INTERNATIONAL STANDARD

ISO
9042

First edition
1988-12-15

INTERNATIONAL ORGANIZATION FOR STANDARDIZATION
ORGANISATION INTERNATIONALE DE NORMALISATION
МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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

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

Reference number
ISO 9042 : 1988 (E)

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for approval before their acceptance as International Standards by the ISO Council. They are approved in accordance with ISO procedures requiring at least 75 % approval by the member bodies voting.

International Standard ISO 9042 was prepared by Technical Committee ISO/TC 17, *Steel.*

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Printed in Switzerland

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

1 Scope

This International Standard specifies a manual 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.

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

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2 Principle

2.1 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.

2.2 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.

3 Symbols and definitions

For the purpose of this International Standard, the following symbols are used.

n = number of fields observed

P_T = total number of points in the grid

P_i = point count on the ith field

$P_P(i)$ = proportion of grid points in the constituent on the ith observed field, expressed as a percentage of the total number of points in the grid

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

$\bar{P}_P$ = arithmetic average of $P_P(i)$

$$\bar{P}_P = \frac{1}{n} \sum_{i=1}^{n} P_P(i)$$

$\hat{s}$ = estimate of the standard deviation (σ)

$$\hat{s} = \left\{ \frac{1}{n-1} \sum_{i=1}^{n} \left[P_P(i) - \bar{P}_P \right]^2 \right\}^{1/2}$$

CI = 95 % confidence interval

$$\mathrm{CI} = \pm 2 \frac{\hat{s}}{\sqrt{n}}$$

V_V = volume fraction of the constituent expressed as a percentage

$$V_V = \bar{P}_P \pm \mathrm{CI}$$

$$\text{Error } \% = \frac{\mathrm{CI}}{\bar{P}_P} \times 100$$

= statistical precision

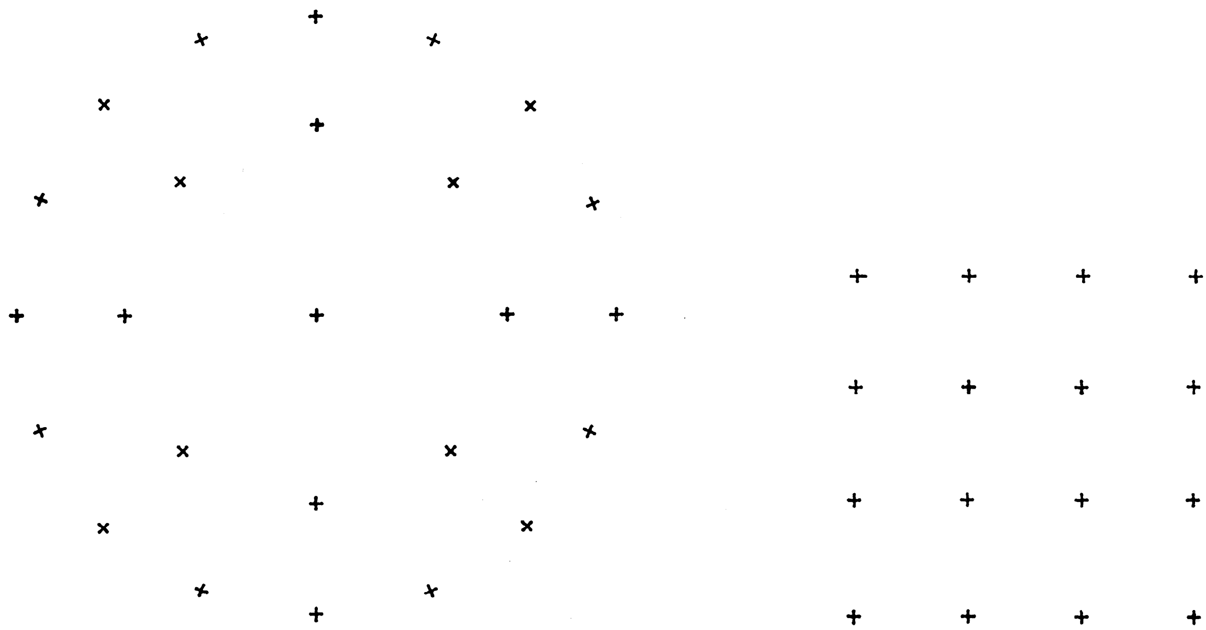
4 Apparatus

4.1 Grid

The grid consists of a specified number of equally spaced points formed by the intersections of very thin lines. The two types of grid (circular or square array) shown in figure 1 are given as examples that can be used.

The grid can be constituted by a reticle placed in the eyepiece of the microscope or reproduced on a transparency which is placed on the viewing screen of the microscope or on micrographs[1)].

1) Since the use of micrographs is time-consuming and more costly, it should be avoided if possible.



Circular grid

Square grid

NOTE — It is possible to use all 25 points, the 16 outer points, or the 9 inner points (circular grid).

Figure 1 — Examples of grids that can be used

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4.2 Means of observation

A microscope or other suitable device with a viewing screen or eyepiece reticle and preferably with an X and Y translation stage is used for the observation of the microstructure.

5 Sample

5.1 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.

5.2 Preparation of samples

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

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

It is recommended that the etching be made as shallow as possible. Colouring-type etchants are generally preferable to those which cause a preferential etching of one or more of the constituents, since the latter type of etchant can introduce an error.

It is important to check the influence of the etching-time on the estimation of the volume fraction.

6 Procedure

6.1 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 1 gives the recommended values of the total number of points of a grid as a function of the 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 precision is optimized in using these values.

Table 1 — Recommended values of the total number of points of a grid

Visual area fraction estimation	Total number of points on a grid P_T
2 % to 5 % inclusive	100
5 % to 10 % inclusive	49
10 % to 20 % inclusive	25
> 20 %	16

6.2 Magnification selection

The selected grid is placed on the image of the structure. The magnification shall be selected as high as possible such that two adjacent grid points do not lie on the same particle or at the same aggregate of particles of the constituent.

6.3 Selection of the number of observed fields

The number of fields to be observed depends on the desired degree of precision for the measurement. Table 2 gives the number of fields to be observed as a function of the selected error and of the amount of the volume fraction. A minimum of 30 fields is recommended to provide an acceptable statistical precision.

When less than 30 fields are used, a multiplier from table 3 shall be used to modify equation (5) in clause 7.

6.4 Array of the fields

When the number of fields (n) is defined, the spacing of the fields to form a regular distribution of the fields on the surface of the sample without any overlapping of the fields is then determined. The movements of the stage in the X and Y directions are based on this spacing of the fields.

Table 2 — Prediction of the number of fields to be observed as a function of the desired precision and of the estimated magnitude of the volume fraction of the constituent

Amount of volume fraction V_V %	Error of 33 %				Error of 20 %				Error of 10 %			
	Number of fields n for a grid of P_T =				Number of fields n for a grid of P_T =				Number of fields n for a grid of P_T =			
	16 points	25 points	49 points	100 points	16 points	25 points	49 points	100 points	16 points	25 points	49 points	100 points
2	110	75	35	(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)

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NOTES

1 The values given in the table above are approximate values based on the equation

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$$n \simeq \frac{4}{E^2} \cdot \frac{100 - V_V}{V_V} \cdot \frac{1}{P_T}$$

where

E = 0,01 × % error;

V_V is expressed as a percentage.

2 For the values indicated in parentheses, the multipliers of table 3 should be used for the calculation of the confidence interval according to equation (5).

Table 3 — Multiplier for calculation of the 95 % confidence interval

Number of observed fields n	Multiplier
5	2,57
7	2,36
9	2,26
11	2,20
13	2,16
15	2,13
20	2,09
25	2,06
30	2,04

NOTE — 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 circular grid or a square grid that is placed at an angle to the microstructure image.

6.5 Examination and estimation of the fields

Each field is observed with the selected grid placed on the image of the microstructure. The movement from one field to the next is 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.

For each field observed, the number of points included in the constituent P_i is 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.

For each field, the proportion of points included in the constituent $P_P(i)$ is calculated according to the equation

$$P_P(i) = \frac{P_i}{P_T} \times 100 \qquad \ldots (1)$$

7 Calculation of the volume fraction V_V

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The arithmetic average $\overline{P}_P$ of the values P_P is calculated according to the equation

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$$\overline{P}_P = \frac{1}{n} \sum_{i=1}^{n} P_P(i) \qquad \ldots (2)$$

The estimated value of the standard deviation about the average is calculated according to the formula

$$\hat{s} = \left\{ \frac{1}{n-1} \sum_{i=1}^{n} \left[P_P(i) - \overline{P}_P \right]^2 \right\}^{1/2} \qquad \ldots (3)$$

The volume fraction V_V is given by the equation

$$V_V = \overline{P}_P \pm \mathrm{CI} \qquad \ldots (4)$$

The 95 % confidence interval is calculated according to the equation

$$\mathrm{CI} = \pm 2 \frac{\hat{s}}{\sqrt{n}} \qquad \ldots (5)$$

NOTE — When a small number of fields (< 30) is used, the multiplier 2 in formula (5) should be replaced by the appropriate value selected from table 3.

The error for the estimation of V_V can be calculated according to the equation

$$\text{\% error} = \frac{\mathrm{CI}}{\overline{P}_P} \times 100 \qquad \ldots (6)$$

NOTE — If an improvement of the obtained precision is wanted, additional measurements can be made by increasing the number of fields; to reduce the % error by 50 %, a total of four times the original number of fields should be measured.

8 Test report

The test report shall mention at least the following information :

a) nature and identification of the examined samples;

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

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

d) number of the observed fields by samples and their spacing;

e) selected magnification;

f) value of the volume fraction and, if required, values of the confidence interval and of the calculated error.

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UDC 669.14 : 620.18

Descriptors : steels, tests, microscopic analysis, determination, microstructure.

Price based on 4 pages