International Standard

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Steels — Point counting method for statistically estimating the volume fraction of a constituent with a point grid

Aciers — Méthode d'estimation statistique de la fraction volumique d'un constituant à l'aide de grilles de points

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Foreword

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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 second edition cancels and replaces the first edition (ISO 9042:1988) which has been technically revised.

The main changes are as follows:

— clearer definition of t multiplier and corrected values in Table 4 (formerly Table 3);

— additional grid examples;

— application to digital images;

— change of terminology: Error % is now E_{rel};

— editorial changes.

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.

Steels — Point counting method for statistically estimating the volume fraction of a constituent with a point grid

1 Scope

This document specifies a point counting method for statistically estimating the volume fraction of a constituent through the microstructure of a steel by means of a point grid.

It applies to constituents which are clearly identifiable.

By default, counting is performed manually, but can be computer assisted or substituted by validated computer algorithms.

NOTE In this document, the word "constituent" can designate a phase as well as a micrographic constituent composed of two or more phases.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

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/

4 Symbols and abbreviated terms

The symbols used are given in Table 1.

Table 1 — Symbols

Symbols	Definition	Value
n	Number of fields observed	—
P_T	Total number of points in the grid	—
P_i	Point count on the i^{th} field	—
$P_P(i)$	Proportion of grid points in the constituent on the i^{th} observed field, expressed as a percentage of the total number of points in the grid.	See Formula (2)
$\overline{P_P}$	Arithmetic mean of $P_P(i)$	See Formula (3)
$\hat{s}$	Estimate of the standard deviation σ of the mean	See Formula (4)
CI	95 % confidence interval	See Formula (5)
$t_{0,05;n-1}$	Student's t-distribution for a two-sided confidence level of 95 % and n observed fields	See Table 4 for values

Table 1 *(continued)*

Symbols	Definition	Value
V_V	Volume fraction of the constituent expressed as a percentage	See Formula (6)
$A_{A,est}$	Estimated area fraction of the constituent expressed as a percentage	—
E_{rel}	Relative error, a measure of the statistical precision, expressed as a percentage; a lower E_{rel} corresponds to a lower error and thus a higher accuracy	See Formula (7)

5 Principle

The basic principle is that a grid with a number of regularly arrayed points, when systematically placed over an image of a micrographic section, can provide, after a representative number of placements on different fields, an unbiased estimation of the volume fraction of the constituent.

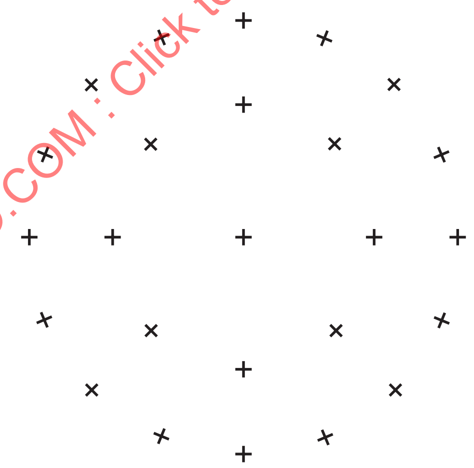
The method consists in superimposing the point grid on a given number of fields of the observed surface and in counting the number of points of the grid included in the constituent and then calculating its volume fraction.

6 Apparatus

6.1 Grid

The grid consists of a specified number of regularly spaced and unambiguously defined points. The grids shown in Figures 1 to 3 are given as examples. Alternately, a grid of randomly distributed points may be used.

The grid can be applied via a reticle placed in the eyepiece of the microscope, a graphical overlay on a digital image, the pixel array of a digital image, or other image analysis procedures. It can also be reproduced on a transparency which is placed on the viewing screen of the microscope or on micrographs.



NOTE It is possible to use all 25 points, the outer points, or the 9 inner points.

Figure 1 — Circular grid

Figure 2 — Square grid

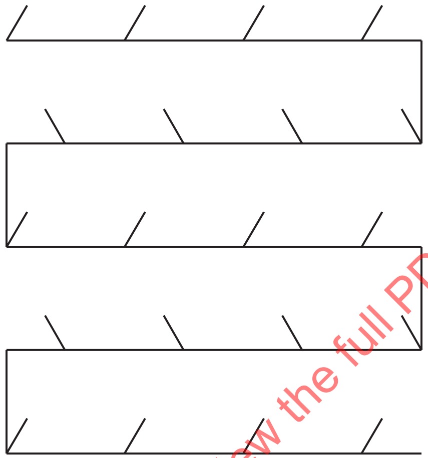


Figure 3 — Hexagonal “Blaschke” grid with serpentine counting aid

6.2 Means of observation

A microscope or other suitable device for magnification, equipped with a suitable means of observation such as a viewing screen, a computer screen, or eyepiece reticle, and preferably equipped with an X and Y translation stage, shall be used for the observation of the microstructure.

7 Sample

7.1 Sampling and number of samples

The sample shall be representative of the microstructure for which the constituent is to be estimated.

The place of sampling, the orientation of the surface observed (e.g. longitudinal or transverse), the number of samples and the surface area to be examined shall be specified in the product standard or agreed upon between the parties.

7.2 Preparation of samples

7.2.1 The sample shall be polished in accordance with metallographic techniques. Care shall be taken during the polishing to avoid any alteration of the microstructure.

7.2.2 When necessary, the sample may undergo a micrographic etching to reveal the constituent to be measured.

NOTE ISO/TR 20580 contains a report of various applicable metallographic preparation techniques. Colouring-type etchants and shallow etching minimize the error introduced by etching.

8 Procedure

8.1 Selection of the grid

A previous visual estimation of the area fraction of the constituent is necessary for the selection of the grid, for example the total number of points in the grid.

Table 2 gives the recommended values of the total number of points of a square grid as a function of the estimated area fraction of the constituent of which the volume fraction is to be determined. These indications do not correspond to theoretical constraints, but empirical observations have shown that the duration of the test for a given relative error is optimized in using these values, especially for methods without computer assistance.

In the case where a structure shows a certain periodicity of the distribution of the constituent being measured, any coincidence of the points of the grid and the structure should be avoided. This can be achieved with either a non-periodic (e.g. circular) grid or a periodic grid that is placed at an angle to the microstructure image.

Table 2 — Recommended values of the total number of points of a square grid

Visual area fraction estimation $A_{A,est}$	Total number of points on a grid P_T [b]
$2\,\% < A_{A,est} \leq 5\,\%$	100
$5\,\% < A_{A,est} \leq 10\,\%$	49
$10\,\% < A_{A,est} \leq 20\,\%$	25
$A_{A,est} > 20\,\%$ [a]	16

[a] For $A_{A,est} > 50\,\%$, it is often easier and more precise to count the minority fractions and determine V_V as a complement to 100 %.

[b] For non-square grids, the recommended values are of the same order of magnitude.

8.2 Magnification selection

The selected grid shall be placed on the image of the structure. The magnification should be selected to optimise according to the following criteria.

— Point designations on the grid should be smaller than the constituents to be identified.

— The field of view should be representative of the sample.

— The distance between the grid points should be the same order of magnitude as the smallest constituent of interest.

8.3 Selection of the number of observed fields

The number of fields to be observed depends on the desired relative error for the measurement. Table 3 gives the number of fields to be observed as a function of the required relative error and of the magnitude of the volume fraction. A minimum of five fields is recommended to provide an acceptable statistical sampling of the specimen, but more fields may be necessary to obtain a specified E_{rel}.

8.4 Array of the fields

When the number of fields *n* is defined, the positions of the fields is then determined, either as a regular array or a random scatter. The pattern shall cover the area of interest without any overlapping of individual fields. The movements of the stage in the X and Y directions are based on these predetermined positions.

Where micrographs are used, they shall be recorded at these locations and printed out in sufficient quality to ensure that the constituent can be clearly identified.

Table 3 — Prediction of the number of fields to be observed as a function of the desired relative error and of the estimated magnitude of the volume fraction of the constituent [a]

Magnitude of volume fraction V_V (%)	Relative error of 30 %				Relative error of 20 %				Relative error of 10 %			
	Number of fields *n* for a grid with a number of points P_T =				Number of fields *n* for a grid with a number of points P_T =				Number of fields *n* for a grid with a number of points P_T =			
	16	25	49	100	16	25	49	100	16	25	49	100
2	110	75	40	20	310	200	105	50	1 250	800	410	200
5	50	30	15	8	125	80	40	20	500	320	165	80
10	25	15	10	4	65	40	20	10	250	160	85	40
20	15	10	5	4	30	20	10	5	125	80	40	20
30	6	4	2	1	15	10	5	3	60	40	20	10
40	4	3	2	1	10	6	4	2	40	25	15	6
50	3	2	1	1	7	4	3	1	25	20	10	4

[a] The values given in this table are approximate values based on Formula (1).

$$n \cong \frac{40\,000}{(E_{rel})^2} \times \frac{100\,\% - V_V}{V_V} \times \frac{1}{P_T} \qquad (1)$$

8.5 Examination and estimation of the fields

Each field shall be observed with the selected grid placed on the image of the microstructure. The movement from one field to the next shall be made without viewing the image, only by moving the stage; this is done to eliminate any possibility of operator bias in the field position with respect to the grid.

Where micrographs are used, the transparency with the grid shall be applied without bias. The same transparency shall only be applied once to each micrograph. Multiple measurements of the same micrograph must avoid bias.

For each field observed, the number of points included in the constituent P_i shall be recorded.

Any points falling on the constituent boundary shall be counted as one-half. In the same way, in order to minimize the error, when there is doubt as to whether a point is inside or outside the constituent boundary it shall be counted as one-half.

NOTE An abundance of half counts can be due to improper magnification selection.

Computer algorithms that completely replace manual discrimination and counting shall be validated.

For each field, the proportion of points included in the constituent P_p *(i)* shall be calculated as a percentage in accordance with Formula (2).

$$P_P(i) = \frac{P_i}{P_T} \times 100 \qquad (2)$$

8.6 Calculation of the volume fraction V_v

The arithmetic mean $\overline{P_P}$ of the values $P_p(i)$ is calculated in accordance with Formula (3).

$$\overline{P_P} = \frac{1}{n}\sum_{i=1}^{n} P_P(i) \tag{3}$$

The estimated value of the standard deviation of the mean shall be calculated in accordance with Formula (4).

$$\hat{s} = \sqrt{\frac{1}{n-1}\sum_{i=1}^{n}\left(P_P(i) - \overline{P_P}\right)^2} \tag{4}$$

The 95 % confidence interval shall be calculated in accordance with Formula (5).

$$CI = t_{0,05;n-1} \times \frac{\hat{s}}{\sqrt{n}} \tag{5}$$

The appropriate values for $t_{0,05;n-1}$ are given in Table 4 as a function of the number of observed fields n.

Table 4 — Selected values of Student's t-distribution for calculation of the 95 % confidence interval [a,b]

Number of observed fields n	Student's t-distribution $t_{0,05;n-1}$	Number of observed fields n	Student's t-distribution $t_{0,05;n-1}$
4	3,182	15	2,145
5	2,776	20	2,093
6	2,571	25	2,064
7	2,447	30	2,045
8	2,365	40	2,023
9	2,306	50	2,010
10	2,262	60	2,001
12	2,201	∞	1,960

[a] For n ≥ 30, a value of $t_{0,05;n-1} = 2$ is acceptable for most practical purposes.

[b] Additional values for $t_{0,05;n-1}$ are accessible online and can be computed by many spreadsheet programs. For n observed fields, the degrees of freedom for looking up $t_{0,05;n-1}$ are n-1.

The volume fraction V_V is given by Formula (6).

$$V_V = \overline{P_P} \pm CI \tag{6}$$

The relative error E_{rel} is given by Formula (7). The relative error should be better than 30 %; a maximum value may be agreed upon between the parties.

$$E_{rel} = \frac{CI}{\overline{P_P}} \times 100 \tag{7}$$

NOTE If an improvement of the obtained relative error E_{rel} is wanted, additional measurements can be made by increasing the number of fields n; as per Formula (1), a reduction of the relative error E_{rel} by 50 % can be achieved by measuring a total of four times the original number of fields n.

NOTE The terminology of the confidence interval CI has been left unchanged from ISO 9042:1988. Many statistics texts refer to this document's confidence interval as "margin of error" and use the term confidence interval to refer to the mathematical interval of the mean ± the margin of error.

9 Test report

The test report shall provide at least the following information:

a) state of the examined samples (e.g. cast, forged, annealed, unknown);

b) specifics for identifying and tracking the sample (e.g. heat, cast, lot, unknown);

c) orientation of the observed surface in the case of anisotropy;

d) type of grid used (array, total number of points P_T);

e) number of the observed fields n per sample and their spacing;

f) selected magnification;

g) value of the volume fraction V_V and, if required, values of the confidence interval CI and of the relative error E_{rel}.