Powder Characterization

The processes of manufacturing P/M articles economically depend largely on the physical and chemical characteristics of the initial metal powders. The characteristics of metal powders depend upon the method used in producing these metal powders.

There are various methods of manufacturing metal powders and consequently there is a wide range in there characteristics. A choice regarding the suitability of manufacturing techniques of metal powders can be made only after considering the required finished product for a specific job. The main purpose of powder testing is to ensure that the powder is suitable for subsequent processing. A sample of metal powder is always selected for the determination of its characteristics and control of these characteristics is necessary for maintaining the (required) uniformity in different powder lots. The basic characteristics of a metal powder are:

1. Chemical composition and purity
2. Particle size and its distribution
3. Particle shape
4. Particle porosity
5. Particle microstructure

The other characteristics which are dependent entirely or a large extent on the above primary properties of metal powders:

1. Specific surface
2. Apparent density
3. Tap density
4. Flow rate
5. Compacting
6. Sintering

Primary properties such as the particle size distribution and the important secondary properties such as apparent density and flow rate are most widely used in specification and control routine. Other properties such as permeability regarding liquids and gases, magnetic properties, electrical and thermal conductivity, etc. are also of importance for special applications of P/M parts.

<u>Chemical composition</u>

The chemical composition of powders is the outstanding characteristic. It usually reveals the type and percentage of impurity and determines the particle hardness and compressibility. The term impurity refers to some elements or compounds which has an undesirable effect. Impurities influence not only the mechanical properties of the powder compacts, but also their chemical, electrical and magnetic properties.

It may also exert a decisive effect on pressing, sintering, and other post- sintering operation which are essential for the production of finished product from powders. Insoluble oxides such as alumina, silica in appreciable amounts give rise to serious troubles such as abrasion of die and poor mechanical properties due to non-uniform sintering. The chemical composition of a powder is determined by the well-established standard techniques of chemical analysis. Oxygen content is determined either by wet analysis or by "loss of weight in hydrogen".

Particle size

The particle size has a great importance in Powder Metallurgy because it affects most of the properties such as

- mould strength,
- density of compact,
- porosity,
- expulsion of trapped (occluded) gases,
- dimensional stability,
- agglomerations
- Flow and mixing characteristics.

Particle size is expressed by the diameter for spherical shaped particles and by the average diameter for non- spherical particles. Average diameter is defined in different ways according to the method employed for size distribution.

- When the method involves sizing, the particle size is measured as the opening of a standard screen which just retains or passes the particle.
- When determined by micro count method, the diameter is measured by averaging several dimensions.
- According to the sedimentation method the particle size is defined as the diameter of the spherical particle having the same specific gravity and settling velocity as the non-spherical particle under test.
- The average diameter in the case of large particle size can be determined by counting and weighing as the cube root of the volume.

In practical P/M metal powders are divided into three distinct classes:

1. Sieve
2. Sub-sieve
3. Sub-micron (or ultrafine)

The screen with the opening of finest standard mesh-sieve for production purposes is the 325 mesh screen having the aperture of 44 micron. Sub- sieve particles are smaller than the aperture of such a screen but greater than 1µ. This class of powder is used for the production of refractory metals, hard carbides and magnetic cores. The sub- micron powder particle size is smaller than 1µ and is used for the manufacture of dispersion strengthened high temperature alloy, bearing and micro porous components, magnetic materials, nuclear reactor fuels. Sieve size powders are used for most ordinary mass production because of their good flow ability and lack of further processing requirement such as granulation. Majority of metal powders employed in powder metallurgy industry vary in size from 4 to 200 microns. Powders of sub-micron size have been developed and used for the production of many powder metallurgical parts particularly dispersion strengthened materials, etc. Metal powder particle sizes in the range of 0.01 to 1 micron are employed for the production of these alloys but their (powders) use poses many problems. For example, apparent density is very low, flow rate is poor, inter particle friction is high, they agglomerate readily, they tend to be pyrophoric, they oxidize quickly with the atmosphere and difficulty reduce without bonding together and they alter their characteristics during their storage. The advantages include:

- Very fine grain sizes possible in the dispersed phase and the good dispersion itself
- Fine powders sinter easily at lower temperatures and in shorter times than are required by the coarse powders.
- Sintering is invariably accompanied by very high shrinkages which cause the dimensional control of parts more difficult.

Particle Size Measurement Technique

There are numerous techniques of particle size measurement. The more common techniques employed in P/M and their ranges of applicability are given in table below.

Method of Analysis	Approximate useful particle size range (microns)
1. Sieving Analysis	
• Sieving using mechanical shaking	44-840
• Micromesh sieve	5-44
2. Microscopic analysis	
• Light Microscopy	0.1-100
• Electron Microscopy	0.001-10
3. Sedimentation Method	
• Sedimentation and decantation Method	2-50
• Pipette Method	2-50
• Gravitational	1-50
• Turbidimetry	0.05-50
• centrifugal	0.05-10
4. Elutriation Method	
• Elutriation	5-100
• Roller Air Analyzer	5-40
5. Permeability Method	
• Permeability	0.5-100
• Fisher Sub-sieve sizer	0.2-50
6. Adsorption Method	
• Adsorption (gases)	0.002-20
7. Electrolytic Resistivity Method	
• Coulter counter	0.3-300

Particle Shape

There are various shapes of metal powders such as:

1. Spherical (carbonyl iron, condensed zinc, lead, atomization, precipitation from aqueous solution by gases)

2. Rounded or droplets (atomized copper, zinc, aluminum, tin, chemical decomposition)

3. Angular (mechanically disintegrated Sb, cast iron, stainless steel obtained by intergranular corrosion)

4. Acicular (chemical decomposition)

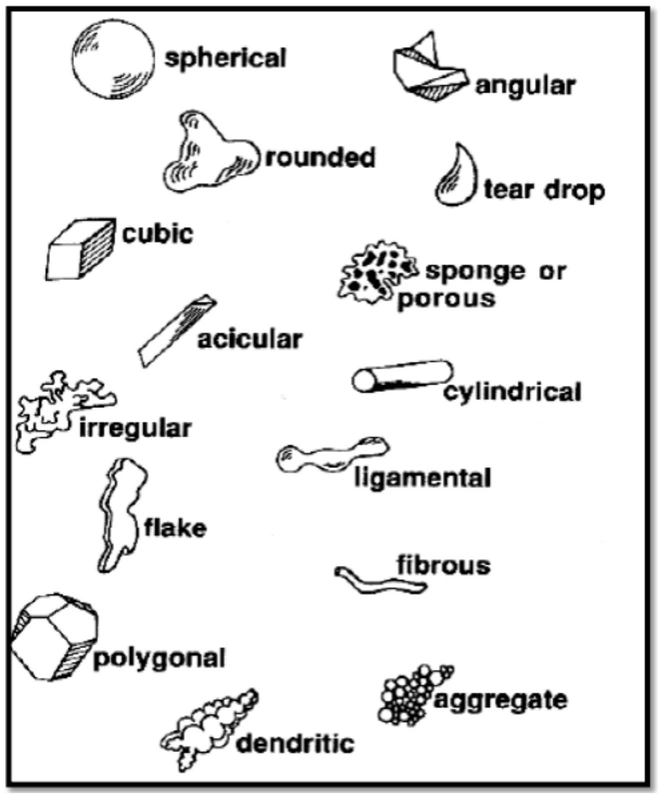
5. Dendritic (electrolytic silver, copper, iron powders)

6. Flakes (ball milled copper, aluminum, and stamped metals)

7. Porous (reduction of oxides)

8. Irregular (atomization, reduction, chemical decomposition)

9. Fragmented



Particle shape has a pronounced effect on the packing of a powder and has an influence on its compacting and sintering properties and the mechanical strength of the sintered product thus; irregularly shaped particles have reduced apparent density and flow rate, good pressing and sintering properties, while spherical particles have maximum apparent density and flow rate but reduced pressing properties and good sintering characteristics. In the same way, dendritic powders result in reduced apparent density and poor flow rate.

Specific Surface

The specific surface of a powder is defined as the total surface area per unit weight (cm^2 /gm). It depends on size, shape, density and surface conditions of the particle. The compacting and sintering properties are considerably influenced by contact between the metal particles. High specific surface not only results in high sintering rate, but also causes entrapment of air and bridging effects thereby causing the compact to crack rather before or during sintering. Coarser powders with smaller contact areas have an inferior sintering characteristic and, therefore, mechanically weak.

Apparent Density

The apparent density of a powder is defined as the mass per unit volume of loose or unpacked powder. Thus it includes internal pores but excludes external pores. It is governed by chemical composition, particle shape, size, size distribution, method of manufacture of metal powders as well as shape and surface conditions which can vary from 20-50% of the theoretical density.

The main factor is not the particle size but the particle size distribution for altering the apparent density. Thus the uniform and identical particles occupy a constant fraction of the available space but various sized particles increase the density to an optimum extent. An increase in apparent density is obtained with additions of fine particles and results from the ability of fines to fill the inter particle voids. Because of their more brittle behavior, oxides present on the surface result in the lowering of strength and altering the apparent density of the metal powder.

Tap Density

Tap density (or load factor) is the apparent density of the powder after it has been mechanically shaken down or tapped until the level of the powder no longer falls. It appears to be widely used for storage, packing or transport of commercial powders and also a control test on mixed powder.

Flow Rate

The flow rate is a very important characteristic of powders which measures the ability of a powder to be transferred. It is defined as the rate at which a metal powder will flow under gravity from a container through an orifice both having the specific shape and finish. The powder filling of die must be rapid and uniform without bridge formation for obtaining a rapid rate of production, consistent compacts and economy.

On the other hand poor flow properties of the powder result in the slow and uneconomical feeding of the die cavity and the possibility during pressing of uneven filling of the die cavity. It is affected only by particle size, size distribution and shape, but also by absorbed air or gas, moisture, lubricant, coefficient of inter particle friction, etc. in general, fine or dendritic, irregular, coarse and spherical powders have poor, reduced, good and maximum flow rates respectively. Flow rate increases with decreased particle irregularly and increased particle size, specific gravity, and apparent density. It can also be increased by tapping or vibrating.

The standard apparatus, known as Hall Flowmeter, is generally used for the determination of flow rate. It consists of a standard and accurately machined conical funnel made of brass with smooth finish having an internal angle of 60º. The orifice situated at the bottom of the funnel is either 1/8" for ferrous powders or 1/10" in diameter for non-ferrous powders. The time required to flow the weighed sample of powder (usually 50 gm) from the funnel into a cup held at a fixed distance below the orifice is a measure of flow rate, which is expressed in seconds or gm/minute in case a non- standard weight of the sample is employed. There is such a close relationship between apparent density and flow properties that is very difficult to vary one without altering the other. Flow rate, apparent density, and tap density are essential processing factors since they affect the transporting and pressing of powders.

Compressibility

Compressibility is one of the most important characteristics of a metal powder since it affects the densification process. It is a measure of the powders ability to deform under applied pressure and is represented by the pressure/ density (or pressure/ porosity) relationship, It is defined as the ratio of: a. The green density of the compact to the apparent density of the powder This ratio is usually termed "compression ratio". The maximum compression ratio is given by the relation:

$$\text{Maximum compression ratio} = \frac{\text{Ultmate or true density of bulk material}}{\text{Apparent density}}$$

Though the compression ratio can be varied from about (2 – 8), in most practical applications, it approaches a value of about 3. Higher values of compression ratio require greater die depths and produce severe complications due to the introduction of friction between the powder and the die walls and internal friction of the powder.

Thus a low compression ratio is preferred because of the reduction of: i. Die depths ii. Breakage and wear of tooling iii. Press movement and thereby it is possible to achieve higher production rate.

Compactibility

Compactibility is indicated by the pressure/green strength relationship. Compactability of powder is defined as the minimum pressure required producing a compact of given "green strength". Both these terms are dependent on particle size, shape, porosity or density and hardness, surface properties, chemical composition and previous history (e.g. hardening, annealing treatment, etc) of the powder.

Green Density

In general, green density has been found to increase with: i. Increase of compaction pressure ii. Increase of particle size or apparent density iii. Decrease of particle hardness and strength iv. Decrease of compacting speed Improvements in green density may also be affected by employing smooth and regularly shaped annealed particles with high particle densities (possessing no internal or interconnected porosity).

Green Strength

The strength of the green compacts is primarily dependent on the consolidation pressure and a rise mainly from cold welding and mechanical interlocking neighboring particles and particle shearing. Particle shape and structure have a great effect upon green strength. Thus, it can be improved by using soft irregular shaped particles with clean surfaces.

Green Spring

Another property of the green compact, associated with the difference between the size of the compact and the tools employed to prepare, it is usually termed green "spring" because the compacts expand both radially and longitudinally on ejection from the tools. During compacting, plastic deformation of the powder particles causes work hardening and an increase in the elastic limit. As the compact leaves the die, there is elastic recovery of the residual stresses and when it exceeds the green strength of the compact, cracking will occur on ejection. For the manufacture of parts with close dimensional tolerance, it is necessary to determine the extent of green spring. In general, the green spring upon ejection amounts to 0.2% on the diameter and 0.5% on the length. It depends on the powder material, compacting pressure, elastic recovery of the tools and design of the dies.