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Method for evaluating the nodularity of spheroidal carbides — Steels for cold heading and cold extruding

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Foreword

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This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 4, *Heat treatable and alloy steels.*

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

The nodularity of spheroidal carbide is an important characteristic of steel for cold heading or cold extruding. However, the degree of spheroidization is assessed with reference to an agreed series of standard images, which does not exist up-to-now as subject of an International standard. Thus, there has been a continuing debate between supply and demand for the determination of the degree of spheroidization.

This document specifies a test method for evaluating the degree of spheroidal carbide in CHQ wire (Cold Heading Quality wire).

Method for evaluating the nodularity of spheroidal carbides — Steels for cold heading and cold extruding

1 Scope

This document specifies a micrographic method based on comparison charts for determining the degree of spheroidisation of carbides after annealing of wire rod, wire or bars made of non-alloy and low alloy steels intended for cold heading and cold extrusion. The range of carbon content is up to 1,20 % C.

In addition, Annex A includes a method based on machine vision for routine measurements.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4954, *Steels for cold heading and cold extruding*

3 Terms and definitions

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

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

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

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

3.1
maximum Féret diameter
maximum length of an object whatever its orientation, as shown in Figure 1

3.2
roundness
area of the carbide particle divided by the area of the circle whose diameter is the maximum Féret diameter of the same carbide particle, calculated according to Formula (1):

$$\text{Roundness} = A/A_m = 4A/\pi{\cdot}l_m^2 \quad (1)$$

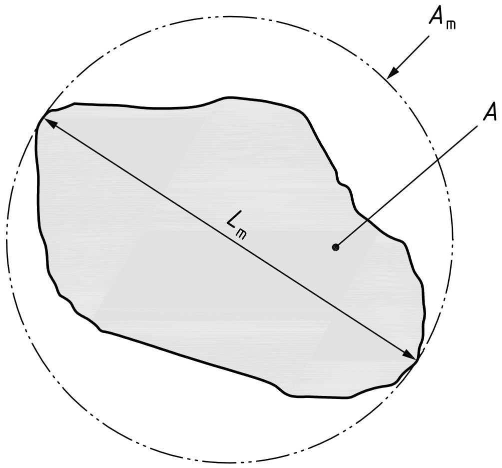


Figure 1 — Illustration of maximum Féret diameter of a carbide particle

where

l_m is the maximum Féret diameter of the carbide particle;

A_m is the area of the circle diameter *l*m;

A is the area of the carbide particle

3.3
area ratio
accepted/all objects area ratio and describes the relative amount of round objects on the image, calculated according to Formula (2):

$$A\% = \frac{A_{\text{accepted}}}{\left(A_{\text{accepted}} + A_{\text{rejected}}\right)} \quad (2)$$

Note 1 to entry: Round (spheroidized) objects on real images are shown in Figure 2 in green colour (bright) whereas rejected (non-round) objects are drawn in red colour (dark). From class 70 to class 90 the area ratio is increased, so 90 class images contain relatively more round particles.

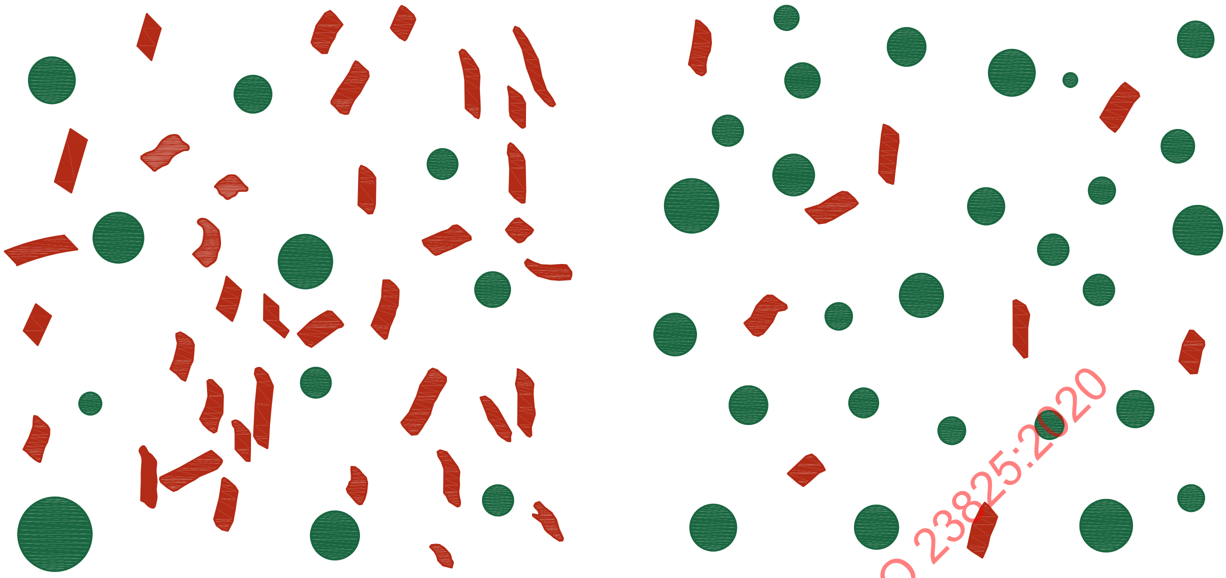


Figure 2 — The ratio of accepted objects to rejected objects on (a) is less than on (b).

3.4
area mean and area standard deviation
objects mean area (A) with its standard deviation (σ_A) describe the particle distribution (see Figure 3) and when considering the particle size from class 70 % to class 90 %, the frequency of inclusions of large particles increases

3.5
spheroidite
characteristic soft microstructure consisting of sphere-like globular cementite particles within a ferrite matrix

Note 1 to entry: see ISO 4885, 3.190.

Note 2 to entry: Carbide particles classified as globular carbide with roundness ≥0,75 to 1,00.

3.6
spheroidizing
annealing just below the A1 temperature of steels with long soaking time to bring the carbides in the form of spheroids

Note 1 to entry: see ISO 4885, 3.191

3.7
nodularity
assessment of the proportion of spheroidal carbide particles in a CHQ Wire (Cold Heading Quality wire) sample, generally expressed as a percentage

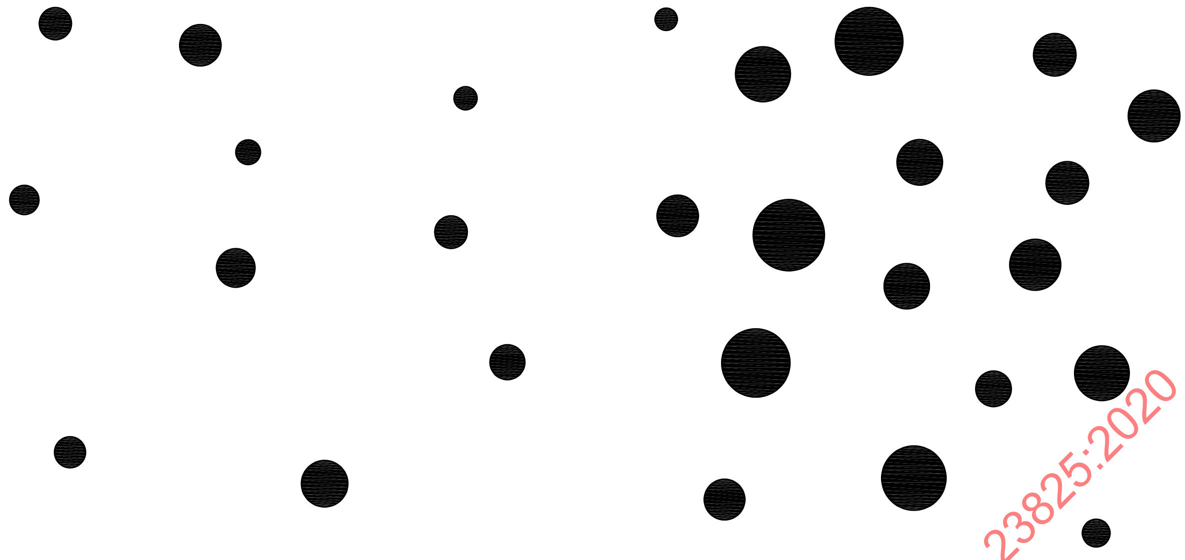


Figure 3 — Round objects area distribution for two different classes

4 Sampling and sample preparation

Sampling and sample preparation are conducted according to ISO 4954. Sampling shall be carried out in such a way that the samples are characteristic of the respective delivery.

The microsection per test piece to be examined shall have a size of 100 mm^2, if possible and practicable. Its position in the cross or longitudinal microsection can differ depending on requirements and should be left to the discretion of the manufacturer.

The test pieces shall be ground and polished. They should be etched in a suitable way, normally with a 2 % Picric acid solution (2 % $C_6H_3N_3O_7$ in C_2H_5OH).

Attention should be paid to a good metallographic preparation to ensure that the microstructure is not altered.

5 Evaluation

5.1 General

To characterize the carbide particles observed, information is generally necessary on the nodularity, the roundness and the distribution of the carbide particles.

5.2 Evaluation methods

This document characterises carbide particles by series of standard images. For routine measurements other methods can be preferred, especially examination by automatized image processing. An example for the automatized image processing is given in Annex A.

In case of dispute, however, the reference method for the characterisation of carbide particles is done by a comparision of images of a test piece under a microscop with a series of standard images.

6 Characterization of carbide particles by means of microscopic examination

6.1 Groups of steel grades and levels of spheroidisation

The volume of carbides depends principally on the carbon content. Other alloying elements of cold heading steel grades have minor influence. The steel grades are classified as presented in Table 1.

Table 1 — Groups of steel grades

Groups of steel grades	Carbon content	
1		≤0,10
2	>0,10	≤0,25
3	>0,25	≤0,40
4	>0,40	≤0,80
5	>0,80	≤1,20

The spheroidisation is characterised as percentage. Partial spheroidisation is for instance ≥70 % and optimal spheroidisation is for instance between 90 % and 100 %. For the spheroidisation four different levels are defined as shown in Table 2.

Table 2 — Different levels of spheroidisation

Abbreviation for the level	Level of Spheroidisation	Characteristic	Microstructure level (Example of heat treatement)
0	S around 0 %	initial structure	not soft annealed/not annealed on spheroidized carbides
70	70 % ≤ S < 80 %	partial globularisation	Spheroidisation of more than 70 % ((S)AIP)
80	80 % ≤ S < 90 %	satisfactory globularisation	Spheroidisation of more than 80 % (SAIP)
90	S ≥ 90 %	optimal spheroidite structure	Spheroidisation of more than 90 % (best possible structure of steel after soft annealed/annealed on spheroidized carbides, PSASAIP)

6.2 Series of diagrams

This image series characterizes the microstructure of a test piece by steel groups and the spheroidisation of carbides. The five steel groups represent different carbon contents.

Table 3 shows the microscopic image series with different levels of perlite and globular carbides for steel grades with four different typical carbon contents. Steels with carbon content ≤ 0,1 % C are not shown in the diagrams, because they are mainly ferritic.

Table 3 — Standard images of spheroidal structures of carbides for steels for cold heading and cold extruding

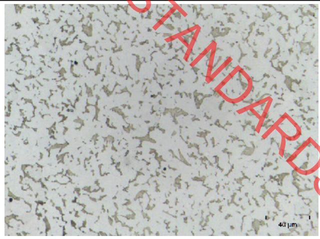
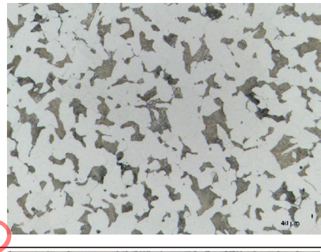
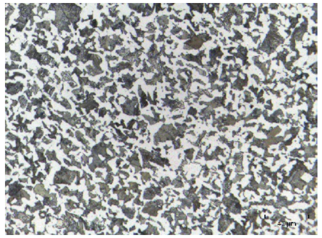
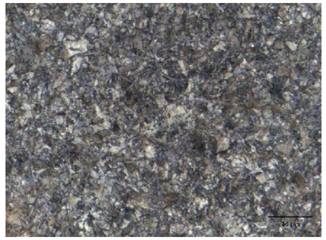
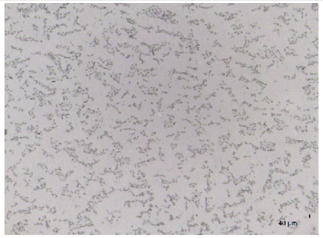
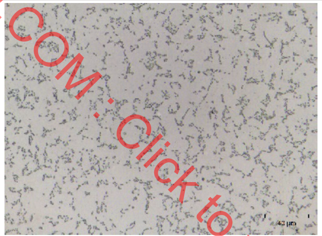
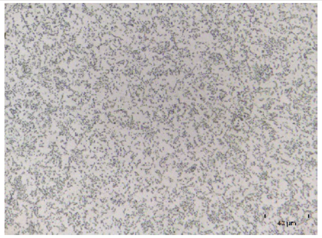
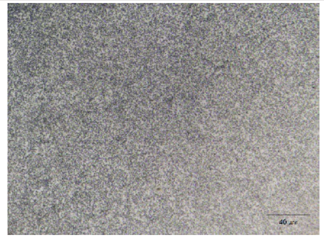
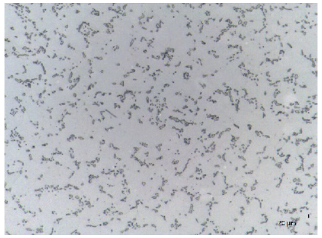
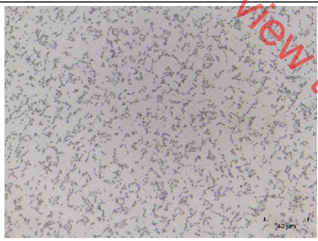
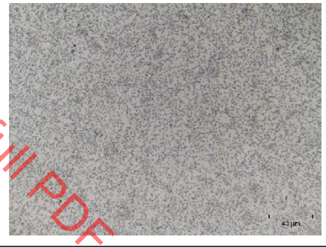
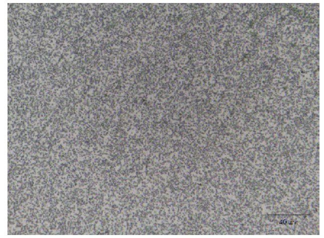
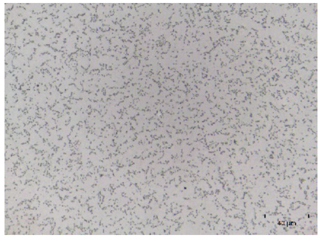
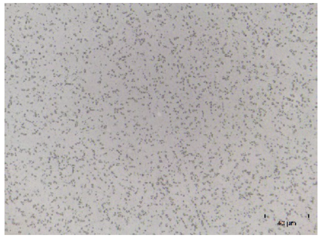
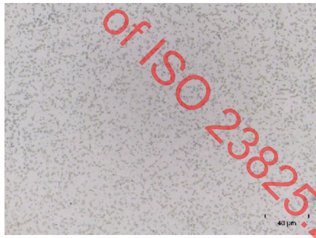
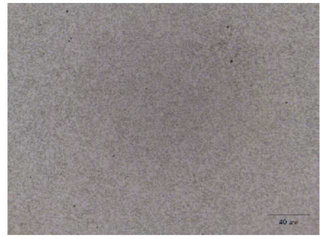
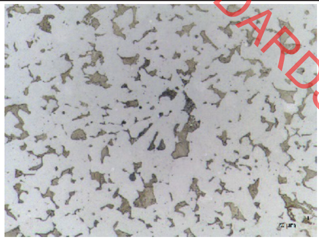
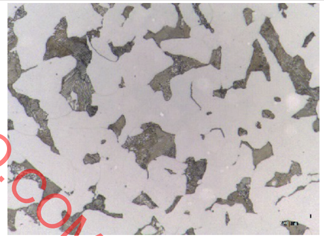
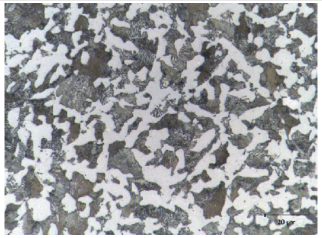
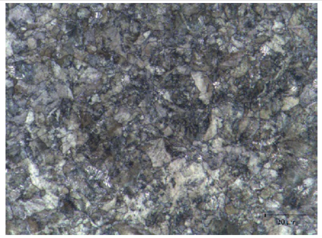
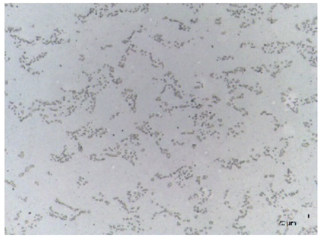
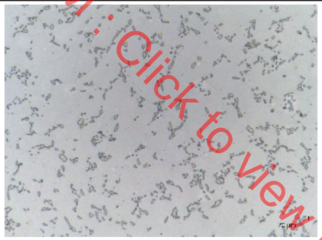
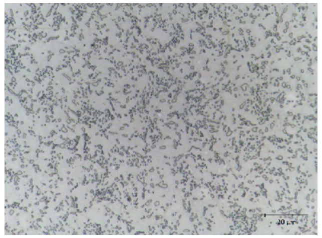
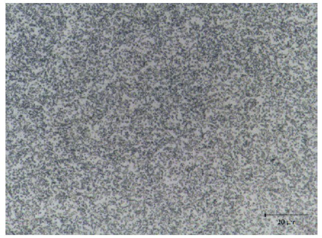
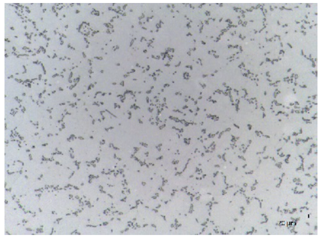
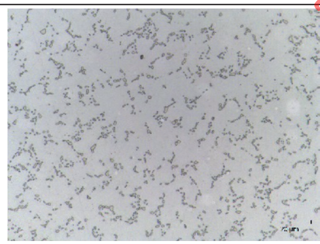
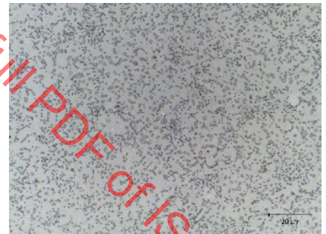
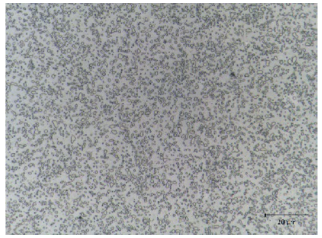
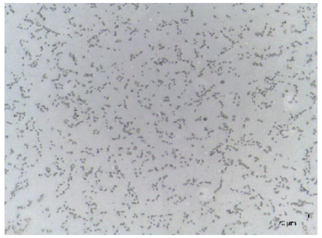
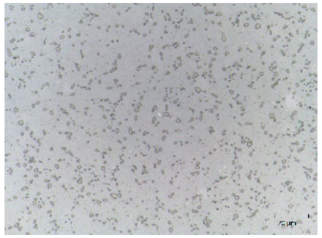
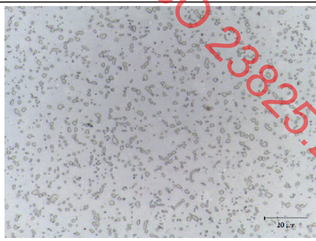
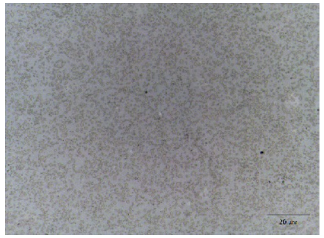
a) Magnification 500:1				
	Steels with 0,10 %< C ≤ 025 % steel group 2	Steels with 0,25 % < C ≤ 0,40 % steel group 3	Steels with 0,40 % < C ≤ 0,80 % steel group 4	Steels with 0,80 % < C ≤ 1,20 % steel group 5
lamellar S around 0 %				
spheroidite S ≥ 70 %				
spheroidite S ≥ 80 %				
Fully spheroidite S ≥ 90 %				

Table 3 *(continued)*

b) Magnification 1000:1				
	Steels with 0,10 %< C ≤ 025 % steel group 2	Steels with 0,25 % < C ≤ 0,40 % steel group 3	Steels with 0,40 % < C ≤ 0,80 % steel group 4	Steels with 0,80 % < C ≤ 1,20 % steel group 5
lamellar S around 0 %				
spheroidite S ≥ 70 %				
spheroidite S ≥ 80 %				
Fully spheroidite S ≥ 90 %				

With both information on the steel group and the level of spheroidisation a code will be established with the first number of the steel group and after a dot a further number for the level of spheroidisation. For a steel grade C45EC and for the image in Table 1 with C > 0,40 % and a spheroidisation level of around 90 % the code will be C45EC - 4.90.

NOTE The real carbon content according to the cast analysis and the apparent carbon content according to Table 1 can be different.

6.3 Procedure for carbide classification

6.3.1 General

Adjust the microscope magnification to match as closely as possible the corresponding scale of the standard images in Table 3. The observation can be conducted either in the eyepiece or the micrograph projected to a ground glass screen. The individual area to be observed should have the same size as the respective comparative areas (about 80 mm diameter). It is appropriate to limit the field of vision to this size by a pitch circle in the eye or on the screen.

The allocation is made by visual comparison between the image of the etched structure of the test piece to be examined in the light microscope and the images of the standard image series. It is necessary first to determine the micrograph of the series which resembles most the observed structure with regard to the respective characteristic. In each case the image should be selected, whose example is most similar to the real microscopic image.

Attention should be drawn to well spheroidised cementite, which is of major importance, but the distribution is not always uniform which is of minor importance. Ununiformity happens when the spheroidizing annealing is made on a former lamellar perlite structure on a matrix of ferrite and the temperature/time are not sufficient to move the carbon from the perlite grains to the ferrite grains. For example, the spheroidization is almost the same, but sometimes some grains are 100 % ferrite, while the cementite remaines in the former lamellar perlitic grains.

Non-round cementite with a ration of b/a of >5 will not be considered if there are only small amounts.

If spheroidisation based on this standard is agreed at the time of enquiry and order, but not the level of spheroidisation, a spheroidisation level of ≥70 % should be assumed.

6.3.2 Procedure

The polished samples shall be scanned under a microscope in such a manner that a representative area is examined. The sample shall first be targeted with 100× magnification to find the location with the lowest spheroidisation and then be analysed. To examine the carbide form and distribution, a ×500 magnification should preferably be chosen.

For a good quantification of the spheroidisation structure it is necessary to record at least five further homogeneously distributed view fields per cut. The mean value of at least five measured view fields is calculated.

If there are two images in the same steel group, e.g. from steel group 4 (C>0,70 %), the levels 1 and 3 (view fields with an spheroidisation of about 70 %, but also about the same number of visual fields with at least 90 % indentation) this results in an average value of 80, means a code of 4,80.

7 Test report

The test report shall contain the following information:

— the designation of the material and the heat treatment condition;

— the identification of the cast;

- the sampling location and the orientation of the test piece;
- the code concerning the steel group, the average level and the minimal level of spheroidisation of the carbide particles;
- the number of tested fields;
- the magnification;
- the name of image analysis software (if analysis was done by automatised image processing)
- date of the test and signature of the person who conducted the tests;
- the name and address of the testing laboratory;
- the name and address of the organization requesting the test;
- any deviation from this test method.

Annex A
(informative)

Automatised image processing for determining the level of carbide spheroidisation

A.1 Automatised image processing for determining the level of carbide spheroidisation

This test method is an evaluation method for measuring quantitative data by carbide spheroidization treatment.

An overview on the examples for the steel types, their carbon range and their levels of spheroidisation is given in Table A.1.

Table A.1 — Steel types, range of carbon and level of spheroidising

Steel type	Carbon content %	Steel group	Level of Spheroidisation %
C18C(SWRCH18A)	0,15 to 0,20	2 (0,10 to 0,25)	as rolled (0)
			70
			80
			90
C25GC(SWRCH25K)	0,16 to 0,27	3 (0,25 to 0,40)	as rolled (0)
			70
			80
			90
C45GC(SWRCH45K)	0,30 to 0,50	4 (0,40 to 0,80)	as rolled (0)
			70
			80
			90

A.2 Procedure for carbide classification — quantitave measurement method

The nodularity classification of the reference image is set in the following way. An important discriminatory feature is the information about the disorder and the neighborhood relationships of the particles. The spatial distribution of particles can be characterized and quantified using a geometrical model namely Delaunay's graph as mentioned in Bertram M.(*). Delaunay triangulation was made through gravity centers of round objects. The mean length of the sides of Delaunay triangles(L) and its standard deviation(σ_L) are used for image classification. Delaunay triangulation for two different classes are shown in Figure A.1.

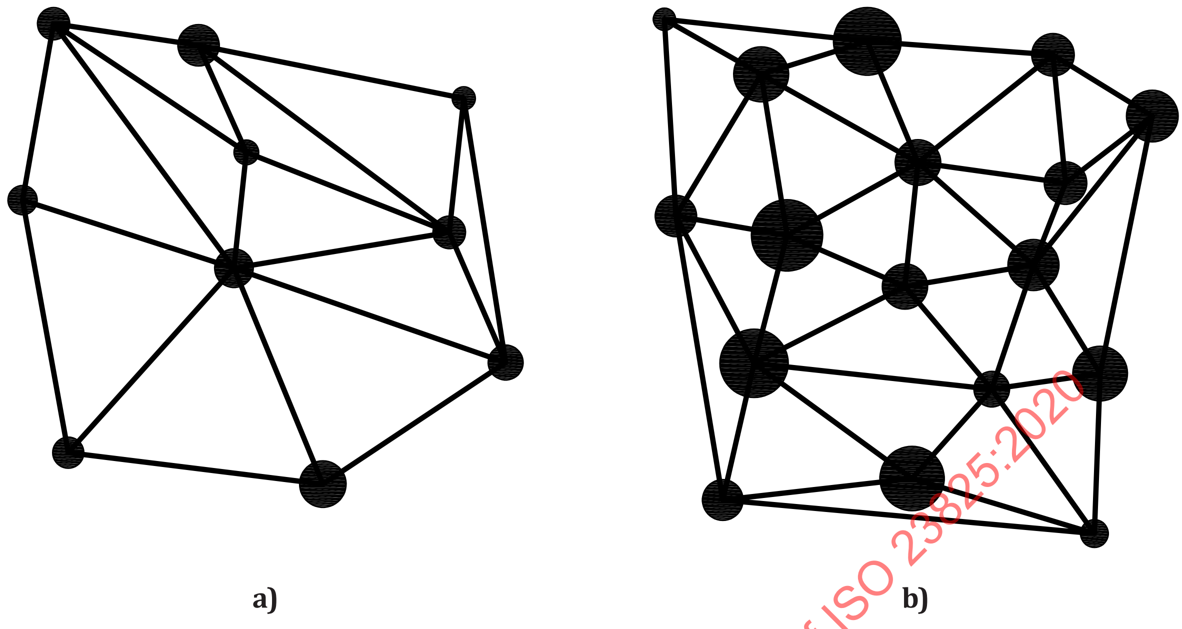


Figure A.1 — More spaces between particles on the (a), whereas particles are more compactly distributed on the (b)

For each input the reference image is calculated according to the Formula (3).

$$f = \left[A\%, \bar{A}, \sigma_A, \bar{L}, \sigma_L \right] \tag{3}$$

The parameters DATA of the reference images calculation in case roundness parameter given as 0.75 ~ 1.0 are shown in from Table A.2 to A.4 in Annex A. For nodularity classification of steel grade, 5 reference images were used for one class. For each cluster, it's mean center ($\bar{f}$) and calculated reference points about each nodularity classification of steel grade.

$$\bar{f} = \left[\mu_1, \mu_2, \mu_3, \mu_4, \mu_5 \right] \tag{4}$$

When the degree of spheroidization of the steel to be tested is given, it is measured for 5 times repeatedly about five parameters like as follows(x). Like the reference image, the roundness parameter is 0.75 to 1.0. For each cluster the nodularity to be measured is expressed as the mean value ($\bar{x}$).

$$x = [\ A\ \%,\ \bar{A},\ \sigma_A,\ \bar{L},\ \sigma_L\ ] \tag{5}$$

Covariance (dispersion) matrix (S)are calculated.

$$S = \begin{bmatrix} \sigma_1^2 & \cdots & \sigma_{15} \\ \vdots & \ddots & \vdots \\ \sigma_{51} & \cdots & \sigma_5^2 \end{bmatrix} \tag{6}$$

The Mahalanobis distance from the observation feature set to reference point ($\bar{f}$) and mean values ($\bar{x}$) of five parameters measured for the nodularity can be calculated by Formula (7).

$$D = \sqrt{\left(\bar{x} - \bar{f}\right)^T S^{-1} \left(\bar{x} - \bar{f}\right)} \tag{7}$$

T is the transformation matrix of $\left(\bar{x} - \bar{f}\right)$

Then the image classification can be done by finding the minimum Mahalanobis distance from the observed image features set to clusters correspondent to each class.

A.3 Characterization of carbide particles

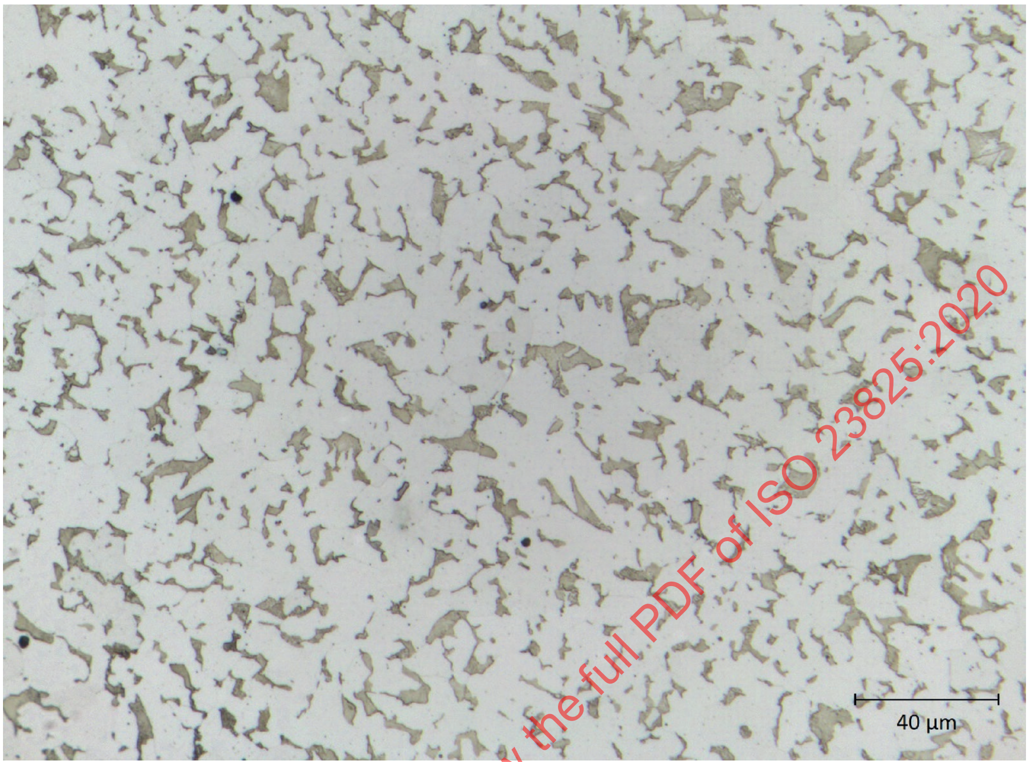
To characterize the carbide particles observed, information is generally necessary on the nodularity, the roundness and the distribution of the carbide particles. For this purpose, the reference images according to the type of steel are as follows.

a) Nodularity is designated by the degree of spheroidization for the C18C(SWRCH18A); see reference images of Figure A.2 in Annex A. The parameters DATA of the reference images are shown in the Table A.2.

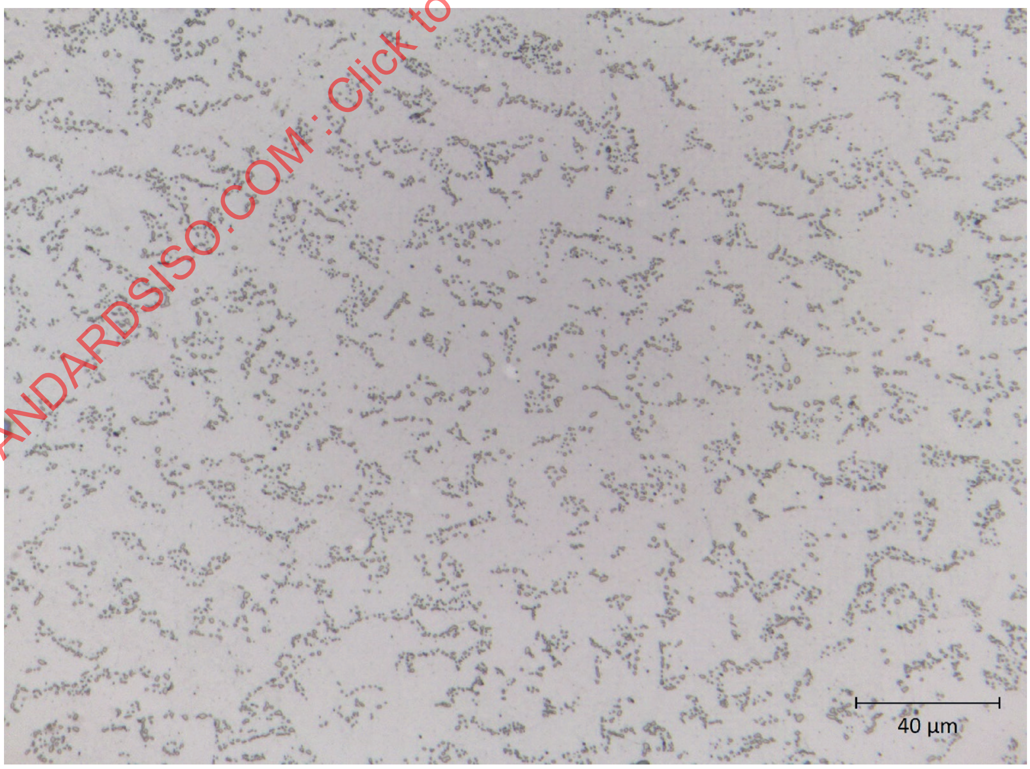
b) In the same manner, the reference images of the C25GC(SWRCH25K) are shown in Figure A.3 in Annex A. The parameters DATA of the reference images are shown in the Table A.3.

c) Respectively, the reference images and the parameters of the C45GC(SWRCH45K) are shown in Figure A.4 and Table A.4.

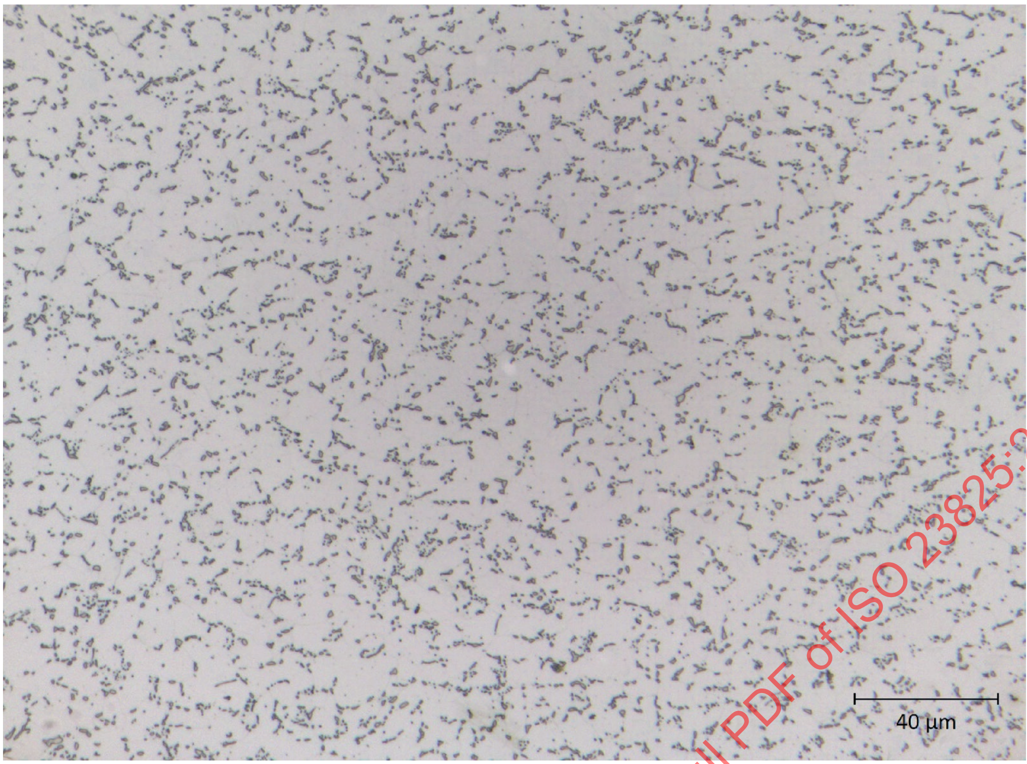
Reference images and table for C18C (SWRCH18A)



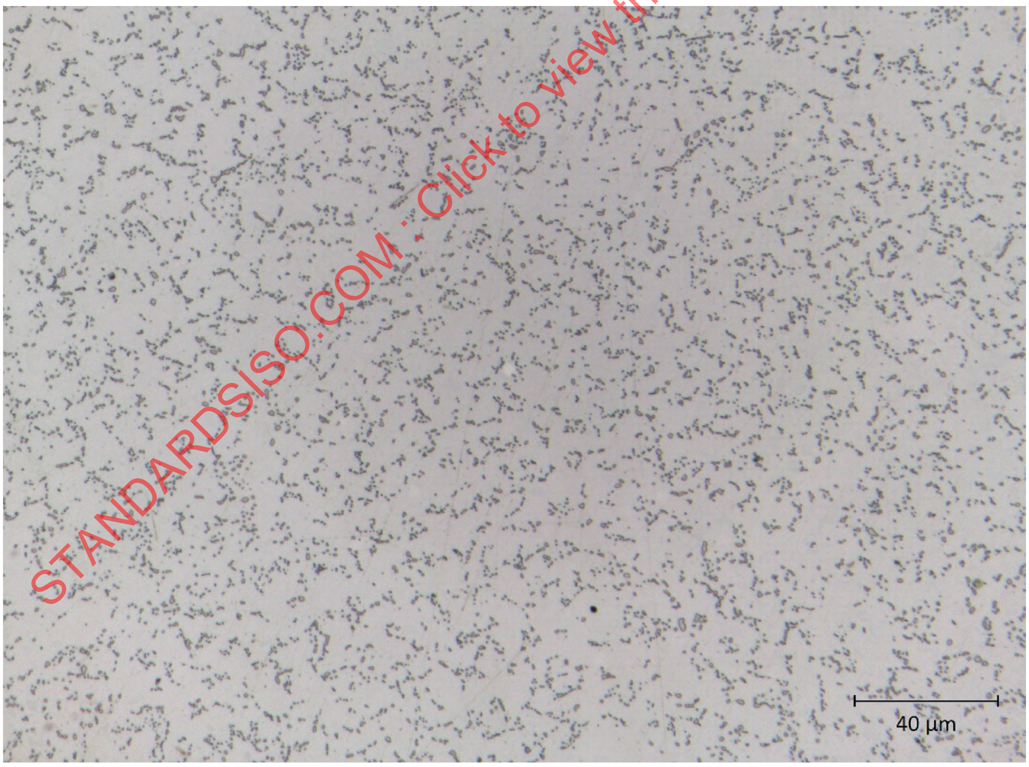
(a) As rolled



(b) Nodularity 70 %



(c) Nodularity 80 %



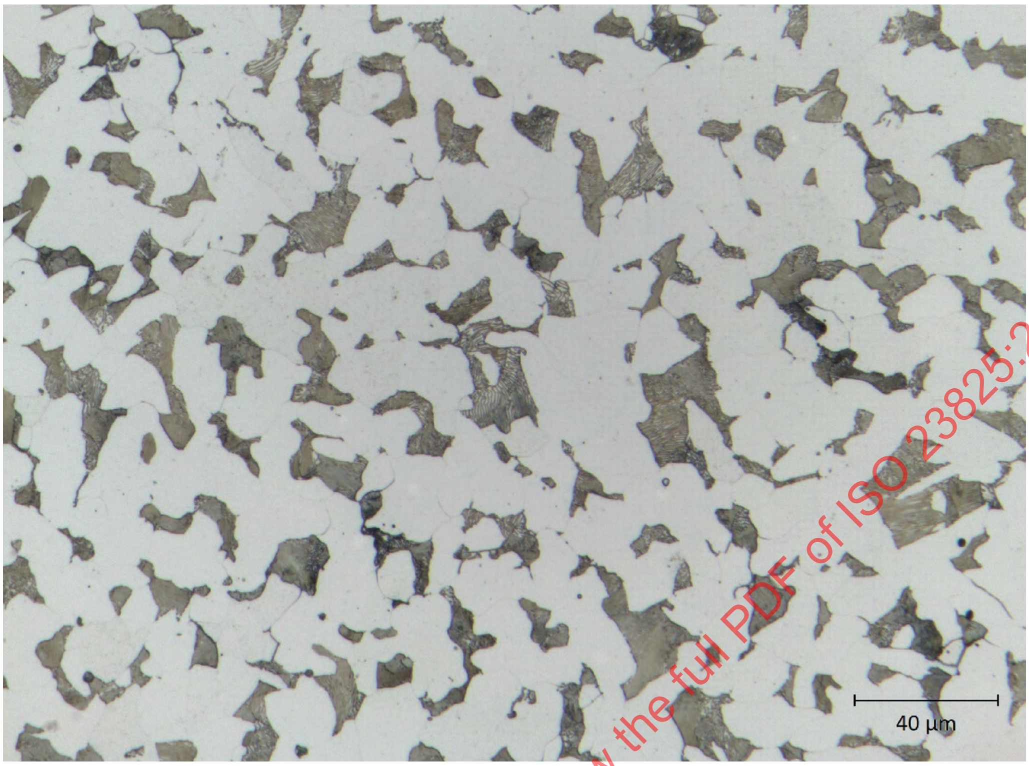
(d) Nodularity 90 %

Figure A.2 — Reference images for the nodularity of C18C (SWRCH18A)

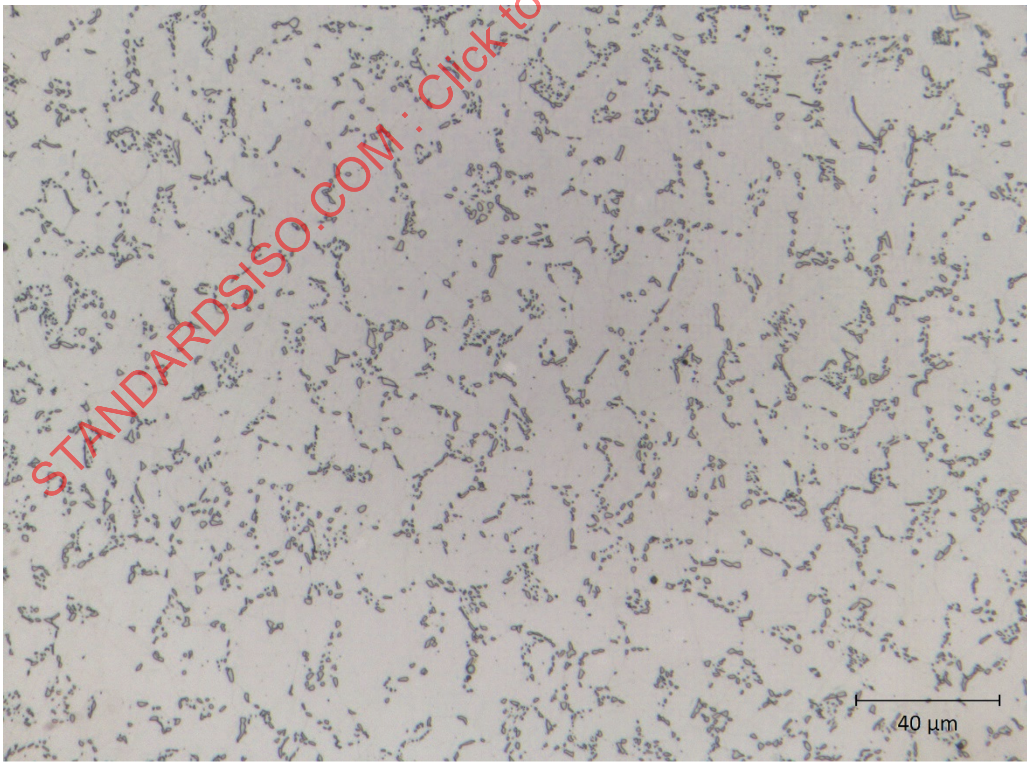
Table A.2 — The parameters DATA of the reference images for the C18C (SWRCH18A)

Class (%)	$A\%$	$\bar{A}$	σ_A	$\bar{L}$	σ_L
70	0,10	36,15	30,44	291,36	969,22
	0,09	39,52	34,26	316,74	994,05
	0,08	36,92	28,34	350,54	1082,63
	0,08	38,50	26,85	332,54	1036,05
	0,11	36,71	29,41	246,87	859,54
80	0,21	33,94	21,13	125,48	609,62
	0,22	32,97	20,19	132,20	625,90
	0,23	33,36	21,59	128,48	615,62
	0,21	33,11	21,65	123,09	580,73
	0,21	32,67	22,89	126,88	604,55
90	0,31	29,68	18,17	126,19	611,45
	0,29	28,51	16,66	124,34	596,96
	0,28	28,95	14,95	124,08	602,18
	0,31	29,70	18,89	113,48	579,37
	0,29	29,78	18,66	126,05	595,14

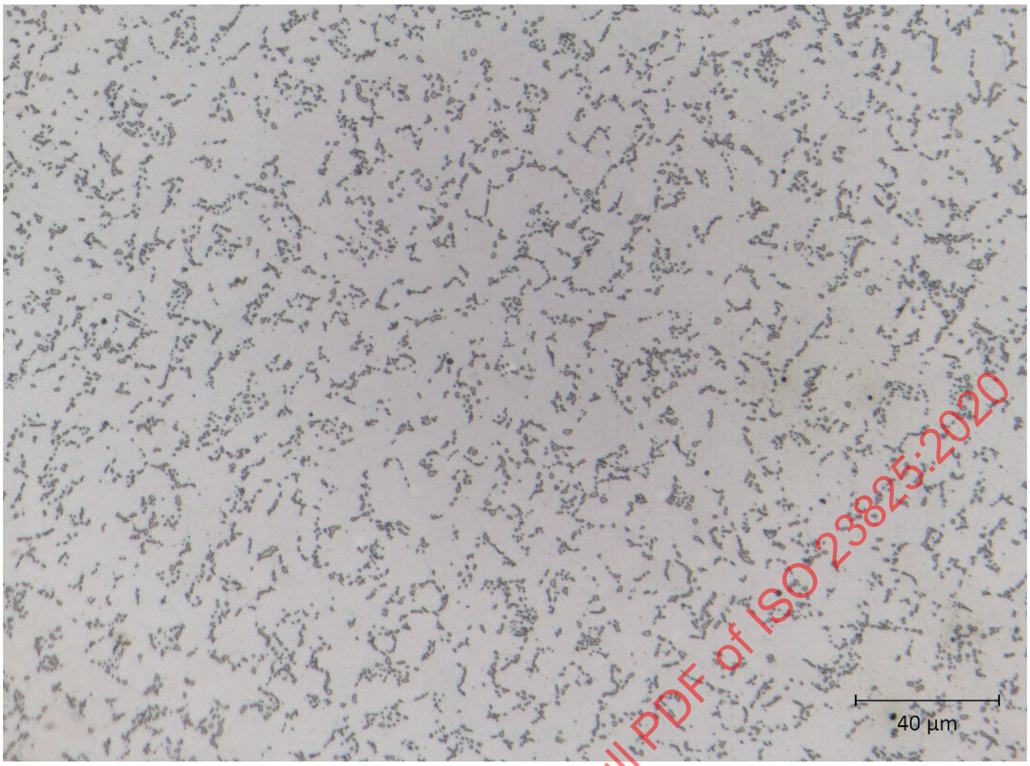
Reference images and table for C25GC (SWRCH25K)



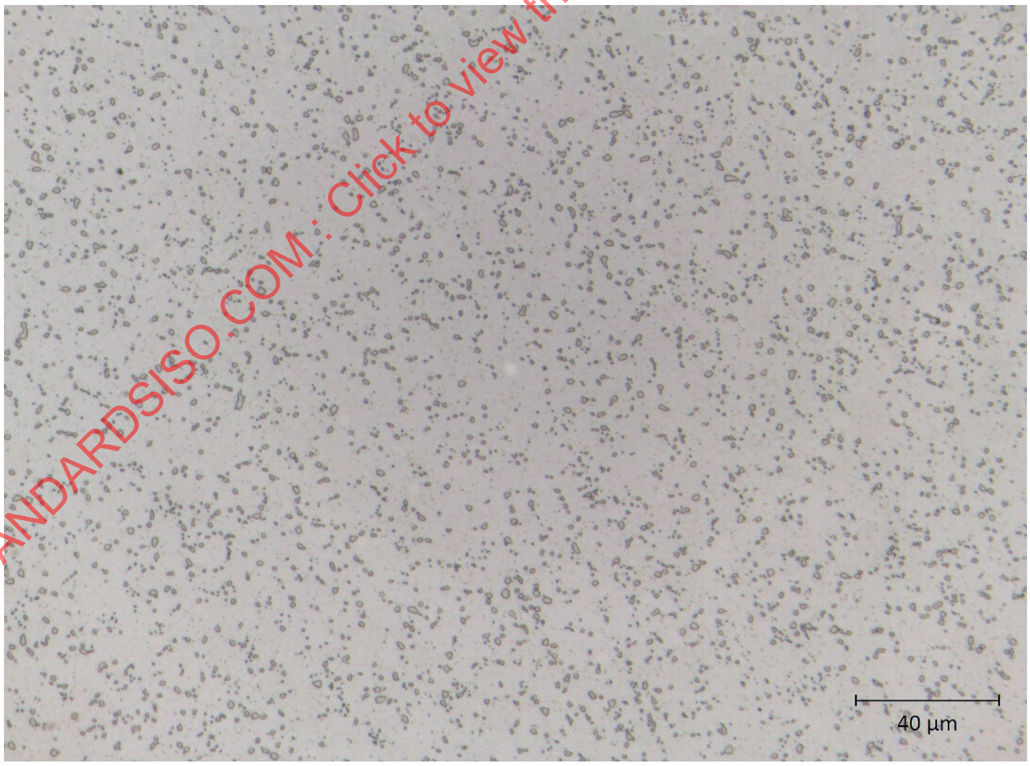
(a) As rolled



(b) Nodularity 70 %



(c) Nodularity 80 %



(d Nodularity 90 %

Figure A.3 — Reference images for the nodularity of C25GC (SWRCH25K)

Table A.3 — The parameters DATA of the reference images for the C25GC (SWRCH25K)

Class (%)	$A\%$	$\bar{A}$	σ_A	$\bar{L}$	σ_L
70	0,10	32,21	26,86	155,36	684,51
	0,11	32,38	20,96	146,37	665,31
	0,11	31,78	21,23	143,86	646,83
	0,12	31,34	21,49	132,23	604,07
	0,11	31,79	17,88	152,67	682,42
80	0,14	31,25	20,06	126,00	600,97
	0,11	29,74	18,39	145,76	645,08
	0,12	29,14	17,96	143,69	662,41
	0,13	31,81	24,58	134,59	612,38
	0,14	30,79	22,29	126,06	601,35
90	0,25	34,46	21,02	100,31	526,47
	0,23	32,73	17,52	109,59	562,95
	0,22	31,88	16,88	96,030	488,92
	0,22	32,75	18,33	111,82	569,06
	0,25	34,01	17,95	113,08	592,30