TECHNICAL REPORT

ISO/TR 945-2

First edition
2011-01-15

Microstructure of cast irons —

Part 2: Graphite classification by image analysis

Microstructure des fontes —

Partie 2: Classification du graphite par analyse d'image

Reference number
ISO/TR 945-2:2011(E)

PDF disclaimer

COPYRIGHT PROTECTED DOCUMENT

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In exceptional circumstances, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example), it may decide by a simple majority vote of its participating members to publish a Technical Report. A Technical Report is entirely informative in nature and does not have to be reviewed until the data it provides are considered to be no longer valid or useful.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO/TR 945-2 was prepared by Technical Committee ISO/TC 25, *Cast irons and pig irons*.

ISO 945 consists of the following parts, under the general title *Microstructure of cast irons*:

— *Part 1: Graphite classification by visual analysis*

— *Part 2: Graphite classification by image analysis* [Technical Report]

Introduction

Image analysis, as well as other testing methods, is part of the general or specific assessment of the quality of castings to be agreed between the manufacturer and purchaser at the time of acceptance of the order.

The characterisation of the graphite particle shape in cast irons is often made visually, using the reference sketches of ISO 945-1. The procedure described in ISO 945-1 has an inherent subjective character that can be overcome by using image analysis and appropriate computer software.

Microstructure of cast irons —

Part 2:
Graphite classification by image analysis

1 Scope

This part of ISO 945 deals with the two-dimensional characterisation of graphite form and size in cast irons.

A standard method for determining graphite form by image analysis had not been developed at the time of this report, but several methods are in use in the industry. The purpose of this part of ISO 945 is to give an illustration of what is possible and to suggest ways in which the technique might develop in the future.

This part of ISO 945 does not apply to the graphite distribution of grey (lamellar graphite) cast iron as defined in ISO 945-1.

This part of ISO 945 describes procedures used to carry out image analysis of graphite form and size and provides a method of comparison to the results obtained using visual analysis techniques.

It does not specify any particular mathematical description of the graphite forms but provides a means for comparison.

NOTE A mathematical description of graphite form is given in ISO 16112. Other mathematical descriptions of graphite forms and information on technical research are given in the Bibliography.

2 Terms and definitions

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

2.1
maximum Féret diameter
maximum length of an object whatever its orientation

NOTE This dimension applies to all graphite forms.

2.2
roundness
area of the graphite particle divided by the area of the circle whose diameter is the maximum Féret diameter of the same graphite particle (X)

NOTE The calculation of roundness is shown in Figure 1.

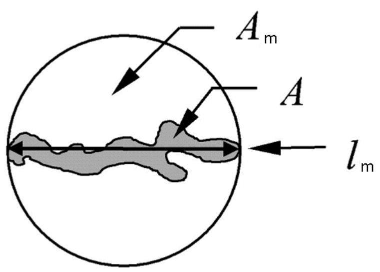


Figure 1 — Roundness

Roundness = $A/A_m = 4A/\pi \cdot l_m^2$

where

l_m is the maximum axis length of the graphite particle in question, which is the maximum distance between two points on the graphite particle perimeter;

A_m is the area of the circle diameter l_m;

A is the area of the graphite particle in question.

2.3
perimeter
P
total length of the object contour

2.4
graphite content rate
graphite content as a percentage of the measured area

3 Designations

3.1 Designation system for classifying graphite in cast irons

When cast iron materials are examined using the image analysis method in accordance with this part of ISO 945, the graphite should be classified by:

a) its form, designated by Roman numerals I to VI, see Annexes A and B (informative);

b) its size, designated by the Arabic numerals 1 to 8, see Table 1.

3.2 Image analysis classification of graphite

The reference images given in Annex A provide a basis for classifying graphite forms. For this purpose, and as examples of actual graphite forms corresponding to these reference images, typical microstructures of graphite in cast irons are arranged in a series of photomicrographs given in Annex B.

The graphite size is determined by measuring the graphite and comparing it with the dimensions and reference numbers given in Table 1.

Because the examined images represent two dimensional cross sections of three dimensional graphite particles, the observed size of any particle will almost always be smaller than its actual size. This fact should be taken into account when evaluating particle size distributions, whether by image analysis methods or by the visual analysis method of ISO 945-1. In ISO 945-1 the determination of graphite size is based on the larger observed particle sizes. Using image analysis, an adjustment to exclude other than the representative larger particle sizes is appropriate.

3.3 Designation of graphite by form and size

3.3.1 Designation by system

To characterise the graphite observed, indications are generally necessary on the form and size of the graphite particles. For this purpose, the following symbols should be used at different positions of the designation:

— Roman numerals of Annex A are used for the graphite form at position 1;

— Arabic numerals of Table 1 are used for the graphite size at position 2.

EXAMPLE 1 For grey cast iron with flake (lamellar) shaped graphite particles of form 1 and size 4, the following designation should be used to describe the structure:

I 4

EXAMPLE 2 For a cast iron with spheroidal graphite particles of form VI and size 4, the following designation should be used to describe that structure:

V1 4

Table 1 — Dimension of the graphite particles forms I to VI

Reference number	Indication of the particle dimension observed at 100 x magnification mm	Actual 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	6 to <12	0,06 to <0,12
6	3 to <6	0,03 to < 0,06
7	1,5 to <3	0,015 to <0,03
8	<1,5	<0,015

NOTE 1 This table is identical to Table 1 ISO 945-1 except for NOTES 2, 3 and 4.

NOTE 2 For determining size ranges 1 and 2, a lower magnification (25 x or 50 x) may be used.

NOTE 3 For determining size ranges 6 to 8, a higher magnification (200 x or 500 x) may be used.

NOTE 4 For determining size ranges 1 and 2, the largest visible graphite particle size is to be retained.

3.4 Designation of intermediate graphite size

If the graphite observed covers two sizes, reference to both is possible.

EXAMPLE 1 3/4

In a given case the predominating size may be emphasised by underlining.

EXAMPLE 2 3/4

This method can be extended to cover structures where more than two sizes are present.

NOTE Other means of expressing graphite size, such as histograms, can be used.

3.5 Designation of mixed graphite forms and sizes

Graphite structures of mixed form and size can be defined by calculating their percentage proportions.

EXAMPLE For a cast iron with graphite area of 85 % spheroidal graphite particles of form VI, and size 4 and 15 % vermicular graphite particles of form III and covering sizes 3 and 4, the following designation shall be used to describe the structure:

85 % VI 4 + 15 % III 3/4

4 Sampling and sample preparation

4.1 Samples taken from castings or cast samples

The location from which samples are taken should be agreed between the manufacturer and purchaser, taking into account any other requirements in the relevant material standard. If an examination report is required, the location from where the sample is removed shall be recorded.

The sample should be of sufficient size to provide a true representation of the graphite structure in the agreed location from which it is taken.

4.2 Sample preparation

4.2.1 General

Attention should be paid to the careful grinding and polishing of samples, so that the graphite structure appears in its original form and size. Inappropriate preparation can cause unacceptable alteration of the microstructure.

Sample preparation should be carried out in three stages:

— 1. Sectioning;

— 2. Grinding;

— 3. Polishing.

The examination of the graphite should be carried out in the un-etched condition.

4.2.2 Sectioning

The sample should be sectioned in the area agreed between the manufacturer and the purchaser. Care should be taken to ensure that the structure is not altered by the sectioning technique.

4.2.3 Grinding

The sample should be ground in the area agreed between the manufacturer and the purchaser. Care should be taken to ensure that the structure is not altered by the grinding technique.

4.2.4 Polishing

Samples should be repeatedly polished and etched until the true graphite content is revealed and the samples should then be examined in the un-etched condition.

5 Binary image preparation

5.1 General

Once the sample is correctly prepared, appropriate images are taken using a metallographic microscope, a digital camera, a computer image caption program and the image analysis programme. This procedure encompasses several repetitive actions which are listed in chronological order. All these factors can influence the relative exposure of matrix and graphite and the sharpness of the transition zone between both phases.

NOTE It is recommended that a magnification is chosen that enables 20 or more graphite particles to be measured per measured field. The number of particles measurable depends on the pixel resolution of the digital camera. For effective image analysis a higher number of particles can be useful.

5.2 Microscope image light setting

The final picture can be substantially influenced by the intensity of the light on the sample. For un-etched samples containing graphite, a high illumination will remove details from the metal matrix, scratches will disappear and the matrix becomes plain white. At the same time, details in the graphite become visible, as for example, stratifications in graphite spheroids. However, thin graphite details, like thin lamellae or small particles, gradually disappear.

5.3 Microscope filters

Light filters can be present which change the colour composition of the illuminating light.

5.4 Camera

A minimum number of pixels per unit length is needed in order to detect fine details of the graphite structure. The pixel resolution directly influences the number of pixels on the edge of a graphite particle and therefore also the particle perimeter. This parameter appears in some shape factors (for example, the sphericity factor) which are used to classify the graphite particles. Images with a lower total number of pixels can detect the same details on the sample if the magnification of the microscope is increased.

NOTE 1 The image acquiring system should be adjusted to eliminate the background noise.

NOTE 2 Provision should be made to prevent the white and black saturation levels from being reached

NOTE 3 A minimum resolution of 1 pixel/µm should be used.

5.5 Binary image

5.5.1 Brightness setting

The effect of a variation of the illumination by the external light source of the microscope can be obtained in a similar way by changing the programme settings of the image capture programme. By adjusting the exposure time setting, the metallic matrix can be overexposed giving a plain white matrix with less fine graphite details.

Computer programs often have an under/over exposure option. With this option, the user can see whether parts of the image are underexposed or overexposed. In between, details of the metallic matrix are clearly visible (for example, scratches).

To avoid over exposure it is suggested that a colour coding is displayed in the live image for every pixel with brightness above a threshold value.

5.5.2 Sharpness setting

The images should be sharp when the picture is taken. However, image capture programmes allow the sharpness between black and white areas to be increased. The grey transition zone becomes smaller. This feature has an important effect on the grey selection value. Images with sharpened edges are less prone to changes in the grey scale threshold value because the image is shifted from a grey scale picture to a black and white picture.

5.5.3 Camera setting "colour" or "grey"

Un-etched cast iron samples do not require colour images. Grey scale images are sufficient to highlight all graphite shape details. If binary pictures are used, i.e. white for metal and black for graphite, comparison between different image analysis programmes is possible using exactly the same conditions.

In order to separate the graphite particles from the matrix, the threshold level should be carefully chosen so that it does not impair the particle contours (halation effect). The operation can be done either automatically or manually. In either case, the operator should carefully check the threshold level result.

5.5.4 Image data format storage type

A graphic type of image should be used which does not eliminate details. Pictures using "bmp" or "tif" file formats do not compress data and preserve the original image details at the expense of large data size. Images should be stored in the same condition before and after image analysis. When transferring images from producer to customer, generally available graphic standards should be used.

5.6 Image analysis computer programme

5.6.1 General

The image analysis computer programme should be able to read in images made by other image capture programmes.

5.6.2 Grey scale threshold value

A very important factor is the selection of the grey scale threshold value. Grey scale images use an integer value from 0 to 255 (i.e. 8 bits or 1 byte) to define a grey scale. Extreme values are white (255) for the metallic matrix and black (0) for the graphite particles. The grey scale threshold value divides pixels into two groups: either metallic (white) or graphite (black). The percentage of graphite in the image changes depending on the grey scale threshold value.

If there are pores or voids, the user or the software should be able to exclude these areas from the graphite measurement.

5.6.3 Magnification factor

Many shape factors are dimensionless parameters not requiring any scale factor. However, thickness, length, perimeter, area or the number of particles per unit surface all require a dimension. Consequently, the magnification factor should be supplied for each picture. It should be given as the number of pixels per unit length. This value should be the same in both the horizontal and the vertical direction.

6 Measurement

Particle measurements are carried out using two frames: one delimits the image field and the other delimits the measurement field. That is to say, the particles intercepted by the two adjacent sides of the frame of the measurement field should be eliminated. Particles intercepted by the two other sides should be retained.

Systematic elimination of all particles intercepted by the frame of the measurement field is necessary to measure particle measurement values. For the determination of the graphite fraction the edge correction should not be used.

Before carrying out an analysis, small sized particles should be eliminated from the assessment.

Graphite should be analysed using the following criteria:

— maximum Féret diameter, ≥ 5 μm;

— area A, ≥ 25 μm^2.

The method to deal with contiguous particles needs to be established and documented.

The method of elimination of non-graphite particles and porosity needs to be documented.

The number of measured fields should be chosen so as to have a minimum of 400 to 1 000 particles, depending on the number of nodules per square millimetre.

The measurement parameters of each graphite particle should include:

— surface area;

— maximum Féret diameter;

— perimeter.

The minimum sizes of the measurement parameters should be documented.

NOTE For identification purposes, it can be helpful for the particles to be colour-coded.

7 Test report

The test report should contain at least the following items:

— laboratory;

— date of testing;

— sample identification;

— cast iron grade;

— location of the surface tested;

— total surface area tested;

— surface area of the measuring field, in mm^2;

— magnification;

— minimum surface of graphite particles taken into account;

— number of particles and the number of particles per field;

— graphite content rate;

— number of particles per surface unit;

and, if necessary:

— table giving the percentage in number or in surface area of the particles, as a function of forms from I to VI;

— identity of the software programme used to carry out the analysis;

— method to separate joined/contiguous graphite particles;

— erosion and dilation to exclude undersized graphite particles;

— number and filtering characteristics of morphological operations;

— details of the presence of any other graphite from not covered by ISO 945-1;

— minimum size of graphite particles considered;

— method used to deal with graphite particles on border of field of view;

— method used to deal with contiguous graphite particles;

— method used to deal with non-graphite particles and porosity;

— graphite particle size using actual range or Reference Numbers from Table 1;

— average surface area of the particles, its range and its standard deviation;

— a histogram of the surface areas of the particles by form and/or size;

— photograph or image identifying the graphite particles using a colour coding to aid identification.

8 Acceptance procedure

8.1 General

The results of image analysis depend upon the software used. It is important that the procedure for acceptance of the image analysis technique compares the results of image analysis with the visual analysis method according to ISO 945-1. The procedure for acceptance of the visual analysis technique should be agreed between the manufacturer and the purchaser. The use of acceptance limits or confidence intervals (see 8.2) for determining conformance should be defined. It is recommended that use is made of acceptance limits as the criteria.

8.2 Proposed procedure for the comparison of the image analysis technique with the visual analysis technique given in ISO 945-1

The following procedure is recommended:

— minimum of 10 samples;

— minimum of 3 locations per sample;

— at least three metallographers should examine the samples;

— samples to be a mixture of normal production samples covering the expected range of results and at least two samples should include out-of-specification results;

— for each test sample the test should be performed in the same location using the same microscope settings (this is to minimize variation of the result depending on the location of the test);

— metallographers should undertake tests independently to prevent influencing test results;

— metallographers should classify the graphite morphology using both the visual and image analysis techniques for determining the form and size of the graphite particles;

— determine whether comparisons are to be made by using a set acceptability level or a confidence level;

— if required, calculate average results for the visual method and determine conformance. Review and investigate any non-conformances and re-determine conformance;

— analysis should include the determination of conformance to the visual method by the metallographer.

NOTE For proficiency testing methods and statistical analysis techniques, see ISO 13528 and ISO/IEC 17043.

Annex A
(informative)

Typical graphite forms in cast iron materials

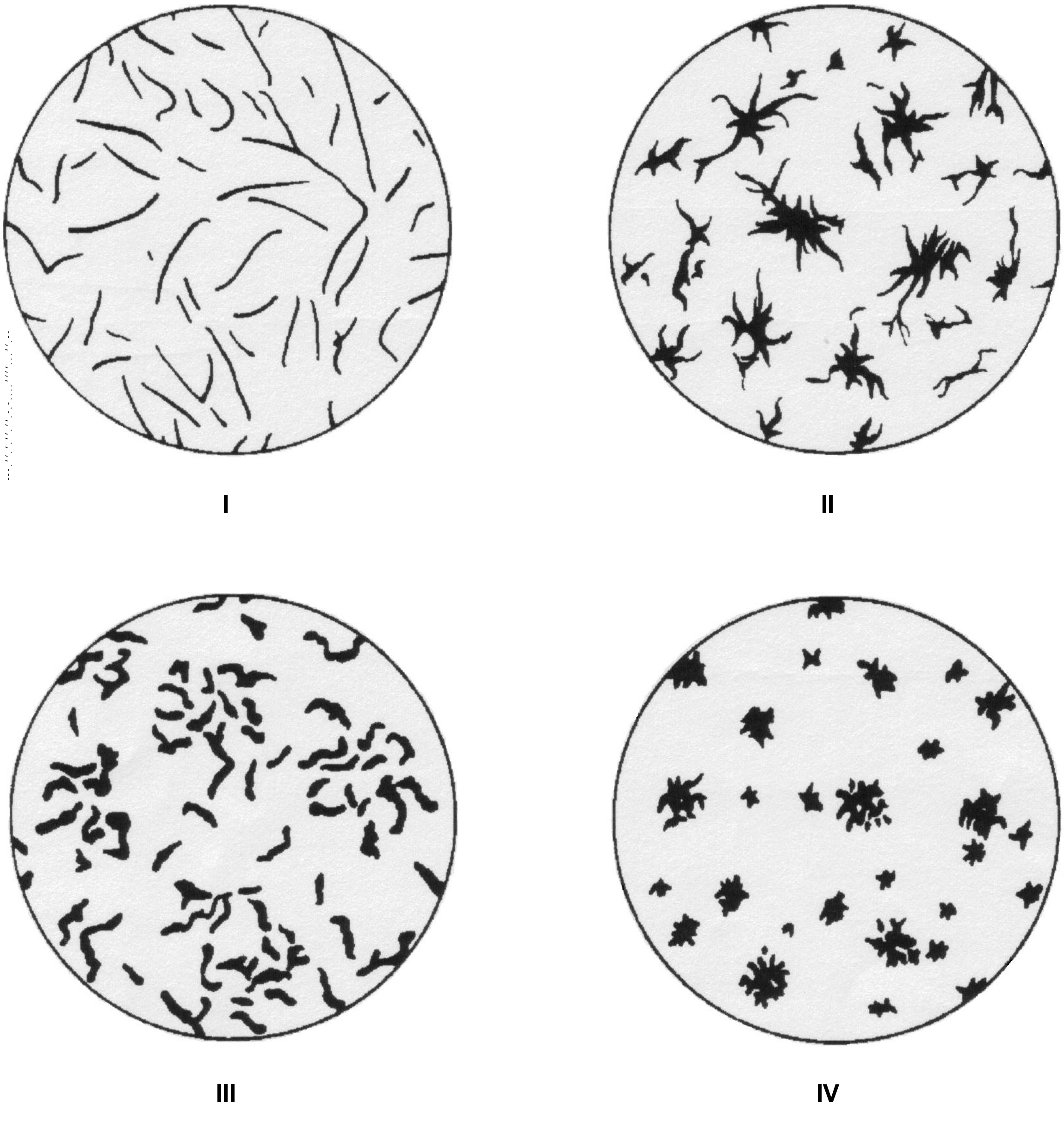
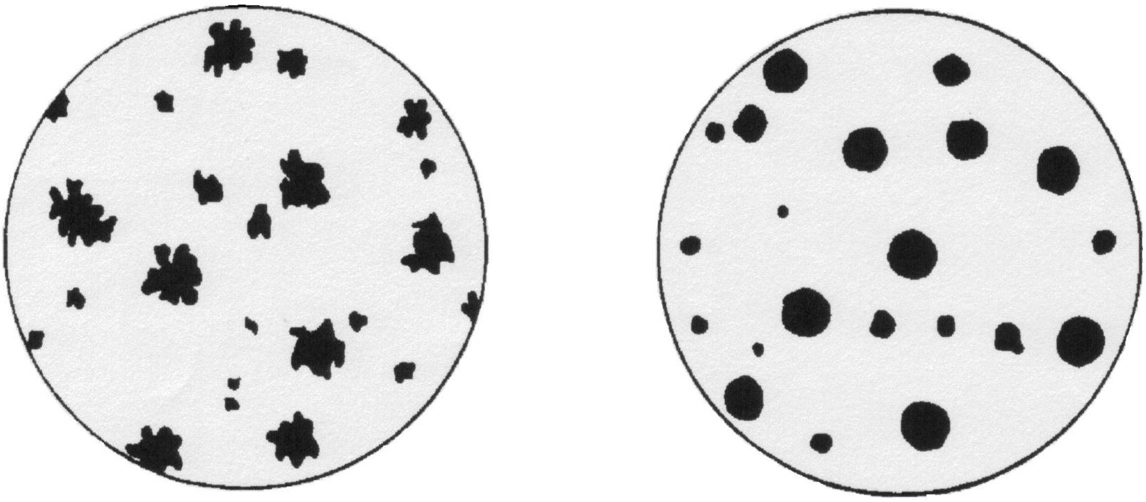


Figure A.1 (*continued*)



V VI

Figure A.1 — Principal graphite forms in cast irons — Reference images

Annex B
(informative)

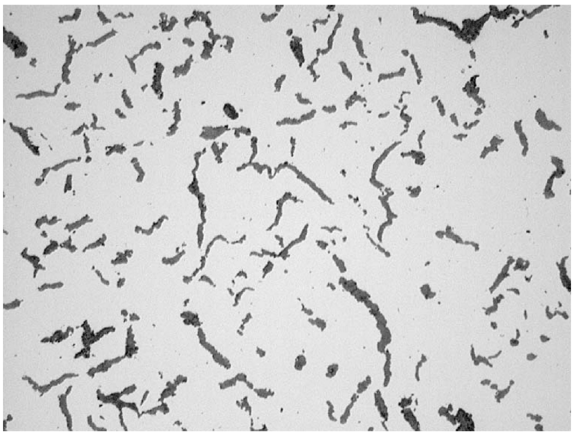
Typical graphite forms in cast iron materials (Examples of photomicrographs)

The following photomicrographs are two-dimensional representations of three-dimensional forms and therefore care has to be taken in assessing their true form.

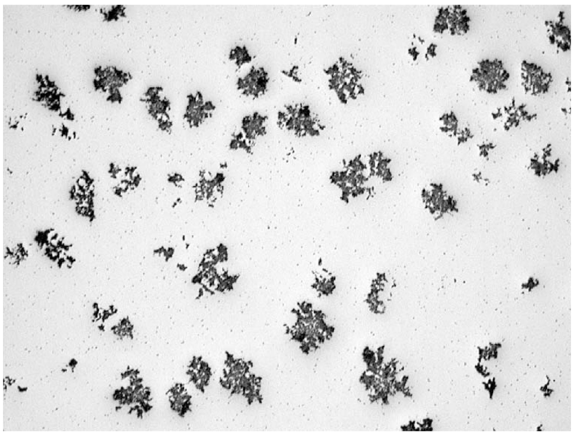
Form I

Form II,
represented by reference image of Figure A.1,
is never encountered alone in cast irons
(see Annex C of ISO 945-1)

Form II

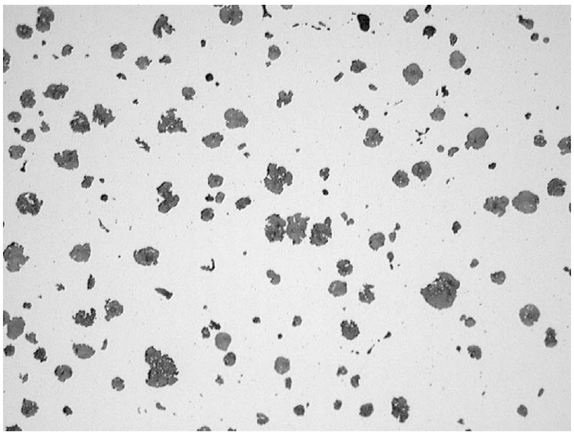


Form III

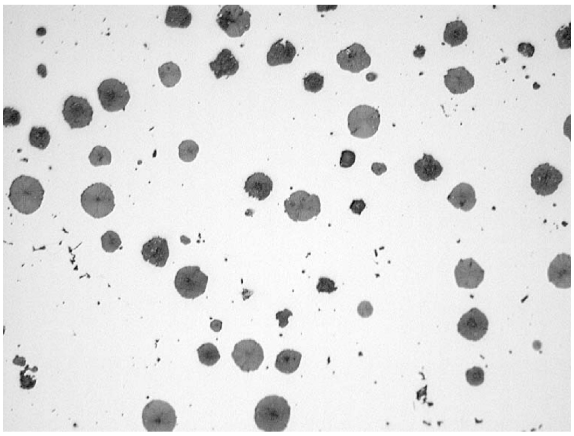


Form IV

Figure B.1 (*continued*)



Form V



Form VI

Figure B.1 — Examples of photomicrographs

Bibliography

[1] ISO 945-1, *Microstructure of cast irons — Part 1: Graphite classification by visual analysis*

[2] ISO 13528, *Statistical methods for use in proficiency testing by interlaboratory comparisons*

[3] ISO 16112, *Compacted (vermicular) graphite cast irons — Classification*

[4] ISO/IEC 17043, *Conformity assessment — General requirements for proficiency testing*

[5] J. Fargues, M. Hecht, M. Stucky, Characterizing the form of graphite in cast irons using an image analyser, Physical metallurgy of cast iron, Advanced Materials Research, vol. 4-5 (1997), 427-432

[6] NF A 04 197, *Spheroidal graphite cast iron casting-Characterization of the shape of the graphite by image analysis*, 2004

[7] F. Mampaey, Image Analysis of Graphite Particles by a Mathematical Description of the particle contour, AFS Transactions, vol. 113, paper no 05-155, 2005

[8] A. Velichko, F Mücklich, Bildanalytische Formanalyse und Klassifizierung der irregulären Graphitmorphologie in Gusseisen, Practical Metallography 43, 2006, 192-207

[9] A. Velichko, C. Holzapfel, F. Mücklich, 3D characterization of graphite morphologies in cast iron, Advanced Engineering Materials, 2007, 9, N° 1-2, 39-45

[10] A. Velichko, K. Schladitz, C Holzapel, F. Mücklich, 3D-Charakterisierung der Graphitmorphologie mit Hilfe der FIB-Nanotomographie, Prakt. Met. Sonderband 38 (2006), 169-176

[11] K. Breitkreutz, W. Kleinkröger, N. Bürger, Zur Revision der Norm DIN EN ISO 945 für die Gefügeanalyse von Gusseisen mit Kugelgraphit, Bedingen und Lösungen, Quality Engineering, 10, 2006, 52-54

ICS 77.080.10

Price based on 14 pages