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МЕЖДУНАРОДНАЯ ОРГАНИЗАЦИЯ ПО СТАНДАРТИЗАЦИИ

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)

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.

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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.

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

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

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

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

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

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

CI = 95 % confidence interval

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

V_V = volume fraction of the constituent expressed as a percentage

$$V_V = \overline{P}_P \pm CI$$

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

= statistical precision

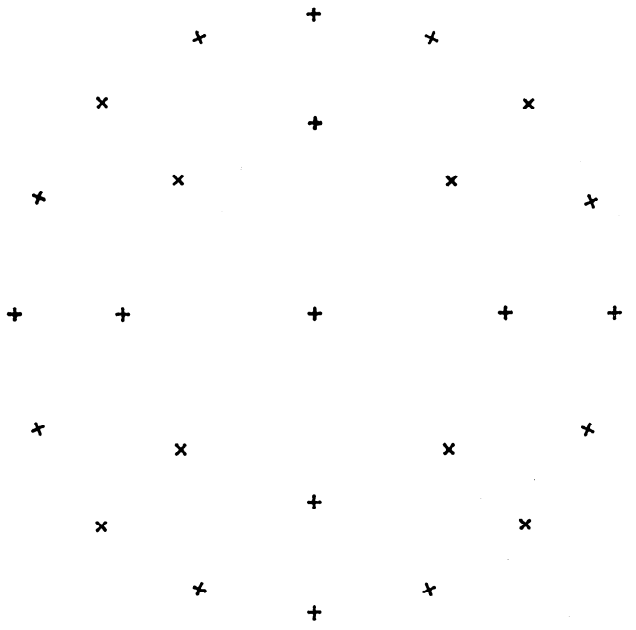
4 Apparatus

4.1 Grid

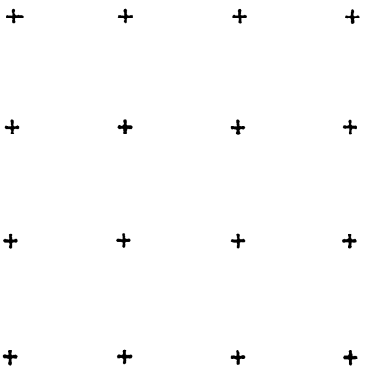
The grid consists of a specified number of equally spaced points formed by the intersections of very thin lines. The two types of grid (circular or square array) shown in figure 1 are given as examples that can be used.

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

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