

Powder production

The production of powders for PM products can be divided into mechanical and chemical methods. The final properties and the price are strongly dependent on the raw-material cost, the production method, particle shape, size and distribution, impurities, and oxygen sensitivity.

Mechanical Methods

Disintegration of solids (without phase change) and liquids (with phase change) can be applied to produce metal powder.

Disintegration without Phase Change

The production of fine chips by machining techniques such as milling or turning is one of the easiest ways to produce powder, but also one of the most expensive ones because the productivity of these processes is relatively low and the danger of contamination is high. Therefore, it is applied only for producing extremely expensive materials and for special purposes.

Milling

The classical approach to break down brittle material is milling in ball mills. The material is stressed by the impact, compression, and shearing of the hard balls, and finally is disintegrated into smaller particles.

The average particle size for most metals produced by milling is $>100\ \mu\text{m}$, and the average particle size approaches a constant value depending on the ductility of the powder and the processing conditions (especially temperature). Brittle ceramics or metals may give particle sizes of $<1\ \mu\text{m}$.

Typical mills are tumbling ball mills, vibratory mills, and attritors. Jet mills are based on the rotating motion of a particle-loaded gas stream at high speed in a round chamber. The high-velocity collision of the particles leads to fragmentation, and the small particles are removed in the center of the chamber whereas large particles are retained in the stream until the desired particle size is reached. The biggest advantage is that the incorporation of impurities is limited because of the absence of milling aids (balls etc.). The milling process finally leads to an increase of the dislocation density up to $10^{15}\ \text{m}^{-2}$ and, therefore, reduces the compactibility. In most cases, subsequent heat treatment is necessary to soft-anneal the powders before use.

Disintegration with Phase Change

Liquids can be disintegrated by using gases, water, oil, or mechanical dispersion.

Atomization: The water jet process is the most modern and productive way to obtain metal powders for PM parts production. The principle of water atomization is to disintegrate a free-falling melt stream, supplied from a nozzle in a tundish, by water jets. The disintegrated melt

solidifies immediately, and the surface oxidizes. The powder–water slurry is collected, dried, and subsequently reduced with hydrogen. The freshly reduced powder will slightly sinter to form a sort of cake that is mildly milled before the final treatment of the powders (magnetic separation, screening, and equalizing). The arrangement of water atomizer illustrated in figure (2).

The method can be used to produce iron and steel powders (alloyed with Mo, Ni, or Cr), stainless steel powders, copper, and copper alloys. The final powders have an irregular shape that provides good green strength by interlocking of the particles. The process involves many variables: the temperature and the amount of superheat of the molten material (related to the composition), the water-to-metal ratio (10–15 L per 1 kg of produced powder), the diameter of the molten-metal stream, the geometry of the nozzle (amount of water jets and angle of incidence between water jet and molten metal stream), and the water pressure.

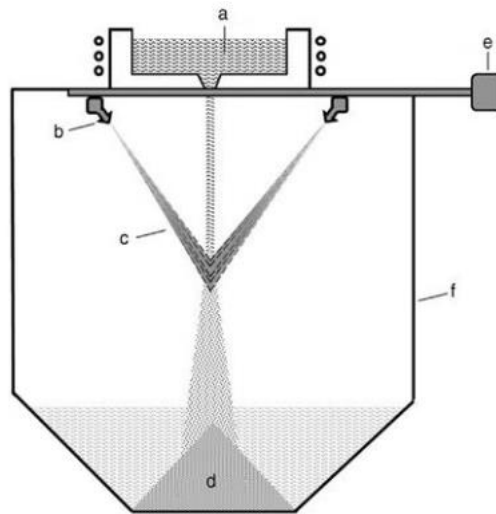


Figure 2 : Principle arrangement of a water atomizer a) Melt; b) Jet; c) Water spray; d) Powder; e) Pressure source; f) Chamber

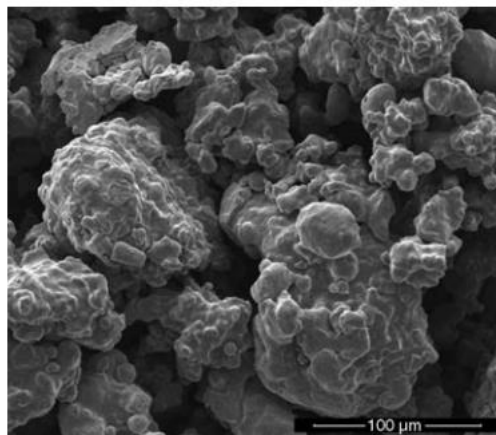


Figure 3: Typical water-atomized powder.

Gas atomization

Atomization by inert gases is used to produce powders from oxygen-sensitive elements if water atomization is not applicable because of the reactivity of the metals. The typical shape of gas-atomized powders is spherical, which is needed for applications where high bulk densities and flow rates are required. The slower cooling rate of 1000 K/s than that of water atomization, in which case 10 000 K/s is possible, allows the droplets to form spheres before solidification. As the energy input of the gas is much lower compared to that of water, the production of fine particles requires special techniques, e.g., by special nozzles that allow extremely high gas speeds. Figure (4) shows the spherical particles which produced by gas atomization.

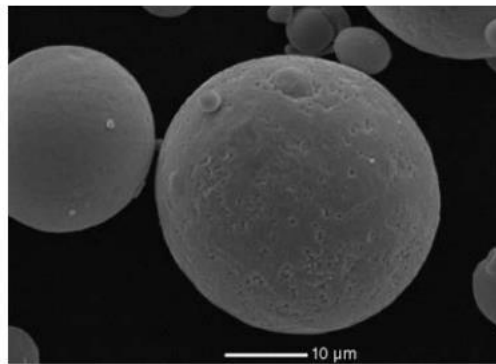


Figure 4: Typical gas-atomized powder

Some materials such as titanium and Ti alloys require techniques where the molten metal must not interact with crucibles. The fragmentation by centrifugal forces from a rotating electrode is used to disintegrate the material without contact to a nozzle. The melting of the material is performed by an electric arc or plasma torch on the front surface of the rotating electrode. The centrifugal forces and slow melt supply lead to fine powders with highest purity as the process is performed in argon. The drawback is the low productivity of these methods. The increasing demand for titanium and Ti alloys with highest quality led to plasma atomization in which a metal wire or ingot is introduced into the atomization chamber and molten at the front face by a plasma torch; an argon stream disintegrates the liquid material. Inductively coupled plasma methods are also used to transform crushed, atomized, and sponged powders into pore-free spherical powders.

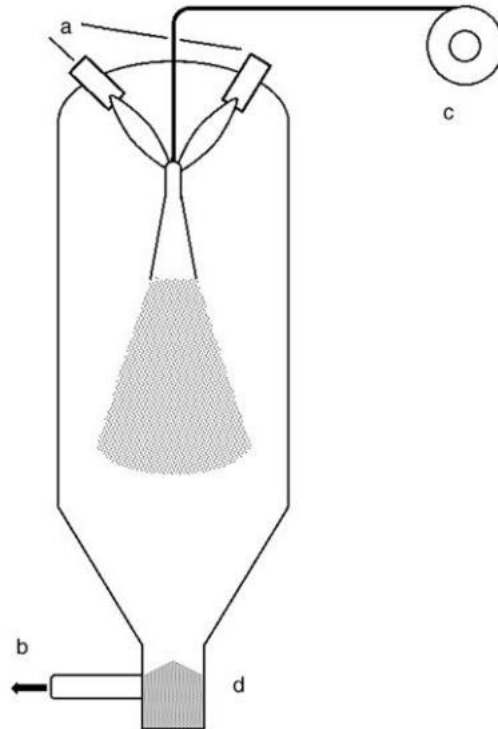


Figure 5: Principle of plasma atomization a) Plasma torches; b) Vacuum pump; c) Titanium spool; d) Powder collection.

Electrolytic Method

It is used extensively in the preparation of copper, beryllium, iron and nickel powders. Adjustment of the chemical and physical conditions during electrodeposition makes it possible to cause the metal to deposit loosely on the cathode of the cell either as a light cake or in flake form. Both are readily crushed to a powder. The method yields a high purity metal with excellent properties for conventional powder metallurgy processing.

The process involves the control and manipulation of many variables and in some cases is significantly more costly than other techniques. The following are the factors promoting powdery deposits: (a) high current densities; (b) weak metal concentrations; (c) additions of colloids and acids; (d) low temperature; (e) high viscosities; (f) avoidance of agitation; (g) suppression of convection.

Chemical Methods

These methods can be further classified as chemical reduction and decomposition.

Chemical Reduction

Chemical reduction involves chemical compound most frequently an oxide, but sometimes a halide or other salt of the metal. This may be carried out:

Oxide reduction:

Reduction of compounds particularly oxides by the use of reducing agent in the form of either solid or gas according to the reaction: This is a convenient, economical and extremely flexible method for controlling the properties of the product regarding size, shape and porosity over a wide range. The production of iron, copper, tungsten, and molybdenum powders from their respective oxides are well-established commercial processes. On a smaller scale, oxide reduction is also used for production of cobalt and nickel powders. This process yields extremely fine powders with irregularly shape particles, good compactibility (high green strength) and sinterability, low final porosity of such powders and relatively low cost.

Precipitation:

The principal of precipitating a metal from its aqueous solution by the addition of a less noble metal which is higher in the electromotive series has been applied in numerous metallurgical processes. Ag powder is produced in quantity from its nitrate solution by adding copper or iron according to the reaction: This method is used for producing metal powders of Ag, Sn, Pt and iron particles coated with copper. Precipitation is also synonymous with the term electrolytic precipitation to coat metals with a corrosion resistance film. The produced powder take the form of spongy mass which crashed into a hard and brittle powder.

Chemical Decomposition of Compounds

Under this category of powder production two methods are very common.

These are : (i) Decomposition of metal hydrides (ii) Decomposition of metal carbonyls

Decomposition of metal hydrides:

This involves first hydriding the refractory metals like Ti, Zr, Hf, V, Th or U by heating the metal in the form of sponge, chip or turnings or even compact metal in hydrogen. TiH_2 is formed from titanium in the temperature range between 300–500°C. These hydrides are quite brittle and can be readily ball-milled into powder of the desired fineness. These may be dehydrided by heating them in a good vacuum at the same temperature at which the hydride was formed. Care must be taken to avoid contamination of O_2 , N_2 and C during hydriding or dehydriding Uranium hydride may serve as intermediate not only in producing uranium metal powder, but also UC and UN powder.

Decomposition of metal carbonyls:

The famous example under this category is iron and nickel powder production. The carbonyls are liquids at normal temperature with a low boiling point. These are formed by reaction of the metal and carbon-monoxide gas under pressure. For example, iron carbonyl

(Fe(CO)₅) is formed at 70–200 atmosphere pressure and a temperature of 200–220°C. The carbonyls can now be decomposed by heating the vapour at atmospheric pressure.