Introduction to Quantitative Metallography

Standard Stereological Terms

The following terminology is established in stereology:

P	Points	A	Flat areas	V	Volume
L	Lines	S	Curved surfaces	N	Number

The terms can be combined. Thus, S_V represents the amount of surface per unit volume, V_V the volume of a particular phase in the total volume (*i.e.* its volume fraction), P_P the number of test points lying in a particular feature as a fraction of the total number of test points *etc.*

Measurement of Volume Fraction

V_V, the volume fraction of a phase α in a microstructure can be measured directly by separation of the phases, *e.g.* the electrolytic extraction of carbides in a steel. However, this method is impractical for most purposes.

A_A, the area fraction presented by α on a planar section can be measured using image analysis. If σ_A is the standard deviation of the areas of individual α particles, then the measurement error E_A is given by

$$E_A^2 = \frac{1}{N}\left[1 + \left(\frac{\sigma_A}{\overline{A}}\right)^2\right] \qquad (1)$$

where N is the number of particles measured and $\overline{A}$ is the mean area measured. The error is minimised by making a large number of measurements (large N) and is expected to be smaller for uniform microstructures (small σ_A).

The line fraction L_L of α in a matrix β can be measured by projecting test lines onto the microstructure and measuring the part that falls in α. The error in such measurements depends on σ_L^α and σ_L^β which represent the standard deviations of the intercepts which fall in α and β respectively:

$$E_L^2 = \frac{1}{N}(1 - V_V)^2 \left[\left(\frac{\sigma_L^\alpha}{\overline{L}_\beta} \right)^2 + \left(\frac{\sigma_L^\beta}{\overline{L}_\beta} \right)^2 \right] \tag{2}$$

By contrast, measurements in which points are projected onto the microstructure to measure the point fraction P_P of α have the simplest error term:

$$E_P^2 = \frac{1}{P} \tag{3}$$

For sufficiently large numbers of measurements, $V_V = A_A = L_L = P_P$. The following experimental errors may also contribute to uncertainty:

1. The sample on which observations are made is unlikely to be flat. After all, observations are frequently made on etched samples. The surface relief can be characterised by the height h (Fig. 1a). Suppose the relief is caused by the etching of grain boundaries, than the measured volume fraction V_V^e may be overestimated compared with the true fraction V_V as follows:

$$V_V^e = V_V + \frac{1}{4} S_V h \tag{4}$$

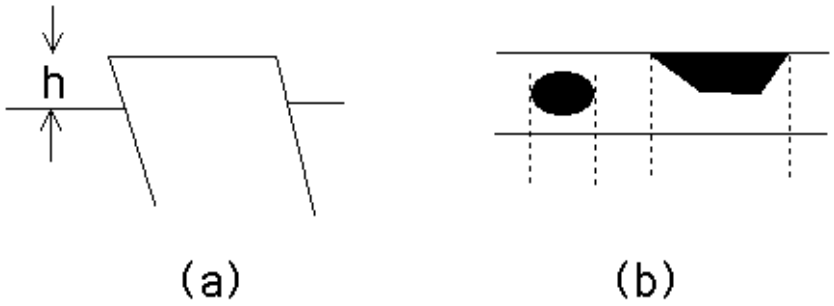

Fig. 1: (a) The surface relief caused by etching and characterised by the height h. (b) Projection of particles in a thin foil.

where S_V is the amount of grain surface per unit volume.

2. There may be errors due to microscope resolution. Interfaces appear as diffuse bands on two–dimensional sections; the thickness of the confused region δ is given by

$$\delta \simeq \frac{\lambda}{2n \sin \alpha}$$

where λ is the wavelength of the light used for imaging, n is the refractive index of the medium between the light and the sample and α is the angle relative to the incident light, by which reflected light is accepted in order to form the image.

3. Errors may be caused by looking at non–representative regions in heterogeneous samples.

Grain Size Determination

A common measure used in industry is the ASTM† grain size number N_g:

$$n = 2^{N_g - 1} \tag{5}$$

where n is the number of grains per square inch as seen in a specimen viewed at a magnification of 100! A large value of N_g implies a small grain size.

N_g	n	N_g	n
1	1	4	8
2	2	5	16
3	4	6	32

It is possible in practice to obtain grids which when inserted into the microscope allow a comparison with the underlying microstructure to directly give an estimate of the ASTM number.

The mean lineal intercept $\overline{L}$ is a much better method of characterising the grain size since it is related fundamentally to the amount of surface per unit volume, $S_V = 2/\overline{L}$. The intercept is measured by imposing test lines onto the microstructure. For a two–phase material,

$$\overline{L}_\alpha = \frac{LV_V^\alpha}{N^\alpha} \qquad\qquad \overline{L}_\beta = \frac{LV_V^\beta}{N^\beta} \tag{6}$$

where L is the total traverse length and N represents the number of grains of the appropriate phase within the total traverse length.

Particles in Thin Foils for Transmission Microscopy

A thin–foil is nevertheless a three–dimensional object and the image obtained is a projection of that object (Fig. 1b). For spherical particles

† American Society for Testing of Materials

of radius r in a foil of thickness t, there will be a projection error which is corrected as follows:

$$V_V = A_A^e \left(1 + \frac{3t}{4r}\right)^{-1} \tag{7}$$

where A_A^e is the projected area fraction. Naturally, the fraction estimated using A_A^e alone will appear larger than the true fraction V_V. Similarly, the number of particles per unit volume is related to the number counted per unit area as follows:

$$N_V = \frac{N_A}{t + 2\overline{r}} \tag{8}$$

where $\overline{r}$ is the mean observed radius.

Representative Samples

It was emphasised earlier that 'representative' samples should be used when making observations. This also means choosing the right technique or set of techniques for the problem concerned. The highest magnification technique is not always the right answer. The following example illustrates this by estimating the amount of metal that has been studied, using thin foil samples, in a transmission electron microscope since its invention:

t	foil thickness, $10^{-7}\,\mathrm{m}$	x	size of micrograph, $0.1 \times 0.08\,\mathrm{m}^2$
z	magnification, 5×10^4	N	number of pictures per year, 10^6
y	number of years, $= 40$	ρ	density, $7 \times 10^6\,\mathrm{kg\,m}^{-3}$

$$\text{mass of material examined} = txNy\rho/z^2 \simeq 10^{-4}\,\mathrm{kg}$$

$$\text{weight of a pin } \simeq 10^{-3}\,\mathrm{kg}$$

Classification of Shape

A shape factor must be dimensionless since shape does not depend on size. The divisor and divident must have the same units, *e.g.* area/length×perimeter, or it may be a number, such as the number of corners per grain. The shape factor must also be independent of the orientation of the feature.

It is useful to define some terminology before discussing shape factors:

A	Area	p	Perimeter
l	Longest Feret	b	Shortest Feret
c	Convex perimeter		

We shall see that the perimeter p should be used with caution since its value depends on the resolution of the measuring instrument. A Feret is the length of an object measured between two parallel rulers (Fig. 2). The convex perimeter c is that measured by tightly wrapping a string around an object and measuring the length of the string on unwrapping (Fig. 2). We now proceed to discuss some shape factors.

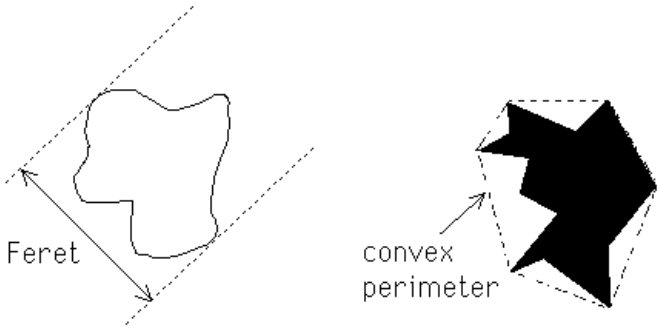

Fig. 2: Definition of a Feret diameter.

$p^2/4\pi A$ This shape factor has a minimum value of 1 for a true circle. It defines *roundness* and can be used to measure departures from circularity or surface smoothness.

$c^2/4\pi A$ ignores any indentations in the surface, so will classify a rough and smooth circle together.

l/b is useful to isolate fibres in the image.

p/c helps identify agglomerated clumps of particles from isolated particles.

Fractal Dimensions

For particles with complex surface topologies, for example clouds or other rough features, measures such as perimeter are not useful since they depend on the resolution of the measuring technique. Thus, a child walking around a coastline will measure a larger perimeter than an adult who has a longer foot–span. The child is able to penetrate nooks and crannies in the perimeter which the adult cannot. Similarly, the surface area of a catalyst as measured using the absorption of an atomic gas on the surface will not be the same as that measured using the absorption of polymer molecules.

Suppose that the perimeter p of the coastline is measured as a function of the resolution η. A plot of $\log p$ versus $\log \eta$ gives a straight line with slope $(1 - D)$, where D is the fractal dimension, which is a unique measure of the ruggedness of the coastline. The perimeter p can then be derived for an arbitrary resolution η.