Designation: E 562 – 99

Standard Test Method for Determining Volume Fraction by Systematic Manual Point Count[1]

This standard is issued under the fixed designation E 562; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This test method may be used to determine the volume fraction of constituents in an opaque specimen using a polished, planar cross section by the manual point count procedure.

1. Scope

1.1 This test method describes a systematic manual point counting procedure for statistically estimating the volume fraction of an identifiable constituent or phase from sections through the microstructure by means of a point grid.

1.2 The use of automatic image analysis to determine the volume fraction of constituents is described in Practice E 1245.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

E 3 Methods of Preparation of Metallographic Specimens[2]

E 7 Terminology Relating to Metallography[2]

E 407 Practice for Microetching Metals and Alloys[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

E 1245 Practice for Determining the Inclusion or Second Phase Constituent Content of Metals by Automatic Image Analysis[2]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, see Terminology E 7.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *point count*—the total number of points in a test grid that fall within the microstructural feature of interest, or on the feature boundary; for the latter, each test point on the boundary is one half a point.

3.2.2 *point fraction*—the ratio, usually expressed as a percentage, of the point count of the phase or constituent of interest on the two-dimensional image of an opaque specimen to the number of grid points, which is averaged over *n* fields to produce an unbiased estimate of the volume fraction of the phase or constituent.

3.2.3 *stereology*—the methods developed to obtain information about the three-dimensional characteristics of microstructures based upon measurements made on two-dimensional sections through a solid material or their projection on a surface.

3.2.4 *test grid*—a transparent sheet or eyepiece reticle with a regular pattern of lines or crosses that is superimposed over the microstructural image for counting microstructural features of interest.

3.2.5 *volume fraction*—the total volume of a phase or constituent per unit volume of specimen, generally expressed as a percentage.

3.3 *Symbols:*

P_T = total number of points in the test grid.

P_i = point count on the i^{th} field.

$P_P(i)$ = $\frac{P_i}{P_T} \times 100$ = percentage of grid points, in the constituent observed on the i^{th} field.

n = number of fields counted.

$\bar{P}_p$ = $\frac{1}{n}\sum_{i=1}^{n} P_p(i)$ = arithmetic average of $P_p(i)$.

s = estimate of the standard deviation (σ) (see (Eq 3) in Section 10).

95 % CI = 95 % confidence limit = $\pm 2.0s/\sqrt{n-1}$ (see Note 1).

V_V = volume fraction of the constituent or phase expressed as a percentage (see (Eq 5) in Section 10).

% RA = % relative accuracy, a measure of the statistical precision = $(95\,\%\ \text{CI}/\bar{P}_p) \times 100$.

NOTE 1—The multiplier is given as 2.0 for purpose of definition. Table 1 gives the appropriate multiplying factors for any number of fields

[1] This practice is under the jurisdiction of ASTM Committee E-4 on Metallography and is the direct responsibility of Subcommittee E04.14 on Quantitative Metallography.

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[2] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 14.02.

TABLE 1 Prediction of the Number of Fields (*n*) to be Observed as a Function of the Desired Relative Accuracy and of the Estimated Magnitude of the Volume Fraction of the Constituent

Amount of volume fraction, V_V in percent	33 % Relative Accuracy				20 % Relative Accuracy				10 % Relative Accuracy			
	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)

NOTE 1—The given values in the table above are based on the formula:

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

where:

E = 0.01 × % RA, and
V_V = is expressed in %.

NOTE 2—For the values indicated in parentheses, the multipliers of Table 4 shall be used for the calculation of the confidence interval according to (Eq 4).

measured.

4. Summary of Test Method

4.1 A clear plastic test grid or eyepiece reticle with a regular array of test points is superimposed over the image, or a projection of the image, produced by a light microscope, scanning electron microscope, or photograph, and the number of test points falling within the phase or constituent of interest are counted and divided by the total number of grid points yielding a point fraction, usually expressed as a percentage, for that field. The average point fraction for *n* measured fields gives an estimate of the volume fraction of the constituent. This method is applicable only to bulk opaque planar sections viewed with reflected light or electrons.

5. Significance and Use

5.1 This test method is based upon the stereological principle that a grid with a number of regularly arrayed points, when systematically placed over an image of a two-dimensional section through the microstructure, can provide, after a representative number of placements on different fields, an unbiased statistical estimation of the volume fraction of an identifiable constituent or phase **(1, 2, 3).**[4]

5.2 This test method has been described **(4)** as being superior to other manual methods with regard to effort, bias, and simplicity.

5.3 Any number of clearly distinguishable constituents or phases within a microstructure (or macrostructure) can be counted using the method. Thus, the method can be applied to any type of solid material from which adequate two-dimensional sections can be prepared and observed.

5.4 A condensed step-by-step guide for using the method is given in Annex A1.

6. Apparatus

6.1 *Test Grid*, consisting of a specified number of equally spaced points formed by the intersection of very thin lines. Two common types of grids (circular or square array) are shown in Fig. 1.

6.1.1 The test grid can be in the form of a transparent sheet that is superimposed upon the viewing screen for the measurement.

6.1.2 *Eyepiece Reticle*, may be used to superimpose a test grid upon the image.

6.2 *Light Microscope*, or other suitable device with a viewing screen at least 100 mm × 125 mm, preferably with graduated *x* and *y* stage translation controls, should be used to image the microstructure.

6.3 *Scanning Electron Microscope*, may also be used to image the microstructure; however, relief due to polishing or heavy etching must be minimized or bias will be introduced as a result of deviation from a true two-dimensional section through the microstructure.

6.4 *Photomicrographs*, of properly prepared opaque specimens, taken with any suitable imaging device, may be used provided the fields are selected without bias and in sufficient quantity to properly sample the microstructure.

7. Sample Selection

7.1 Samples selected for measurement of the phase or constituent should be representative of the general microstructure, or of the microstructure at a specified location within a lot, heat, or part.

7.2 A description of the sample locations should be included as a part of the results.

7.3 Any orientation of the prepared section (that is, whether longitudinal or transverse) can be used. However, it should be recorded since it may have an effect upon the precision obtained.

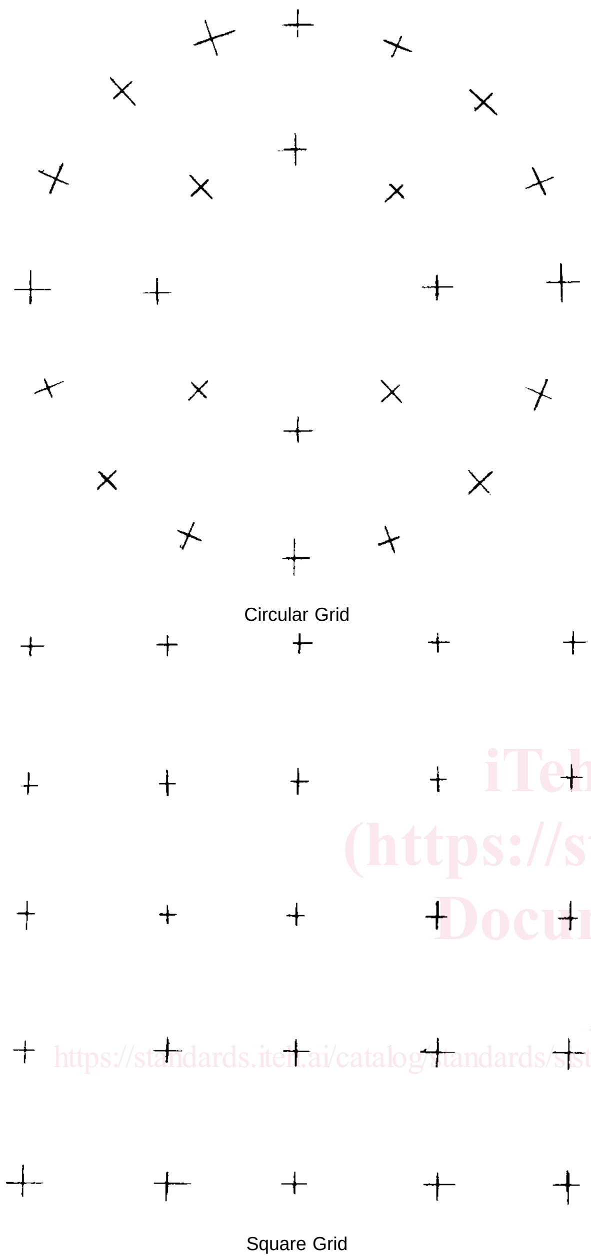
7.4 If the sample microstructure contains gradients or inhomogeneities (for example, banding) then the section should contain or show the gradient or inhomogeneity.

8. Sample Preparation

8.1 The two-dimensional sections should be prepared using standard metallographic, ceramographic, or other polishing procedures, such as described in Methods E 3.

8.2 Smearing or other distortions of the phases or constituents during preparation of the section or sections should be minimized because they tend to introduce an unknown bias

[4] The boldface numbers in parentheses refer to the list of references at the end of this standard.



NOTE 1—The entire 24 points can be used, or the outer 16, or the inner 8 points.

FIG. 1 Examples of Possible Grid Configurations That Can Be Utilized

into the statistical volume fraction estimate.

8.3 Etching of the sections, as described in Test Methods E 407, should be as shallow (that is, light) as possible because deviations from a planar two-dimensional section will cause a bias toward over estimation of the volume fraction.

8.4 Stain- or coloring-type etchants are preferable to those that cause attack of one or more of the constituents or phases.

8.5 Description of the etchant and etching procedure should be included in the report.

8.6 If etching is used to provide contrast or distinguishability of constituents then the volume fraction estimates should be obtained as a function of etching time to check the significance of any bias introduced.

9. Procedure

9.1 *Principle*:

9.1.1 An array of points formed by a grid of lines or curves is superimposed upon a magnified image (that is, a field of view) of a metallographic specimen.

9.1.2 The number of points falling within the microstructural constituent of interest is counted and averaged for a selected number of fields.

9.1.3 This average number of points expressed as a percentage of the total number of points in the array (P_T) is an unbiased statistical estimation of the volume percent of the microstructural constituent of interest.

9.1.4 A condensed step-by-step description of the procedure is provided in Annex A1.

9.2 *Grid Selection*:

9.2.1 The grid should consist of equally spaced points formed by the intersection of fine lines. Diagrams of two possible grids, one with a circular pattern and one with a square pattern, which are recommended for use, are shown in Fig. 1.

9.2.2 Determine the number of points (that is, the grid size, P_T) from a visual estimate of the area fraction occupied by the constituent of interest. Table 2 provides guidelines for this selection. The values in Table 2 do not correspond to theoretical constraints; but, by using these values, empirical observations have shown that the method is optimized for a given precision.

9.2.3 Superimpose the grid, in the form of a transparency, upon a ground glass screen on which the section image is projected.

9.2.4 A grid in the form of an eyepiece reticle may also be used.

9.2.5 The use of photomicrographs for point counting is not recommended because of the amount of effort required to obtain an adequate number of fields, but they can be used under certain circumstances (see 6.4).

9.2.6 If the constituent areas form a regular or periodic pattern on the section image, avoid the use of a grid having a similar pattern.

9.3 *Magnification Selection*:

9.3.1 Select the magnification so that it is as high as needed to clearly resolve the microstructure without causing adjacent grid points to fall over the same constituent feature.

9.3.2 As a guideline, choose a magnification that gives an average constituent size that is approximately one half of the grid spacing.

TABLE 2 Guidelines for Grid Size Selection[A]

NOTE 1—A grid size selection which gives a significant number of fields having no grid points on the constituent of interest should be avoided.

Visual Area Fraction Estimate Expressed as a Percentage	Grid Size (Number of Points, P_T)
2 to 5 %	100
5 to 10 %	49
10 to 20 %	25
>20 %	16

[A] These guidelines represent an optimum for efficiency for the time spent counting and for the statistical information obtained per grid placement.