International Standard

ISO 4967

Steel — Determination of the non-metallic inclusion content — Micrographic method

Acier — Détermination de la teneur en inclusions non métalliques — Méthode micrographique

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis)*.

This fourth edition cancels and replaces the third edition (ISO 4967:2013), which has been technically revised.

The main changes are as follows:

— added the mandatory clauses normative references (see Clause 2) and terms and definitions (see Clause 3), and renumbered the subsequent clauses;

— modified the proximity conditions for stringers (allowing for legacy conditions): the new transverse conditions mirror the conditions used longitudinally and remove ambiguity;

— changed the width definition (allowing for legacy/alternative definitions): the new definition avoids the sensitivity to misalignment of the bounding box and the underestimation of the “largest particle” approach;

— added further illustrations of width definitions, including the largest particle approach to inclusions with overlapping particles;

— clarified the treatment of inclusions intersecting the field of view, particularly for long inclusions (allowing for legacy treatment);

— clarified the treatment of B/C hybrid stringers;

— modified Tables 2 and 3;

— added sampling specifications and the possibility to use stacking and/or rectangular fields for cross-section thicknesses under 0,71 mm;

— included the DS subgroup into D thick rating;

— clarified the treatment of DS inclusions in Method B;

— clarified the averaging of cross sections in Method A;

— modified most of the chart diagrams;

— replaced the global metrics for Method B (allowing for legacy metrics);

— added more analysis examples.

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

Every routine inclusion rating by necessity applies the analysis of an incomplete sample to an entire heat. On the one hand, only a very small fraction of the total material volume is analysed, and on the other hand the analysis is performed on a two-dimensional section of three-dimensional inclusions. Standards like this document cannot eliminate the associated statistical uncertainties but can strive to add as little uncertainty as possible by defining the process as clearly as possible.

Despite the statistical shortcomings, methods like those described in this document are widely used to assess the suitability of a steel product for a given use. However, since it is difficult to achieve reproducible results owing to the distributional randomness of non-metallic inclusions, even with a large number of specimens, precautions should be taken when using the method.

One way to reduce the scatter inherent to the method is to avoid relying on subjective human judgment. Image analysis has shown itself to be a useful tool to improve reproducibility — if the hardware is appropriately configured and if the rules in the standard are indisputably clear for the software developer. This document addresses the minimal system requirements for resolution and reduces ambiguity in its rules compared to the previous revision.

However, it is acknowledged that neither steel producers and customers can instantly change specifications, nor can software developers immediately change the rules for evaluation. To allow for an adaptation period, where methods and definitions have changed, it is permitted to continue to use the methods and definitions that have been established in the past. This document refers to such methods and definitions as well as derived interpretations as “legacy.” Because of the ambiguities of previous editions, there is no one legacy approach, but instead a variety of legacy approaches.

Another clarification relative to the 2013 revision concerns the DS inclusions. There was much ambiguity surrounding them because they were presented as another type of inclusions. This made it unclear whether large globular particles were part of the D rating as well, since one important rule of inclusion rating is to rate every inclusion once and only once. With the redefinition of DS as a subgroup of type D designed for easy rating and reporting of oversized type D inclusions it is clear that every DS particle is rated in the D_{thick} rating, just as every oversized sulfide is rated in the A_{thick} rating.

Historically, ISO 4967 has always shown a significant similarity to the ASTM E 45 standard. With the revised definitions, particularly those defining proximity limits, there is a greater separation between the standards, though due to the inherent statistical uncertainties the ratings will correlate in most instances. However, these increased differences convinced the ISO/TC 17/SC 7 to continue using the terms “fine” and “thick” in order to more clearly distinguish ISO 4967 results from ASTM E 45 results.

Revisions always take place on a strict timeline and often the deadline forces the publication of a standard that is good enough, but not yet perfect. Topics that further revisions can address include the treatment of particle clusters, easier oversized reporting for Types A to C, and more guidelines on computer-assisted rating.

It is worth remembering that the changes in this inclusion rating method do not change a good steel into a bad steel, but serve the goal of a clearer, more differentiated description of the steel.

Steel — Determination of the non-metallic inclusion content — Micrographic method

1 Scope

This document 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 the images of a standard reference chart or direct measurement by image analysis technologies.

The standard reference chart described in this document is not entirely applicable for certain types of steel (e.g. free cutting steels).

2 Normative references

There are no normative references in this document.

3 Terms and definitions

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

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

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

— IEC Electropedia: available at www.electropedia.org

3.1
particle
single precipitate, in general non-metallic

3.2
stringer
arrangement of at least 2 particles for type A and C inclusions and 3 particles for type B, aligned in a plane parallel to the hot working axis and offset by no more than 10 µm, with a separation of no more than 40 µm between any two nearest neighbour particles

3.3
inclusion
general designation of a rateable feature composed of at least one particle, defined by the size and proximity of its constituents

Note 1 to entry: The inclusion can describe a single particle or a single stringer.

3.4
length
l
dimension of a particle or an inclusion in the main deformation direction, usually larger than the width

3.5
width
w
largest local dimension of a particle or an inclusion measured perpendicular to the main deformation direction (calliper width), as shown in Figure 1

Note 1 to entry: The calliper width clarifies an ambiguity in previous editions. A "largest particle" and a "bounding box" approach were among the most frequent alternatives. Further illustrations are shown in Annex D.

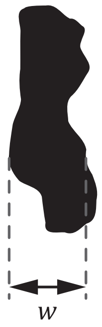


Figure 1 — Schematic of particle and inclusion width

3.6
aspect ratio
ratio of length to width

3.7
diameter
d
dimension of a globular particle or inclusion in the main deformation direction

3.8
worst-field rating
rating in which the specimen is rated for each group and subgroup of inclusion by assigning the value for the highest severity rating observed of that inclusion group and subgroup anywhere on the evaluated area of the specimen

3.9
type
categorization of inclusions according to morphology, colour and proximity

3.10
class
categorization by width or diameter within types (fine and thick)

3.11
group
categorization by type and class

3.12
globular particle
particle with an aspect ratio less than 3

3.13
elongated particle
particle with an aspect ratio more than or equal to 3

3.14
hybrid stringer
stringer consisting of both globular and elongated particles

3.15
reduction ratio
ratio between original and final cross-sectional area after rolling or forging

3.16
calibration factor
parameter indicating the actual size on the specimen surface corresponding to the pixel length

4 Symbols

The symbols used are given in Table 1.

Table 1 — Symbols

Symbols	Definitions	Values
A	Area of an individual inclusion A bar above the symbol indicates an average. Superscripts used: variable index i, "excess", variable class Subscripts used: selected type/class/group, "tot" (total), S (specimen), "ox" ("oxidic"), "glob" (globular), fixed index (e.g. "i = 0,5")	$A=\frac{\pi}{4}\left(l\times w\right)$
t	Wall thickness, as shown in Figure 9	
C_{i}	Weighted global severity indices Superscripts used: selected type/class/group, "tot" (total) The subscript "i" stands for "inclusion" and is not the index number.	
d	Dimension of a globular particle or inclusion in the main deformation direction A bar above the symbol indicates an average. Superscripts used: variable class c, variable index i Subscripts used: "min", "max", and selected type/class/group	
e	Longitudinal distance between particles as shown in Figures 11 to 12	
f	Inclusion area fractions Subscripts used: "tot" (total), "ox" ("oxidic"), "glob" (globular)	
f_{number}	Inclusion number frequency Superscripts used: selected type/class/group	
i	Index	
l	Dimension of a particle or an inclusion in the main deformation direction, usually larger than the width A bar above the symbol indicates an average. Superscripts used: index i Subscripts used: "min", "max"	
n	Number of type D globular oxides per field A bar above the symbol indicates an average. Superscripts used: index i Subscripts used: "min"	
N	Number of rated fields	
r	Width of plate as shown in Figures 4 to 6	

Table 1 *(continued)*

Symbols	Definitions	Values
R	Relative area fractions Superscripts used: selected type/class/group or combination thereof such as “ox” (“oxidic”) Subscripts used: Ratio expressed, e. g. “B:C”, “f:t” (fine to thick), “G:nG” (globular to elongated)	
s	Transverse distance between particles as shown in Figures 11 to 12	
S	Specimen area	
w	Dimension of a particle or an inclusion perpendicular to the main deformation direction A bar above the symbol indicates an average. Superscripts used: class c, index i Subscripts used: “min”, “max”, selected type/class/group	
x	Inclusion interdistance Subscripts used: selected type/class/group	
Legacy global metrics		
C_t	Cleanness index (“t” stands for “total”)	$C_t = \left(\sum_{i=0} n_i \times F_i\right) \times \frac{1\,000}{S}$
F_i	Weighting factor of index *i*, always rounded to one significant digit	$F_i = 10^{\frac{i-2,5}{1,5}}$
i_{moy}	Mean index for the entire assessed surface	$i_{moy} = \frac{i_{tot}}{N}$
i_{tot}	Total index for the entire assessed surface	$i_{tot} = \sum_{i=0} i \times n_i$
n_i	Total number of fields (A, B, C, and D) and inclusions (DS) rated as index *i*	

5 Principle

5.1 The method consists of comparing the observed field to the chart images defined in this document and taking in consideration separately each group and subgroup of inclusions. In the case of image analysis, fields are rated according to Table 2, Table 3, and the relationships given in Annex F. Automatic image analysis can be used provided that the accuracy of the method has previously been validated. Digitized images should have a calibration factor of 1 µm/pixel or preferably finer.

5.2 The chart images 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 with bright field illumination at 100×.

5.3 According to the grey level, shape, and distribution of the inclusions, the chart images are divided into four main types, bearing the reference A, B, C, and D. These four types represent the most commonly observed inclusion types and morphologies:

— **Type A (“sulfide” type)**: highly malleable, individual light grey elongated particles with a wide range of aspect ratios and generally rounded ends;

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

— **Type C (“silicate” type):** highly malleable, individual black or dark grey elongated particles with a wide range of aspect ratios (≥ 3) and generally smooth outlines, often with sharp ends;

— **Type D (globular “oxide” type):** non deformable, angular or circular, low aspect ratio (< 3), black or bluish, randomly distributed particles.

NOTE The chemical composition of the inclusions present in a steel sample cannot be determined with the methods described in this document. The apparent chemical names attributed to the types A, B, C, and D derive from the typical composition historically found when analysing inclusions of such morphology and colour. The same is true for the non-traditional inclusion types in the paragraphs that follow.

5.4 Non-traditional inclusion types may also be rated based on their morphology compared to the above four types and a statement about their apparent chemical nature. As an example, globular sulfides would be rated as a D type and denoted with a descriptive subscript (e.g. D_{sulf}) defined in the test report. Examples of subscripts include D_{cas} (globular calcium sulfides); D_{RES} (globular rare earth sulfides); and D_{Dup} (globular duplex inclusions, such as calcium sulfide surrounding an aluminate). The treatment of complex inclusions should be separately agreed between the parties. Examples are given in Annex C.

5.5 Types of precipitate, such as borides, carbides, carbonitrides or nitrides may also be rated based on their morphology compared to the above four types and a statement about their apparent chemical nature as described in 5.4. Examination at a magnification greater than 100 × may be used to identify the nature of the non-traditional inclusions before performing the test.

5.6 The categorization of inclusions into types A, B, C and D is based on their grey level and then, after forming stringers, on their morphologies. Each of the four types shall be further categorized into two classes based on their width or diameter, as specified in Annex A, with six images representing increasing inclusion content. The images for indices 3,5 and higher and the limit values for indices 5,5 and higher are not given in this document. For simplifying the frequent oversized reporting for Type D inclusions with a diameter > 13 µm an additional subgroup DS is defined. Oversized Type D inclusions are rated as D_{thick} and also as DS.

5.7 The chart images in Annex A carry an index number, *i*, from 0,5 to 3, the numbers increasing with the inclusion lengths (Types A, B, C) or by the number (Type D) or by the diameter (Subgroup DS), as defined in Table 2, and categorized by thickness, as defined in Table 3. The total length of inclusions, or number of inclusions, or diameter of the inclusion in each chart image is the lower boundary value of Table 2. For example, the images for A 2 depict inclusions with a morphology in accordance with type A and with the-total length corresponding to the lower boundary value of *i* = 2. For indices larger than 3,0, the rating is performed by comparing measured values with Table 2.

Table 2 — Inclusion rating limits

Index *i*	Type				Subgroup
	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
1,0	≥ 127	≥ 77	≥ 76	≥ 4	≥ 19
1,5	≥ 261	≥ 184	≥ 176	≥ 9	≥ 27
2,0	≥ 436	≥ 343	≥ 320	≥ 16	≥ 38
2,5	≥ 649	≥ 555	≥ 510	≥ 25	≥ 53
3,0	≥ 898	≥ 822	≥ 746	≥ 36	≥ 76
3,5	≥ 1 181	≥ 1 147	≥ 1 029	≥ 49	≥ 107

Table 2 *(continued)*

Index *i*	**Type**				**Subgroup**
	A total length µm	**B** total length µm	**C** total length µm	**D** count number	**DS** diameter µm
4,0	≥ 1 498	≥ 1 530	≥ 1 359	≥ 64	≥ 151
4,5	≥ 1 848	≥ 1 973	≥ 1 737	≥ 81	≥ 214
5,0	≥ 2 230 (< 2 641)	≥ 2 476 (< 3 042)	≥ 2 163 (< 2 639)	≥ 100 (< 121)	≥ 303 (< 429)
⋮	⋮	⋮	⋮	⋮	⋮

Table 3 — Inclusion width and diameter parameters

Type	**Fine class**		**Thick class**	
	µm	µm	µm	µm
A (width)	≥ 2	≤ 4	> 4	≤ 12
B (width)	≥ 2	≤ 9	> 9	≤ 15
C (width)	≥ 2	≤ 5	> 5	≤ 12
D (diameter)	≥ 2	≤ 8	> 8	≤ 13
NOTE Type D inclusions with a width less than 2 µm are also not included in the inclusion rating.				

6 Sampling

6.1 The shape of the inclusion depends to a large extent on the reduction ratio of the steel; therefore, comparative measurements should only be carried out on prepared specimens taken from samples with a similar reduction ratio.

6.2 Unless something else is defined in the product standard or agreed by the parties involved, the polished surface of the specimen used to determine the content of inclusions should be about 200 mm^2.

6.3 When the cross-section thickness is insufficient to prepare a single specimen of 200 mm^2, more than one specimen shall be taken from the same sampling location to conform to 6.2. Where reaching 200 mm^2 or more is onerous, the total length of the longitudinal pieces taken from each sampling location shall not be less than 100 mm. These specimens shall be analysed as one whole specimen.

6.4 In general, single sections less than 0,71 mm in thickness are not analysed using this document, since this is restricted by the field side length. However, as part of a mutual agreement, narrower sections may be assessed by

a) stacking two or more sections together and thereby creating a thicker aggregate section, making sure that no small gaps or edge artefacts between the stacked sections are categorised as inclusions

b) using a rectangular field as long as the field area is 0,5 mm^2, e.g. 1,0 mm×0,5 mm, provided that the rectangular field fits within the visual field of the microscope at the applied magnification.

6.5 The method of sampling, including the sampling location and the number of sampling locations, shall be defined in the product standard or subject to agreement between the parties.

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

— bar, wire rod, or wire 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 conform to 6.2 (see Figure 2);

— bar, wire rod, wire, or billet with a diameter greater than 25 mm and less than or equal to 40 mm: the surface to be examined consists of at least half the diametral section (from the centre to the edge of the sample) (see Figure 3);

— bar, wire rod, wire, 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 4);

— 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 5);

— plate with a thickness greater than 25 mm and less than or equal to 40 mm: the surface to be examined consists of at least half the thickness from the surface to the centre and is located at the quarter of the width (see Figure 6);

— plate with a thickness greater than 40 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 7);

— tube or pipe with a wall thickness less than or equal to 25 mm: the surface to be examined consists of the full diametral section of a length sufficient to obtain a sufficient surface, and, for welded products, located far away from the welding bead (see Figure 8);

— tube or pipe with a wall thickness greater than 25 mm: the surface to be examined consists of a part of the diametral section located halfway between the outer diameter and the inner diameter, and, for welded products, far away from the welding bead (see Figure 9).

6.7 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.

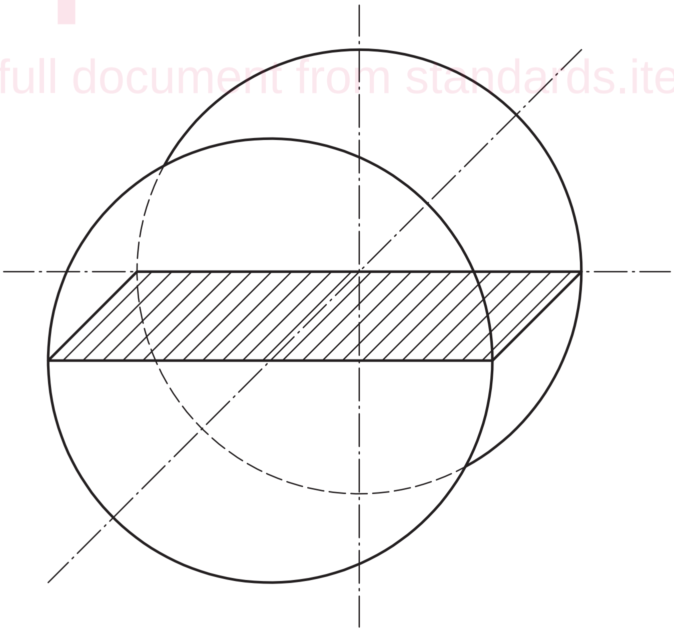


Figure 2 — Sample from bar with a diameter ≤ 25 mm

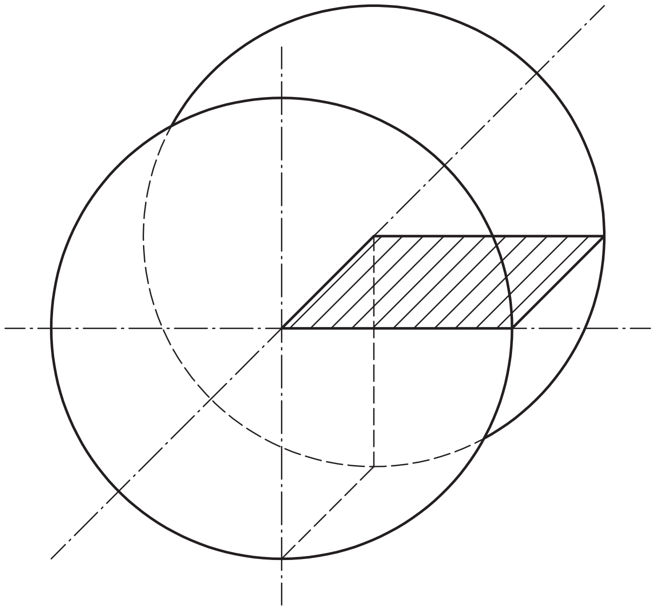


Figure 3 — Sample from bar or billet with a diameter or length of side > 25 mm and ≤ 40 mm

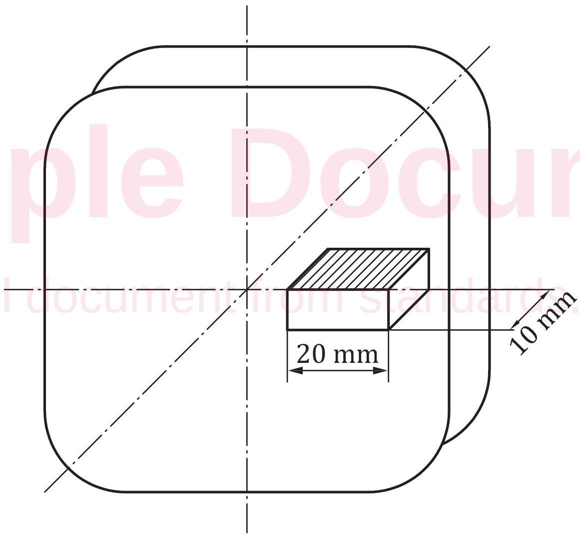
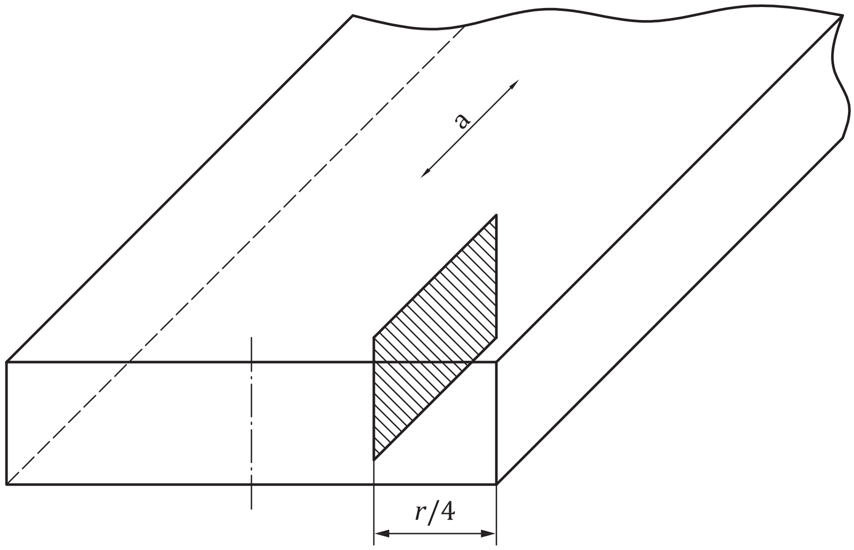


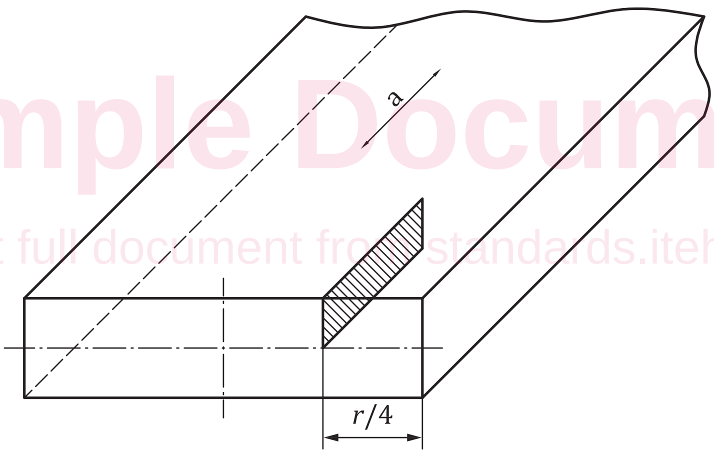
Figure 4 — Sample from bar or billet with a diameter or length of side > 40 mm



Key

r	width
a	rolling direction

Figure 5 — Sample from plate with thickness ≤ 25 mm



Key

r	width
a	rolling direction

Figure 6 — Sample from plate with thickness > 25 mm and ≤ 40 mm