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Steel — Determination of content of nonmetallic inclusions — Micrographic method using standard diagrams

Aciers — Détermination de la teneur en inclusions non métalliques — Méthode micrographique à l'aide d'images types

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Foreword

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The committee responsible for this document is ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This third edition cancels and replaces the second edition (ISO 4967:1998), of which it constitutes a minor revision.

Steel — Determination of content of nonmetallic inclusions — Micrographic method using standard diagrams

1 Scope

This International Standard specifies a micrographic method of determining the non-metallic inclusions in rolled or forged steel products having a reduction ratio of at least 3 using standard diagrams. This method is widely used to assess the suitability of a steel for a given use. However, since it is difficult to achieve reproducible results owing to the influence of the test operator, even with a large number of specimens, precautions should be taken when using the method.

NOTE For certain types of steel (e.g. free cutting steels), the standardized diagrams described in this International Standard may not be applicable.

This International Standard also provides for the determination of non-metallic inclusions by image analysis technologies (see Annex D).

2 Principle

The method consists of comparing the observed field to the chart diagrams defined in this International Standard and taking in consideration separately each type of inclusion. In the case of image analysis, fields will be rated according to the relationships given in Annex D.

The chart pictures correspond to square fields of view, each with an area of 0,50 mm^2, as obtained with a longitudinal plane-of-polish and as observed at 100 ×.

According to the shape and distribution of the inclusions, the standard diagrams are divided into five main groups, bearing the reference A, B, C, D and DS.

These five groups represent the most commonly observed inclusion types and morphologies:

— **Group A (sulfide type)**: highly malleable, individual grey particles with a wide range of aspect ratios (length/width) and generally rounded ends;

— **Group B (aluminate type)**: numerous non deformable, angular, low aspect ratio (generally < 3), black or bluish particles (at least three) aligned in the deformation direction;

— **Group C (silicate type)**: highly malleable, individual black or dark grey particles with a wide range of aspect ratios (generally > 3) and generally sharp ends;

— **Group D (globular oxide type)**: non deformable, angular or circular, low aspect ratio (generally < 3), black or bluish, randomly distributed particles;

— **Group DS (single globular type)**: circular, or nearly circular, single particle with a diameter > 13 µm.

Non-traditional inclusion types may also be rated based on their morphology compared to the above five types and a statement about their chemical nature. As an example, globular sulfides would be rated as a D type and a descriptive subscript (e.g. D_{sulf}) defined in the test report. D_{cas} would indicate globular calcium sulfides; D_{RES} would indicate globular rare earth sulfides; D_{Dup} would indicate globular duplex inclusions, such as calcium sulfide surrounding an aluminate.

Types of precipitate such as borides, carbides, carbonitrides or nitrides may also be rated based on their morphology compared to the above five types and a statement about their chemical nature as described in the previous subclause.

NOTE Examination at a magnification greater than 100 × may be used to identify the nature of the non-traditional inclusions before performing the test.

Each main group on the chart consists of two subgroups, each made up of six diagrams representing increasing inclusion content. This division into subgroups is merely intended to give examples of different thicknesses of non-metallic particles.

The diagrams on the chart are given, by inclusion group, in Annex A.

These chart diagrams carry an index number, *i*, from 0,5 to 3, the numbers increasing with the inclusion or stringer lengths (Groups A, B, C) or by the number (Group D) or by the diameter (Group DS), as defined in Table 1, and by thickness, as defined in Table 2. For example, the diagram A 2 indicates that the shape of the inclusions observed under the microscope is in accordance with group A and that their distribution and quantity are in accordance with number 2.

Table 1 — Rating limits (minimum values)

Chart diagram Index *I*	Inclusion group				
	A total length µm	**B** total length µm	**C** total length µm	**D** count number	**DS** diameter µm
0,5	37	17	18	1	13
1	127	77	76	4	19
1,5	261	184	176	9	27
2	436	343	320	16	38
2,5	649	555	510	25	53
3	898	822	746	36	76
	(< 1 181)	(< 1 147)	(< 1 029)	(< 49)	(< 107)

NOTE The above length values for the A, B and C groups have been computed from the equations given in Annex D and then rounded off to the nearest whole number.

Table 2 — Inclusion thickness parameters

Group type	Fine		Thick	
	Minimum width µm	Maximum width µm	Minimum width µm	Maximum width µm
A	2	4	4	12
B	2	9	9	15
C	2	5	5	12
D	2	8	8	13

NOTE For type D, the maximum dimension is defined as the diameter.

3 Sampling

The shape of the inclusion depends to a large extent on the degree of reduction of the steel; therefore, comparative measurements may only be carried out on prepared sections taken from samples with a similar amount of deformation.

The polished surface of the specimen used to determine the content of inclusions shall be approximately 200 mm^2 (20 mm × 10 mm). It shall be parallel to the longitudinal axis of the product and shall be located halfway between the outer surface and the centre.

The method of sampling shall be defined in the product standard or subject to agreement between the parties. The test surface, in the case of plates, shall be approximately at a quarter of the width.

In the absence of such specifications, the sampling procedure shall be as follows:

— bar or billet with diameters greater than 40 mm: the surface to be examined consists of a part of diametral section located halfway between the outer surface and the centre (see Figure 1);

— bar with a diameter greater than 25 mm and less than or equal to 40 mm: the surface to be examined consists of half the diametral section (from the centre to the edge of the sample) (see Figure 2);

— bar with a diameter less than or equal to 25 mm: the surface to be examined consists of the full diametral section of length sufficient to obtain a surface of about 200 mm^2 (see Figure 3);

— plate with a thickness less than or equal to 25 mm: the surface to be examined consists of the whole thickness, and located at the quarter of the width (see Figure 4),

— plate with a thickness greater than 25 mm and less than or equal to 50 mm: the surface to be examined consists of half the thickness from the surface to the centre and is located at the quarter of the width (see Figure 5);

— plate with a thickness greater than 50 mm: the surface to be examined consists of quarter the thickness and is located halfway between the outer surface and the middle of the thickness and at the quarter of the width (see Figure 6).

The number of samples to be taken is defined in the product standard or by special agreement.

For any other product, the sampling procedures shall be subject to agreement between the parties.

Dimensions in millimetres

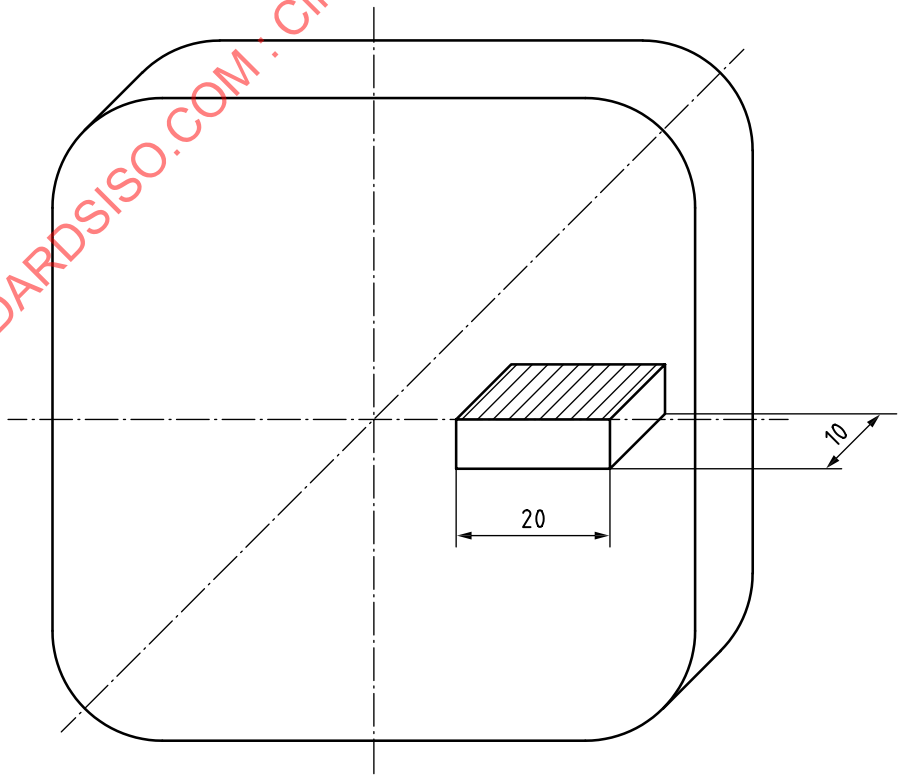


Figure 1 — Sample from bar or billet with a diameter or length of side > 40 mm

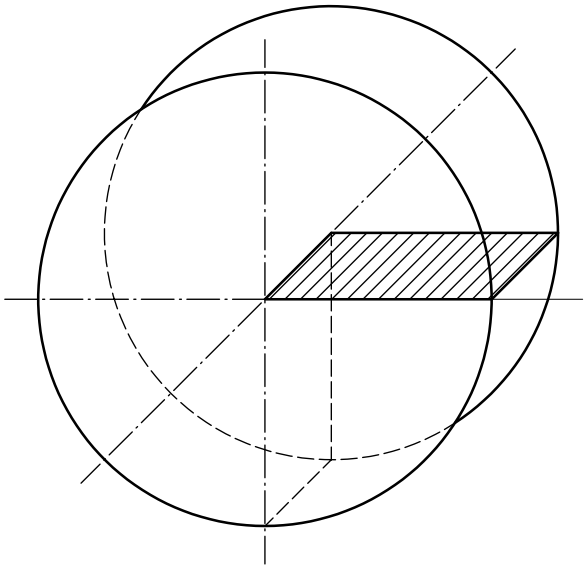


Figure 2 — Sample from bar or billet with a diameter or length of side > 25 mm and ≤ 40 mm

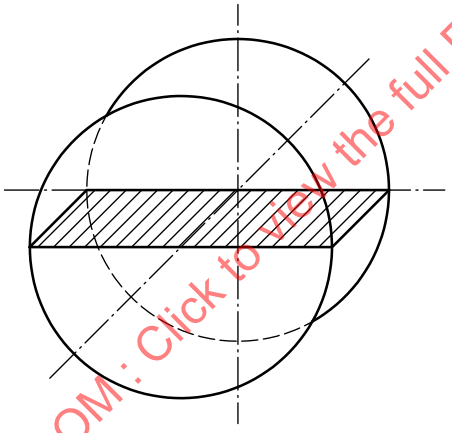
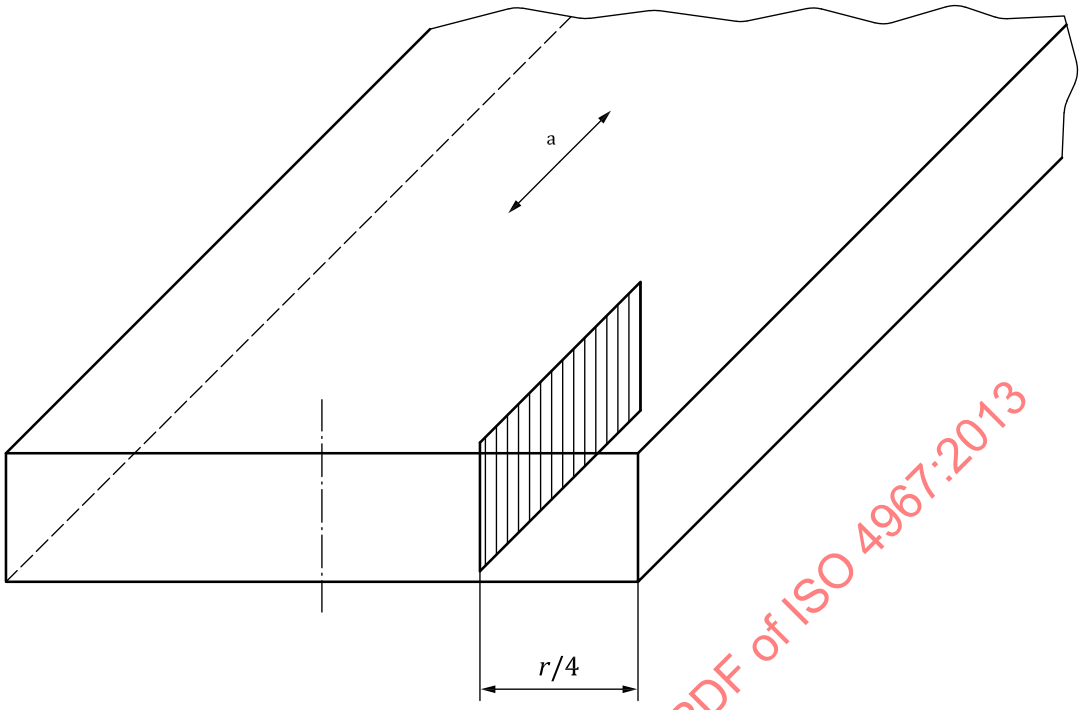


Figure 3 — Sample from bar with a diameter ≤ 25 mm

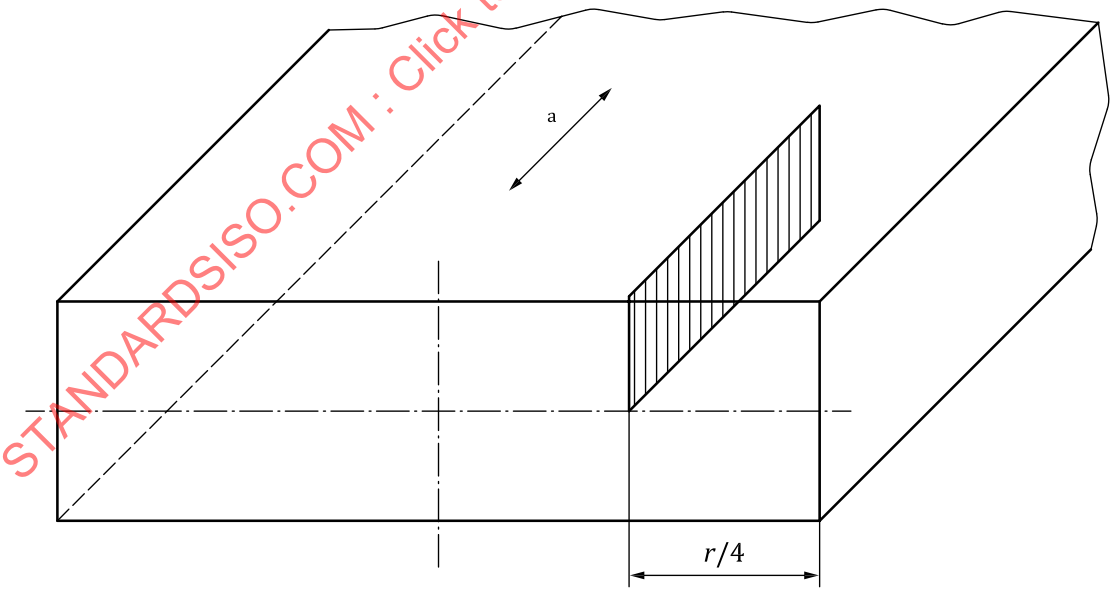


Key

r = width

a rolling direction

Figure 4 — Sample from plate with thickness ≤ 25 mm

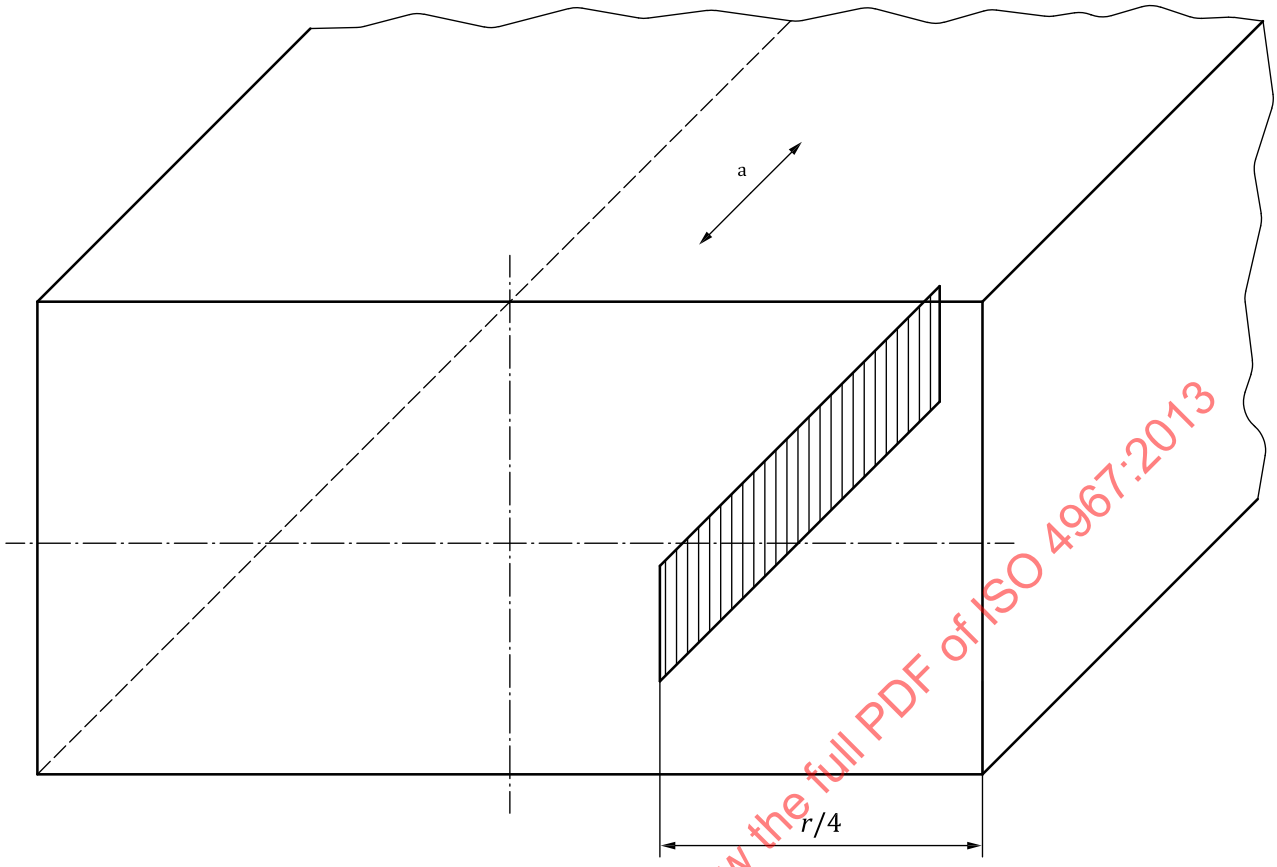


Key

r = width

a rolling direction

Figure 5 — Sample from plate with thickness > 25 mm and ≤ 50 mm



Key

r = width

a rolling direction

Figure 6 — Sample from plate with thickness > 50 mm

4 Preparation of specimens

The specimen shall be cut so as to obtain a surface for examination. In order to achieve a flat surface and to avoid rounding the edges of the specimen when polishing, the specimen may be held mechanically or may be mounted.

When polishing specimens, it is important to avoid any tearing out or deformation of the inclusions, or contamination of the polished surface, so that the surface is as clean as possible and the shape of the inclusions is not affected. These precautions are of particular importance when the inclusions are small. It is advisable to use diamond paste for polishing. In certain cases it may be necessary for the specimen to be heat treated before polishing in order to give it the maximum possible hardness.

5 Determination of the content of inclusions

5.1 Method of observation

Examination with the microscope may be by one of two methods:

— by projection on to ground glass,

— by observation by means of an eyepiece.

The method of observation chosen shall be maintained throughout the test.

If the image is projected onto a ground glass or similar device, the magnification shall be 100 × ± 2 × on the ground glass. Place a clear plastic overlay (Figure 7) of a 71 mm square (0,50 mm^2 true area) over or behind the ground glass projection screen. The image within the test square is compared to the standard pictures on the chart (Annex A).

If the inclusions are examined through the microscope eyepieces, insert a reticle with the test pattern shown in Figure 7 in the microscope at the appropriate location so that the test grid area is 0,50 mm^2 at the image plane.

NOTE In special cases, a magnification greater than 100 may be used, provided that the same magnification is applied for the standard diagrams, and shall be recorded in the test report.

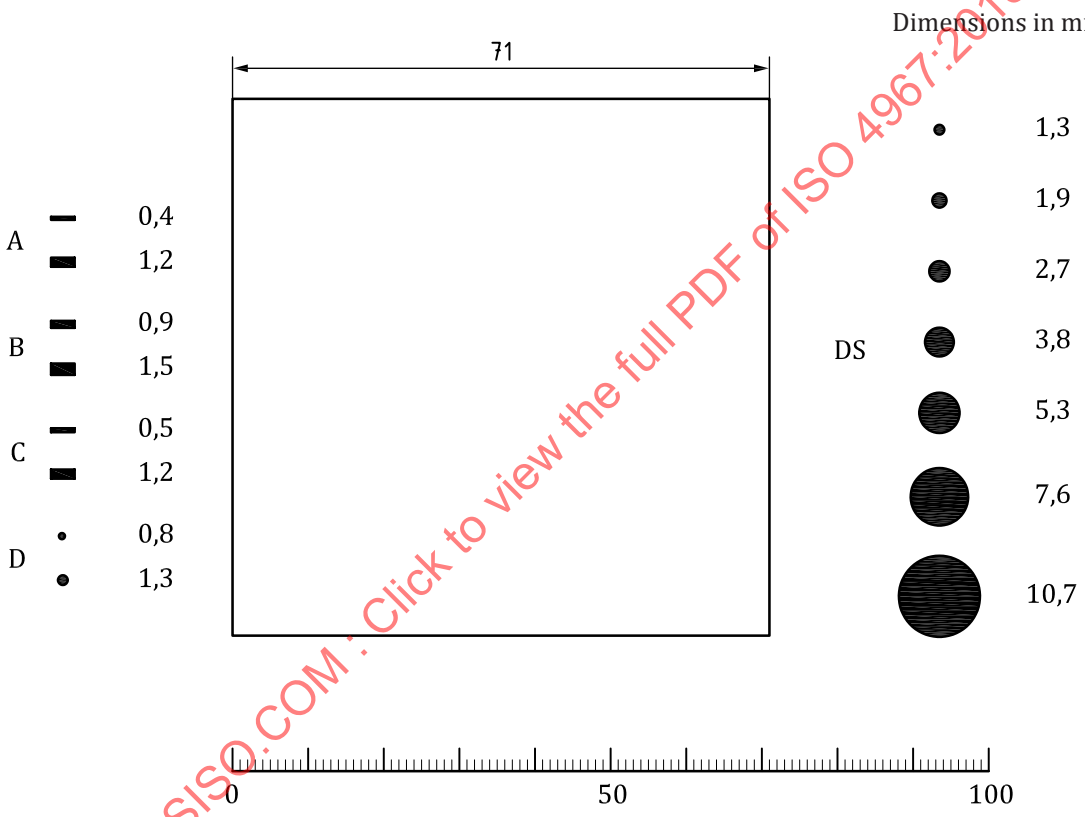


Figure 7 — Test pattern for grid overlays or reticles

5.2 Actual examination

Two methods are defined as described in 5.2.1 and 5.2.2.

5.2.1 Method A

The entire polished surface is examined and, for each type of inclusion, a note is made of the reference number which lies to the side of the standard diagram which corresponds to the worst field examined, in the fine and thick series.

5.2.2 Method B

The entire polished surface is examined and each field of the specimen is compared with the standard diagrams. The reference number of the field (indicated to the side of the standard diagrams) which best corresponds to the field examined for each type of inclusion is noted, in the fine and thick series.

In order to minimize the cost of examination, it may be agreed upon to make a partial examination of the specimen by studying a reduced number of fields, distributed in accordance with a fixed scheme. Both the number of fields examined and their distribution shall be arranged by prior agreement.

5.2.3 General rules for methods A and B

Each field observed is compared with the standard diagrams. If a field of inclusions falls between two standard diagrams, it is rated following the lower diagram.

Individual inclusions or stringers that have a length greater than the field width (0,710 mm) or a width or diameter greater than the thick series maximum (see Table 2) will be rated as oversized by length, width or diameter. The oversized dimensions of the inclusion or stringer shall be noted separately. However, these inclusions shall still be part of the overall rating of that field.

Reproducibility of measurements is improved if actual measurements (stringer lengths of A, B or C types, diameter of DS type) and counts (D types) are made. Use a grid overlay or reticle, as shown in Figure 7, the measurement limits in Tables 1 and 2, and the morphological descriptions in Clause 2, as illustrated in the chart.

Non-traditional inclusion types are rated according to the chart group (A, B, C, D, DS) that best corresponds to their morphology. Compare the length, number, thickness or diameter of the inclusions to each group shown in Annex A or determine their total length, number, thickness or diameter, and use Tables 1 and 2 to assign the appropriate inclusion content number and thickness class (fine, thick or oversize). Then indicate the nature of the non-traditional inclusion with a subscript to the group type. Define the subscript in the test report.

For type A, B and C inclusions, two individual inclusions or stringers of lengths l_1 and l_2 and which are, or are not, in line are considered as one inclusion or stringer if the distance d is lower than or equal to 40 µm and if the distance s, (the distance between centres of inclusions/stringers) is lower than or equal to 10 µm (see Figures 8 and 9).

In the case of a stringer with inclusions of different width, the width to be considered is the width of the biggest inclusion.

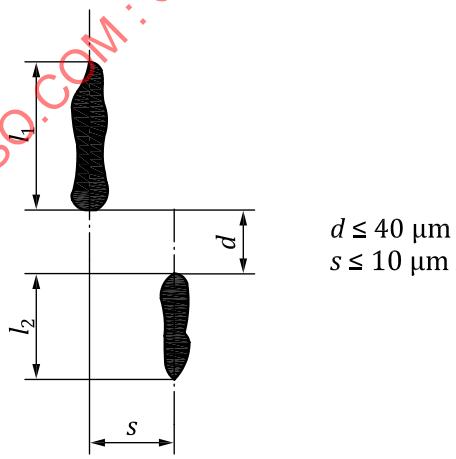


Figure 8 — Type A and C inclusions

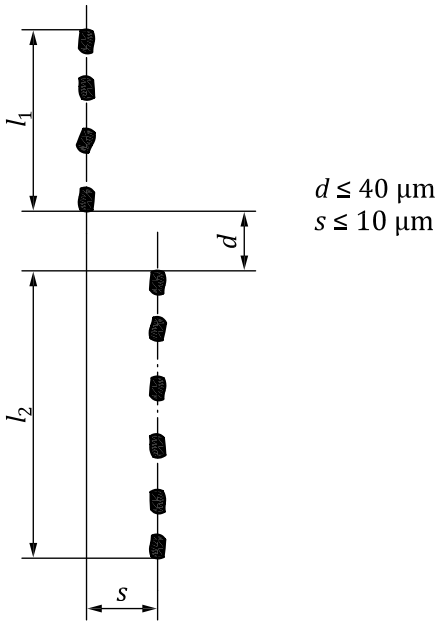


Figure 9 — Type B inclusions

6 Expression of results

6.1 General

Unless otherwise stated in the product standard, the results may be expressed as described in 6.2 and 6.3 depending on the method used (method A or method B).

6.2 Case of method A

For each type of inclusion and for each thickness series (see Annex B), the reference symbol for the group of inclusions shall be followed by the reference number of the worst field, the presence of thick inclusions being indicated by the letter e, the presence of inclusion oversized (see 5.2.3) being indicated by the letter s.

EXAMPLES A 2, B 1e, C 3, D 1, B 2s, DS 0,5.

Any subscripts used to identify non-traditional inclusion types shall be defined.

On the basis of the index numbers relating to each specimen and on their basis, an arithmetic mean may be assessed per cast for each type of inclusion and for each thickness series.

6.3 Case of method B

The total number of fields shall be indicated for a given index, per type of inclusion and per thickness series for a given number of fields observed (N).

The full set of total numbers of fields for a given index relative to the various types of inclusions may be used in special methods for expressing results, such as total index, i_{tot}, or mean index, i_{moy}, subject to agreement between the parties.

EXAMPLE:

For type A inclusions taking

n_1 as the number of fields of index 0,5,

n_2 as the number of fields of index 1,

n_3 as the number of fields of index 1,5,

n_4 as the number of fields of index 2,

n_5 as the number of fields of index 2,5,

n_6 as the number of fields of index 3,

then

$$i_{tot} = (n_1 \times 0{,}5) + (n_2 \times 1) + (n_3 \times 1{,}5) + (n_4 \times 2) + (n_5 \times 2{,}5) + (n_6 \times 3)$$

$$i_{moy} = \frac{i_{tot}}{N}$$

where N is the total number of fields observed.

A typical result is given in Annex C.

7 Test report

The test report shall contain the following information:

a) a reference to this International Standard, i.e. ISO 4967;

b) the steel grade and the cast number;

c) the nature of the product and its dimensions;

d) the type of sampling and position of the area examined;

e) the method selected (method of observation, method of examination, method of expressing results);

f) the magnification if greater than 100 ×;

g) the number of observed fields or the total area examined;

h) the results of the examination (including the number, size and type of oversize inclusions or stringers);

i) statement of subscripts used to define any non-traditional inclusion type;

j) report number and date;

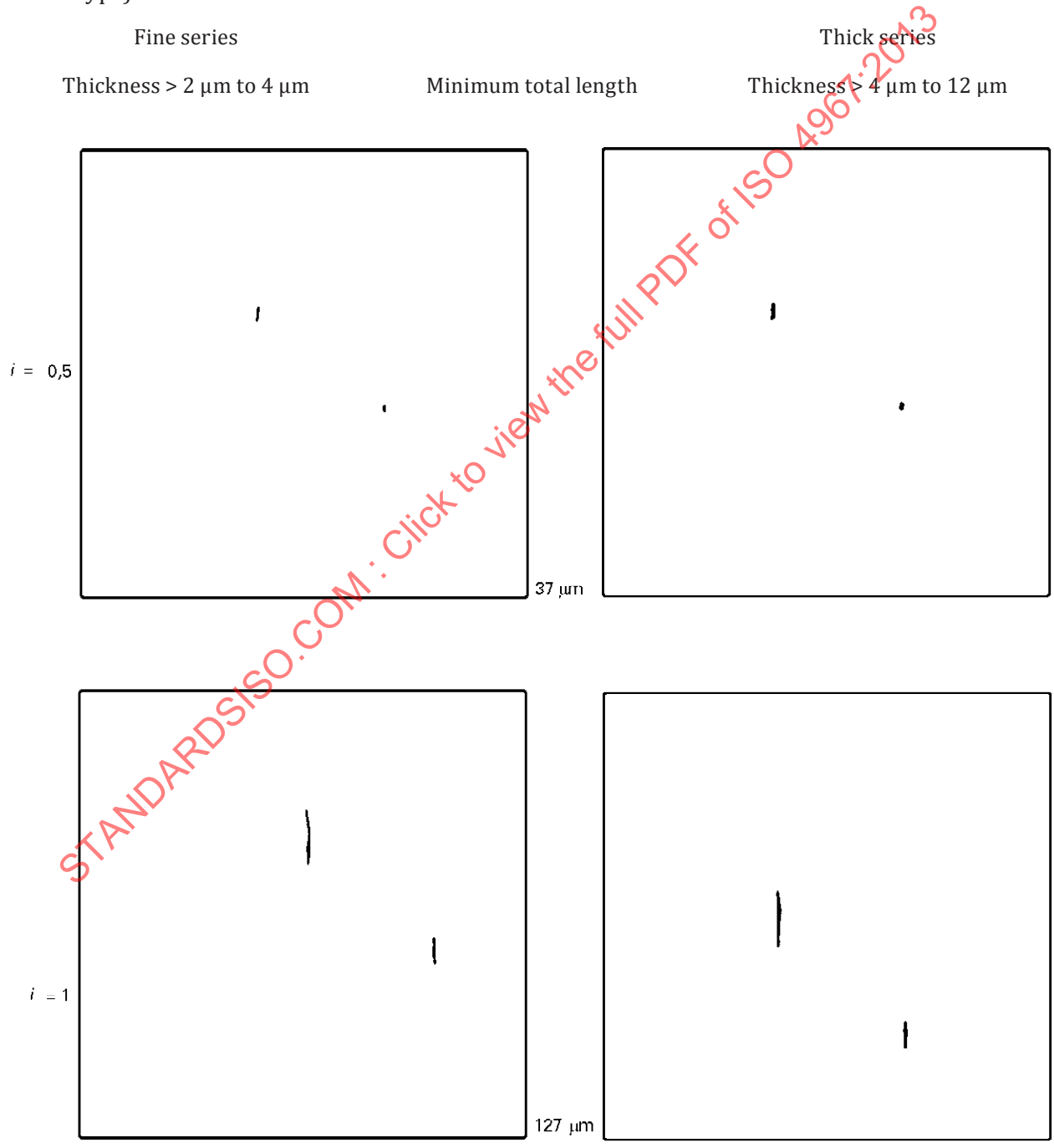
k) name of operator.

Annex A
(normative)

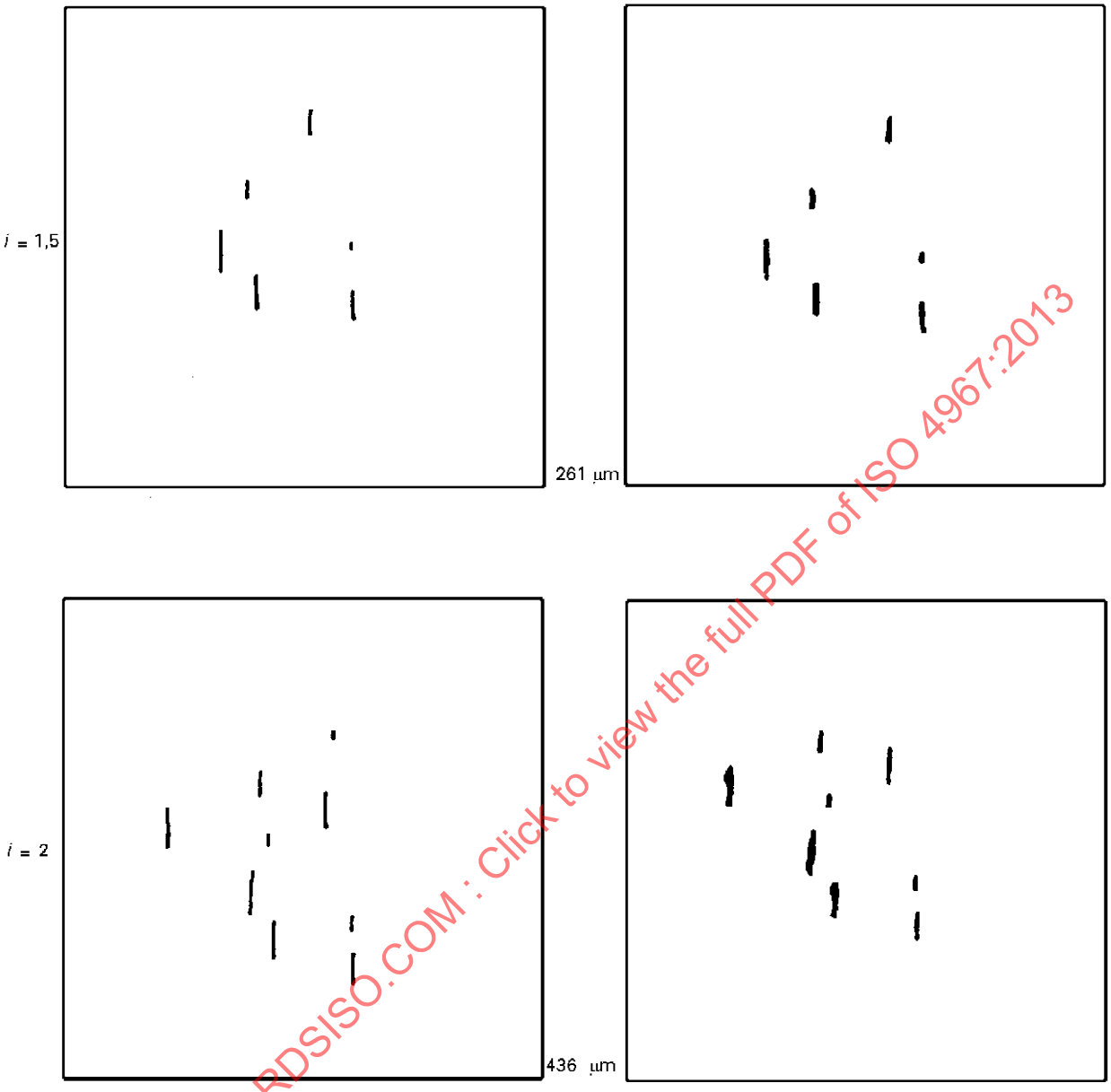
ISO Chart diagrams for inclusion groups A, B, C, D and DS

A

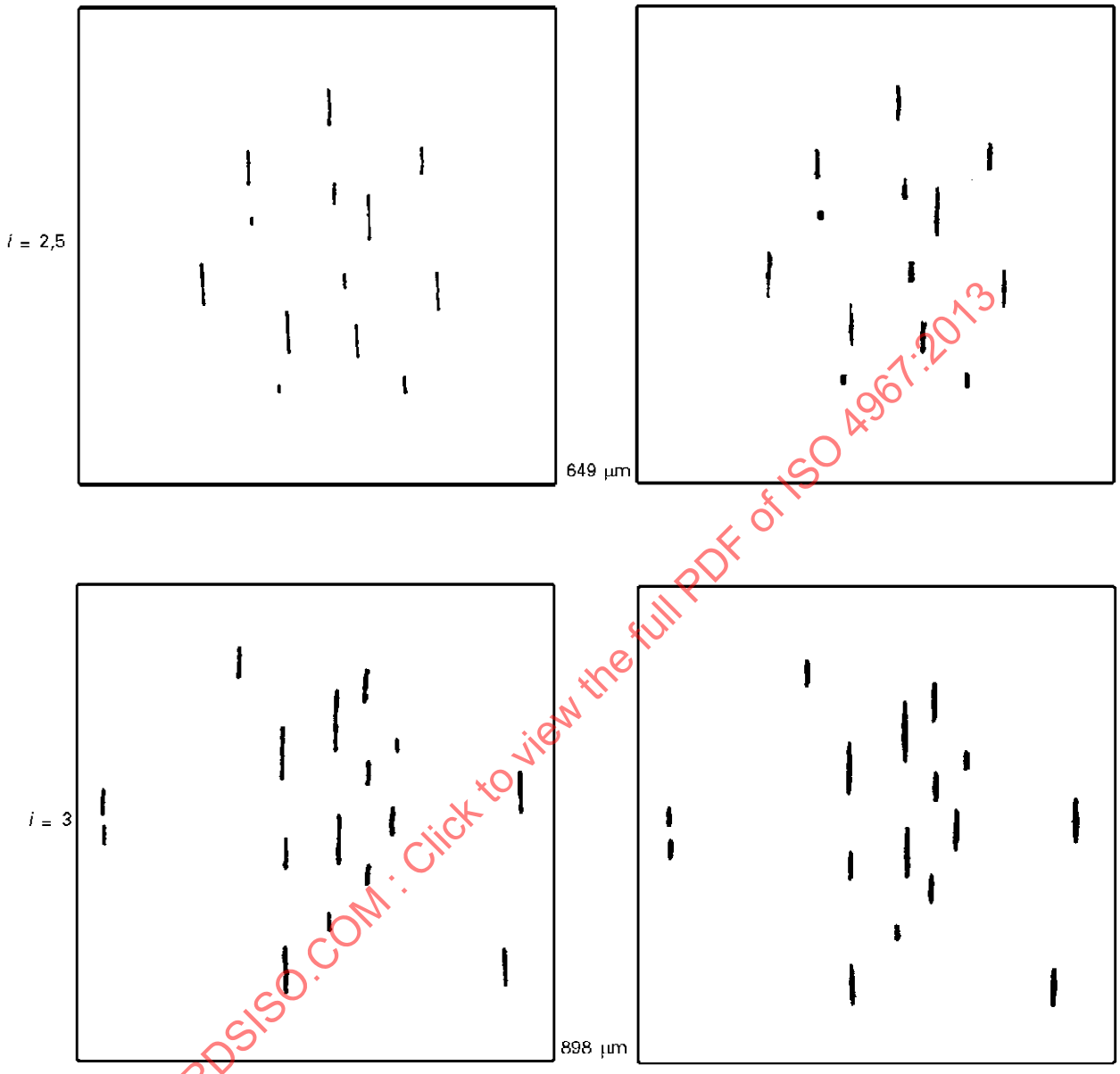
(Sulfide type)



Magnification = 100 ×



Magnification = 100 ×

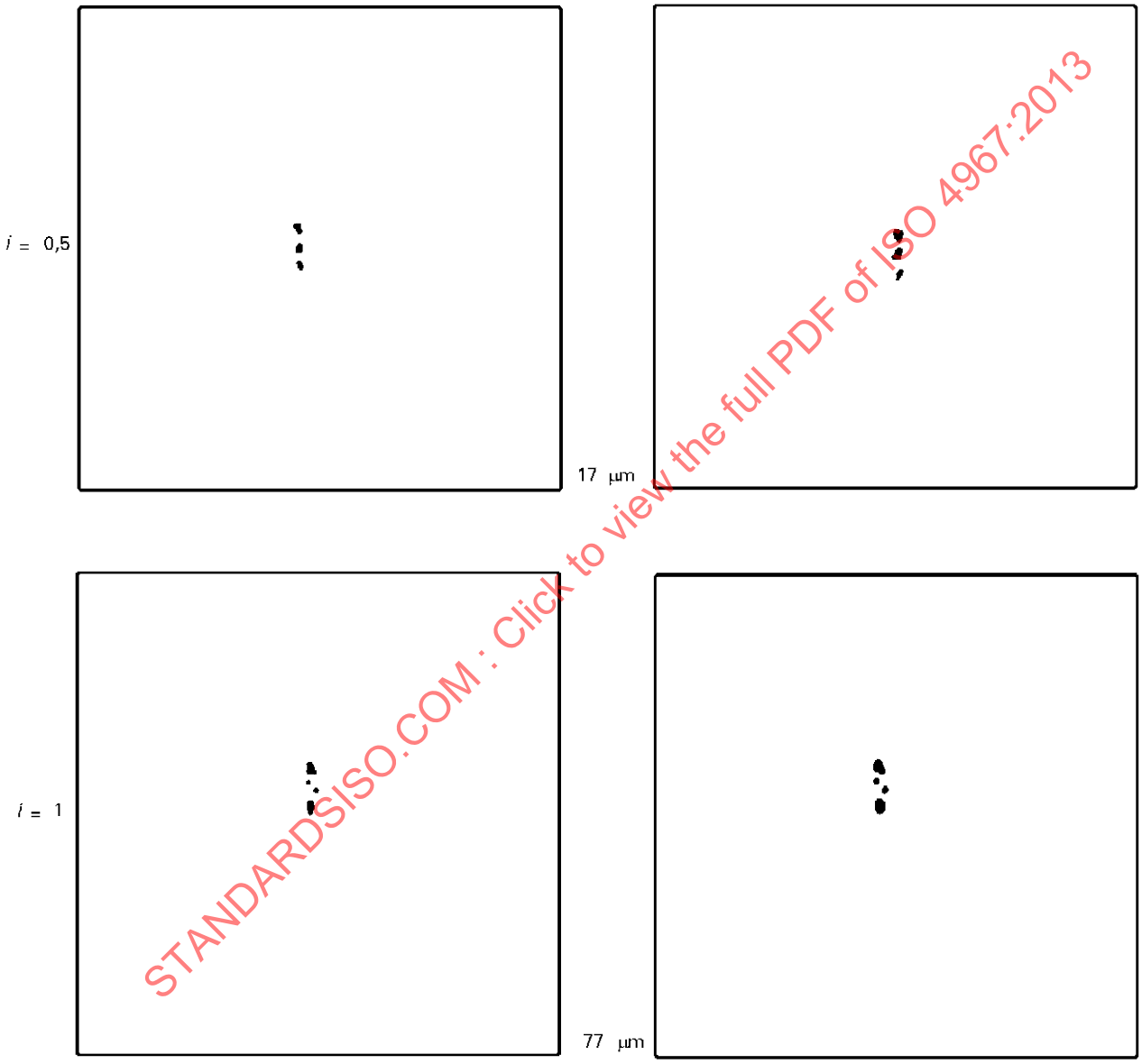


Magnification = 100 ×

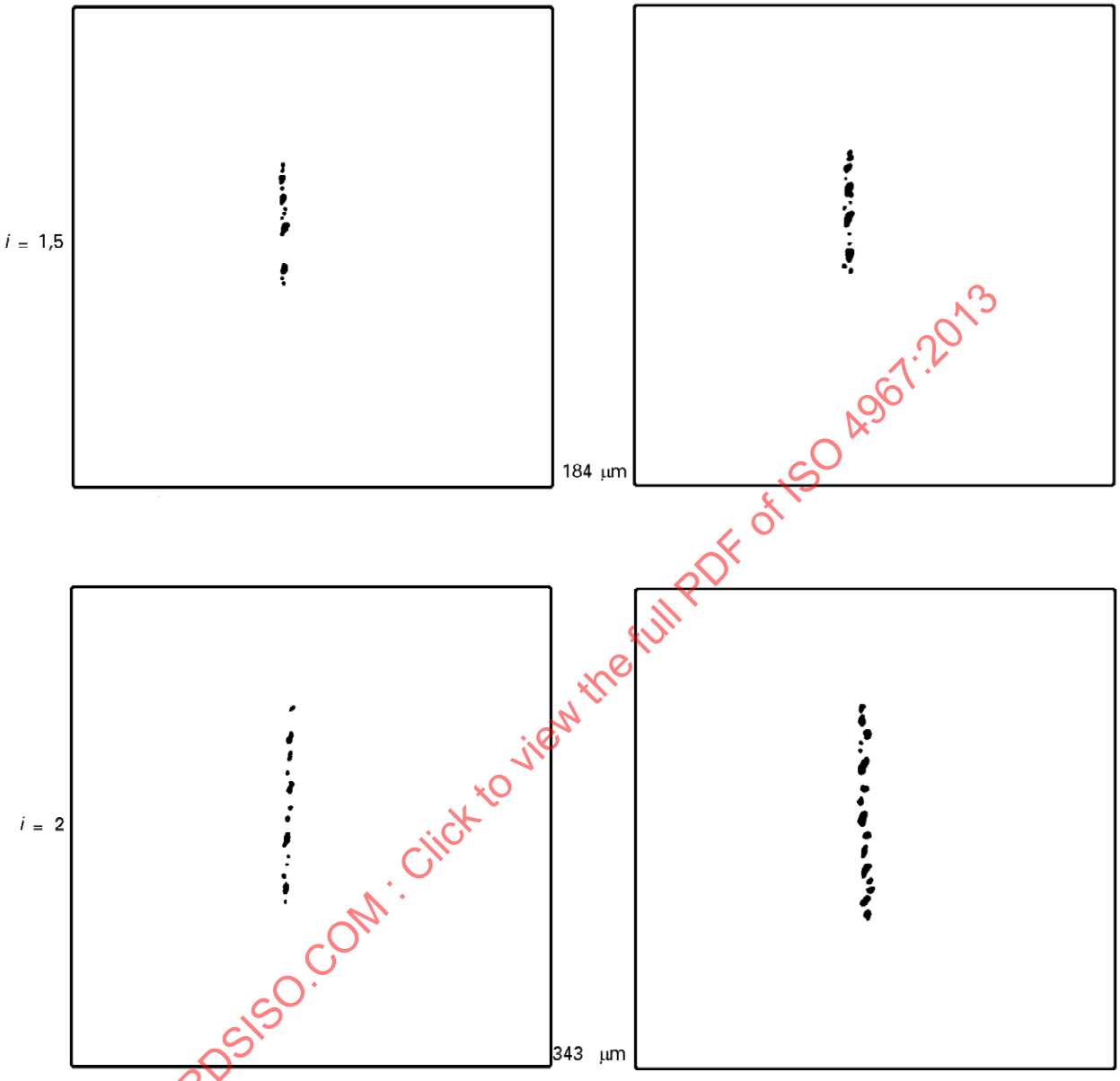
B

(Aluminate type)

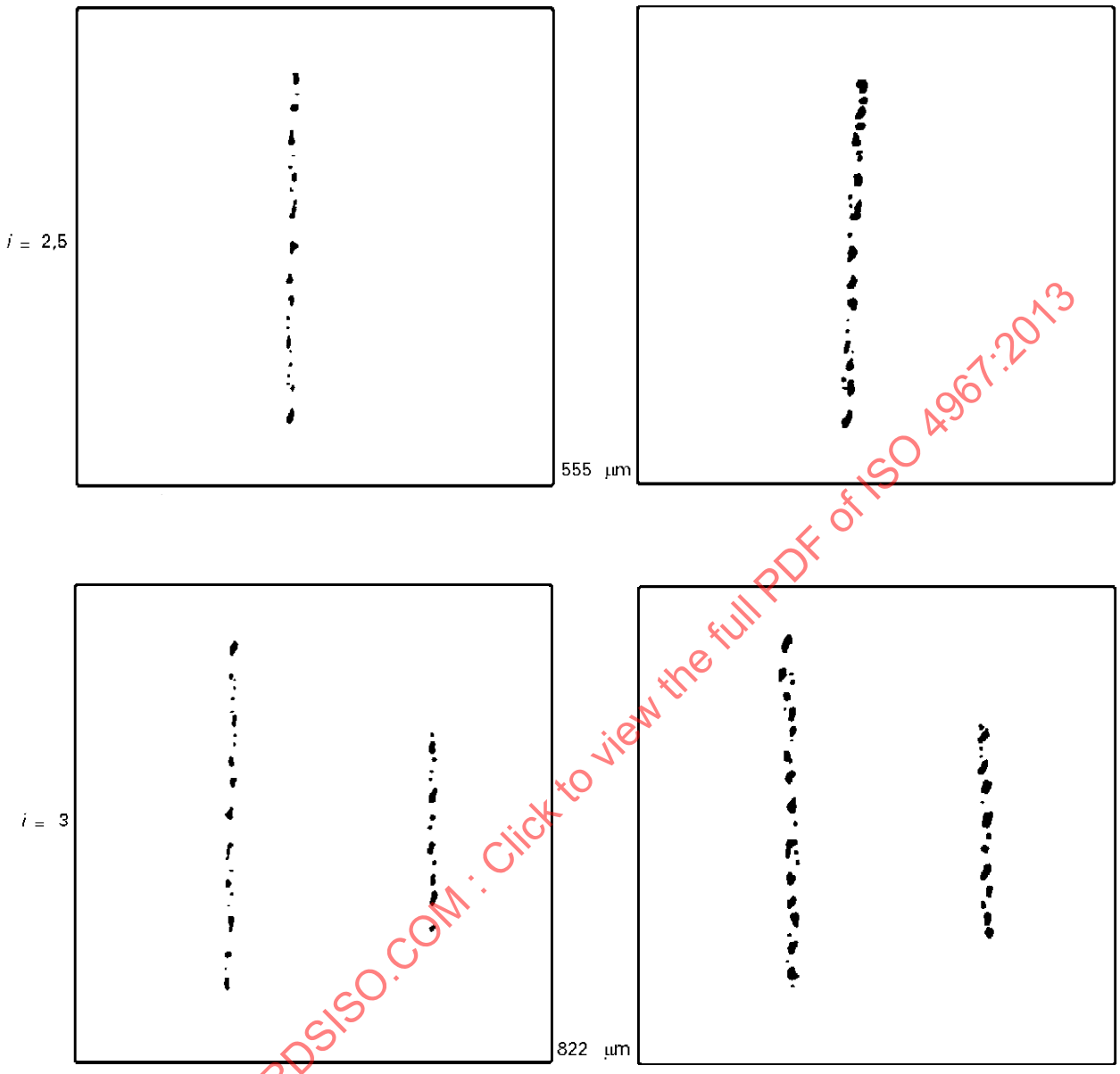
Fine series		Thick series
Thickness > 2 µm to 9 µm	Minimum total length	Thickness > 9 µm to 15 µm



Magnification = 100 ×



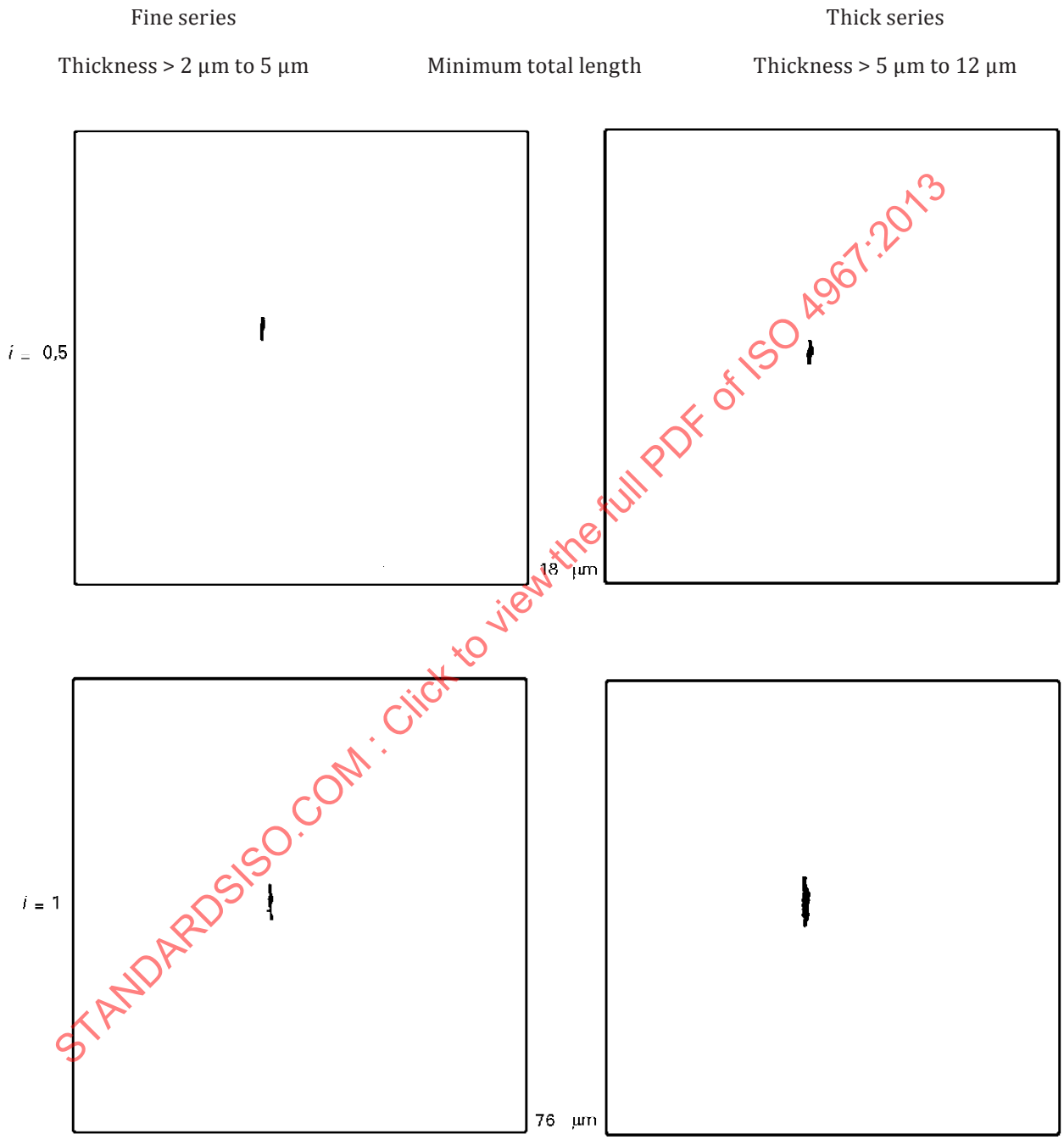
Magnification = 100 ×



Magnification = 100 ×

C

(Silicate type)



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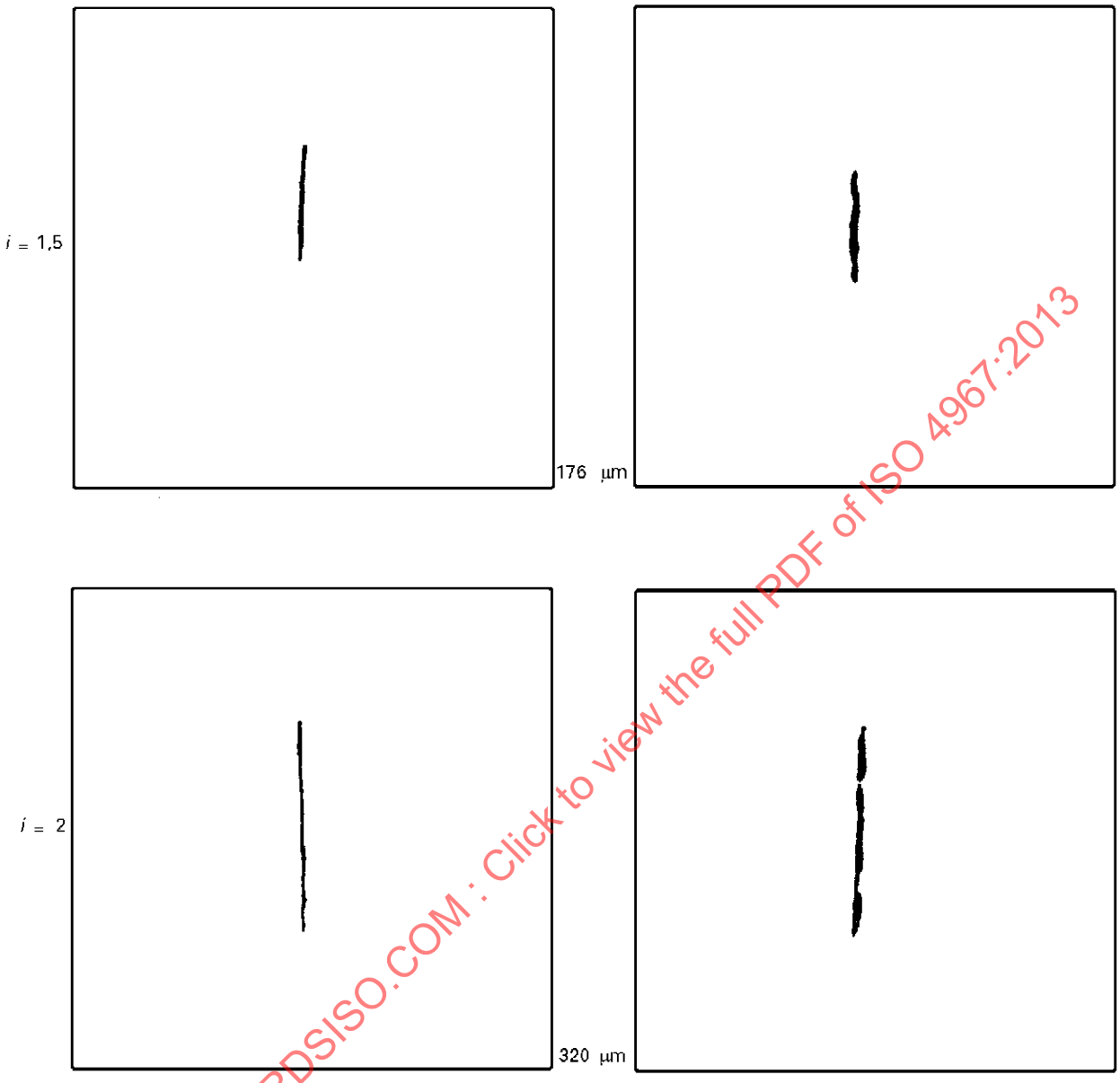
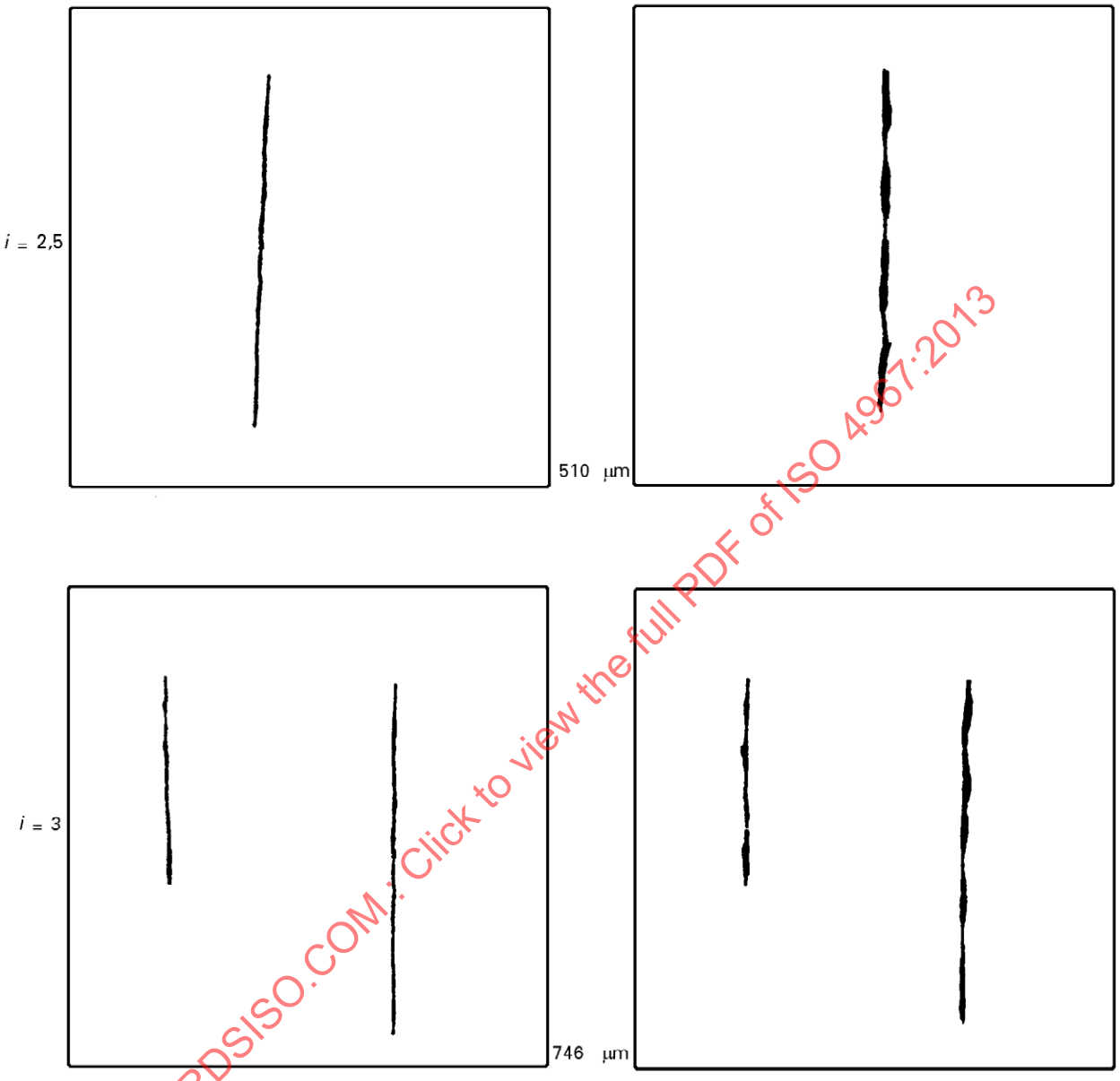


Magnification = 100 ×

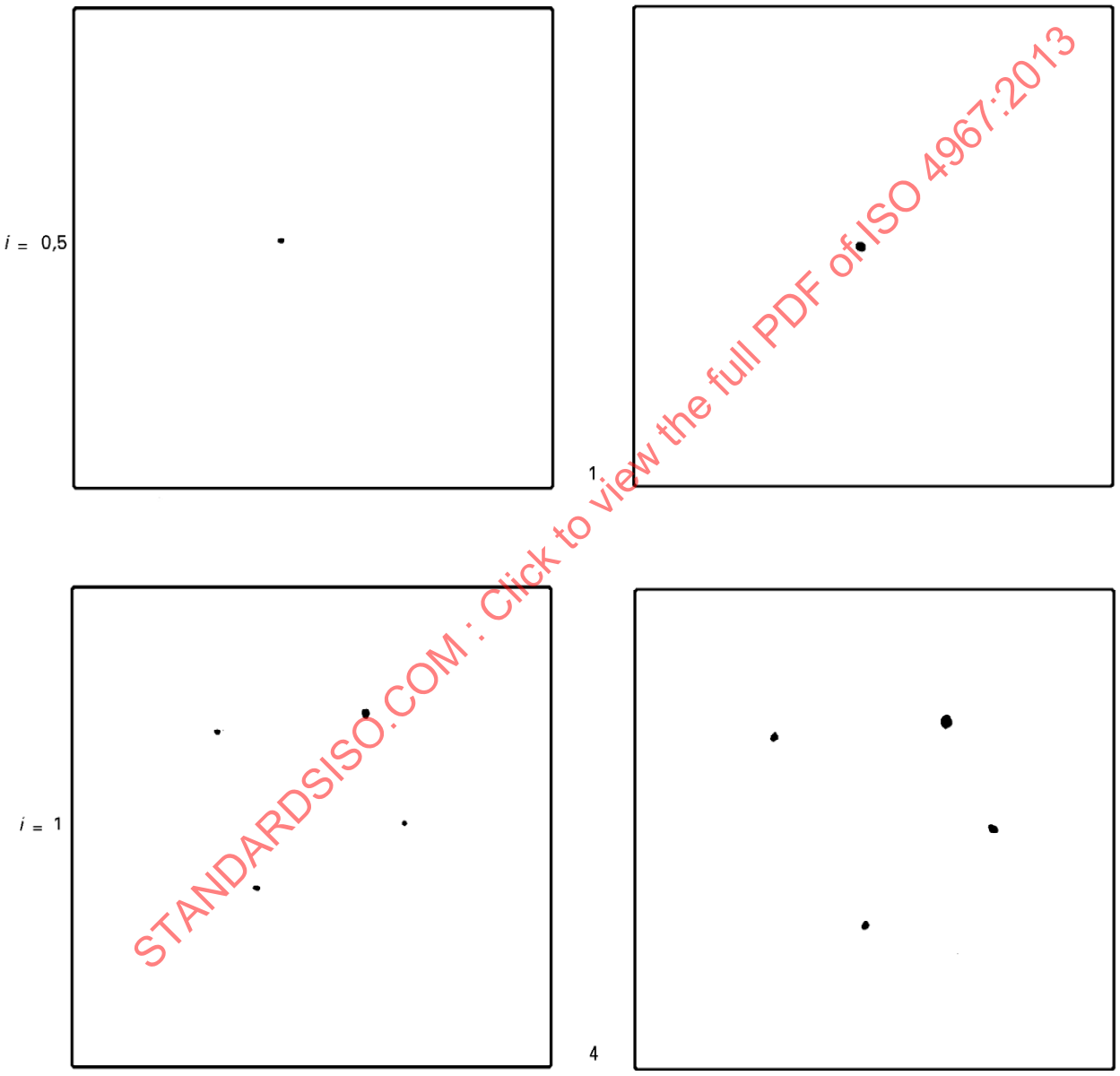


Magnification = 100 ×

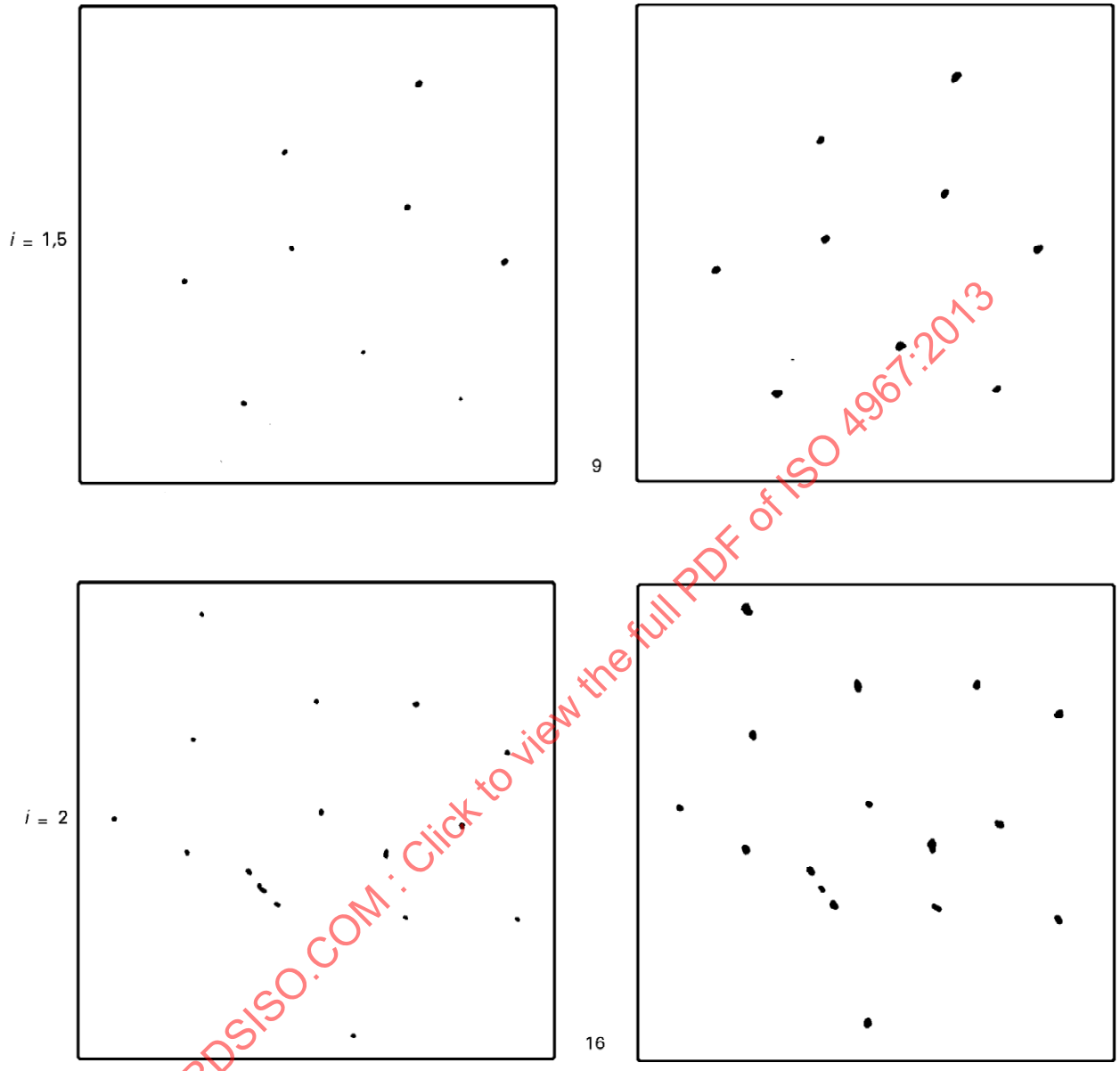
D

(Globular type oxides)

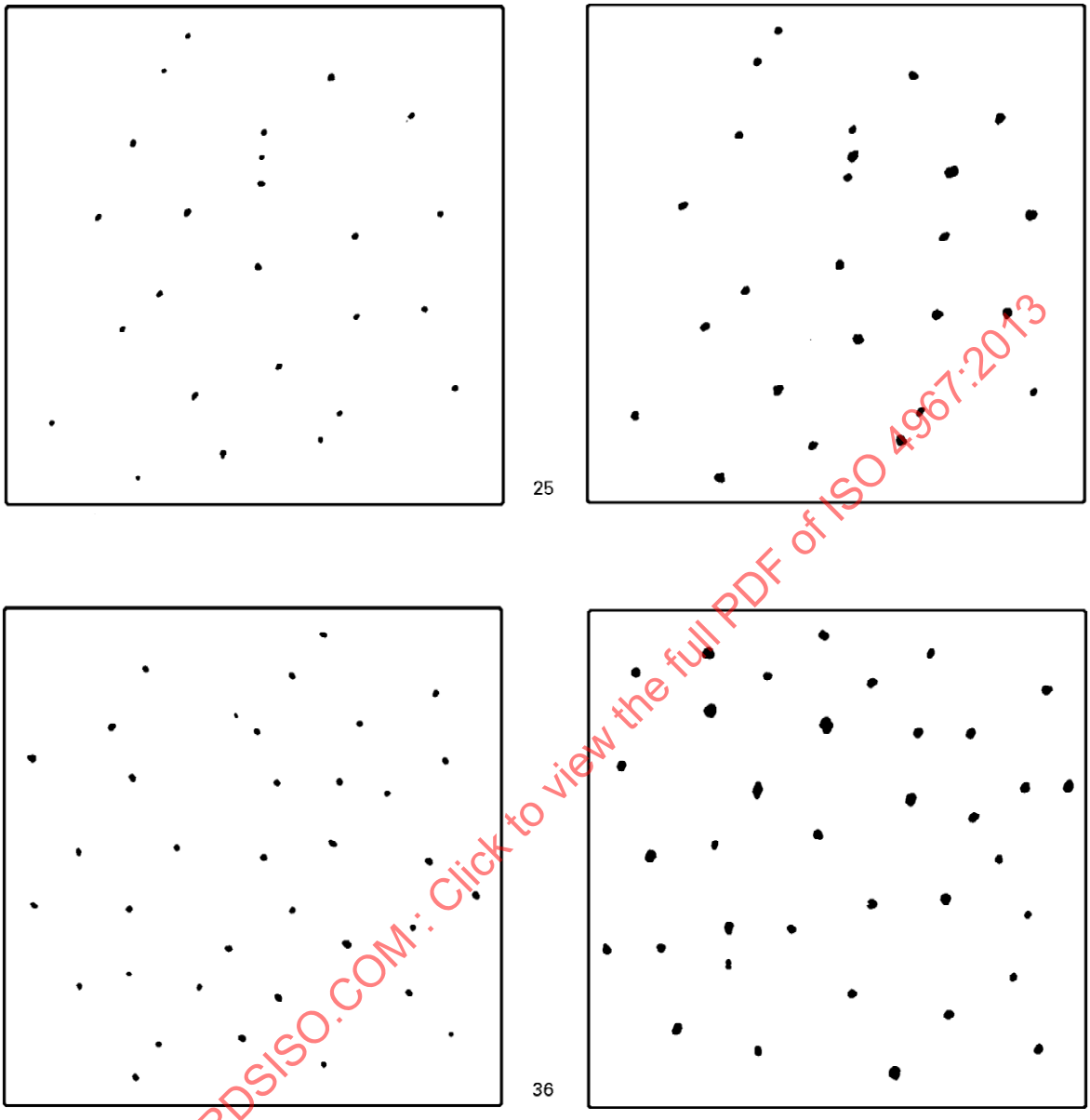
Fine series		Thick series
Thickness > 3 µm to 8 µm	Minimum number of inclusions	Thickness > 8 µm to 13 µm
i = 0,5	1	
i = 1	4	



Magnification = 100 ×



Magnification = 100 ×



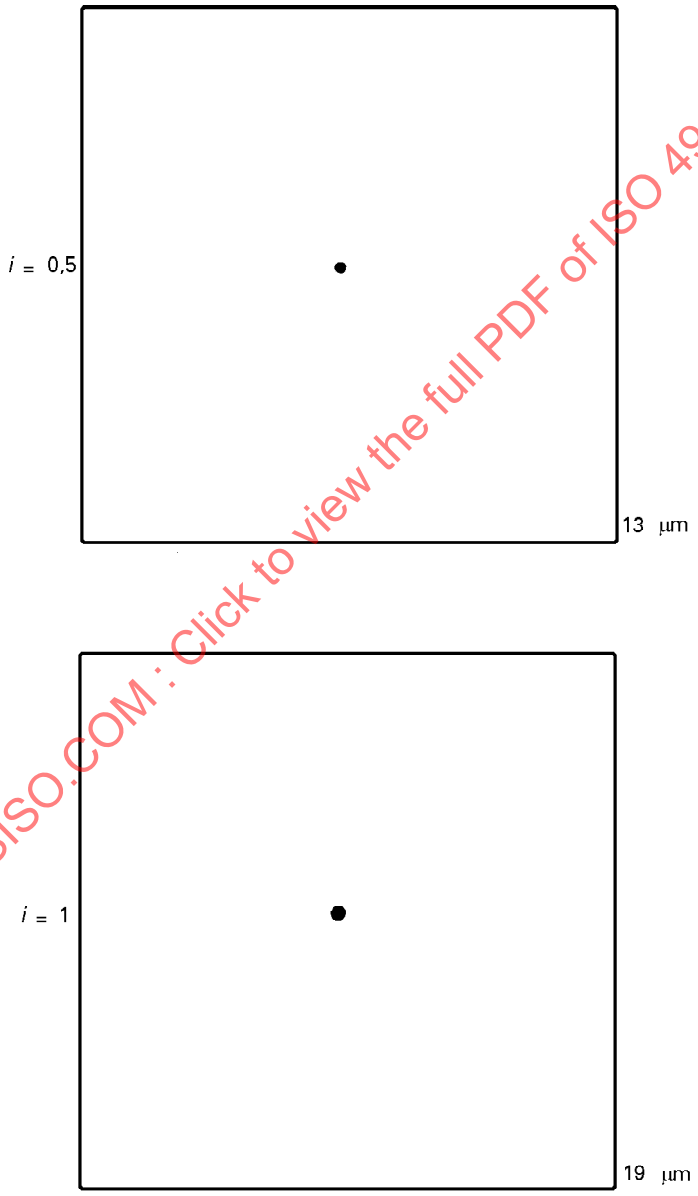
Magnification = 100 ×

DS

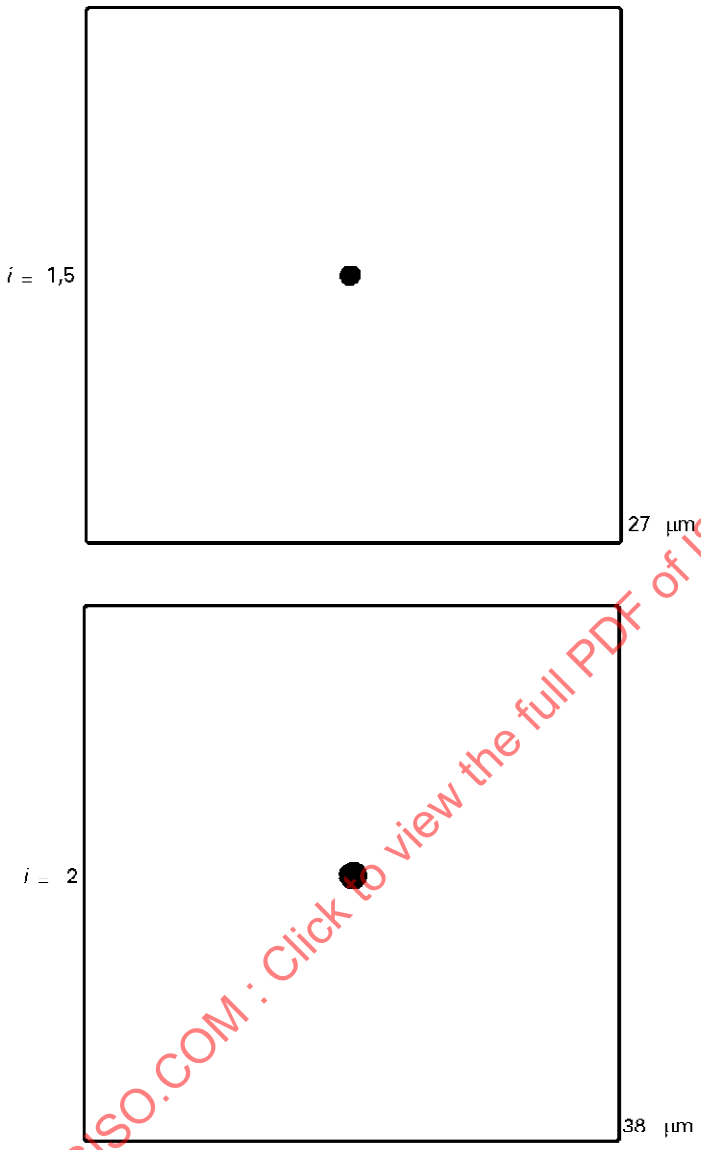
(Globular type oxides)

Diameter > 13 µm to 76 µm

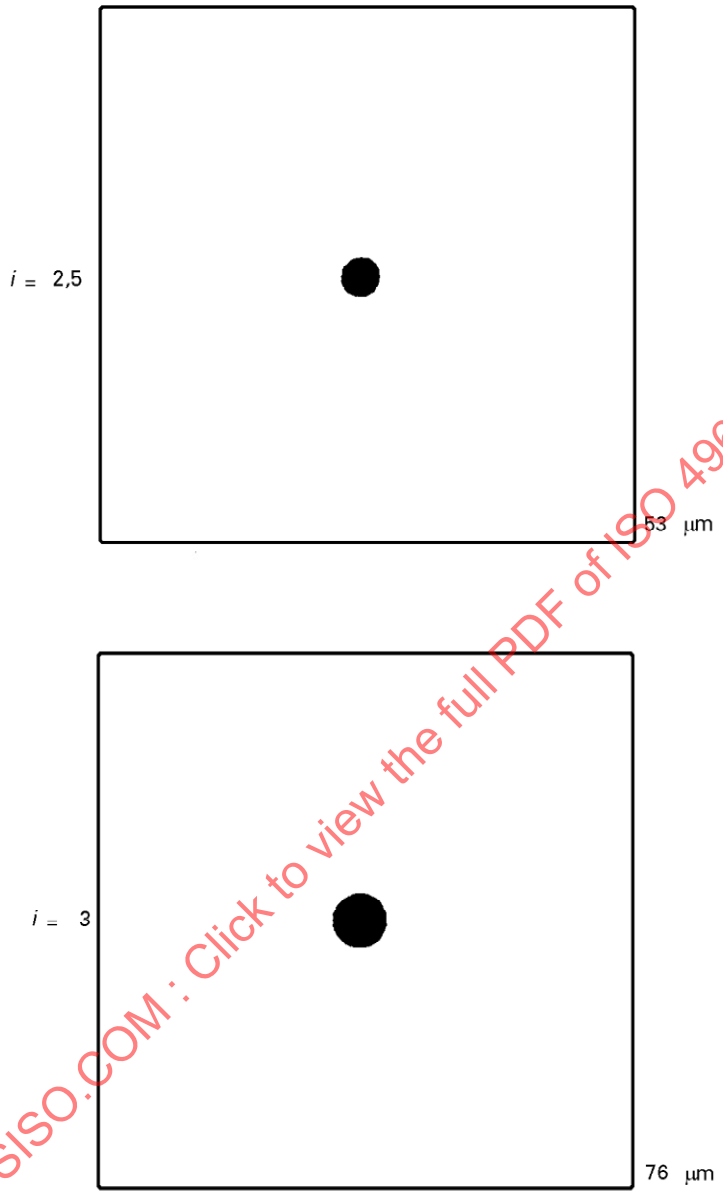
Minimum diameter



Magnification = 100 ×



Magnification = 100 ×

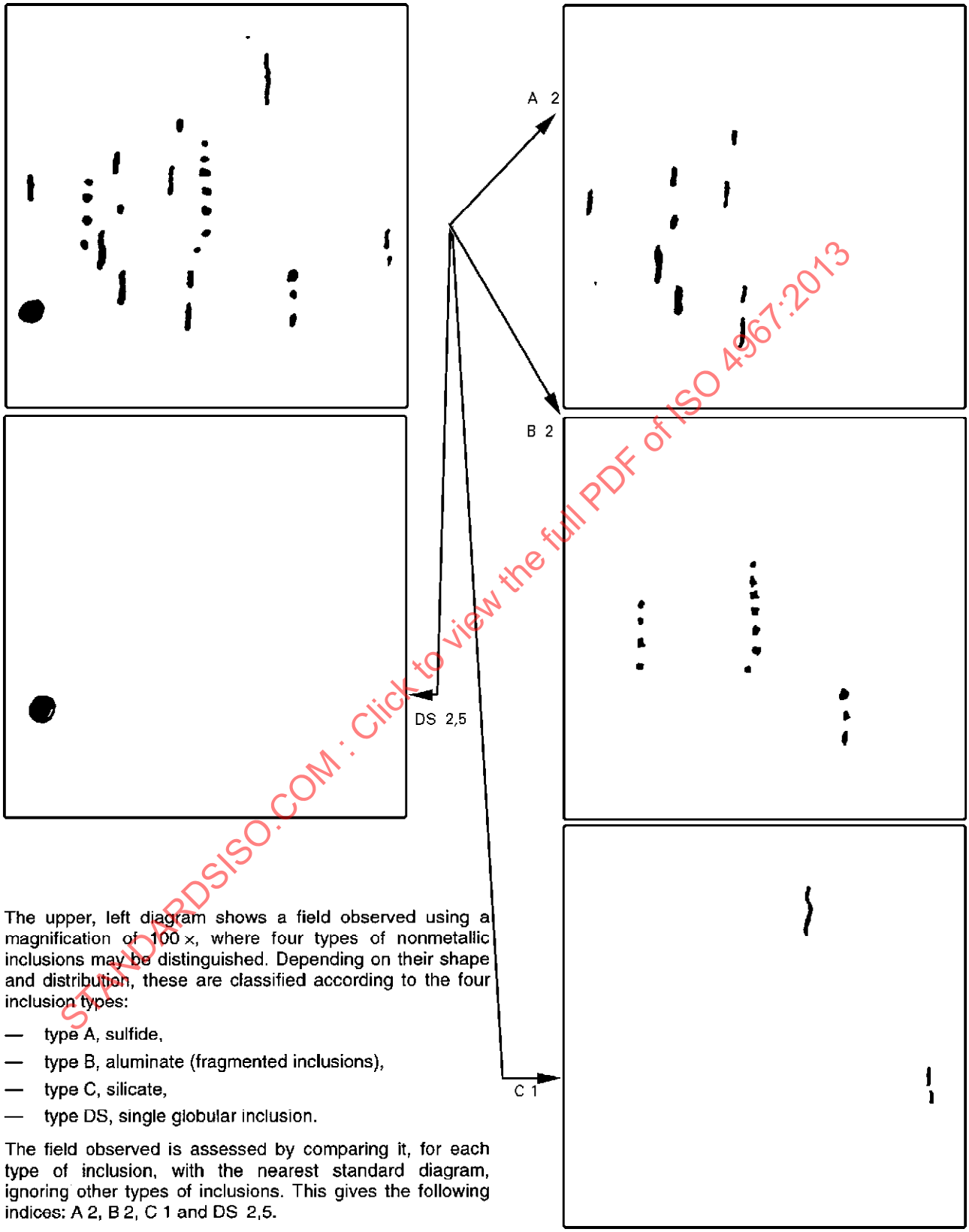


Magnification = 100 ×

Annex B
(informative)

Assessment of a field and of oversized inclusions or stringers

B.1 Example of assessment of a field



The upper, left diagram shows a field observed using a magnification of 100 ×, where four types of nonmetallic inclusions may be distinguished. Depending on their shape and distribution, these are classified according to the four inclusion types:

— type A, sulfide,
— type B, aluminate (fragmented inclusions),
— type C, silicate,
— type DS, single globular inclusion.

The field observed is assessed by comparing it, for each type of inclusion, with the nearest standard diagram, ignoring other types of inclusions. This gives the following indices: A 2, B 2, C 1 and DS 2,5.

Figure B.1 — Field assessment

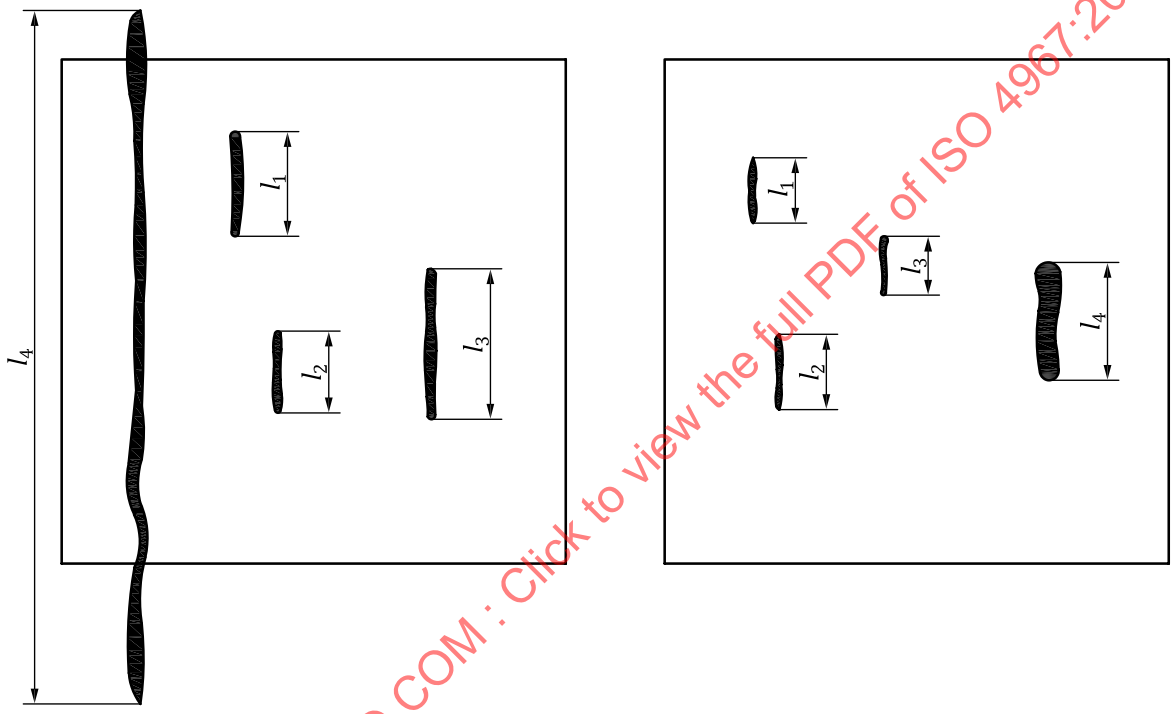
B.2 Example of assessment of oversized inclusions or stringers

If the inclusion or stringer is oversized by length only, the portion of the inclusion or stringer within the field for method B, or 0,710 mm for method A, will be added to the length of any other inclusions of the same type and thickness in the same field (see Figure B.2 a).

If the inclusion or stringer is oversized by width or diameter (type D inclusions), the inclusion should be part of the thick series rating for that field (see Figure B.2 b).

For the type D inclusion, if the number of particles is greater than 49, the index can be calculated from the equation given in Annex D.

For a DS type inclusion with a diameter greater than 0,107 mm, the index can be calculated from the equation given in Annex D.



The field rating is based on the total length L

$L = 0{,}71 + l_1 + l_2 + l_3$

with separate indication of oversized length l_4

a) For inclusions or stringers oversized by length only

The field rating is based on the total length L

$L = l_1 + l_2 + l_3 + l_4$

with separate indication of oversized width l_4

b) For inclusions or stringers oversized by width or diameter

Figure B.2 — Field assessment with oversized inclusions on stringers

Annex C
(informative)

Typical example of results (the total number of fields showing the index, by type of inclusion, for a given number of fields observed)

C.1 Indices according to field and type of inclusion

Table C.1 gives an example of the results of an assessment of this type, for a total of 20 observed fields, in order to simplify the lay-out of the examples. In general, a minimum of 100 fields are examined.

Table C.1 — Indices

	Type of inclusion								
Field	**A**		**B**		**C**		**D**		**DS**
	Fine	thick	fine	thick	fine	thick	fine	thick	
1	—	0,5	1	—	0,5	—	—	—	—
2	0,5	—	—	—	0,5	—	—	—	—
3	0,5	—	0,5	—	—	0,5	—	—	0,5
4	1	—	—	0,5	1,5	—	—	0,5	—
5	—	—	—	1,5	—	1	—	—	—
6	1,5	—	—	—	—	—	0,5	—	1
7	—	1s	1,5	—	—	0,5	—	—	—
8	—	—	—	1	1	—	—	1	—
9	0,5	—	0,5	—	0,5	—	—	—	—
10	—	0,5	1	—	0,5	—	—	—	—
11	1	—	0,5	—	—	0,5	—	—	1
12	0,5	—	—	—	—	—	—	1s	—
13	—	—	—	0,5	—	1,5	1	—	—
14	2	—	—	1	—	—	—	—	—
15	—	—	—	—	0,5	—	—	—	—
16	0,5	—	1	—	—	1	—	—	—
17	0,5	—	0,5	—	—	—	—	0,5	1,5
18	—	—	—	1,5	1	—	—	—	—
19	—	2	—	3	0,5	—	0,5	—	—
20	—	—	0,5	—	—	0,5	—	—	—

C.2 Total number of fields per index according to type of inclusion

On the basis of these results it is possible to determine the total number of fields for the various indices and inclusion types. Table C.2 below gives the values for the total number of fields.

Table C.2 — Total number of fields

Index	Type of inclusion								
	A		B		C		D		DS
	fine	thick	Fine	thick	fine	thick	fine	thick	
0,5	6	2	5	2	6	4	2	2	1
1	2	1	3	2	2	2	1	2	2
1,5	1	0	1	2	1	1	0	0	1
2	1	1	0	0	0	0	0	0	0
2,5	0	0	0	0	0	0	0	0	0
3	0	0	0	1	0	0	0	0	0

NOTE Inclusions having a length greater than the dimension of the field, or a width or a diameter greater than indicated in Table 2 shall be rated using the standard diagrams and reported separately in the test report.

C.3 Calculation of total index, i_{tot}, and mean index, i_{moy}

Using the total numbers of fields given in Table C.2, it is possible to calculate the corresponding total and mean indices for each inclusion type and each series.

C.3.1 For type A inclusions

a) Fine series

$i_{tot} = (6 \times 0{,}5) + (2 \times 1) + (1 \times 1{,}5) + (1 \times 2) = 8{,}5$

$$i_{moy} = \frac{i_{tot}}{N} = \frac{8{,}5}{20} = 0{,}425$$

where N is the total number of fields observed (see 6.3).

b) Thick series

$i_{tot} = (2 \times 0{,}5) + (1 \times 1) + (1 \times 2) = 4$

$$i_{moy} = \frac{4}{20} = 0{,}20$$ with indication of 1 s

C.3.2 For type B inclusions

a) Fine series

$i_{tot} = (5 \times 0{,}5) + (3 \times 1) + (1 \times 1{,}5) = 7$

$$i_{moy} = \frac{7}{20} = 0{,}35$$

b) Thick series

$i_{tot} = (2 \times 0{,}5) + (2 \times 1) + (2 \times 1{,}5) + (1 \times 3) = 9$

$$i_{moy} = \frac{9}{20} = 0{,}45$$

C.3.3 For type C inclusions

a) Fine series

$i_{tot} = (6 \times 0,5) + (2 \times 1) + (1 \times 1,5) = 6,5$

$$i_{moy} = \frac{6,5}{20} = 0,325$$

b) Thick series

$i_{tot} = (4 \times 0,5) + (2 \times 1) + (1 \times 1,5) = 5,5$

$$i_{moy} = \frac{5,5}{20} = 0,275$$

C.3.4 For type D inclusions

a) Fine series

$i_{tot} = (2 \times 0,5) + (1 \times 1) = 2$

$$i_{moy} = \frac{2}{20} = 0,10$$

b) Thick series

$i_{tot} = (2 \times 0,5) + (2 \times 1) = 3$

$$i_{moy} = \frac{3}{20} = 0,15$$ with indication of 1 s

C.3.5 For type DS inclusions

$i_{tot} = (1 \times 0,5) + (2 \times 1) + (1 \times 1,5) = 4$

$$i_{moy} = \frac{4}{20} = 0,20$$

C.4 Weighting factor

It is possible to use a weighting factor for each index number in order to calculate global cleanness index based on the amount of inclusions.

The weighting factors given in Table C.3 may be used: