

### *Metal Powder Production*

gas flow systems which remove a desired size fraction of particles. Other types of mills have been described in Chapter 4, where they are used as secondary processes. A disadvantage of mechanical method for powder production is the potential contamination from the balls and mill walls.

Flake metal powders are produced by flattening equiaxed particles produced by another method. These may employ ball, hammer or roll mills. When milling the original particles lubricants are added to prevent them from welding or sticking to each other. Aluminium, copper, bronze, silver, gold, iron and stainless steel flake powders are produced commercially by the milling method.

#### **2.4 Selection of Metal Powder Production Method**

Selection of the production method for a particular metal powder would depend on:

- (a) Raw Material Available
- (b) Type of End Application

Table 2.1 gives the list of specific equipment required for various methods of copper powder production. It confirms the simplicity in the case of atomization which has one handling in contrast to the cementation process, which has four. The process selection according to end applications depends on the powder characteristics. A detailed description of metal powder characterization shall be given in the next chapter.

#### **References**

1. A. Lawley (1978), Preparation of Metal Powders, In 'Annual Review of Materials Science', **8**, 49–71.
2. P.U. Gummesson (1972), High Pressure Water Atomization, in 'Powder Metallurgy for High Performance Application' Ed. J.J.Burke and V. Weiss, Syracuse University Press, Syracuse, 27.
3. P.R. Roberts (1984), Commercial Atomization by the Rotating Electrode Process, in 'Atomization Processes', Current and Future', P/M 84 Seminar Preprints, MPIF, Princeton, 51.

## **3 METAL POWDER CHARACTERISTICS**

### **3.1. Introduction**

The success of any powder metallurgical process depends to a great extent on the complete characterization and control of the metal powders. The method of powder production influences particle chemistry and structure, apart from the precise nature of particle size distribution. These properties also influence the behaviour of the powder during compaction and sintering, and the composition, structure and properties of the sintered material. In the present chapter, various characteristics of the powder, some interrelated, are considered. Table 3.1 gives a brief summary of principles involved in the characterization of various powder properties which shall be described in subsequent sections.

### **3.2 Chemical Composition and Structure**

The levels of impurity elements in metal powders can be very significant to both the processing and properties of the final product. It is necessary to know whether such elements are present in their elemental form or whether

**Table 3.1** Powder properties and methods of measuring them

Particle size and size distribution	Sieve analysis; Permeability; Sedimentation; electrical resistance; Light obscuration; Light scattering; Microscopy; Surface area.
Particle shape [external]	SEM; Shape parameters; Morphological analysis; Fractals.
Particle shape [external and internal]	Stereology; Mercury Porosimetry; Gas absorption.
Particle density	Pycnometry; Mercury porosimetry.
Specific surface area	Gas absorption; Permeametry.
Surface chemistry	X-ray photoelectron spectroscopy (ESCA); Auger electron spectroscopy; Secondary ion mass spectroscopy; Ion scattering spectroscopy.
Alloy phases and phase distribution	Optical metallography; Stereology; Electron microscopy; EDAX; X-ray diffraction.
Quality of mixing [segregation]	Macroregion: Variability coefficient (by chem.anal.); Microregion: Variability coefficient (2 <sup>nd</sup> comp. >5%); Homogeneity coefficient (2 <sup>nd</sup> comp. <5%); by metallography

### *Metal Powder Characteristics*

they are present in the form of a chemical compound. For example, in reduced iron powder silicon is present as impurity in the form of silica. Other ceramic and rather inert compounds may exist; these may be reported in terms of an acid insoluble figure. The effect of impurity elements on the hardness of the particles and the degree of chemical reactivity during sintering will differ widely, depending on the actual form they are in.

The hydrogen loss is a common parameter to know the level of oxygen impurities in those metal powders whose oxides are easily reducible by hydrogen, e.g. iron, tungsten, copper, nickel, etc. However, this value can be in error due to incomplete reduction of oxides, and some oxides may not be reduced at all. The annealing of the powder in a reducing atmosphere is an effective way of reducing oxygen contents. Details of procedures for determining these parameters can be had from the standards of Metal Powder Industries Federation (MPIF) and the American Society for Testing and Materials (ASTM).

Practically any metal powder adsorbs significant quantities of gases and water vapour from the atmosphere during storage. Such adsorption can lead to the formation of surface oxides on metals which may interfere with compaction and sintering and possibly remain in the sintered material. The amount of such contamination increases with decreasing particle size and with increasing chemical activity of the surface.

The microstructure of the crystalline powder has a significant influence on the behaviour of powder during compaction and sintering and on the properties of the final product. Fine grain size is always desirable, as it improves the mechanical properties apart from the sinterability and the uniformity of dimensional changes.

The grain size can be dependent on the powder particle size. The particular powder production method, e.g. rapidly cooled powder, would naturally give rise to small particles and also small grain sizes.

Prealloyed powders may contain various phases depending on their exact composition, the appropriate phase diagram, their thermal history and the method of powder production used. Multiphase microstructures may result from alloying such as those in steels, cast irons and superalloys. In the case of water atomized solid-solution type alloys, the microstructure, like that of the chilled structure, consists of a cored structure. Microporosity associated with entrapped gases is also common. A cold worked powder, e.g. ball milled, exhibits a high dislocation density which could be lowered by annealing. Such imperfections influence the compaction and sintering response of the concerned powders.

### **3.3 Particle Size and Shape**

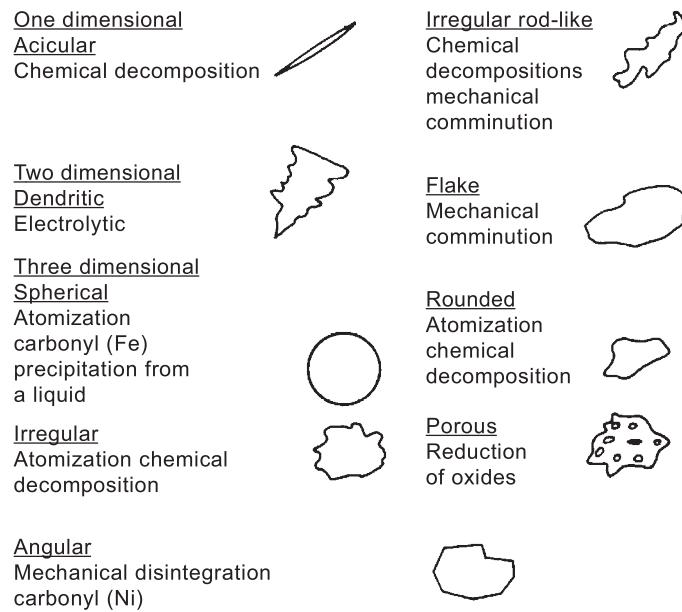
The shape of the powder is characterised by the dimensionality of the particle and its contour surface. An ideal system of shape characterisation is given in Fig.3.1, together with the major manufacturing techniques which produce such shapes. Most powder particles are three-dimensional in

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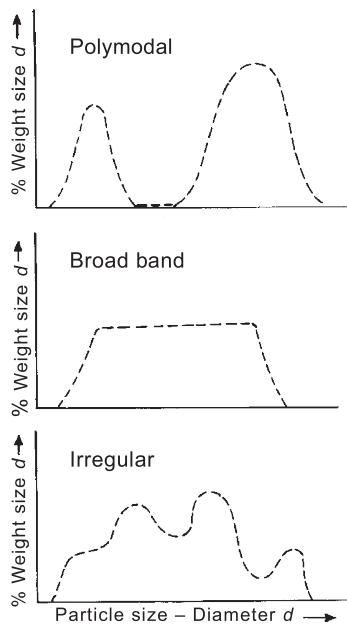
nature and they may be considered as being somewhat equiaxed. Spherical particles represent the simplest and ideal example of this shape. Porous particles differ from irregular ones because of the presence of the porosity, which itself may be very irregular in both size and shape. A large amount of porosity makes any shape characterisation very difficult.

In a real mass of powder, all prepared in the same manner, all the particles will not have the same exact size, even though the shape may be essentially the same. Consequently, we must deal with size distribution when accurately describing powders. There are various methods one may use to calculate average diameters. Particle size, therefore, is not a concise quantity but for any given nonspherical particle may have several values with different meanings, depending on the sizing method used.

Particle size distribution curves relate the particle size to the corresponding fraction of the powder with that size. Figure 3.2 illustrates various size distributions. In unimodal distribution, there is one high point or maximum amount of a certain critical size. The polymodal distribution consists of two or more narrow bands of particle sizes, each with a maximum, with virtually no particles between such band. The broad band distribution simply corresponds to a uniform concentration of particle sizes over a rather broad size interval with virtually no particles having sizes outside this range. The irregular distribution represents a continuous and finite variation of particle sizes within a relatively broad range. It can be thus concluded that the particle size distribution is necessary for a complete char-



**Fig.3.1** System of particles shape characterization.



**Fig. 3.2** Schematic illustrations of some realistic particle size distributions.

acterization instead of an average value or even maximum or minimum values of size.

There are a number of particle size measurement techniques available in powder metallurgy, each having their own limitations. Table 3.2 classifies some of the common methods of particle size determination and their limits of applicability.

Out of all the methods, sieving is technologically most satisfactory for reporting and plotting particle size distribution, in which the successive sizes form a geometrical series. The reference point for their scale has become 75 micrometer which is the opening of the 200-mesh woven wire screen standardized by the National Bureau of Standards. Because of the widespread use of this method, one distinguishes between particles which are larger than 44 micrometers and fines or subsieve powder that is smaller than 44 micrometers. Table 3.3 gives some of the most pertinent data for both the Tyler standard and United States sieve series. Micromesh sieves are also available with openings down to at least five micrometers, but are rather difficult to use and maintain, are very fragile and have low load capacities. These sieves are produced by electrodeposition of nickel or copper on to photosensitized machine ruled lines.

Another advantage of screening is that one may synthesize a desired distribution, according to the type of blend required by the manufacturer. Conventional sieving requires a sample of 50 g for accurate analysis and this becomes difficult in analysis of expensive metal powders.

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**Table 3.2** Common particle size determination methods and their limits of applicability<sup>2</sup>

Class	Method	Approximate useful size range (microns)
Sieving	Sieving using mechanical agitation or ultrasonic induced agitation and screens	44–800
Microscopy	Micromesh screens	5–50
	Visible light	0.2–100
	Electron microscopy	0.001–5
Sedimentation	Gravitational	1–250
	Centrifugal	0.05–60
Turbidimetry	Turbidimetry (light intensity attenuation measurements)	0.05–500
Elutriation	Elutriation	5–50
Electrolytic resistivity	Coulter counter	0.5–800
Permeability	Fisher sub-sieve sizer	0.2–50
Surface area	Adsorption from gas phase	0.01–20
	Adsorption from liquid phase	0.01–50

**Table 3.3** Information on sieves used in powder metallurgy

Mesh designation number	Sieve opening (μm)		
	New US series	Old US series	Tyler series
20	850	841	833
35	—	—	417
40	425	420	—
60	250	250	295
80	180	177	175
100	150	149	147
140	106	105	—
150	—	—	104
200	75	72	74
230	63	63	—
250	—	—	63
325	45	44	44

This is now overcome with the development of small sieves. A Ro-tap type machine is a common sieve shaker used in size analysis. Sieve shakers with different shaking mechanisms are used which depend on the shape of powder. A swirling motion is effective for round particles but is extremely slow for elongated particles. A jumping motion is very effective for elongated particles because it throws them into the air and allows them to rotate and perhaps land point first in a hole and pass through it. This means that a very consistent shaking motion is needed.

Another important industrial method for measuring the subsieve particle size is by what is known as the Fisher Subsieve Sizer. This is very much common in refractory metal powder and cemented carbide industries. In this case, the measured surface area is converted into an equivalent spherical surface diameter, which is only an approximate measurement tool. The technique does not measure the surface-connected porosity. A preweighed amount of powder is exposed to a known flow rate and the pressure drop is measured to determine permeability. From the knowledge of powder porosity and theoretical density, the surface area is calculated. The advantage of this method is that it is a direct reading one, which is very convenient for quick industrial quality control of fine powders. It must be clearly recognised that the value of specific surface obtained from a permeability experiment is representative of the ‘friction’ surface presented by powder mass to the flowing fluid.

For the details of other methods of particle size determination, one can refer to the book by German.<sup>1</sup>

### **3.4 Particle Surface Topography**

The nature of the surface of the individual particles is also an important powder characteristic. A spherical particle may appear smooth, but on a closer examination at high magnifications the surface may actually consist of many protuberances. Reduced metal powder has a highly roughened surface. Atomized metal powders, on the other hand, have finer degree of surface roughness, which are of rounded type rather than sharp and irregular. Scanning electron microscope is a powerful tool for examining surface topography. Surface contamination of particles and agglomeration of fine particles can also be studied by this technique.

The exact nature of surface topography will influence the frictional forces between particles. These are important in the case of bulk movement of the particles, when the powder is flowing, settling or during compaction. The extent of actual particle-to-particle contact during sintering will also be affected by the nature of the surface roughness. Chemical reactivity of the powder will also tend to increase with increasing surface roughness, especially the irregular type.

### **3.5 Surface Area**

The actual amount of surface area per unit mass of powder is of great sig-

nificance. Any reaction between the particles or between the powder and its environment starts at these surfaces. This affects sinterability. For a very irregular shaped particle with a high degree of surface roughness, the specific surface area can be very high.

The surface area of a given powder is measured by the BET method, in which an adsorption of a species in solution may be used to obtain a value of specific surface ( $S_w$ ) if the surface is completely covered by a monomolecular layer of the solute. From a knowledge of the area occupied by one molecule, the total area of the powder sample and, finally,  $S_w$  can be obtained. The amount of gas adsorbed in a monomolecular layer in  $\text{m}^2$  is calculated from an adsorption isotherm, i.e. a series of measurements of the volume  $V$  of gas adsorbed as a function of pressure  $p$ .

The BET method of determining the specific surface is widely used for catalysts. Its use for metal powder is primarily for very fine powders, particularly those of the refractory metals and for characterizing the total surface area of porous powders.

### 3.6 Apparent and Tap Density

The apparent density of a powder refers to the mass of unit volume of loose powder usually expressed in  $\text{g}/\text{cm}^3$ . It is one of the most critical characteristics of a powder, because of following reasons:

- (a) It determines the size of the compaction tooling and the magnitude of press motions necessary to compact and densify the loose powder;
- (b) It determines the selection of equipment used to transport and treat the initial powder;
- (c) It influences the behaviour of the powder during sintering;

Other characteristics which have direct bearing on apparent density are the density of the solid material, particle size and shape, surface area, topography and its distribution.

Apparent density is determined by the Hall flowmeter, where a container of known volume (25 ml) is completely filled by flowing metal powder through a Hall funnel (Fig.3.3).

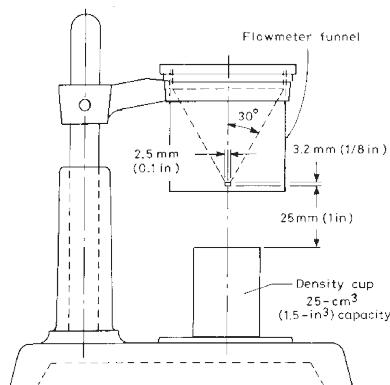


Fig.3.3 Hall flowmeter.