fast rates economically. Consequently, in many cases the deposit is a solid and must be pulverized, for example iron. Electrolytic deposits, powders or solids are usually very reactive and brittle. For both these reasons the material may be given a special annealing treatment. Powders formed during electrolysis have a characteristic dendritic shape; however, this could be changed substantially due to subsequent processing.

The electrolytic process for copper powder is similar to that in copper refinery. However, instead of using impure cast copper anodes, electrolytically refined copper anodes are used. The cathode is generally cast antimonial lead. The electrolyte concentration is 50 g/l of $CuSO_4 \cdot 5\ H_2O$ (in contrast to 150 g/l for refining) while the current density is 535 A/m^2 (in contrast to 107–215 A/m^2 for refining). The copper powder is washed and filtered and finally given an annealing and reducing treatment at temperatures between 500–800^{o}C in a belt furnace with an atmosphere of partially combusted hydrocarbon gas. Powder properties, particularly its apparent density, are primarily controlled by the reducing treatment after electrolytic deposition. The reduced powder forms a cake which must be broken up into powder by a hammer mill.

In contrast to copper powder, electrolytic iron is not deposited as powder but as a brittle lightly adhering sheet like deposit on stainless steel cathodes. The anodes are usually Armco iron or low carbon steel. The electrolyte is a chloride or sulphate solution. The brittle nature of the deposit is because of hydrogen and or oxygen as impurities and can be readily milled into powder in ball mills. Powder must be annealed so as to make it soft and pure and the resulting cake is again ground into powder in a hammer mill. Electrolytic iron powder is a premium product because of its purity and its better compressibility (higher green density) compared with reduced or atomized powder and demands a price much higher than that of reduced powder. Figure 2.2 shows the schematic of the electrolytic process for making metal powders.

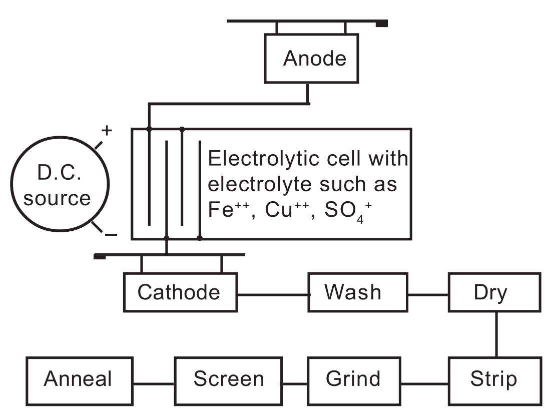

Fig. 2.2 Schematic of the electrolytic process for making metal powders.

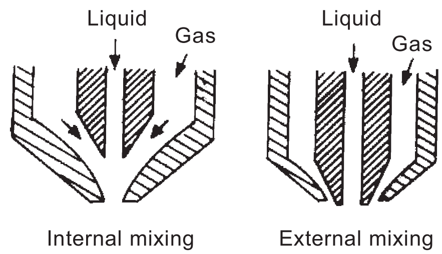

Fig. 2.3 Two fluid atomization design.

2.2.2 Atomization

Atomization has been classified into categories, namely gas, water and centrifugal.[1] Virtually, any material that can be melted can be made into powder by disintegration of the liquid. Aside from chemical reactivity, which may necessitate specific atmosphere or materials, the process is independent of the normal physical and mechanical properties associated with the solid material. The method is being widely adopted, especially because of the relative case of making high purity metals and prealloyed powders directly from the melt. The basic procedure employed is to force a liquid through an orifice, possibly at a bottom of a crucible and impinge a gas or liquid stream on the emerging melt. A great deal depends on the exact design of the orifice. It may induce turbulence in the melt which atomizes the material directly and allows the impinging gas or liquid to reduce the size of the particle still faster.

Gas atomization: The general atomizing media are nitrogen, argon or air. Various atomization geometries are used in commercial practice. In what is known as 'external mixing' (Fig.2.3), contact between the atomizing medium and melt takes place outside the respective nozzles. This type of mixing is used exclusively for the atomization of metals. 'Internal mixing' (Fig.2.3) is quite common for the atomization of materials which are liquid at room temperature. The axes of the gas jets are equally inclined to the melt stream axis and intersect this axis at the geometrical impingement point. The process is governed by a number of interrelated operating parameters. Controllable variables include jet distance, jet pressure, nozzle geometry, velocity of gas and metal, and melt superheat.

Gas atomized powders are typically spherical, with relatively smooth surfaces. Higher pressure and/or a smaller jet distance produce finer powder. Gas atomization pressures are typically in the range 14×10^5 Pa to 42×10^5 Pa at gas velocities from 50 $m\cdot s^{-1}$ to 150 $m\cdot s^{-1}$; under these conditions, the particle quench rate is ~10^2 $K\cdot s^{-1}$. Such production method is used for preparing powders of the superalloys, titanium, high speed steel and other reactive metals. The method suffers from a very low overall energy efficiency (~3 %) and is expensive if inert gases other than nitrogen have to be used.

Water atomization: In water atomization, a high pressure water stream is forced through nozzles to form a disperse phase of droplets which then impact the metal stream. In this method, large quantities of energy are required to supply the water at high pressure. It is estimated that the overall energy process efficiency is ≤4 %. This production method is significant for low and high alloy steels, including stainless steel. Because of oxide formation, water atomization is not likely to be used in the atomization of highly reactive metals such as titanium and the super alloys. In general, water atomized powders are irregular in shape, with rough oxidized surfaces.

The advantages of atomized powders and more specifically, of high pressure water atomized powders are summarized by Gummeson.[2] These are:

1. Freedom to alloy
2. All particles have the same uniform composition
3. Control of particle shape, size and structure
4. Higher purity
5. Lower capital cost.

Fine particle sizes are favoured by:

(a) Low metal viscosity
(b) Low metal surface tension
(c) Superheated metal
(d) Small nozzle diameter, i.e. low metal feed rate.
(e) High atomizing pressure
(f) High atomizing agent volume
(g) High atomizing agent velocity
(h) High atomizing agent viscosity
(i) Short metal stream (*F*)
(j) Short jet length (*E*)
(k) Optimum apex angle (α^{o}).

Figure 2.4 illustrates some major variables in the atomizing process.

Particle shapes of atomized powders can be modified from almost perfectly spherical to highly irregular, by controlling the processes which take place in the interval between disintegration of the liquid metal stream and the solidification of the drop. Sphericity of a metal powder is favoured by:

(a) High metal surface tension
(b) Narrow melting range
(c) High pouring temperature
(d) Gas atomization, especially inert gas
(e) Low jet velocity
(f) Long apex angles in water atomization
(g) Long flight paths.

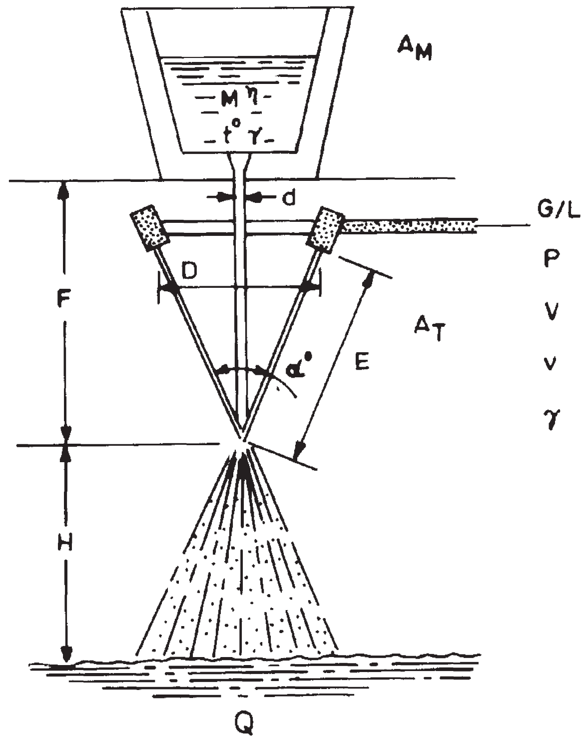

Fig. 2.4 Major variables in the atomizing process.[2]

In water atomization, water pressures are commonly in the range of 35×10^5 Pa to 210×10^5 Pa with associated water velocities from 40 $m\cdot s^{-1}$ to 15 $m\cdot s^{-1}$. The particle cooling rate is ~ 10^3 $K\cdot s^{-1}$ to 10^4 $K\cdot s^{-1}$.

The surface tension of liquid metals is high and a droplet once formed tends to assume the spherical shape. The higher the viscosity of the atomizing medium, the greater is the deformation of the droplet. The higher the cooling rate, the shorter is the time during which the surface tensional forces can operate to spheroidize the droplet and, therefore, the more irregular the particle shape. Impurities and alloying elements in the metal or reactions on the surface of the droplets that decrease the surface energy, will promote irregular particle shapes. Small quantities of phosphorus in copper lead to formation of a P_2O_5 film at the particle surfaces which increase the surface energy and results in the formation of spherical droplets. The existence of a solid oxide film such as ZnO acts in the opposite fashion, tending to give less rounded particles. The addition of silicon is a well-known method of influencing the particle shape of atomized stainless steel powder.

While the particle shape is not appreciably influenced by metal pouring temperatures in gas atomization, it is in water atomized powders. At higher pouring temperatures, there remains enough superheat after atomization to allow surface tension forces to create spheroids. Higher water pressure results in more irregular particle shapes due to greater impact forces and to larger volumes of water with resultant more rapid quench.

Another aim during atomization of particularly multiphase alloy systems is the elimination of gross metal segregations. The particle structure is therefore, a function of the solidification rate. A fine microstructure particle is promoted with water atomization as opposed to gas atomization, by lower metal pouring temperatures, higher atomizing agent pressure, flow rate and viscosity and by shorter particle flight paths.

Liquid gas atomization: The Krupp Company (Germany) introduced a novel version of atomization in which the melt is atomised with cryogenic liquid gas (argon or nitrogen) at –200°C. During the process, the pressure of the liquid gas is increased up to 300 bar, while a recooling unit prevents the temperature from rising in spite of compression and prevents the cryogenic liquid from vaporizing instantaneously at the jet opening. An even stream is generated, which atomizes the melt comparably to water atomization and which cools rapidly. Since the atomization liquid vaporises completely, gas and powder can be easily separated in the cyclone. The resulting powder has the following properties:

– It is much purer than the powder atomized with water and can be compared to the quality of the gas atomized powder.

– The cooling rate is ten times higher than in gas atomization and almost reaches the quality of water atomization. Particles of 100 µm in diameter, for example, are quenched for approximately 10^6 K/s.

– The powders are, as in gas atomization, spherical and have an average size of 6–125 µm. Due to the distinctive presence of satellites mainly low alloy powders show satisfactory results in cold forming, while having a good flowability. Gas atomized powders, on the other hand, have poor green strength.

Centrifugal atomization: The basis of centrifugal atomization is the ejection of molten metal from a rapidly spinning container, plate or disc. The rotating electrode process (REP) is a further example of centrifugal atomization.[3] The material in the form of a rod electrode is rotated rapidly while being melted at one end by an electric arc. Molten metal spins off the bar and solidifies before hitting the walls of the inert gas filled outer container (Fig.2.5). The process was developed primarily for the atomization by high purity low oxygen content titanium alloys and superalloys. Powder particles are smooth and spherical with an average diameter of ~200 µm; the size range is 50–400 µm. Typically, yields run to ~75% for –35 mesh powder.

Tungsten contamination from the stationary electrode is a limitation of REP powders. To eliminate this, the PREP (Plasma Rotating Electrode Process) method has been commercialised (Fig.2.6). It is important that the electrodes are precisely dimensioned and straight. This can be achieved by

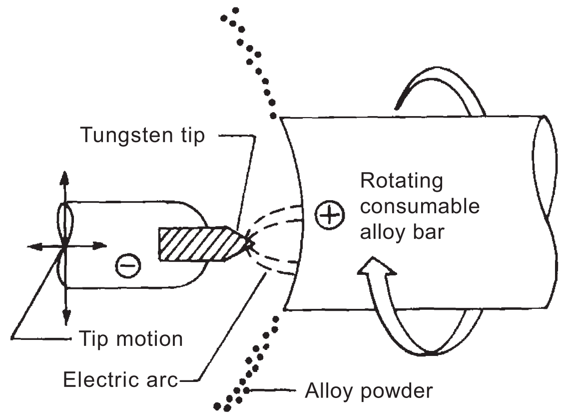

Fig. 2.5 Rotating electrode process – schematics [3].

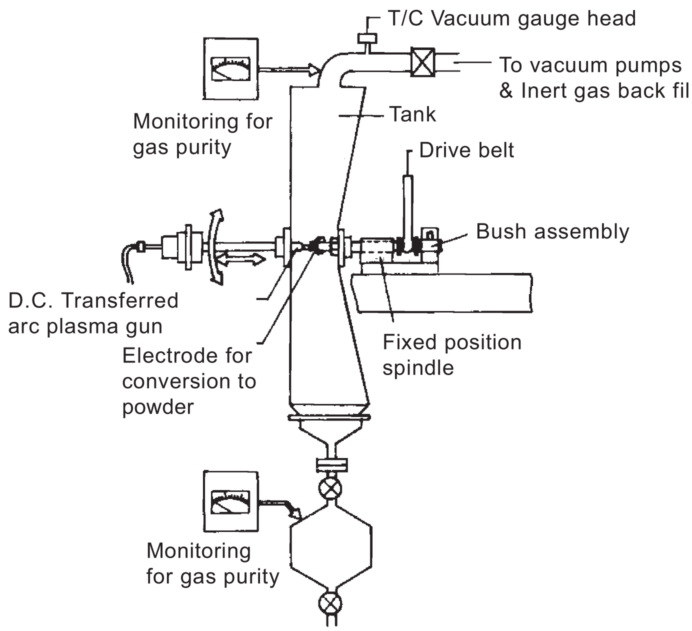

Fig. 2.6 Short bar PREP machine.

subjecting cold drawn rod to cross roll straightening.

Resonance effects are experienced in the REP. The natural frequency experienced is a function of the modulus of elasticity (E), the sectional moment of inertia (I), the beam length (l) and the beam weight (w) or weight per unit length (m) and is given by the relationship:[3]

$$f = C_n\sqrt{\frac{EI_g}{Wl^3}} \text{ or } C_n\sqrt{\frac{EI_g}{ml^4}}$$

where C_n is a constant depending upon beam support conditions and mode number of vibration and g is the gravitational constant (9.81 m/s^2).

Accurately controlled rotation of the anode is important so as to obtain a desired range of particle size distribution. The molten droplet diameter in a given material is determined by such parameters as the surface tension of the liquid metal, centrifugal forces (related to rotation speed) and to some extent by the 'aerodynamics' of the droplets trajectory through the inert cover gas.

The design of REP or PREP equipment includes sufficient damping to suppress or withstand resonant vibrations of moderate amplitude. However, when the electrodes are marked by out of tolerance for straightness, severe loading is imposed on the spindle and seal mechanism.

Spherical metal powders made by either REP or gas atomization are not well suited for cold pressing into green compacts to be followed by sintering. They are used in more specialized applications where consolidation is achieved by hot isostatic pressing (HIP) or some other high temperature method in which inter particle voids are more readily closed.

There are various mechanical atomization methods like Roller atomization, vibratory electrode atomization and ultrasonic atomization. An excellent review has been made by Lawley.[1] The details of such methods are out of scope of the present book.

Vacuum atomization: Vacuum or soluble gas atomization is a commercial batch process based on the principle that when a molten metal supersaturated with gas under pressure is suddenly exposed to vacuum, the gas expands, comes out of solution, and causes the liquid metal to be atomized. Alloy powders based on nickel, copper, cobalt, iron and aluminium can be vacuum atomized with hydrogen. Powders are spherical, clean and of a high purity compared to powders produced by other processing methods. The process was developed and patented by Homogeneous Metal, Inc. Figure 2.7 illustrates a schematic of the equipment used for the atomization process. The principal use of powder made by vacuum atomization has been for the production of gas turbine disks and intricate parts by injection moulding.

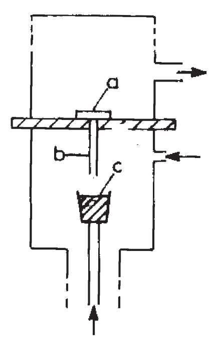

Fig. 2.7 Schematic representation of vacuum atomization. (a) trap door, (b) transfer tube, (c) molten metal.

Table 2.1 P/M grade copper powder: Specific equipment requirements

Process	Cementation	Electrolysis	Oxide reduction	Water atomization
Equipment	Reaction cells	Melting furnace (for melting anodes)	(Drying)	Melting and atomization device and pump
	Purification device	Electrolysis cells and current rectifier	Magnetic separator	
	Cementation cells	Washing and neutralizing device	Mill air separator	
	Washing device		Atmosphere generator	
Number of handling	4	3	2	1

2.3 Mechanical Methods

These processes are not much used as primary methods for the production of metal powders. Mechanical comminution is possible by methods such as impact, attrition, shear and compression. The formation of metal powders by mechanical methods relies on various combinations of these four basic mechanisms. Such methods have been used as the primary process for the following cases:

– materials which are relatively easy to fracture such as pure antimony and bismuth, relatively hard and brittle metal alloys and ceramics.

– reactive materials such as beryllium and metal hydrides.

– common metals such as aluminium and iron which are required sometimes in the form of flake powder.

A common method is the use of a ball mill consisting of a rotating drum with hard wear resistant balls. The critical factor is the speed of the drum's rotation. A very high speed will cause the material and the ball to be pressed against the walls of the drum, because of the centrifugal forces and prevent relative motion between the material and the balls. Too low a speed will result in an insignificant amount of movement in the lower part of the drum. The optimum speed corresponds to a situation in which some amount of ball and material is lifted up to the top of the drum and falls down on the remaining material.

Another type of mill is a vortex mill in which particles of materials to be ground are fractured by mutual contact or collision. Such mills consist of two or more very rapidly rotating propellers within the mill casing and