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Fifth edition
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Steels — Micrographic determination of the apparent grain size

Aciers — Détermination micrographique de la grosseur de grain apparente

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Contents

Page

iTeh Standards
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Document Preview
ISO 643:2024
https://standards.iteh.ai/catalog/standards/iso/d0973674-eda1-4a15-989c-c00528a5a1f3/iso-643-2024

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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 document 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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This document was prepared by Technical ISO/TC 17, *Steel*, Subcommittee SC 7, *Methods of testing (other than mechanical tests and chemical analysis),* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 459, *ECISS - European Committee for Iron and Steel Standardization,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This fifth edition cancels and replaces the fourth edition (ISO 643:2019), which has been technically revised.

The main changes are as follows:

— the test temperature of McQuaid-Ehn method has been modified for case hardening steels to 950 °C (see A.4);

— subclause 7.2 has been modified with reference to new Annex B and amended Table 2;

— Annex B from the third edition (ISO 643:2012) has been reinstated, now with new ISO grain size charts instead of ASTM charts;

— parts of the old Annex B (evaluation method) have been revised and moved to the main body of the standard (subclause 7.3) and the remainder of the annex has been renumbered as Annex C;

— new Annexes D and E have been added.

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Document Preview
ISO 643:2024
https://standards.iteh.ai/catalog/standards/iso/d857b6f4-eda1-4af5-9b9e-c0602b9a3a13/iso-643-2024

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Steels — Micrographic determination of the apparent grain size

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1 Scope

This document specifies micrographic methods of determining apparent ferritic or austenitic grain size in steels. It describes the methods of revealing grain boundaries and of estimating the mean grain size of specimens with unimodal size distribution. Although grains are three-dimensional in shape, the metallographic sectioning plane can cut through a grain at any point from a grain corner, to the maximum diameter of the grain, thus producing a range of apparent grain sizes on the two-dimensional plane, even in a sample with a perfectly consistent grain size.

2 Normative references

There are no normative references in this document.

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3 Terms and definitions

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

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

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

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

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3.1 Grains

3.1.1
grain
closed polygonal shape with more or less curved sides, which can be revealed on a flat section through the sample, polished and prepared for micrographic examination

Note 1 to entry: In ISO 4885[1] grain is defined as “space lattice formed by atoms with regular interstices".

Note 2 to entry: If any other constituent (e.g. pearlite) of similar dimensions to the grains of interest is present, that constituent can be counted as grains of interest.

3.1.2
austenitic grain
crystal with a face-centred cubic crystal structure which may, or may not, contain annealing twins

3.1.3
ferritic grain
crystal with a body-centred cubic crystal structure which never contains annealing twins

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3.2 General

3.2.1
index
positive, zero or possibly negative number G which is derived from the mean number m of *grains* (3.1.1) counted in an area of 1 mm^2 of the section of the specimen

Note 1 to entry: By definition, $G = 1$ where $m = 16$; the other indices are obtained by Formula (1).

3.2.2
intercept
N
number of *grains* (3.1.1) intercepted by a test line, either straight or curved

Note 1 to entry: See Figure 1.

Note 2 to entry: Straight test lines will normally end within a grain. These end segments are counted as 1/2 an intercept. $\bar{N}$ is the average of a number of counts of the number of grains intercepted by the test line applied randomly at various locations. $\bar{N}$ is divided by the true line length, L_T usually measured in millimetres, in order to obtain the number of grains intercepted per unit length, $\bar{N}_L$.

3.2.3
intersection
P
number of intersection points between *grain* (3.1.1) boundaries and a test line, either straight or curved

Note 1 to entry: See Figure 2.

Note 2 to entry: $\bar{P}$ is the average of a number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations. $\bar{P}$ is divided by the true line length, L_T usually measured in millimetres, in order to obtain the number of grain boundary intersections per unit length, $\bar{P}_L$.

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4 Symbols

The symbols used are given in Table 1.

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Table 1 — Symbols

Symbols	Definition	Value
$\bar{a}$	Mean area of grain in square millimetres	$\bar{a} = \frac{1}{m}$
A_B	True area of the test box	mm^2
A_C	True area of the test circle	mm^2
A_F	Apparent area of the test figure in square millimetres	—
$\bar{d}$	Mean grain diameter in millimetres	$\bar{d} = \frac{1}{\sqrt{m}}$
D	Diameter of the circle on the ground glass screen of the microscope or on a photomicrograph enclosing the image of the reference surface of the specimen	79,8 mm (area = 5 000 mm^2)
g	Linear magnification (to be noted as a reference) of the microscopic image	In principle 100
G	Equivalent index of grain size	$G = \log_2 m - 3$
l	Mean lineal intercept length, generally expressed in millimetres	$l = 1/\bar{N}_L = 1/\bar{P}_L$
l_0	Mean lineal intercept length for $G = 0$, in millimetres	0,32
L_T	True length of the test line divided by the magnification, in millimetres	—
a The method for designating the direction conforms to ISO 3785[2].		

Table 1 *(continued)*

Symbols	Definition	Value
m	Number of grains per square millimetre of specimen surface in the area examined	$m = n_t/A_C$ $m = n_t/A_B$
M	Number of the closest standard chart picture where g is not 100	—
n_e	Number of grains completely inside the circle of diameter D	—
n_i	Number of grains intersected by the circle of diameter D	—
n_t	Total equivalent number of grains examined on the image of diameter D	—
$\bar{N}$	Mean number of grains intercepted per unit length L	—
$\bar{N}_L$	Mean number of grains intercepted per unit length of the line	$\overline{N_L} = \bar{N} / L_T$
N_x	Number of intercepts per millimetre in the longitudinal direction[a]	—
N_y	Number of intercepts per millimetre in the transverse direction[a]	—
N_z	Number of intercepts per millimetre in the perpendicular direction[a]	—
$\bar{P}$	Mean number of counts of the number of grain boundaries intersected by the test line applied randomly at various locations	—
$\bar{P}_L$	Mean number of grain boundary intersections per unit length of test line	$\overline{P_L} = \bar{P} / L_T$
Q	Correction factor for non-standard magnification	$Q = 2log_2\left(\frac{g}{100}\right)$

a The method for designating the direction conforms to ISO 3785[2].

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5 Principle

This document is applicable to grain structures that have a unimodal size distribution. The apparent grain size is determined by micrographic examination of appropriately prepared sections of the specimen.

The following principal methods are available to obtain an index representing the mean value of the grain size:

a) comparison method using standard charts (see 7.2);

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b) planimetric method counting grains to determine the mean number of grains per unit area, (see 7.3);

c) intercept method counting the number of grains or grain boundaries along a line of a known length (see 7.4).

All methods give comparable results.

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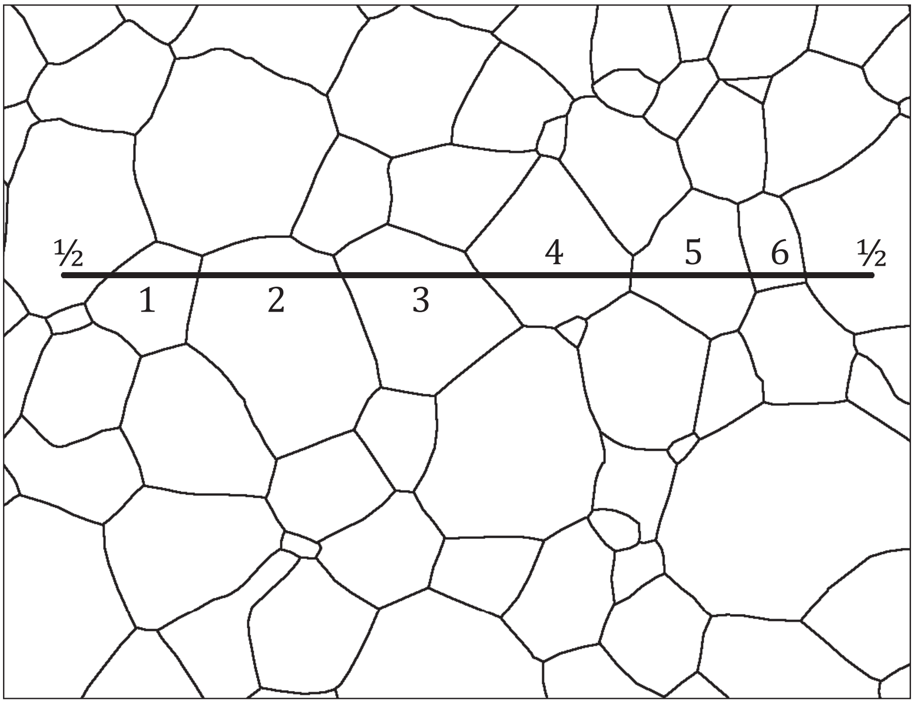

Figure 1 — Example of intercept, *N*

Intercept, *N*, grain counts for a straight line on a single-phase grain structure. Six intercepts and two line segments ending within a grain equals 2 × 1/2 + 6 = 7.

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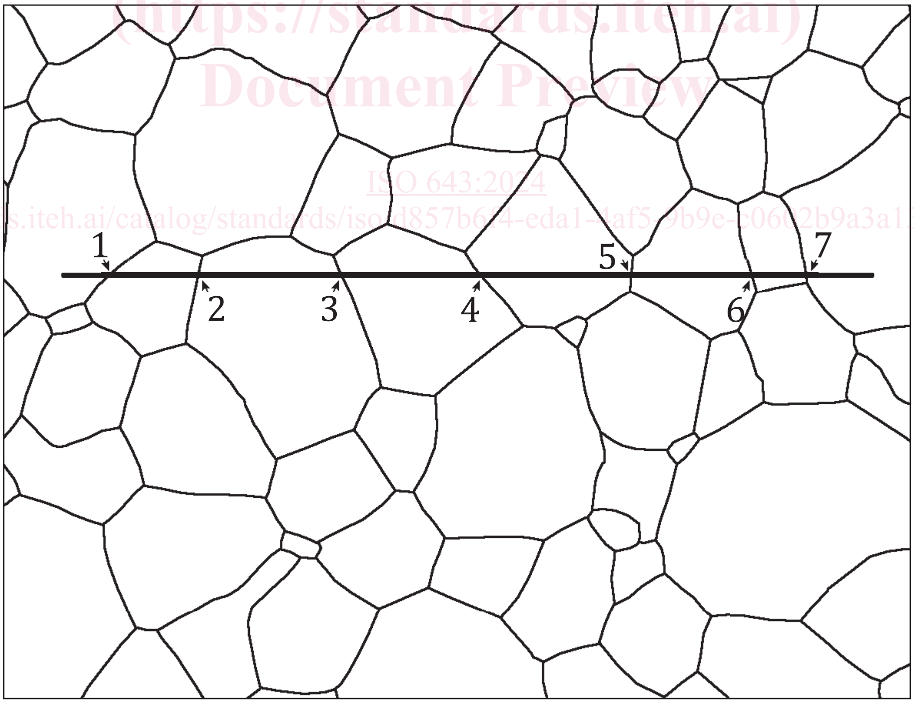

Figure 2 — Example of intersection, *P*

Intersection, *P*, counts for a straight test line placed over a single-phase grain structure where the arrows point to 7 intersection points and *P* = 7.

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6 Selection and preparation of the specimen

6.1 Test location

If the order, or the standard defining the product, does not specify the number of specimens and the point at which they are to be taken from the product, these are left to the manufacturer, although it has been shown that precision of grain size determination increases the higher the number of specimens assessed. Care shall be taken to ensure that the specimens are representative of the bulk of the product (i.e. avoid heavily deformed material such as that found at the extreme end of certain products or where shearing has been used to remove the specimen, etc.). The specimens shall be polished in accordance with the usual methods.

Unless otherwise stated by the product standard or by agreement with the customer, the polished surface can be randomly selected for the specimens with equiaxial grains. The polished surface shall be parallel to the principal axis of deformation in wrought products, for the specimens with deformed grains.

NOTE Measurements of the grain size on a transverse plane will be biased if the grain shape is not equiaxial.

6.2 Revealing ferritic grain boundaries

The ferritic grains shall be revealed by etching with nital [ethanolic 2 % to 3 % (by volume) nitric acid solution], or with another appropriate reagent.

6.3 Revealing austenitic and prior-austenitic grain boundaries

6.3.1 General

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In the case of steels having a single-phase or dual-phase mainly austenitic structure (delta ferrite grains in an austenitic matrix) at ambient temperature, the grains shall be revealed by an etching solution. For single phase austenitic stainless steels, the most commonly used chemical etchants are glyceregia, Kalling's reagent (No. 2) and Marble's reagent. The best electrolytic etch for single or two-phase stainless steels is aqueous 60 % nitric acid at 1,4 V d.c. for 60 s to 120 s, as it reveals the grain boundaries but not the twin boundaries. Aqueous 10 % oxalic acid, 6 V d.c., up to 60 s, is commonly used but is less effective than electrolytic 60 % nitric acid.

For other steels, one or other of the methods specified below shall be used depending on the information required:

— "Bechet-Beaujard" method by etching with aqueous saturated picric acid solution (see A.2);

— "Kohn" method by controlled oxidation (see A.3);

— "McQuaid-Ehn" method by carburization (see A.4);

— grain boundary sensitization method (see A.7);

— other methods specially agreed upon when ordering.

NOTE The first three methods are for prior-austenitic grain boundaries while the others are for austenitic Mn or austenitic stainless, see Annex A.

If comparative tests are carried out for the different methods, it is essential to use the same heat treatment conditions. Results may vary considerably from one method to the other.

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7 Characterization of grain size

7.1 General

7.1.1 Characterization methods

The apparent grain size can be determined by three micrographic methods: comparison method, planimetric method and intercept method.

7.1.2 Formulae

The index is defined by Formula (1):

$$m = 8 \times 2^{G} \tag{1}$$

This formula may be stated as Formula (2):

$$G = \log_2 m - 3 \tag{2}$$

NOTE An alternative system of grain size definition is known as the ASTM grain size (see C.2).

7.1.3 Accuracy of the methods

In general, the comparison method allows for an accuracy of 0,5; the planimetric and intercept segment methods allow for an accuracy of 0,1, see Reference [3]. For comparison between methods, the indexes obtained are usually rounded to multiples of 0,5.

Due to the randomness of the spatial position in which each grain is cut through by the sectioning plane and due to the measurement error, no determination of apparent grain size can be an exact measurement. Therefore, for planimetric and intercept methods it can be of interest to calculate the 95 % confidence interval of the grain size measurement result and adjust the number of fields inspected according to the percentage relative accuracy, % RA, of counting corresponding to the uncertainty of ±0,25 grain size units, taking into account that for a symmetric error of G the % RA of the measured quantity is not symmetric, see Annex D.

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The methods described in this document yield representative results for specimens with a unimodal grain size distribution. Applying them to specimens with bimodal (or more complex) size distributions will yield an average value that likely has no meaningful relationship with the various grain populations but may still represent the specimen on average. ISO 14250[4] may be the more appropriate standard for characterizing these specimens, see Annex E.

7.2 Comparison method

7.2.1 The image examined on the screen (or on a photomicrograph) shall be compared with a series of standard charts presented in Annex B or overlays (using eye-piece graticules designed for grain size measurement can be used provided these are traceable to national or international standards). The standard charts at a magnification of 100:1 are numbered from -1 to 10 so that their number is equal to the index G. Images for grain sizes -1 to 3 are included in the chart but when determining grain sizes in this range it is recommended for reasons of accuracy to reduce the operating magnification of the microscope, in combination with index conversion according to Table 2.

Using ASTM E112 charts gives substantially the same results as using the comparison charts of Annex B, see C.2.4.

7.2.2 The standard chart with the grain size closest to that of the examined fields of the specimen can then be determined. A minimum of three randomly selected fields shall be assessed on each specimen.

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7.2.3 Where the magnification g of the image on the screen or photomicrograph is not 100:1, the index G shall be equal to the number M of the closest standard chart, modified as a function of the ratio of the magnifications, as given by Formula (3):

$$G = M + 2\log_2 \frac{g}{100} = M + 6{,}64\lg \frac{g}{100} \qquad (3)$$

Table 2 gives the relationship between the indices for the usual magnifications.

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Table 2 — Relationship between indices for the usual magnifications

Standard chart no. *M*		1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10
Magnification of the image *g*	Q [a]	Grain size [b]																		
25	−4	−3	−2,5	−2	−1,5	−1	−0,5	0	0,5	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6
50	−2	−1	−0,5	0	0,5	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8
100	0	1	1,5	2	2,5	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10
200	+2	3	3,5	4	4,5	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12
400	+4	5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14
500	+4,5	5,5	6	6,5	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14	14,5
800	+6	7	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14	14,5	15	15,5	16
1 000	+6,5	7,5	8	8,5	9	9,5	10	10,5	11	11,5	12	12,5	13	13,5	14	14,5	15	15,5	16	16,5

[a] The values for g = 500 and g = 1 000 are rounded to the nearest multiple of 0,5.

[b] EXAMPLE: At a magnification g = 50 the standard chart number M = 3 corresponds to a grain size of G = 1.

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7.2.5 For the comparison method, if the difference between the maximum index G_{max} and the minimum index G_{min} determined is less than three (e. g. range from G = 6 to 8,5), compute the test result as the arithmetic mean of the found indices. If the indices are calculated using Formula (3), the arithmetic mean is to be calculated after the modification for non-standard magnification. If this condition is not fulfilled, the operator may perform an additional series of at least six determinations of the grain size. If the difference between the maximum index and the minimum index determined in this new series is less than three, then compute the test result as the arithmetic mean of all determinations of both the first and second series. If this latter condition is not fulfilled, note the spread and a comment on the findings in the final report. Alternatively, ISO 14250 should be considered. For a more elaborate discussion on specimens of non-unimodal distribution, see Annex E.

The calculated arithmetic mean of indices shall be rounded to the nearest multiple of 0,5.

7.3 Planimetric method

7.3.1 Historically a circle measuring 79,8 mm in diameter (A_F = 5 000 mm^2), see Figure 3, was drawn or superimposed over a micrograph or a live image on a ground glass projection screen. The magnification was then adjusted so that the circular area contained at least 50 grains in order to minimize the counting error associated with a circular test pattern. The following procedure and formulae are magnification neutral.

NOTE The circle referenced an apparent size specifically at 100:1 magnification as perceived by an operator at a microscope using ×10 oculars and ×10 objective. This reference also was, and still is, used in other applications such as inclusion assessments. It is moreover a component of the recommended concentric circle grid used in the intercept method, as well as one of the reference circles used in the comparison method.

However, more recent tools like image software allow for optimizing the combination of circle diameter and magnification to facilitate the count and to make sure that the number of grains within the circle reach at least 50. Therefore, it is no longer always relevant to reference a specific circle size at a specific magnification.

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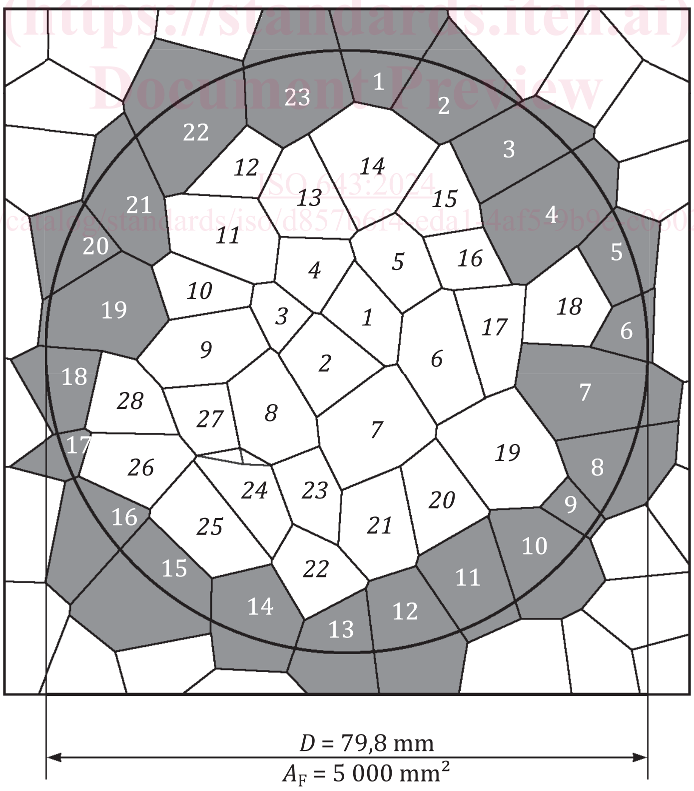

Figure 3 — Evaluation of the number of grains in an area enclosed by a circle

7.3.2 Two counts are made; the number of grains completely enclosed within the test circle of any given size, n_e, and the number of grains intercepted by the test circle, n_i.

The total number of equivalent grains, n_t, is calculated using Formula (4):

$$n_t = n_e + \frac{n_i}{2} \tag{4}$$

The number of grains per mm^2, m, is calculated using Formula (5):

$$m = \frac{n_t}{A_C} \tag{5}$$

where A_C is the true area of the circle.

7.3.3 The planimetric approach assumes that on average, half of the grains on the test circle are within and half are outside test circle. This assumption is only fully valid along a straight line through a grain structure, but not for a curved line, including a circle. The bias created by this assumption increases as the number of grains inside the test circle decreases. If the number of grains within the test circle is at least 50, the bias becomes about 2 %.

7.3.4 A simple way to avoid this bias, irrespective of the number of grains within the test figure, is to use a square or a rectangle (see Figure 4). However, the counting procedure then shall be modified.

First, it is assumed that the grains on each of the four corners are, on average, one fourth within the figure and three-fourths outside. These four corner grains together equal one grain within the test box. Ignoring the four corner grains, a count is made of the grains completely enclosed by the test box, n_e, and the grains intercepted by the four sides of the box, n_i, (see Figure 4). The total number of grains is calculated as in Formula (6):

$$n_t = n_e + \frac{n_i}{2} + 1 \tag{6}$$

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