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Micrographic examination of the non-metallic inclusion content of steels using standard pictures

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Metallographische Prüfung des Gehaltes nichtmetallischer Einschlüsse in Stählen mit Bildreihen

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Détermination micrographique de la teneur en inclusions non-métalliques des aciers a l'aide d'images-types

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English Version

Micrographic examination of the non-metallic inclusion content of steels using standard pictures

Détermination micrographique de la teneur en inclusions
non-métalliques des aciers à l'aide d'images-types

Metallographische Prüfung des Gehaltes nichtmetallischer
Einschlüsse in Stählen mit Bildreihen

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
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Foreword

This document (EN 10247:2007) has been prepared by Technical Committee ECISS/TC 2 “Steel - Physico-chemical and non-destructive testing”, the secretariat of which is held by AFNOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by October 2007, and conflicting national standards shall be withdrawn at the latest by October 2007.

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

This document supersedes ENV 10247:1998.

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Cyprus, Czech Republic, Denmark, Estonia, Finland, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland and United Kingdom.

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Introduction

This document establishes procedures for the assessment of inclusions in steels, based on their morphology using standard pictures.

These procedures include principles that are coherent with physical results obtained from inclusion measurements.

The results are in physical units: length in $\mu\text{m}/\text{mm}^2$, number/ mm^2 , areas in $\mu\text{m}^2/\text{mm}^2$. In comparison to other inclusion rating standards, in this standard the order of the classification begins with the length (row index q). These results can be transposed into other standard's ratings for comparison purposes.

The conditions of assessments, for instance the rules to scan fields on the specimen, are defined such that there is an optimization between magnification and the number of fields to be assessed. The same precision level is achieved by using the same method in manual evaluation and computer controlled measurements.

The chart of standard pictures is derived from mathematical principles.

The results and their precision may be directly computed from field assessments.

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1 Scope

This European Standard defines a method of microscopic non-metallic inclusion assessment using picture charts.

The method does not apply to particles of a length less than 3,0 µm or a width smaller than 2,0 µm. Defined by a product standard or agreement between the involved parties for certain special products, inclusions with a width below 2,0 µm can only be evaluated according to their length. Elongated inclusions with a length above 1 410 µm are counted separately and are beyond the upper application limit of this standard. Globular inclusions with a diameter of 3,0 µm and above are included in the assessment.

It is assumed, if particles are elongated or if there are stringers of particles, that they are parallel to each other. Other arrangements are not covered by this standard. This European Standard applies to samples with a microscopic precipitation approaching random distribution.

From the data of measurements obtained by this method, evaluation according to other standards can be established.

This European Standard does not apply to free cutting steels.

NOTE The basic principle of this European Standard allows the determination of non-metallic inclusion content by image analysis techniques.

2 Normative references

The following referenced documents are indispensable for the application 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.

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories* (ISO/IEC 17025:2005)

3 Principles

This method consists of a comparison between inclusions observed in a field of view with chart pictures. The chart pictures defined by this European Standard are based on the shape of inclusions and for each shape on length, width and area, for columns 1 to 10 and number for column 11.

This standard employs an ellipse as a basic shape with the circle as a special case of an ellipse (see Figure 1a). Inclusions with a shape like a rectangle or square are treated as ellipses or circles as their areas are not significantly different for the purpose of this method.

The pictures are arranged in rows and columns. The length changes from row to row, the shape factor changes from column to column. This standard is principally concerned with the morphology and arrangement of inclusions. It does not provide information relevant to crystal structure or chemical constitution of measured inclusions.

General practice usually requires a differentiation between inclusions of different chemical composition. The definition of the types should be defined by the product standards. Should no standard be available, then the definition of characteristic morphologies shall be, by agreement, between the involved parties.

To make description easier, a tree of specific terms is given in Annex A, Figure A.1.

The chart pictures represent the upper limits of classes. The length L_x is classified in row q if:

$$L_{q-1} < L_x \leq L_q \text{ µm} \quad (1)$$

The width w_x is classified in column k if:

$$w_{k-1} < w_x \leq w_k \quad \mu\text{m} \quad (2)$$

In Figure 5 the first row on the top without number and the first column on the left without number and a thicker surrounding contain the lower limiting pictures. Inclusions with a length shorter than that given in that row or a width smaller than that given in that column are not taken into account for classification.

Inclusions classified as columns 6 and 11 are called globular.

The parameters measured are number, length, width and area. The results of the evaluations can give expressions of worst inclusion, worst field or an average field value, all of which have physical dimensions. In addition to these values, this method gives an estimation of distribution of the inclusions within the test specimens.

The entire chart is mathematically based. It has a limited number of pictures, which limits choice and hence improves reproducibility when used in a manual method. The mathematical basis permits use by manual and image analysis methods providing potential for higher statistical precision. The data produced gives a wide range of features for cleanliness definition. The chart employs different shapes and magnifications allowing an application to cleaner steels where shape control is of interest.

This European Standard contains several different methods of evaluation. The choice of method shall be defined by the product standard or be agreed between the involved parties.

By default, the methods of evaluation used are the worst inclusion and the average field method; parameters are given in Annex B.

The methods P_L , P_d ; K_n , K_L and K_n , K_d are proposed as standard methods.

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4 Terms and definitions

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

4.1 General

4.1.1

particle

single precipitate, in general non-metallic

4.1.2

inclusion

general designation of particles in association, defined by the size and proximity of particles. It describes a single, separated particle as well as an arrangement of at least 2 particles (see Figure 2a) if the distance t is $\leq 10 \mu\text{m}$ and the distance e is $\leq 40 \mu\text{m}$ and the main axis of the particles are parallel within $\pm 10^\circ$. For an arrangement of only two globular particles, each particle shall be considered as an individual inclusion.

An inclusion can also be formed by several stringers if the distances t and e are $\leq 10 \mu\text{m}$ and $\leq 40 \mu\text{m}$ (see Figure 2b). Particles with $L < 3 \mu\text{m}$ or $w < 2 \mu\text{m}$ are not taken into account (see Figure 2c).

Special cases:

If elongated and more or less spherical particles are combined, see Figure 2d, in general it is treated as one inclusion. In case 4 the width of the largest particle is consulted for the width of the inclusion. If in this case $w_1 > 3 w_2$ then the particles w_1 and w_2 are treated separately. For an example see Figure 2e.

Some examples for inclusions are given in Figure 2f

4.1.3**stringer**

arrangement of at least 3 particles, normally aligned, forming an inclusion (see Figures 2b, 2f), For examples see Annex C and Figure 2f

4.1.4**test area**

area on the polished surface of the specimen to be evaluated

NOTE In general, the size of the test area is 200 mm².

4.2 Proximity**4.2.1****distances between particles**

distance e between the particles in the direction of main deformation and distance t in the direction perpendicular to it (see Figure 2a)

4.2.2**distance between stringers**

similar to that for the distance between particles (see Figure 2b)

4.2.3**scattered**

random arrangement of particles.

NOTE For example see Annex C. This is defined in one field of view

4.3 Parameters**4.3.1****length**

dimension of an inclusion in the main direction of deformation, always assumed to be greater than the width

4.3.2**diameter**

maximum dimension of inclusion classified according to column 6 (globular inclusion)

4.3.3**width**

Maximum width perpendicular to the direction of principal deformation. This is the width of the ellipse inscribed to the confining rectangle and having the same length as the inclusion.

For manual evaluation this value can only be estimated. The width is the maximum width perpendicular to the direction of principal deformation for inclusions with only one particle.

The width w of an inclusion with 2 particles is given by the largest particle (see Figure 2a).

Width of a stringer (see Figure 1b):

The width of a stringer is defined as the width of an ellipse inscribed to the confining rectangle and having the same length as the stringer

Width of an inclusion out of several stringers (see Figure 2b).

The distance t between two stringers is defined as the shortest distance between the confining rectangles in a transverse direction. The distance e is defined as the shortest distance between the confining rectangles in a machine direction.

Case a) for $0 \leq e \leq 40 \mu\text{m}$, $t \leq 10 \mu\text{m}$: is the width of an inclusion out of two stringers defined as the width of the widest stringer. ($w_{\text{total}} = w_1$, $w_1 > w_2$) (see Figure 2b, a)).

Case b) for $e < 0 \mu\text{m}$, $t \leq 10 \mu\text{m}$: is the width of an inclusion out of two stringers defined as the sum of the stringers' widths and the distance t ($w_{\text{total}} = w_1 + w_2 + t$) (see Figure 2b, b).

The width of an inclusion, which consists of several stringers, is determined by the width of the widest stringer. This is identified under consideration of the neighboring stringers corresponding to case a) and b), see Figure 2b, c)

4.3.4

area

area of the ellipse inscribed to the confining rectangle and having the same length as the inclusion (see 4.3.3 and Figures 1a, 1b)

4.3.5

shape factor

exponent f in the equation

$$\frac{L^2}{a} = \frac{4}{\pi} \left(\frac{L}{c} \right)^f \quad (3)$$

NOTE For details see Annex D

4.4 Classes

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4.4.1

elongated particles

particles with elliptical shape (see Figure 1a)

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4.4.2

globular particles

circular or rectangular particles classified as column 6

4.4.3

type

types of inclusions are separated according to their colour, shape and arrangement and not by chemical composition (see Annex A).

4.5 Others

4.5.1

lot

unit of material processed at one time and subject to similar processing variables

4.5.2

restricted values

values of the average field assessment restricted to inclusions greater than a defined length, shape factor or area

5 Symbols and abbreviations

Symbol	Unit	Designation
a	μm^2	area of inclusions
b		width of the plate
c	μm	factor, 1 μm
d	μm	diameter of inclusions
e	μm	interparticle distance (elongation axis)
f		shape factor
b		black coloured
g		grey coloured (as sulphides)
h		coloured (pink or yellow) (as nitrides)
i		inclusion index
j		field index
k		column number
m		type of inclusion index
\max		index of maximum value of n , L , w , d , a (in j or s)
n		number of assessed particles, inclusions
n_s		number of assessed inclusions per specimen
o		black coloured (as oxides)
p		particle index
q		row number
s		specimen index
t	μm	interparticle distance (transverse axis)
u	μm	scale unit in microscope eyepiece
v		width of polished surface
w	μm	width of inclusions
x		variable
$\text{av or } -$		average value of n , L , w , a

α		scattered, elongated inclusion type
α_g		scattered, elongated, grey coloured inclusion type
α_b		scattered, elongated, black coloured inclusion type
α_{gb}		scattered, elongated, grey / black coloured inclusion type
β		aligned, globular inclusion type
β_b		aligned, globular, black coloured inclusion type,
γ		aligned, elongated inclusion type
γ_b		aligned, elongated, black coloured inclusion type
γ_g		aligned, elongated, grey coloured inclusion type
δ		scattered, globular inclusion type
δ_b		scattered, globular, black coloured inclusion type
δ_g		scattered, globular grey coloured inclusion type
δ_{gb}		scattered, globular, grey / black coloured inclusion type
A	μm^2	area of field of view on the specimen
B		polished surface
D		diameter of product
MD		main direction of deformation (e. g. rolling direction)
E	mm	length of test area
G		magnification
H	μm	length of measuring frame on the specimen
I		length of an stage micrometer
K	- , μm , $\mu\text{m}^2/\text{mm}^2$	average field assessment
L	μm	length of inclusions
M	- , μm , $\mu\text{m}^2/\text{mm}^2$	worst field assessment
N_j		number of fields
N_s		number of specimens
P		worst inclusion assessment
Q		factor for K-assessment
R		restricted values

W mm width of test area (see Figure R.1)

Combined symbols can be written as index or on one line.

EXAMPLE K_L, KL average field assessment for length;

n_j, nj number of inclusions in a field;

$\overline{n_j}, \overline{nj}$ average number of inclusions per field.

6 Sampling

6.1 General

Unless otherwise specified in the technical delivery conditions, the following requirements apply.

6.2 Minimum reduction

The shape of the inclusions depends, to a large extent, on the degree of reduction of the steel. The chart can only be used if the shape of inclusions in the specimen can be compared with that given in the pictures of the chart.

NOTE It is recommended that products should have a minimum reduction by a factor of five. If the deformation is less than a factor of five, care should be taken to differentiate between porosity and inclusions, both of which may be present.

6.3 Size and location of test area

The polished surface of the specimen used to determine the content of inclusions shall be a minimum of 200 mm² with a minimum length greater than 20 mm and a minimum width greater than 10 mm (e. g. 25 mm × 20 mm). It should be possible within this area to define a rectangular test area of 200 mm² for evaluation with a length to width ratio of 2 (e. g. 20 mm × 10 mm). The longer side of the test area shall be parallel to the direction of the main deformation (e. g. rolling direction).

The sampling and the number of specimens shall be specified in the product standard or shall be subject to agreement between parties.

In the absence of an agreement, the sampling procedure shall be as follows, see Figure 3:

- bar or billets with a diameter above 50 mm: the test area shall be located halfway between the outer surface and the centre (see Figure 3a);
- bar with a diameter greater than 25 mm and less than or equal to 50 mm: the surface to be examined consists of half the diametral section (from the center to the edge of the specimen) (see Figure 3b);
- bar with a diameter less than or equal to 25 mm: the surface to be examined consists of the full diametral section of sufficient length to obtain a total surface of 200 mm² (see Figure 3c);
- plates with a thickness less than 25 mm: the specimen contains the whole thickness (see Figure 3d);
- plates with a thickness between 25 mm and 50 mm: the specimen contains half the thickness, position between surface and centre;
- plates with a thickness greater than 50 mm: the specimen contains one quarter of the thickness. The position is not defined.

The positions of the measuring planes for tubes are given in Figure 3e.