MEASURING VOLUME FRACTION

Mathematical Basis

Sampling a Two-Dimensional Structure Using Test Points

The probability that one of an infinite number of test points falls on a group of particles (phase) contained in a sample area L^2 is

$$P = \frac{A}{L^2} = A_A \tag{1}$$

where A is the total area covered by the particles and A_A is the area fraction of particles. This relationship is the basis of the point-count method which is used to estimate the volume fraction of particles or second phase in a microstructure. This is possible because the volume fraction is equal to the area fraction.

General Methods

Volume Fraction, Areal Analysis

1. Trace the structure onto paper, weight the paper, cut out the parts containing the phase of interest and weigh these parts. The weight fraction equals the area fraction of phase in this section. The average of a number of area fraction measurements gives one the volume fraction of phase.

2. Etch the specimen to color each phase differently. Use a computer to measure the area of each phase (color). The average of a number area fraction measurements is equal to the volume fractions of phases.

Volume Fraction, Lineal Analysis

Using lines uniformly distributed but randomly oriented, the average length of the line segments crossing the selected phase is equal to the area fraction of the phase viewed in the planar section. The average of a number area fraction measurements is equal to the volume fractions of phases.

Volume Fraction, Point Count

The fraction of a number of points which fall on the selected phase is equal to the area fraction of that phase. The average of a number area fraction measurements is equal to the volume fractions of phases.

Recommended Method

Measurement of Volume Fraction of Phases Using the Systematic Point Count Method

This method is based on the fact that the average fraction of points which fall on grains of a particular phase is equal to the area fraction of the phase in the polished section and that the average of these area fractions is equal to the volume fraction of the phase.

This method entails selecting a suitable array of points, overlaying it on the specimen and counting the points which fall on a the selected phase, selecting a new are and counting again, and again until

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the desired error is obtained. The procedure is as follows:

Selection of grid

1. The most efficient method will be that which requires the least effort per observation.
2. Using a reticule grid and observing the polished sample is more efficient than using a micrograph.
3. A square array of points is simple, symmetric and easy to obtain.
4. The optimum number of points in the grid large to minimize the effort of changing views (or micrographs) and minimum to reduce the effort of counting.

Specifying the Maximum Allowable Error

5. Bear in mind that the effort involved varies inversely with the square of the error.
6. Specify the confidence level and the allowable confidence interval.
7. Obtain the variance from the confidence interval.

Counting

8. Select an area on the specimen where counts are to be made. This selection should be made at random or per a predefined pattern so as to minimize bias.
9. Count each point which falls on the selected phase.
10. Assign a count value of ½ to points which fall on interphase boundaries.
11. After making a few preliminary counts you can estimate the number of points that must be counted to give the desired error. This can be estimated using the following equation

$$P = \frac{1}{\sigma^2(V_V)}\left[V_V(1 - V_V)\right] \tag{2}$$

where $F^2(V_V)$ is the variance derived from the specified confidence interval.

Tabulate the Results

12. Compute the mean and the confidence interval.
13. Divide these by the number of points in the grid and multiply them by 100. This gives the results in terms of the volume percent of phase.

Example: An eyepiece reticule which has a 5 x 5 grid etched in it is used in this example. Twenty counts are made, the results of which are shown below:

	i	x_i	x^2	f_i	f_i^2
	1	5	25	0.20	0.040
	2	7	49	0.28	0.078
	3	5	25	0.20	0.040
	4	9	81	0.36	0.130
	5	2	4	0.08	0.006
	6	5	25	0.20	0.040
	7	6	36	0.24	0.058
	8	6	36	0.24	0.058
	9	4	16	0.16	0.026
	10	6	36	0.24	0.058
	11	5	25	0.20	0.040
	12	8	64	0.32	0.102
	13	6	36	0.24	0.058
	14	2	4	0.08	0.006
	15	7	49	0.28	0.078
	16	6	36	0.24	0.058
	17	6	36	0.24	0.058
	18	4	16	0.16	0.026
	19	5	25	0.20	0.040
	20	5	25	0.20	0.040
Sum		109	649	4.3600	1.038
Mean		5.45	32.45	0.2180	0.052
Standard Deviation		1.66		0.0663	
Standard Error of the Mean		0.37		0.0148	

The average fraction of points falling on the phase is 5.45/25 = 0.218, which means that the volume fraction of this phase is 21.8%. At a 95% confidence level the confidence interval for the point count is 2×SE = 2×0.37 = 7.4% while the confidence interval for the volume fraction of phases is 2×0.148 = 2.97%. [1]

References

1. R.T. Dehoff and F.N. Rhines, Quantitative Microscopy, McGraw-Hill, New York, 1968.