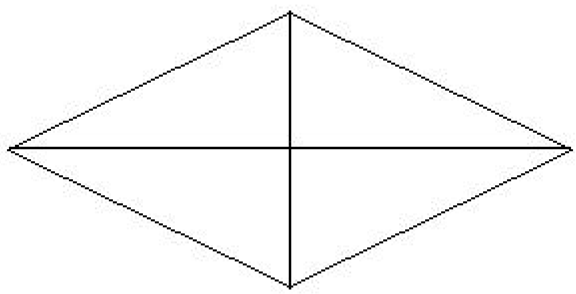
Equation 53. Calculation of Vickers Microhardness

$$HV = 1854.4\, P_1 / d_1^2$$

P_1 is the load in gf and d_1 is the diagonal length in microns.

The Knoop test uses a diamond indenter, that is not square. The Knoop hardness is calculated as follows using measurements of the long diagonal only.

Figure 7. Knoop indentation



Equation 54. Calculation of Knoop hardness

$$HK = 14229\, P_1 / d_1^2$$

P is the load gf, and d is the length of the long diagonal.

Measurements of the diagonal lengths should be made to 0.25 microns or 0.4%, whichever is larger. If one of the diagonal lengths is longer than the other by 20 % and the ends of the diagonal are not both in the field of focus, the surface may be misaligned and another indentation should be made.Tables are provided for the estimation of the Vickers and Knoop hardness.

Reporting Results.

The report should include the following.

- Hardness number (HV or HK), if Knoop hardness method is used. The load in grams force in subscript notation e.g. 400 HK_{100}.
- Test load.
- Time of full load application if other than 10 to 15 seconds.
- Magnification
- Any unusual conditions encountered during the test.

SAE J417 DEC83 Hardness Tests and Hardness Number Conversions.

Method

This standard refers to the ASTM standards for Vickers and Brinell hardness tests. Conversion tables are provided for the estimation of other hardness numbers, Vickers and Brinell hardness to each other and also Rockwell and Shore hardness tests.

These conversion tables are of restricted use. The conversion tables are based on extensive tests on carbon and alloy steel, mostly in the heat treated condition. They have been found to be reliable on practically all constructional alloy steels and tool steels. Special cases, such as high manganese steel, other austenitic steels, constructional and tool steels in the cold worked condition may not conform to the relationship given; at leat not to the same degree of accuracy as steel for which they are intended. Conversion tables should be used with care and the precautions outlined in the various test methods should be followed. The surfaces must be flat and in the case of Vickers test, the surfaces must be very smooth for accurate results.

Reporting Results

Results of conversions should be reported in the format described in the relevant ASTM standard.

ISO / DIS 6507 -1996 Metallic Materials Vickers Hardness Test.

Within this document, the metallic materials Vickers hardness test has been reorganised into one standard with three parts. This is a revision of ISO 6507 parts 1,2,3 and ISO 409 Parts 1, 2 and 3.

- Part 1: Test method
- Part 2: Verification of testing machines.
- Part 3: Calibration of the reference blocks

Part 1: Test Method

The test piece is supported on a rigid platform. A square pyramidal diamond indenter is used to make an indentation in the clean surface of the specimen, using a known test load applied for a known duration (usually 10 to 15 seconds).

Equation 55. Vickers hardness value can be calculated from the measurement of the two diagonals of the test indentation d.

$$HV = \frac{2P\sin\left(\frac{a}{2}\right)}{d^2} = \frac{1.8544P}{d^2}$$

Tables for estimating Vickers hardness results are provided.

Reporting Results

The report should include the following.

- Reference to this international standard.
- All details necessary for identification of the test piece.
- The result obtained.
- All operations not specified by this international standard or regarded as optional.
- Details of any occurrence not specified by this international standard.
- The temperature of this test if outside the specified range 10°C to 35°C under controlled temperature conditions 23°C +-5°C.

Notes

A strict comparison of hardness values is only possible at identical test forces. There is no general process for accurately converting Vickers hardness into other scales of hardness, or into tensile strength. Such conversions therefore should be avoided unless reliable basis for conversion can be obtained.

It should be noted that for anisotropic materials, for example, those which have been heavily cold worked, there will be a difference between the lengths of the two diagonals of the indentation. Where possible the indentation should be made so that the diagonals of the indentation are inclined at approximately 45° to the direction of cold working. The specification may indicate limits to which the length of the two diagonals may differ.

There is evidence that some materials are sensitive to the rate of strain, which cause small changes in the value of yield stress. The corresponding effect on the termination of the formation of an indentation can make alterations in the hardness value.

Part 2 Verification of Testing Machines.

This part of the standard provides methods for the verification of the Vickers test machine, used to make indentations.

Part 3 Calibration of the Reference Blocks

This part of the standard provides methods for the calibration of test blocks for testing the Vickers hardness machine.

JIS Z 2251 Method of Knoop Hardness Test.

Method

The testing machine needs to comply with JIS B7734. The diagonal length should be measured to 0.4%, or 0.2 micron, whichever is the greater. The measurement of a standard indention may be made first, in order to calculate compensation for the measurements.

Equation 56. Calculation of Knoop Hardness value.

$$HK = 1.451\frac{F}{d^2}$$

F is the test force N, d is the diagonal length of the long diagonal.

The thickness of the test piece should be at least 0.3 times the longer diagonal length of the indentation.

Reporting Results

Indication of Knoop hardness

For example:

400 HK 0.05

Knoop hardness value 400 and testing load 0.4903N

JIS Z 2243 Method of Brinell Hardness Test.

Applicable standards include the following:

- JIS B7724 Brinell, hardness testing machines.
- JIS B7736 Standardised blocks of Brinell hardness
- JIS Z 8401 Rules for rounding off numerical values
- ISO 6506 1981 Metallic materials hardness test: Brinell test

Method

The Brinell hardness is calculated from the diameter of the test indentation , using the two diameters at right angles to each other and the equation below.

Equation 57. Calculation of Brinell hardness

$$HB = 0.102 \times \frac{2F}{pD(D - \sqrt{D^2 - d^2})}$$

A steel ball indenter should not be used for material exceeding 350 HB and a cemented carbide alloy indenter should not be used for materials exceeding 650Hb in hardness. HBS indicates the use of a steel indenter, whilst HBW indicates the use of a cemented carbide indenter.

The selection of test load and indenter size can be determined using the table below.

Table 29 Testforce and indeter size for rages of hardness values

Hardness Symbol	Ball diameter D mm	$0.102F/D^2$	Test Force F
			Nominal Value
HBS(HBW) 10/3/3000	10	30	29.42 kN
HBS(HBW) 10/1500	10	15	14.71 kN
HBS(HBW) 10/1000	10	10	9.807kN
HBS(HBW) 10/500	10	5	4.903kN
HBS(HBW) 10/250	10	2.5	2.452kN
HBS(HBW) 10/125	10	1.25	1.226kN
HBS(HBW) 10/100	10	1	980.7kN
HBS(HBW) 5/750	5	30	7.355kN
HBS(HBW) 5/250	5	10	2.452kN
HBS(HBW) 5/125	5	5	1.226kN
HBS(HBW) 5/62.5	5	2.5	612.9N
HBS(HBW) 5/31.25	5	1.25	306.5N
HBS(HBW) 5/25	5	1	245.2N
HBS(HBW) 2.5/187.5	2.5	30	1.838kN
HBS(HBW) 2.5/62.5	2.5	10	612.9N
HBS(HBW) 2.5/31.25	2.5	5	306.5N
HBS(HBW) 2.5/15.625	2.5	2.5	153.2N
HBS(HBW) 2.5/7.8125	2.5	1.25	76.61N
HBS(HBW) 2.5/6.25	2.5	1	61.29N
HBS(HBW) 2/120	2	30	1.177kN
HBS(HBW) 2/40	2	10	392.3N
HBS(HBW) 2/20	2	5	196.1N
HBS(HBW) 2/10	2	2.5	98.07N
HBS(HBW) 2/5	2	1.25	49.03N
HBS(HBW) 2/4	2	1	39.23N
HBS(HBW) 1/30	1	30	294.2N
HBS(HBW) 1/10	1	10	98.07N
HBS(HBW) 1/5	1	5	49.03N
HBS(HBW) 1/ 2.5	1	2.5	24.52N
HBS(HBW) 1/1.25	1	1.25	12.26N
HBS(HBW) 1/1	1	1	9.807N

Reporting Results

The report should include the following.

- Indication of hardness.:
- 250HBS10/3000: Hardness value 250, indenter: steel ball diameter 10mm and test load 29.42 kN.
- 350HBW10/3000: Hardness value 350, indenter: hard metal ball diameter 10mm and test load 29.42 kN.

JIS Z 2244 Method of Vickers Hardness Test.

Applicable standards are:

- JIS B 7725 Vickers hardness test machines
- JIS B 7734 Microhardness testing machines for Vickers and Knoop hardness
- JIS B 7735 Standardised blocks of Vickers hardness
- JIS Z 8401 Rules for rounding off numerical values
- ISO 650-7 Parts 1,2,3. Metallic materials hardness test Vickers test.

Method

The two diagonals of the test indention are measured and the arithmetic mean calculated (d). The Vickers hardness HV is calculated as in the following equation. The measurement should be to 0.4% or 0.25 micron, whichever is the greater.

$$HV = 0.1891 \times \frac{F}{d^2}$$

Where F is the test force in N and d is mean length of the diagonals of the indentation measured.

The thickness of the test piece should be at least 1.5 times the diagonal length of the indentation. The temperature for the test should generally be 10°C to 35°C, but 23°C +-5°C for material sensitive to temperature fluctuations. Hardness correction coefficients are provided in tables for concave and convex surfaces, spheres and cylinders.

Reporting Results

The report should include the following.

- Indication of hardness.
- The Vickers hardness should be indicated as follows below.
- 700HV 0.1 Vickers hardness value of 700 and test load of 0.9807N
- 300HV30 Vickers hardness value of 300 and test load 294.2N

Particle sizing

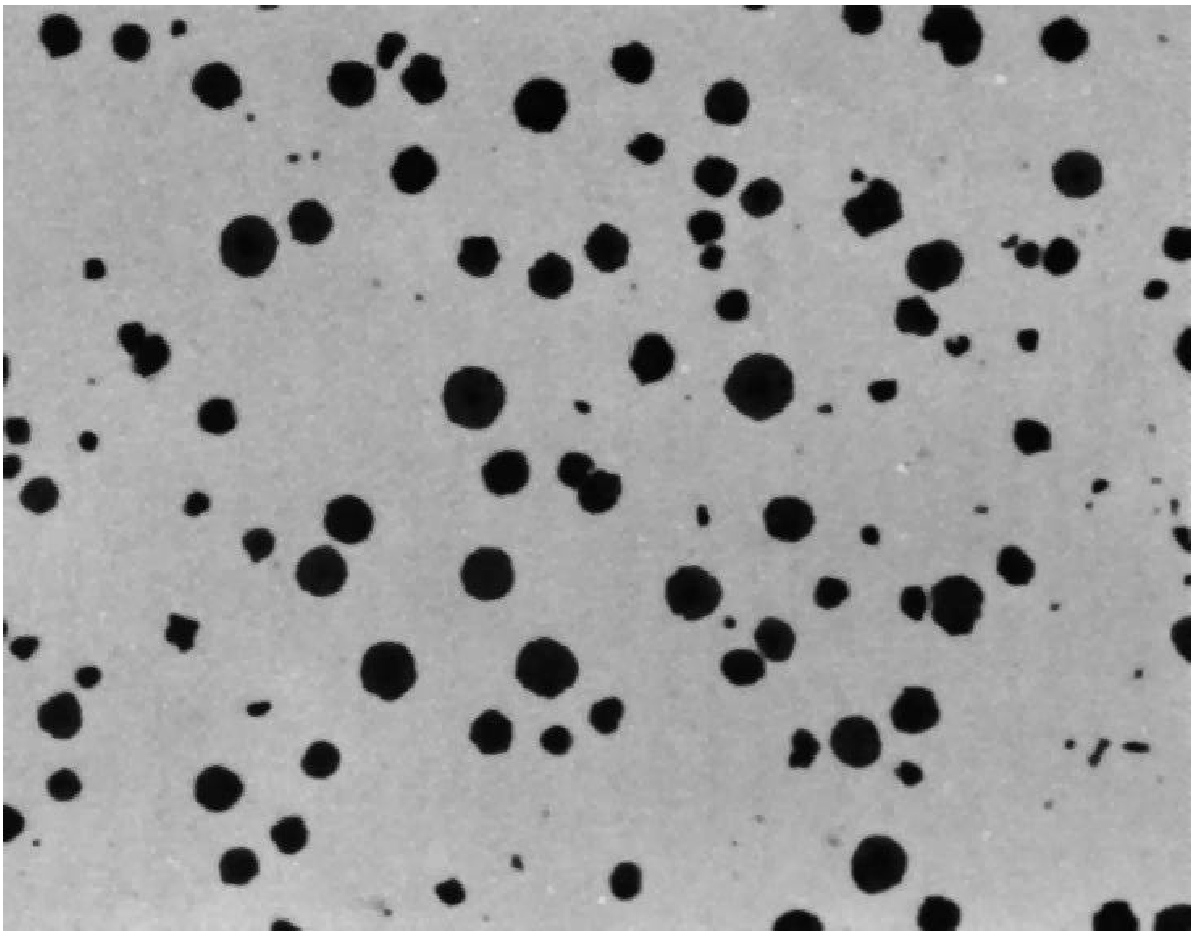
The standards relating to measurement of particle sizes are listed in the table below.

BS 3406 part4	Methods for Determination of Particle Size Distribution
BS 3625	Specification for Eyepiece and Graticules for the Determination of Particle Size of Powders.
BS 3406 part1	Determination of Particle Size Distribution. Guide to Powder Sampling
BS 2955	Terms Relating to Particle Technology.
ISO 9276-1	Representation of Results of Particle Size Analysis-Graphical Method.
ISO/DIS 9276-1	Draft Discussion Document, Representation of Results of Particle Size Analysis-Graphical Method.
ASTM F660	Comparing Particle Size in the Use of Alternative Types of Particle Counters
ASTM E1617	Standard Practice for Reporting Particle Size Characterisation Data.

Introduction to Particle Sizing

Probably the simplest use of image analysis is to provide measurement statistics of small particles. Applications for particle analysis are many and varied, including quality assurance for powder products, particulate contamination detection, and grade measurement.

Figure 8 An image of some particles for measurement.



BS 3406:Part 4 Methods for Determination of Particle Size Distribution Part 4. Guide to Microscope and Image Analysis Methods.

This standard provides procedures for the use of microscopes to measure particles in the range 2nm to 1mm using light microscopes and electron microscopes, both with and without the use of automated image analysis.

Definitions

The definitions in BS2955 apply, additionally there are a number of image analysis terms which are defined below.

Abbreviations and Symbols

The nomenclature of the measured results and calculations is defined. The standard nomenclature should be used when referring to parameters in calculations and results.

Sample Preparation

Samples are collected as described in BS3406 part 1. Mounting of samples can be done in various different ways.

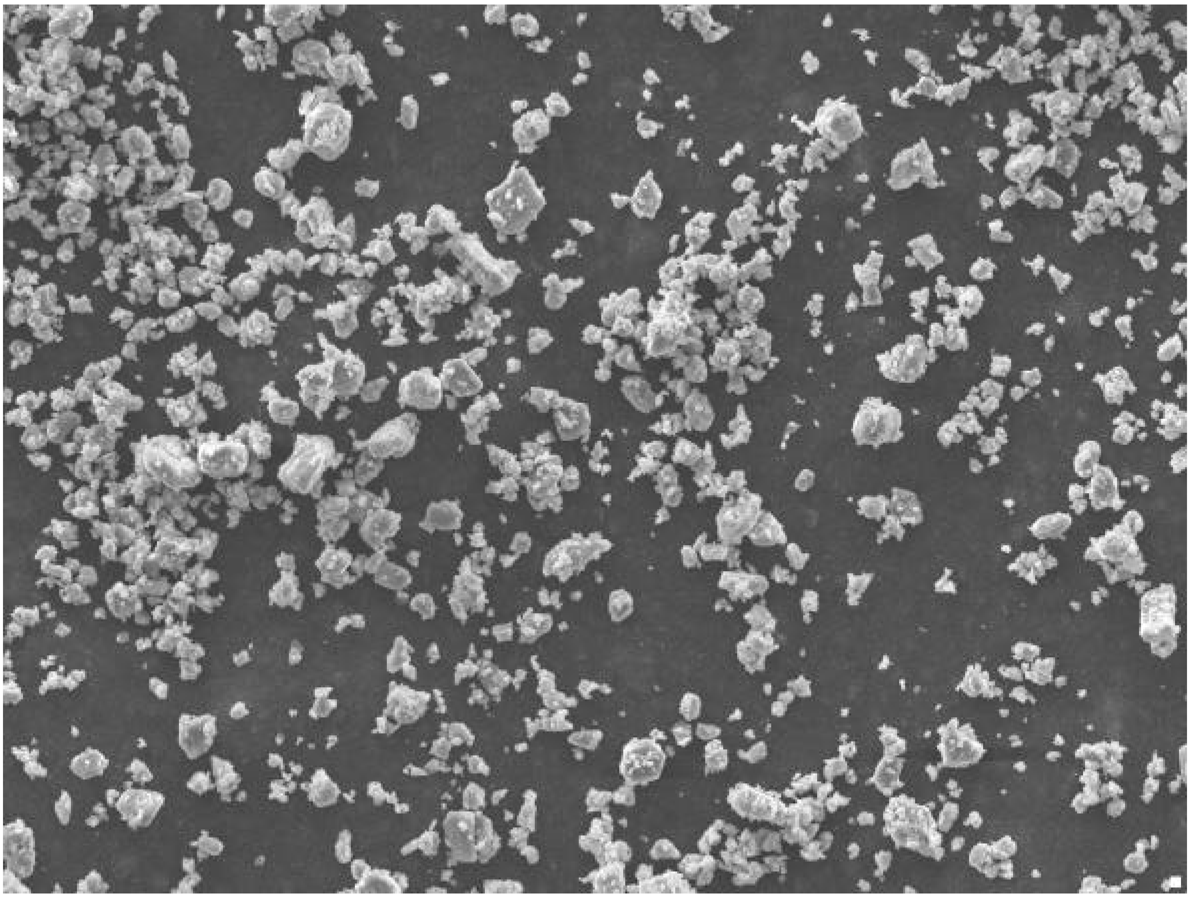
Mounting by filtration, the particles are suspended in a fluid which is then forced though a paper filter. The particles are left on the filter and can be observed under a microscope.

Mounting by straight-sided cavity slide, the particles are suspended in a cavity slide and a cover slip is placed over the slide. The particles are then viewed though the cover slip under a microscope.

Mounting by a drop of suspension on a microscope slide, a drop of the suspension containing the particles is dropped on to a glass slide and covered by a cover slip. It is important to observe sectors of the cover glass because of the potential for errors; due to the radial distribution of particle numbers and size. This technique is suitable for image analysis.

Mounting in a paste, this method is similar to the one mentioned above.

Figure 9 A good example of the application of particle sizing to measurement of powder particle size, this is an image of a powder sample.



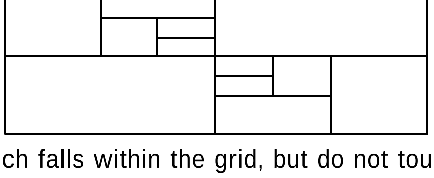
Measurement.

Measurement can be obtained using Transmission Electron Microscopes, Scanning Electron Microscopes, and Light Microscopes.

Manual Measurement Method

A graticule consisting of a number of rectangles of different sizes and circles of different sizes (representing the different classes of particles differentiated by area) is used. See BS 3625 eyepiece and screen graticules for the determination of particle size of powders. The sample area is divided into a number of smaller areas, the measured fields are located in the centre of these areas. The number of fields necessary to provide the required level of accuracy is determined by experimentally gathering data from a few fields and determining the number of additional fields to complete the scan. When particles are few and far between, 100 fields or more may be required.

Figure 10 Example of a graticule containing areas that are 2, 4, 8, 16, 32, 64 times as small as the whole.



Classification of each particle which falls within the grid, but do not touch the bottom and left hand margins of the grid comparison area, is made with the circles, using the filled and unfilled circles depending upon which is most appropriate for the particle in question. The areas over which particles are measured should be recorded.

The population in each class is counted. The selection of appropriate classes can be affected by the distribution of particle sizes in the powder. Most powders are subjected to some comminution process and have size distributions covering a very large range of particle sizes. The distributions are typically displaced markedly to the lower size limit. Such distributions can often be satisfactorily represented using a logarithmic form of the normal distribution. However, where the laboratory sample has been drawn from some pre-classified material then it would be more appropriate to use a linear progression of classification limits.

Measurement Using Image Analysis.

The image analysis system should be programmed to measure the area of individual particles. The results of several fields should be accumulated and the distribution of grain area values should be divided into classes using a geometric progression of size limits. The number of fields measured can be limited by area or by a particle count.

The range of the distribution may be greater than can be measured at a single magnification. If this is the case, once a significant area of the slide has been measured, an array of fields should be measured at a different magnification. The same series of absolute size limits should then be extended to include a few size ranges made common to both ranges in order to provide an overlap. The final results are expressed on a scale of equivalent circle diameter. See BS 3406 table for details of the minimum particle size allowed for some typical objectives. Detection can be performed using simple grey level thresholding or auto-delineation. A guard region should be used when determining particles to include in a measurement.

Finding the correct threshold level can be done by setting the best detection level for a medium size particle in the distribution, and subsequently making a measurement. A smaller particle can then be detected and the threshold level optimised to make a measurement, this can be repeated when the setting is selected for the larger particle. The last measurement should be made and repeated with the particle place near the four corners of the measure field. The detection of the small particle is satisfactory if the spread of its measurements made at the threshold chosen for the medium size particle does not exceed ½ a size class. The spread should be taken as the ratio of the largest to the smallest area measurement, the square root of this measurement shall be taken as the diameter ratio.

Distribution Determination, Accuracy and Precision.

The measurements should be expressed as equivalent diameters. The size distribution should be expressed as counts per unit area of the slides surface and as the total number of particles counted in each class.

The size limits should be separated by a logarithmic series of limits with adjacent limit ratio of 1/2c where c is an integer which may be chosen to give a sensible number of size classes over the expected distribution range.

Equation 58 Calculation of Number size proportion.

$$p_r = \frac{(m_r / A_r)}{\sum_{i=1}^{j} (m_i / A_i)}$$

100p_r is the number percentage of particles per unit area.

Equation 59 Calculation of volume size distribution

First calculate:

$$m_r d_r^3 / A_r$$

Then calculate:

$$q_r = \frac{(m_r d_r^3 / A_r)}{\sum_{i=1}^{j}(m_i / A_i)}$$

$100q_r$ is then the volume percentage of particles in the r^{th} class.

The standard error and confidence intervals can be calculated. The amount of specimen area to be covered can be calculated using the confidence limit to determine whether sufficient fields have been measured to produce results of the required accuracy.

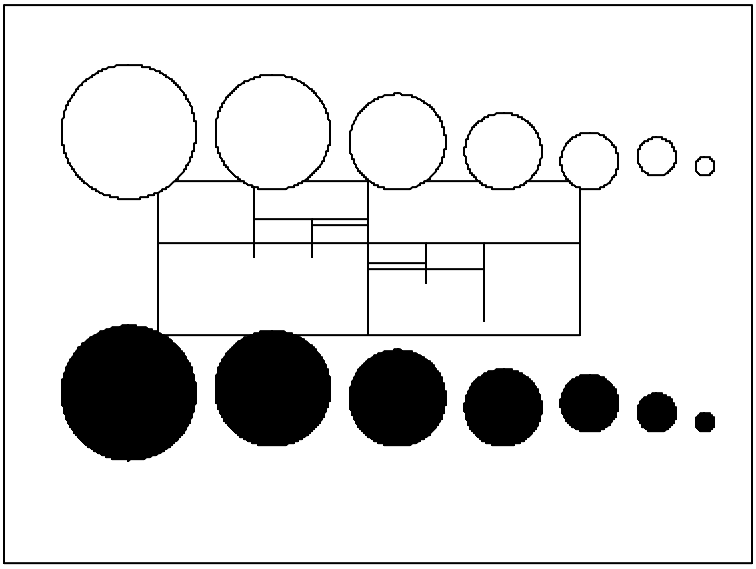
Reporting Results

All details of the analytical conditions used in obtaining them should be included to allow the analysis to be reproduced under identical conditions. Full details including instrument calibrations should be included.

BS3625 Specification for Eyepiece and Screen Graticules for the Determination of Particle Size of Powders.

This standard provides a specification for the graticules used in particle sizing. For example:

Figure 11 This is the grid graticule use in the BS 3406 part 4, the dimensions of each component are defined.



BS3406 part 1 Determination of Particle Size Distribution. Guide to Powder Sampling.

This standard defines the procedures for collecting and sampling the powder samples used in part four of the same standard, it provides an insight into the technology of powder size measurement.

BS 2955 Glossary of Terms Relating to Particle Technology.

This standard provides an agreed nomenclature for the technology of characterisation of particle sizes. This includes a section containing image analysis terms.

ISO 9276-1 Representation of Results of Particle Analysis.

This standard provides rules for standard nomenclature and graphical representation, in histograms, distributions and cumulative distributions of the dimensional statistics of particles.

There is no single definition of particle size x, different methods of analysis are based on measurement of different physical properties. In most cases the particle size is defined as the diameter of a sphere having the same physical properties - this is known as the equivalent spherical diameter. Other definitions are possible, such as the opening in a sieve or a statistical diameter feret diameter measure measured by image analysis.

The nomenclature for the parameters described in this standard is summarised as follows.

- x or d represent particle size. The independent variable is usually plotted on the abscissa.
- Q represents Cumulative measures
- q representa density measures

Figure 12 Arrangement of axes

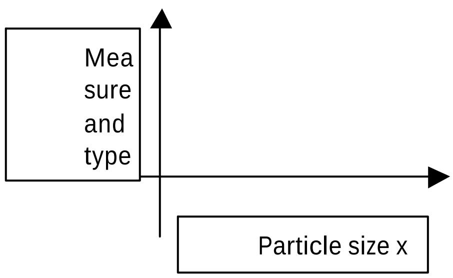


Table 30 Symbols for distributions.

Type	Density distribution	Cumulative distribution
Distribution by : number	$q_0(x)$	$Q_0(x)$
Distribution by : length	$q_1(x)$	$Q_1(x)$
Distribution by : area	$q_2(x)$	$Q_2(x)$
Distribution by : volume or mass	$q_3(x)$	$Q_3(x)$
General symbol	$Q_r(x)$	$Q_r(x)$

Graphical Representation.

The physical property chosen to characterise the particle size is plotted on the abscissa. An example is given in ISO 9276 part 1 draft for public comment. This is an amended version of the standard that is up for public discussion. It includes a sample data set for demonstration purposes. This data was gathered from a sieve, based on examination of the particle size.

Example

Here the classes of particles are defined as the contents of each sieve. In the separation procedure, The quantities of each size are evaluated by the mass of material left in the sieve.

Table 31 Data for example particle size graphical representation to ISO 9276 Part 1

i	x_i (mm)	$\Delta Q_{3,i}$	Δx_i (mm)	q_{3i}	$Q_{3,i}$	Q^*_{3i}
0	0.063	0	0	0	0	0
1	0.09	0.001	0.027	0.037	0.001	0.0028
2	0.125	0.0009	0.035	0.0257	0.0019	0.0027
3	0.18	0.0016	0.055	0.0291	0.0035	0.0044
4	0.25	0.0025	0.07	0.0357	0.006	0.0076
5	0.355	0.005	0.105	0.0476	0.011	0.0143
6	0.5	0.011	0.145	0.0759	0.022	0.0321
7	0.71	0.037	0.21	0.0857	0.04	0.0513
8	1	0.037	0.29	0.1276	0.077	0.108
9	1.4	0.061	0.4	0.1525	0.138	0.1813
10	2	0.102	0.6	0.17	0.24	0.28
11	2.8	0.16	0.8	0.2	0.4	0.4755
12	4	0.21	1.2	0.175	0.61	0.5888
13	5.6	0.24	1.6	0.15	0.85	0.7133
14	8	0.125	2.4	0.0521	0.975	0.3505
15	11.2	0.024	3.2	0.0075	0.999	0.0713
16	16	0.001	4.8	0.0002	1	0.0028

The graphs A1 A2 show the plots of

Q_3 Cumulative distribution by volume or mass and

$\overline{q_3^*}$ Average density distribution in a representation with logarithmic abscissa.

Against x the measured parameter, in this case, is the sieve hole size. Note the way in which the points representing the smaller particles are close together.

Figure 13 Density Distribution v Particle Size.

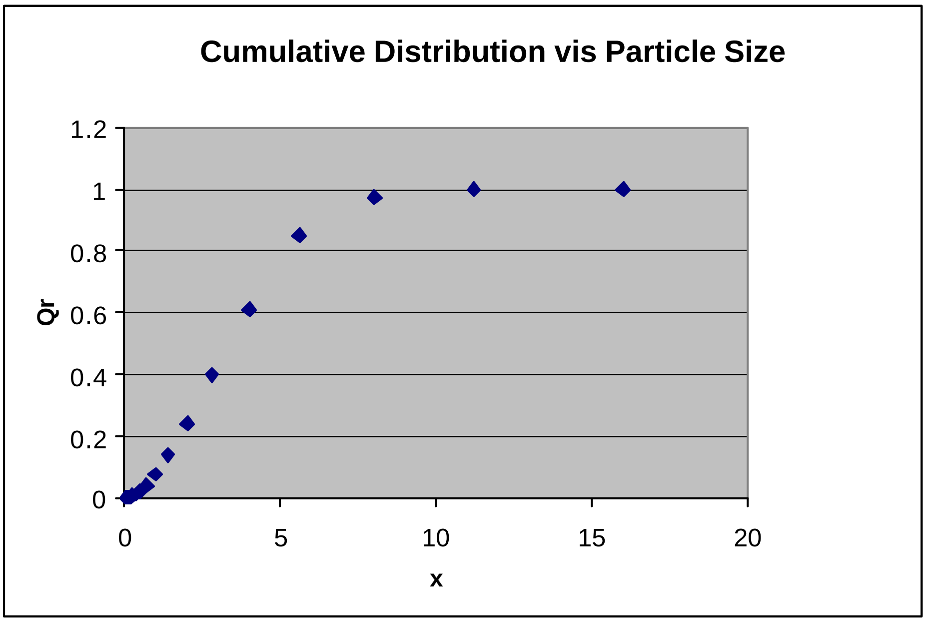
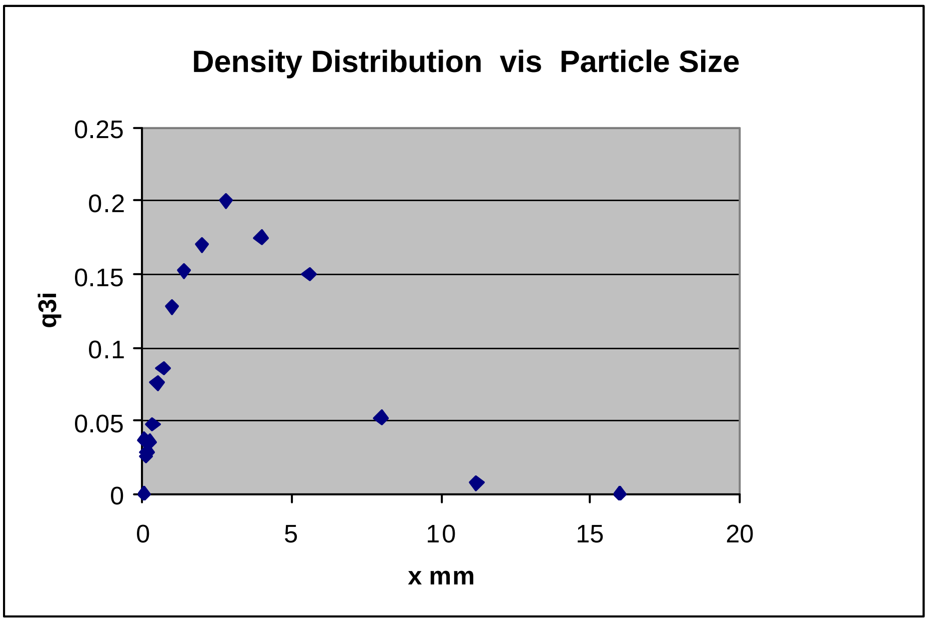


Figure 14 Cumulative distribution v Particle size



The same data is plotted below using logarithmic scale on the abscissa. This improves the graphical representation of the data, since the distribution is biased toward smaller particles. The points on the plot representing smaller particles are separated and made more visible to the reader when the log plot is used.

Figure 15 Density distribution v log Particle Size

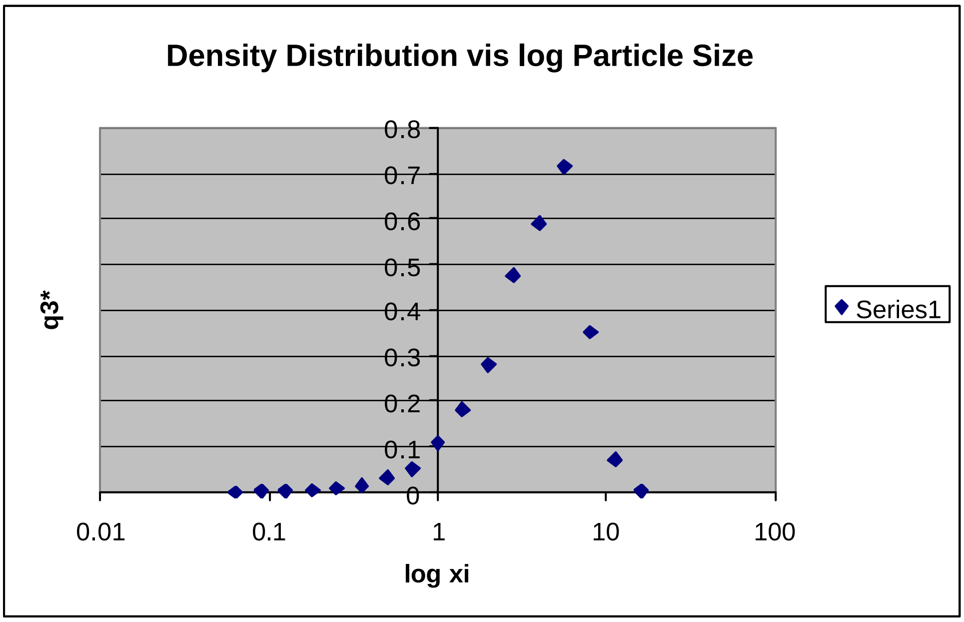
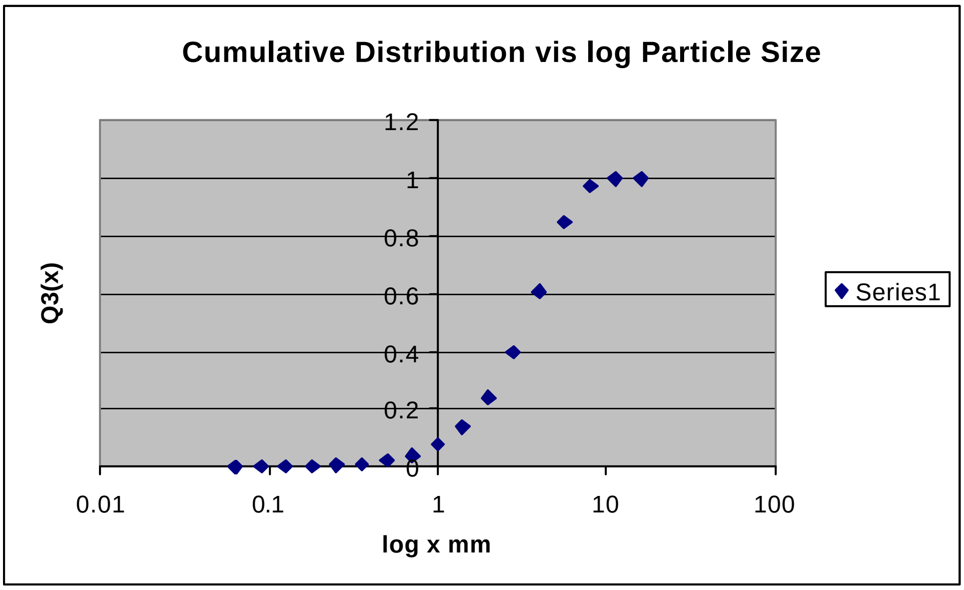


Figure 16 Cumulative distribution v log particle Size



Note: Since we can produce normal distribution by using functions of log x t is possible to calculate confidence limits from the processed data similar to other standards.

Reporting Results

Standards Draft BS standard ISO/DIS 9276-1 and ISO 9276-1. Provide rules for the nomenclature and methods of illustrating the size distribution of particles, by graphical means. The graphical description of the measured particle size data is expressed using numerical analysis. Cumulative distribution, Density distribution. Using linear and logarithmic abscissas.

ASTM F660-83 Comparing Particle Size in the Use of Alternative Types of Particle Counters.

This standard is intended to be used to compare different methods of particle size distribution measurement. This is done by calibrating the individual systems using particles of known size, and then comparing the size distribution curve of the different systems. The curves of each system are fitted to each other to calculate the necessary offset, to apply when comparing the different systems results.

The particle size systems include image analysers, optical counters, electrical resistance counters.

ASTM E1617 Standard Practice for Reporting Particle Size Characterisation Data.

This standard covers the reporting of particle size measurement data.

Reporting Results

Level one applies when only basic information about the sample is required. This is the minimum amount of information that is acceptable. It is applicable where detailed knowledge of the methodology is not required

Level two includes all the information in level one plus information on methodology.

Level three of the reporting standard should include written procedures to allow duplication of the measurement.

Report Guidelines

The report parameters are listed below for each level, note the levels are built up incrementally with more parameters added at each level.

Table 32 ASTM E1617 Report parameters

Particle size characterisation data sheet - Level 1	
	This ASTM designation i.e. ASTM E1617.
	Material.
	Source.
	Lot number
	Size parameters.
	Geometric mean diameter.
	Modal diameter.
	Median diameter
	Other defined size related parameter
	Data bias frequency mass etc
	Measurement range.
	Measurement principle.
Particle size characterisation data sheet - Level 2	
	This ASTM designation i.e. ASTM E1617.
	Material.
	Source.
	Lot number
	Size parameters.
	Geometric mean diameter.
	Modal diameter.
	Median diameter
	Other defined size related parameter
	Data bias frequency mass etc
	Measurement range.
	Measurement principle.
	Instrument and model
	Measurement conditions instrument operational parameters
	Software version number
	Calculation method
	Basic statistics, refer to E456
	Standard deviation
	Degrees of freedom and confidence level

	Confidence level
	Sample preparation
	Dispersion medium
	Dispersion steps
	Dispersion verification
Particle size characterisation data sheet - Level 2	
	This ASTM designation i.e. ASTM E1617.
	Material.
	Source.
	Lot number
	Size parameters for example
	Geometric mean diameter.
	Modal diameter.
	Median diameter
	Other defined size related parameter
	Data bias frequency mass etc
	Measurement range.
	Measurement principle.
	Instrument and model
	Measurement conditions instrument operational parameters
	Software version number
	Calculation method
	Basic statistics, refer to E456
	Standard deviation
	Degrees of freedom and confidence level
	Confidence level
	Sample preparation
	Dispersion medium
	Dispersion steps
	Dispersion verification
	Analytical procedure and number
	Precision and bias (refer to E177 and E691)
	Test or assay numbers
	Calibration or standardisation procedure.

Phase Percent

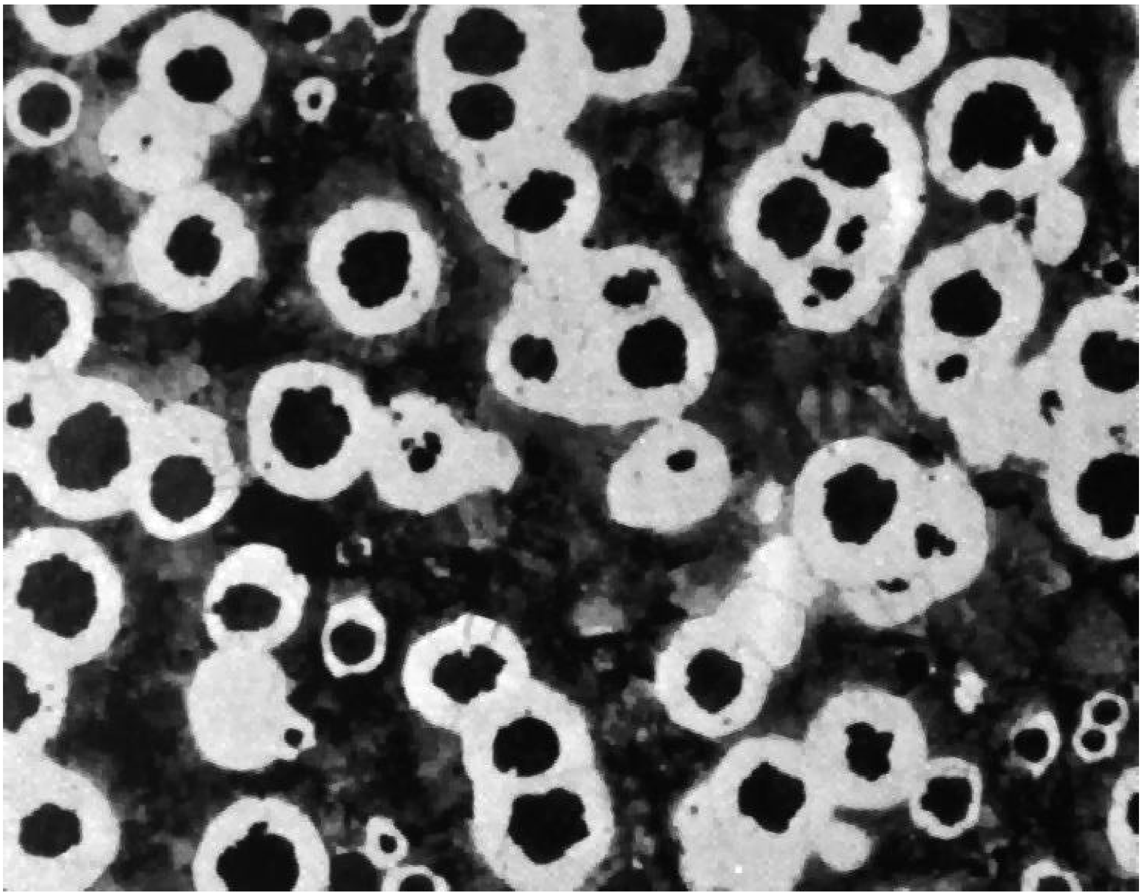
The standards relating to measurement of phase percent are listed in the table below.

BS 7590	Statistically Estimating the Volume Fraction of Phases and Constituents by Systematic Manual Point Count.
BS 7590A	Worksheet for the Determination of Volume Fraction by Systematic Manual Point Count.
ASTM E562	Standard Test Method for Determining Volume Fraction by Systematic Point Count.

Introduction to Phase Volume Fraction Measurement.

Phase volume percentage analysis is used to determine the volume fraction of a single phase in a microstructure, in which two or more phases are present and where the phase of interest can be segregated using grey scale thresholding. This kind of analysis can be used for quality analysis or research measurements in metals or other materials where multiple phases are present. It can also be used for straightforward area measurement. The following standards dictate measurement requirements and results to be reported.

Figure 17 An example image for Phase percentage volume analysis



BS 7590 Statistically Estimating the Volume of Phases and Constituents by Systematic Manual Point Counting with a Grid.

This British standard describes a method for statistically estimating the volume fraction of clearly defined constituents.

The method is based on the principal that when an array of points is systematically placed over a two dimensional section of a microstructure, the proportion of points that fall within a randomly distributed second phase is equal to the area fraction and volume fraction of the second phase in the microstructure. This mathematical relationship is shown below.

Equation 60 Principle of estimation of volume fraction.

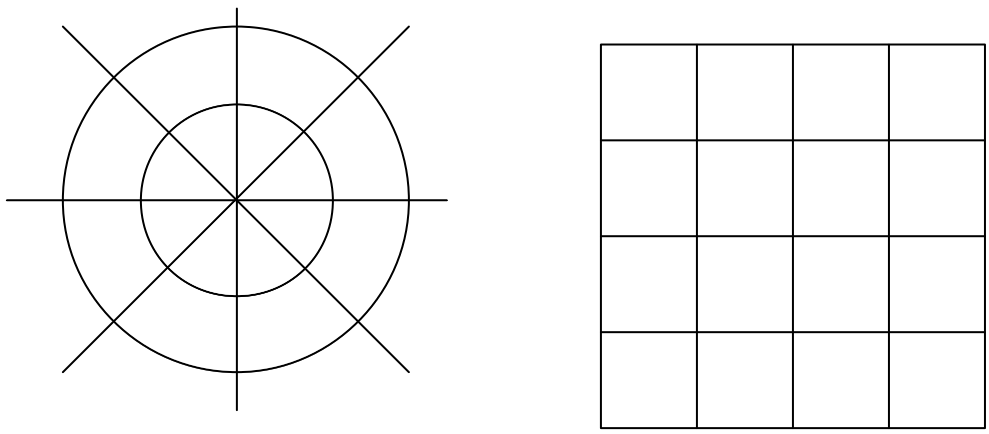
$$V_V = \frac{V_a}{V} = \frac{A_a}{A} = \frac{P_a}{P}$$

V_V is the volume fraction of the alpha phase. $V\alpha$ is the volume of the α phase in the sample. V is the total volume of the three dimensional sample. $A\alpha$ is the area of the α phase in a random planar section. A is the total area of the random planar section. $P\alpha$ is the number of points falling in the α phase in a random planar section. P is the total number of points laid down in the random planar section.

Method

The method consists of superimposing a point grid over a number of different representative fields on the microstructure. Counting the number of points included in the constituent or phase. Calculating the volume fraction of the phase.

Figure 18 Grids used for point counting circular and square.



Grid selection

Make an initial visual estimation of the volume fraction. Guidelines for grid size select a number of points on a grid.

Table 33 Guidelines for grid size selection (total number of points on grid) for visually estimated area fractions.

Visual estimate of area fraction	Recommended grid size P_T number of points on grid
2% to 5%	100
5% to 10%	49
10% to 20%	25
> 20%	<=16
A grid size that gives a significant number of fields with no grid points in the constituent of interest.	
These guidelines represent optimum efficiency for time spent counting.	

Selection of the Number of Fields Observed.

If 30 fields or more are available examine 30 fields minimum to provide acceptable statistics. If less than 30 fields are available for examination take the appropriate multiplier from the table below and use it in the following equation.

Equation 61 Calculation of confidence interval

$$CI = \pm \frac{2s}{\sqrt{n}}$$

(Equation 5 BS 7590)

CI is the confidence interval s is the standard deviation and n is the number of fields.

It is possible to define the acceptable relative error of the measurement and then calculate the number of fields required to produce the required accuracy. See tables 2 to 5 of BS 7590 and A.3.5.

Instead of the multiplier, two in the calculation of confidence limit.

Table 34Multiplier for substitution in equation

Number of fields examined	Multiplier for substitution in equation(5)
5	2.57
7	2.36
9	2.26
11	2.2
13	2.16
15	2.13
20	2.09
25	2.06
29	2.04

Reporting Results

The following information should be included in the report.

- Sample identification
- Orientation of the observed surface
- Number of fields observed and a description of their spacing
- Description of the grid type used and number of points
- Magnification used
- Estimated volume fraction for each section 95% confidence interval and the relative error.

Note: The original data should be recorded but not necessarily included in the test report. It may also be practical to record the visual estimation

A worksheet is provided to illustrate the format of results from this standard procedure.

ASTM E562 Determining Volume Fraction by Systematic Point Count.

This test method describes a systematic point count procedure for statistically estimating the volume fraction of an identifiable constituent or phase from a section though the microstructure using a point grid.

This test method is based on the stereological principle that when a grid is systematically placed over an image of a two dimensional section of microstructure, it can be used to produce an unbiased statistical estimation of the volume fraction of an identifiable constituent or phase.

Method

Sample Selection

Samples selected should be representative of the microstructure. If the sample shows inhomogeneities then the inhomogeneities should be included.

A test grid is superimposed upon image produces by a light or electron microscope. The number of points falling within the phase or constituent are counted and divided by the total number of grid points. This is a point fraction usually expressed as a percentage, for that field. The average point fraction for n measured fields. This method is applicable to opaque planar sections viewed with reflected light microscope.

The average number of points expressed, as a percentage of the total number of points in the array P_T is an unbiased statistical estimation of the volume percent of the microstructural constituent of interest.

Grid Selection

The grid should consist of equally spaced points formed by the intersection of lines.

Determine the number of points required in the grid in order to evaluate the fraction of identified phase in the field. By estimating the area fraction occupied by the phase and using Table 35

Table 35 Number of points required for different estimation of area fraction

Visual area fraction estimate expressed as a percentage	Grid size number of points P_T
2 to5%	100
5 to 10%	49
10 to 20%	25
>20%	16
These guidelines represent an optimum for efficiency for the time spent counting and for the statistical information obtained per grid placement	

Superimpose the grid in the form of a transparency or eyepiece reticule.

Magnification Selection

Select the magnification so that it is high enough to clearly resolve the microstructure without causing adjacent grid points to fall over the same constituent feature. As a guide, choose the magnification so that the average constituent size is approximately half the grid spacing. As the magnification increases the field size decreases and the field to field variability increases, and so the number of fields required to deliver the same degree of precision increases.

Counting

Count and record for each field the number of points falling on the constituent of interest. Count any points falling on a boundary as one half. Any point where it is doubtful whether it is inside or outside the constituent should be counted as one half. The number P_i divided by the total number of points on the grid

P_T times 100 gives the percentage of grid points on the constituent $P_p(i)$ for that field. The value represents a single statistic to be used with other statistics to estimate the average

$$\overline{P_P}$$

and the standard deviation s from a number of fields.

Selection of the number of fields.

The number of fields to measure depends on the desired degree of precision for the measurement. (Table 1E562) gives a guide to the number of fields to be counted as a function of P_T the selected relative accuracy (statistical precision), and the magnitude of the volume fraction. A minimum of 30 fields should be measured in order to calculate a confidence interval 95%CI for the average

$$\overline{P_P}$$

Using the following equation:

Equation 62 Calculation of 95% confidence interval

$$95\%CI = 2.0s \big/ \sqrt{n-1}$$

2.0 can be used as a multiplier when the number of fields is >30. When the number of fields is <30 the values in (ASTM E562 table 3) should be used.

Selection of the array of fields

Use a uniformly spaced array of fields to obtain the estimated value of Pp and s. Where gradients, inhomogeneities, or periodicity of microstructure are present, a random grid pattern may be more appropriate. Use of a random test pattern should be noted in the report. In the case of periodicity a circular grid or a square grid at an angle to the microstructure may be used to avoid coincidence grid points with the periodic distribution.

Grid Positioning

Position the grid over the field without biasing the results i.e. at random.

Improving measurement precision.

It is recommended that the user attempts to sample more of the microstructure either by using multiple specimens or repeating the preparation of the same sample.

Calculation of the Volume Percentage Estimate and % Relative Accuracy

The average percentage of grid points on the features of interest provides an unbiased statistical estimator for the volume percentage within the three dimensional microstructure.

The average percentage of grid points

$$\overline{P_P}$$

,s the standard deviation estimator and the 95% CI should be calculated and recorded for each set of fields. The equations for calculating these values are as follows:

Equation 63 Calculation of average percentage of grid points.

$$\overline{P_P} = \frac{1}{n}\sum_{i=1}^{n} P_P(i)$$

Equation 64 Calculation of standard deviation

$$s = \left[\frac{1}{n-1} \sum_{i=1}^{n} \left[P_P(i) - \overline{P_P} \right]^2 \right]^{1/2}$$

Equation 65 Calculation of 95% confidence interval.

$$95\%CI = 2.0s / \sqrt{n-1}$$

Equation 66 Calculation of relative accuracy.

The value 2.0 is acceptable when the number of fields is greater than 30

The relative accuracy is.

$$\%RA = \frac{95\%CI}{\overline{P_P}} \times 100$$

The Relative Accuracy should always be calculated (and not taken from tables).

Improving the volume fraction estimate.

If additional fields are measured to improve the relative accuracy, the following rule applies. To reduce the RA by 50% the number for fields must be increased by four times the original number.

Combining sets of data.

Data from separate specimens of the same microstructure can be combined by taking a grand mean of the set of means. If no sample heterogeneity is present i.e. the confidence intervals about the mean for each set of data overlap, then the 95% CI can be calculated from the standard deviation using the data from all sets of data. i.e. pooling the data and calculating a mean SD and 95%CI.where the 95% CI about the mean does not over lap for the different sets. Then a statistically significant difference between sample sections may be present. In this case more rigorous significance tests should be considered.

See ASTM E562 for a simplified definition of the procedure.

Reporting Results

Report the following information:

- Raw data.
- Scan pattern type
- Estimated Volume % Pp +- % CI
- % Relative Accuracy (calculated value not one estimated from table 2 E562).
- Number of fields per metallographic section.
- Number of sections.
- Sample Description and preparation including etchant if used.
- Section orientation
- Location of the specimen within the product
- Magnification.
- Grid description
- Field array description and spacing
- List of volume % estimates for each metallographic sections +- 95 % CI

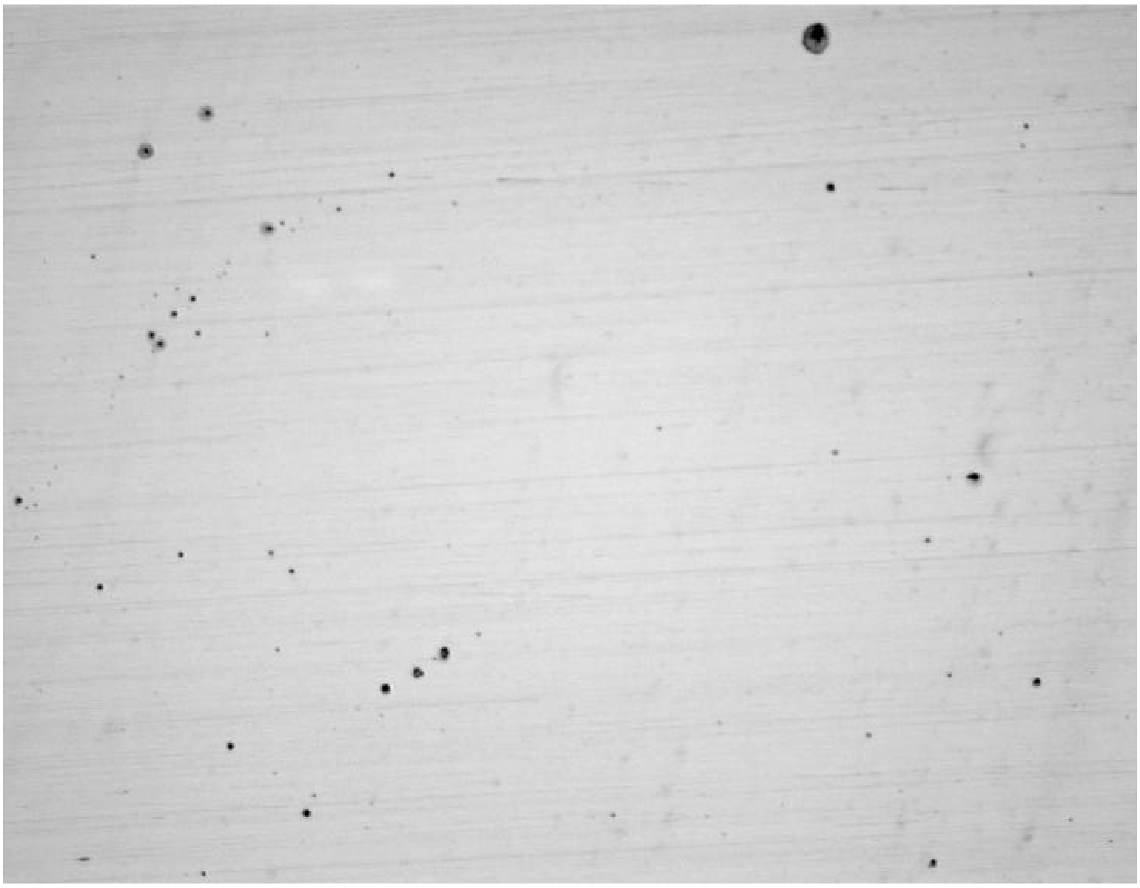
Steel Inclusion Analysis (Manual ASTM E45)

The standards relating to measurement of steel inclusions are listed in the table below.

ASTM E45	Standard Test Method for Determining the Inclusion Content of Steel.

Introduction to Steel Inclusion Analysis.

Figure 19 An Image of Inclusion s in a polished steel sample.



The ASTM E45 standard is a popular standard for the rating of inclusions found in steel. The standard provides manual methods for operators to use to rate inclusions of the four principal types. The methods are described briefly below.

ASTM E45 Standard Test Method for Determining the Inclusion Content of Steel.

Method

The test methods described in this standard are intended for the manual rating of inclusions. est methods are divided into macroscopic methods and microscopical methods. The Macroscopical methods include the macro etch method, the fracture test and magnetic particle method. Microscopical methods are those that can be emulated by image analysis.

These methods ASTM E45 A, B, C, D, E. are described below

Preparation of specimens.

Specimens are prepared from samples of the product. The specimen is cut ground and polished so that a microscopically flat section is produced which shows the inclusions size and shapes accurately. The surface should be clean and free of scratches and artefacts. The surface should be in the polished condition for examination, any evidence of etching prior to polishing should be removed, inclusions should not be pitted or dragged.

Classification of inclusions.

Inclusions are divided into four types based on their morphology rather than the chemical composition.

- Sulphide type A
- Alumina type B
- Silicate type C
- Globular oxide D

Each of these types is classified as thin or heavy according to stated dimensional limits. Differentiation between inclusion types is made upon grey scale and morphology.

Inclusion types are defined in ASTM E7

Sulphide inclusions type A.

Sulphide type inclusions in steel non-metallic composed essentially of manganese sulphide solid solutions (Fe Mn)S. They are characterised by plasticity at hot rolling and forging temperatures. In the hot worked product they appear as dove grey elongated inclusions varying from thread like to oval outline, selinide inclusions may behave similarly.

Sulphide type A and Silicate type C inclusions are very similar in morphology both being elongated and stringered. The two types can be differentiated by greyscale. Sulphide inclusions are dove grey and silicate like all other oxide inclusion types (which appear black).

Alumina Inclusions, type B.

Oxide compounds occurring as non-metallic inclusions in metals usually occur as a result of deoxidising additions e.g. aluminium producing Alumina inclusions. In wrought products made from steel they may occur as stringer formation composed as distinct granular or crystalline appearing particles.

Silicate inclusions type C.

Inclusions composed essentially of silica glass normally plastic at forging and hot rolling temperatures, will appear in steel in the wrought condition as small elongated inclusions usually dark in colour under reflected light as normally observed.

Globular oxide inclusions type D.

Inclusions which are isolated relatively non deformed inclusion with an aspect ratio not in excess of 5:1 in other methods oxides are divided into deformable and non deformable types.

Stringer.

An individual inclusion which is highly elongated in the deformation direction or three or more type B or C inclusions aligned in a plane parallel to the hot working axis, and offset by no more than 15microns with separation of less than 40 microns (0.0016in) between the two nearest neighbour inclusions.

Discontinuous Stringer.

Three or more type B or C inclusions aligned in a plane parallel to the axis of hot working and offset by no more than 15 microns with separation of less than 40 microns between any two nearest neighbour inclusions.

ASTM E45 Method A(Worst Fields)

The test methods require a survey of $160mm^2$ ($0.25in^2$) of the specimen at 100x magnification. The field size shall be equivalent to a field size of 0.5 mm^2 ($0.000779in^2$) as defined by a square 0.71mm by 0.71mm. The specimen is scanned in an S scan pattern. Each 0.5 mm^2 field is compared to the square fields depicted in Plate1-r ASTM E45, and a rating given according to the comparison made. The highest severity rating for each inclusion type A, B, C, D. for both thin and heavy series. The severity level of these worst fields shall be reported for every specimen examined.

Where the severity falls between two ratings it is rounded down to the lower severity level.

Table 36 Minimum for severity level numbers (Methods A D and E.)

Severity Level	Type A	Type B	Type C	Type D
	Total length in one field at 100x min mm (in)	Total length in one field at 100x min mm (in)	Total length in one field at 100x min mm (in)	Count
½	3.7(0.15)	1.7(0.07)	1.8(0.07)	1
1	12.7(0.5)	7.7(0.30)	7.6(0.3)	4
1 ½	26.1(1.03)	18.4(0.72)	17.6(0.69)	9
2	43.6(1.72)	34.3(1.35)	32.0(1.26)	16
2 ½	64.9(2.56)	55.5(2.19)	51.0(2.01)	25
3	89.8(3.54)	82.2(3.24)	74.6(2.94)	36
3 ½	118.1(4.65)	114.7(4.52)	102.9(4.05)	49
4	149.8(5.90)	153.0(6.02)	135.9(5.35)	64
4½	189.8(7.47)	197.3(7.77)	173.7(6.84)	81
5	223.0(8.87)	247.6(9.75)	216.3(8.52)	100

Table 37 Inclusion width and diameter parameters methods A and D

Inclusion type	Thin series width min microns	Thin series width max microns	Heavy series width min microns	Heavy series width max microns
A	2	4	>4	12
B	2	9	>9	15
C	2	5	>5	12
D	2	8	>8	13

Classify discontinuous type stringers of inclusion types B and C as two distinct inclusions when they are separated by at least 450 microns (0.0016in) or offset by more than 15 micron on the specimen surface. If two or more inclusions of the same type A, B or C appear in one microscope field their summed length determines the severity level number. Comparison with the standard Plate 1-r is usually sufficient to determine the rating.

The standard plate is intended to illustrate specific parameters of the different inclusion types. These are as follows. Total length of Type A inclusions. Total stringer length of type B and C inclusions. The number of D type inclusions. The plate divides the inclusion into thin and heavy types according to the width and diameter limits.

ASTM E45 Method B

This test method requires a survey of 160 mm^2 of the test specimen at 100x magnification. The field size shall be equivalent to a square field size of 0.5 mm^2. Any inclusion whose length is equal or longer than 0.127mm should be individually tallied.

This method uses a pattern of parallel lines spaced out so that the distance between each line is 0.127 mm on the specimen surface at x100. A graticule or other device is used to superimpose this pattern on the image. The pattern is then applied to a series of fields as follows. The 160mm^2 test area is marked on the specimen with a scribe. The field of view is placed in one of the corners of the test area One unit equals 0.127mm on the specimen surface.

Measure and record all inclusions one unit in length and longer. Inclusions separated by a distance greater than one unit shall be regarded as being two separate inclusions and not classified as a stringer. The length of an inclusion shall be rounded down to the next whole unit, only whole units shall be recorded. If part of an inclusion lies outside the current field i.e. part of its length lies in what will become field 2, move the field slightly in order that the full length can be measured. Repeat this procedure for all fields in a 's' scan pattern until the entire 160mm^2 area is covered.

Expression of Results.

The length of the longest inclusion shall be recorded first. The description of inclusion width is a superscript T or H representing thin or heavy. A thin inclusion is defined as being 10µm or less in width over 50% or more of its length. Likewise a heavy inclusion must have a thickness 30µm or more over the majority of its length inclusions falling in between these two values shall not be represented by a T or H superscript.

Superscripts d disconnected, vd very disconnected, and g grouped. May also be used. Definitions of these different types can be can be seen in ASTM E45 Fig 8. When required a series of comparison images can be used which illustrate all other non-metallic particles present. These are labelled A, B, C in order of ascending inclusion population. A suitable series of photomicrographs shall be agreed by interested parties e.g. A series of four micrographs of low carbon steel published by ASTM.

The average of all inclusions equal or longer than one unit in length excluding the longest inclusion shall be averaged. The following is an expression of results for this method.

6^d-2^3-A

This indicates that the longest inclusion was six units long, that three other inclusions whose average length was 2 units and that background inclusions were similar to those illustrated in photomicrograph A. The results from a lot shall be tabulated if required; the predominant type of inclusion shall be recorded.

ASTM E45 Method C

This test method requires a survey of 160 mm^2 of the test specimen at 100x magnification. A rectangular mask is used 0.79 x 1.05 mm on the specimen surface. Each field shall be examined for Alumina and Silicate inclusions and rated according to Plate II. NB sulphides are not rated by these methods. The longer side of the rectangle shall be parallel to the rolling direction.

To begin the test outline the required test area. Start the examination with the first field in one of the corners of the test area. Compare the content of the field of view with plate II and select the number of the frame, which most closely represents the oxide and silicate stringers. If an inclusion size falls between two numbers then it should be rounded down to lower value.

Stringered inclusion shall be classified as two distinct inclusions if separated by more than 40µm or offset by more than 15µm. Comparison with plate II is repeated for each field in the scan. It may be necessary to adjust the mask so that the entire stringer is contained within the mask. The rater's objective is to find the longest silicate and oxide inclusions in the specimen. Therefore in practice the rater is scanning the specimen and stopping only when a potential longest stringer is in view.

Expression of results.

A specimen may be classified according to the silicate and oxide photomicrographs in Plate II. E.g. a sample may be classified O-5 (oxide) S-4 (silicate) to indicate that the longest silicate is comparable with silicate micrograph 4 and the longest oxide with oxide micrograph 5. Modification such as suffix numerals may be added to indicate the number of long inclusions noted or the exact length of a particular inclusion when it is over the maximum length indicated in the photomicrographs.

ASTM E45 Method D

This test method is intended for application to steel with low inclusion contents. This test method requires a survey of 160 mm^2 of the test specimen at 100x magnification. The field size shall be equivalent to a square field size of 0.5 mm^2. There is no flexibility in the scan pattern in this method. This is an all field method unlike method A.

The test area should be outlined and the scan started with the field of view in one corner of the test area. Compare the fields with the micrographs on Plate I-r record the inclusion severity level number for each inclusion type A, B, C, and D. that most resembles the field under observation. Do this for both thin and heavy series. See Table 37 Inclusion width and diameter parameters methods A and D.

Report all fields with inclusion above the 0.5 severity level. Type A, B or C inclusion wider than those depicted in the heavy series of plate I-r shall be noted and recorded separately. Their lengths still contribute to the rating of the field. Inclusions with severity falling between two values should be rounded down to the nearest severity level.

Similarly for D type globular oxides, record separately any larger than those shown in the heavy series in plate I-r. Type D globular oxide may not exceed an aspect ratio of 5:1. The minimum inclusion lengths are shown in Table 36 Minimum for severity level numbers (Methods A D and E.) Stringered inclusion shall be classified as two distinct inclusions if separated by more than 40µm or offset by more than 15µm

Inclusion should be classified as thin or heavy depending upon which is most appropriate for each inclusion. If two or more stringered inclusions appear in one field their combined lengths determine the inclusion rating.

Expression of results

The number of fields of each inclusion types of plate I-r for both thin and heavy series shall be recorded in terms of the severity level 0.5 to 3.0 any exceeding the limits of severity level 3 it shall be recorded separately. Inclusions exceeding the limits of the heavy series shall also be recorded separately.

To average the results of more than one specimen the average of the number of fields found for each inclusion rating and type in the various specimens examined may be calculated.

ASTM E45 Method E (SAM Rating)

This test method requires a survey of 160 mm^2 of the test specimen at 100x magnification. The field size shall be equivalent to a square field size of 0.5 mm^2. Outline the test area and start the examination in one of the corners. Compare each field with the images in Plate I-r rate only the B and D type inclusions. Record all B thin fields at severity 1.5 or higher and all B heavy fields at severity 1 or higher. For this method b heavy inclusion are defined as inclusion wider than 30µm. Round intermediate fields down to the lower value.

Stringered B type inclusion shall be classified as two distinct inclusions if separated by more than 40µm or offset by more than 15µm. If two or more b type inclusions appear in one field then their summed length determines the severity rating. A rating of D type inclusions is obtained by recording all D type heavy oxides with a rating greater than 0.5. For this method D type oxides are determined as particles measuring 13µm or larger at their widest point. The minimum inclusion numbers are shown in Table 36 Minimum for severity level numbers (Methods A D and E.).

If any inclusions are present that are longer than the fields shown then they should be recorded separately. Oversize B and D inclusions still contribute to the severity rating of the field.

Expression of results

Results are expressed in terms of two rating numbers type B and D heavy inclusion contents.

The number of B type fields recorded at each severity level is summed and normalised by dividing the total rated area in inches of all samples, the nearest whole number is recorded as the rating.

The number of D units is summed and normalised by dividing by the total rating area in square inches of all samples, the nearest whole number is recorded as the rating. All over size B or D type inclusions will be reported along with their actual lengths or widths or both.

Reporting Results

The following should be reported.

- Date of test
- Rater name
- Plant location
- Heat number
- Specimen number and any other information uniquely identifying the specimen. This can provide traceability within the seller's organisation.
- The average of the ratings for each inclusion type over all the specimens in the lot is calculated