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

Metallographische Prüfung des Gehaltes nichtmetallischer Einschlüsse in Stählen mit Bildreihen

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

Metallographische Prüfung des Gehaltes
nichtmetallischer Einschlüsse in Stählen mit Bildreihen

This draft European Standard is submitted to CEN members for enquiry. It has been drawn up by the Technical Committee ECISS/TC 101.

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EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

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European foreword

This document (prEN 10247:2016) has been prepared by Technical Committee ECISS/TC 101 "Test methods for steel (other than chemical analysis)", the secretariat of which is held by AFNOR.

This document is currently submitted to the CEN Enquiry.

This document will supersede EN 10247:2007.

The many changes in the current revision result from only a few basic adjustments. The length to width limit ratio for globular inclusions has been changed from 1,3 to 3 (Annex I), and the mathematical principles underlying the chart have been more clearly defined (Annex H). These two changes have led to many numerical changes in Table 2 and Figure 11, where moreover some classes have been deleted and others added. The rules of assessment have changed, most notably to allow stringer formation from two particles upward (Subclause 3.1.2, Annex B), to exclude stringer formation between a stringer and a single particle (Subclause 3.1.2), and to consistently define the classification of inclusions by shape, arrangement, and colour (Clause 4, Annexes A and B). Finally, the assessment and recording sheets have been redesigned to simplify manual use (Annexes K, L, and M).

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 chart of standard pictures is derived from mathematical principles. In distinction to other inclusion rating standards, in this standard the order of the classification begins with the length (row index q).

The results may be directly computed from field assessments. The same precision level is achieved by using the same method in manual evaluation and computer controlled measurements.

The results are in physical units: length in $\mu m/mm^2$, number/mm^2, areas in $\mu m^2/mm^2$.

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1   Scope

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

The method does not apply to particles of a length or diameter less than 3,0 μm or a width smaller than 2,0 μm. If defined by a product standard or agreement between the involved parties for certain special products, inclusions with a width below 2,0 μm can be evaluated by length alone.Inclusions with dimensions exceeding the upper limits in Table 2 are evaluated as belonging to the maximum class and noted separately with their true dimensions (see 7.5.6).

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 draft standard. This draft 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 draft European Standard does not apply to free cutting steels.

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

2   Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. 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)*

ISO 9042, *Steels — Manual point counting method for statistically estimating the volume fraction of a constituent with a point grid*

3   Terms and definitions

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

3.1   General:

3.1.1
particle
single precipitate, in general non-metallic

3.1.2
stringer
arrangement of at least 2 particles, normally aligned, that meet the proximity conditions $e \leq 40$ μm and $t \leq 10$ μm (see Figure 4 for proximity conditions, Annex C for examples)

Note 1 to entry: Particles with $L < 3$ μm or $w < 2$ μm are not taken into account (see Figure 5).

3.1.3
inclusion
general designation of a ratable feature composed of at least one particle, defined by the size and proximity of its constituents. It can describe a single particle; a single stringer; or an agglomeration of stringers

Note 1 to entry: Stringers that meet the proximity conditions $e \leq 40$ µm and $t \leq 10$ µm form an agglomeration of stringers (see Figure 4). Formation of inclusions by combining stringers and single particles is not permitted.

Note 2 to entry: If elongated and globular particles are combined (see Figure 6), in general the result is treated as one inclusion.

3.1.4
test area
area on the polished surface of the specimen to be evaluated

3.2 Proximity:

3.2.1
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 3)

3.2.2
distance between stringers
similar to that for the distance between particles (see Figure 4)

3.2.3
scattered
random arrangement of particles

Note 1 to entry: For example see Annex C. This is defined in one field of view.

3.3 Parameters:

3.3.1
length
dimension of an inclusion in the main deformation direction, usually larger than the width

3.3.2
diameter
maximum dimension of inclusion classified according to column 6 (globular inclusion)

3.3.3
width
dimension of inclusion perpendicular to the direction of principal deformation

Note 1 to entry: For inclusions consisting of a single particle, the width is the maximum dimension perpendicular to the main deformation direction (see Figure 1).

For inclusions consisting of a single stringer, the width is the width of the confining rectangle (see Figure 2).

For inclusions consisting of an agglomeration of stringers, three cases apply:

— Case a) two stringers for which $0 \leq e \leq 40$ µm, $t \leq 10$ µm: the width of this inclusion is the width of the widest stringer ($w_{total} = w_1$; $w_1 > w_2$; see Figure 4 a)).

— Case b) two stringers for which $e < 0$ µm, $t \leq 10$ µm: the width of this inclusion is the sum of the stringers' widths and the distance t ($w_{total} = w_1 + w_2 + t$; see Figure 4 b)).

— Case c) an agglomeration of more than two stringers: the width of this inclusion is the widest width obtained by applying the rules in case a) and b) (see Figure 4 c))

3.3.4
area

area of the equivalent ellipse, calculated as $a = \frac{\pi}{4} \times L \times w_{total}$, or, in the case of globular particles,

$a = \frac{\pi}{4} \times d^2$ (see 3.3.3 and Figures 1 and 2)

3.3.5
shape factor
exponent f in the formula:

$$\frac{\pi/4 \times L^2}{a} = \left(\frac{L}{1\mu m} \right)^f \tag{3}$$

Note 1 to entry: For details see Annex D.

3.4   Classes:

3.4.1
elongated particles
particles with a ratio $L/w \geq 3$ (see Figure 1)

3.4.2
globular particles
particles with a ratio $L/w < 3$ (see Figure 1)

3.4.3
type
distinction of inclusions according to their shape, arrangement, and colour, and, if desired, by their apparent chemical composition (see Annex A)

3.5   Others:

3.5.1
lot
unit of material processed at one time and subject to similar processing variables

3.5.2
restricted values
values of the average field assessment restricted to inclusions greater and/or smaller than a defined length/diameter or area, or restricted to inclusions of specified types

4   Symbols and abbreviations

For the purposes of this document, the following symbols and abbreviations apply.

Symbol	Unit	Designation
a	μm^2	area of inclusions
b	mm	width of the plate
b		black
c		coloured (pink or yellow) (typically nitrides)

d	µm	diameter of inclusions
e	µm	interparticle distance (elongation axis)
f		shape factor
g		grey (typically sulphides)
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
p		particle index
q		row number
s		specimen index
t	µm	interparticle distance (transverse axis)
u	µm	scale unit in microscope eyepiece
v	mm	width of polished surface
w	µm	width of inclusions
x		variable
av or $^-$		average value of n, L, w, a ....
α		scattered, elongated inclusion type
α_b		scattered, elongated, black inclusion type
α_c		scattered, elongated, coloured inclusion type
α_g		scattered, elongated, grey inclusion type
β		aligned, globular inclusion type
β_b		aligned, globular, black inclusion type,
β_c		aligned, globular, coloured inclusion type
β_g		aligned, globular, grey inclusion type
γ		aligned, elongated inclusion type
γ_b		aligned, elongated, black inclusion type
γ_c		aligned, elongated, coloured inclusion type
γ_g		aligned, elongated, grey inclusion type
δ		scattered, globular inclusion type
δ_b		scattered, globular, black inclusion type
δ_c		scattered, globular, coloured inclusion type

δ_g		scattered, globular grey inclusion type
A	μm^2	area of field of view on the specimen
B	mm^2	polished surface
D		diameter of product
MD		main deformation direction (e.g. rolling direction)
G		magnification
H	μm	length of measuring frame on the specimen
K	-, μm, $\mu m^2/mm^2$	average field assessment
L	μm	length of inclusions
M	-, μm, $\mu m^2/mm^2$	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

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.

5 Principles

This standard consists of a comparison between inclusions observed in a field of view and chart pictures. The chart classifies inclusions into four different types according to their shape (7.4 / Annex H). The minimum requirements for applying this method are a square measuring frame of 0,71 mm x 0,71 mm overlaid on the viewfield at a magnification of 100:1 (see Figure 9), along with the chart – or alternatively a measuring scale and Table 2 of this draft standard.

An inclusion according to this standard can consist of a single particle, a stringer of particles, or an agglomeration of stringers. All inclusions are treated as ellipses (see Figure 1), with special rules for agglomerations of stringers (3.1.2 / Figure 4). Inclusions are classified according to shape, arrangement, and size (Annex A). A classification by colour is also permissible in order to differentiate apparent chemical composition (Annex A), although this classification provides no information on crystal structure or actual chemical composition.

The length and width of an inclusion are estimated by the class values of its corresponding row and column in the picture chart. The chart pictures depict the upper class boundaries. Upon classification, all further calculations refer to the class values in Table 2.

This standard yields different results depending on the chosen method: the largest inclusions (worst inclusion method), the largest inclusion parameters per field of view (worst field method), and an averaged inclusion content (average field method). If not determined by product standards, the involved parties shall agree on the preferred method for their steel grade. The default rating methods

are the worst inclusion method ($P_{L/d}$) and the average field method (K_n, $K_{L/d}$). All results have physical dimensions, regardless of the method.

Annexes M, N, and P include examples for recording and for calculating results. The following section contains a brief practical guide to the evaluation specified in this standard.

6 Brief practical guide

6.1 Basic rules for evaluation

a) **Preparing the measurement:**

1) Take and prepare specimens according to Clause 7, Sampling.

2) Define the test area and a starting point for the measurement.

b) **Examining the test area:**

1) Scan the entire test area at the selected magnification (usually 100:1).

2) Evaluate all inclusions using the measurement frame or a measuring scale.

c) **Exclusion of particles outside of the scope:**

1) Exclude from evaluation all particles with a length or diameter < 3 µm or a width < 2 µm (see 3.1.3, inclusion).

d) **Rules for ascertaining inclusions:**

1) Distinguish globular from elongated particles using the length-to-width ratio. According to 3.4 Classes, particles with $L/w \leq 3$ are globular and particles with $L/w > 3$ are elongated.

2) Inclusions can consist of a single particle, a stringer of particles, or an agglomeration of stringers.

3) The proximity conditions for joining together particles or stringers are $e \leq 40$ µm and $t \leq 10$ µm (see 3.1.3, inclusion).

4) Particles that do not meet the proximity conditions are rated as individual inclusions.

5) A stringer is formed and rated as an inclusion when at least two particles meet the proximity conditions.

6) Stringers that meet the proximity conditions are joined to form an agglomeration of stringers. The agglomeration (and not the individual stringers) is rated as an inclusion.

7) Inclusions consisting of different types of particles are classified according to the areally predominant shape and subsequently, if necessary, according to the areally predominant colour.

8) Inclusions longer than the field of view are rated according to their total dimensions and only rated once.

9) Inclusions longer or wider than the classes in the scope of this standard (oversized) are rated as belonging to the largest possible class and reported separately with their actual dimensions.

e) Rating the inclusions:

1) Using a measuring scale and the chart (or Table 2), the observed inclusions are classified according to shape, arrangement, and size, and rated according to the parameters of the chosen method (see Clause 8, Types of assessment).

2) The inclusions are assigned to the types α, β, γ and δ according to their shape and arrangement. Where a differentiation by colour is required, inclusions are assigned to the types EA, EC, EAB, EB, EAD, and ED (see Annex A).

3) The length and width of an inclusion are estimated by the class values of its corresponding row and column in the picture chart. The chart pictures depict the upper class boundaries.

4) An inclusion is classified in row q if for its length L_x:

   $L_{q-1} < L_x \leq L_q$ [µm]

5) An inclusion is classified in column k if for its width w_x:

   $w_{q,k-1} < w_x \leq w_{q,k}$ [µm]

Upon classification by length and width, all further calculations (e.g. for inclusion area) refer to the class values in Table 2.

6.2 Evaluation according to the default rating methods

— **Method P_L, P_d – Ascertaining the largest inclusions by length and diameter (see Subclause 8.1, method P):**

For every inclusion type (α, β, γ, δ or EA, EAD, EB, EAB, EC, ED), rate and record the inclusion with the largest value for the chosen parameter (L or d). The recording sheet K.1 can be used for documentation (see Annex K).

The result is the average of the individual values for each specimen.

— **Method K_n, K_L and K_n, K_d – Ascertaining the mean inclusion content by number, as well as by length and diameter (see Subclause 8.3 and Annex M):**

Rate and record every inclusion of the chosen types (e.g.: α, β, γ, δ or EA, EAD, EB, EAB, EC, ED) according to the chosen parameter (L oder d). Take care that each inclusion is recorded once and only once. Record the examined area. The recording sheet M.2 can be used for documentation (see Annex M).

The result is an averaged inclusion content. The computation sheet M.3 can be used to obtain this result (see Annex M).

7 Sampling

7.1 General

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

7.2 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   The lower the degree of reduction, the likelier it is that porosities remain in the steel. Failing to properly differentiate them from inclusions will affect the final result.