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فهرست مراجع

- ❑ An Introduction to Powder Metallurgy; F. THUMMLER and R.OBERACKER, © 1993 The Institute of Materials.
- ❑ ASM Handbook; Volume 7, Powder Metal Technologies and Applications.
- ❑ Presentation files related to each topics

شیوه ارزیابی

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An Introduction

Definition of Powder Metallurgy

Powder metallurgy may defined as, “the art and science of producing metal powders and utilizing them to make serviceable objects.”

OR

It may also be defined as “material processing technique used to consolidate particulate matter i.e. powders both metal and/or non-metals.”

OR

is a process whereby a solid metal, alloy or ceramic in the form of a mass of dry particles is converted into an engineering component of predetermined shape and possessing properties which allow it to be used in most cases without further processing.

An Introduction

Powder metallurgy is a forming and fabrication technique consisting of three major processing stages.

- First, the primary material is physically powdered, divided into many small individual particles.
- Next, the powder is injected into a mold or passed through a die to produce a weakly cohesive structure (via cold welding) very near the dimensions of the object ultimately to be manufactured.
- Finally, the end part is formed by applying pressure,

An Introduction

Powder Metallurgy

➤ Subject Matter

- Powder metallurgy (PM) is the art and science of manufacturing complex shaped objects from powders
- It includes all manufacturing steps to produce the final product
 - Powder production
 - Blending
 - Shaping and compaction
 - Sintering
- This course covers all these aspects of this manufacturing process with emphasis on structure-processing-properties relationships.

➤ Objectives

- To learn the technology
- To gain insight into scientific principles underlying design and operation of PM processes
- To be able to select and design the optimal processing route for any given product properties.

An Introduction

Metal processing technology in which parts are produced from metallic powders.

PM parts can be mass produced to net shape or near net shape, eliminating or reducing the need for subsequent machining.

PM process wastes very little material ~ 97% of starting powders are converted to product.

PM parts can be made with a specified level of porosity, to produce porous metal parts.

Examples: filters, oil-impregnated bearings and gears.

Certain metals that are difficult to fabricate by other methods can be shaped by PM.

An Introduction

PM vs. Other Fabrication Methods (casting, stamping or machining)

- PM is the choice when requirements for strength, wear resistance or high operating temperatures exceed the capabilities of die casting alloys.
- PM offers greater precision, eliminating most or all of the finish machining operations required for castings.
- It avoids casting defects such as blow holes, shrinkage and inclusions. Powder injection molding is coming out as a big challenge for investment casting.
- However the PM process is economical only when production rates are higher, since the tooling cost is quite appreciable.

An Introduction

- ❑ The powder Metallurgy methods starts with powders and the properties of the manufactured parts depend to a large extent on the properties of the initial powders. Amongst powder properties, composition, size, form and structure of particle, specific surface, porosity and volume characteristics, fluidity, strength, hardness, permeability regarding liquids and gases, electric conductivity, compressibility and sinterability are of great importance in powder metallurgy.
- ❑ Metal powders consist of separate small bodies—the so- called particles—from $0.1\text{ }\mu$ up to several millimeters in size.
- ❑ In the majority of powders the size of particle varies from several microns to 0.5 mm . Particles usually have internal pores, cracks and impurities.

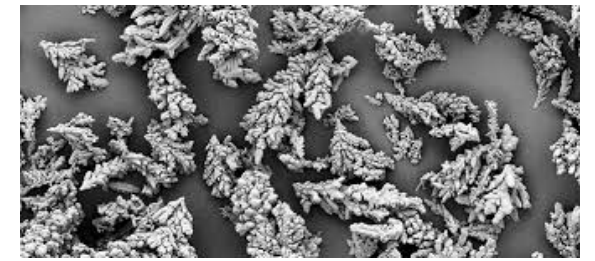
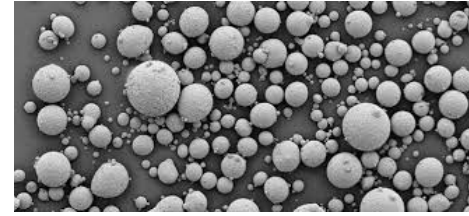
An Introduction

Particle shape is widely varied and determined by the methods of production; the desired particle size is obtained by regulating the conditions of manufacture.

Particles are divided according to their shape into three basic groups:

- (1) hair or needle-shaped, the lengths of which considerably exceed their other dimensions;
- (2) flat (flakes or leaves) whose length and breadth are many times greater than their thickness;
- (3) equiaxed, which are roughly identical in all dimensions.

An Introduction



Powders of the same chemical composition, but with different physical characteristics, are sharply distinguished by technological properties, i.e. by their behaviour during processing. Production methods and the fields of application for powders are determined with powder properties (shape, size)

Example: copper powder with particles in the form of flakes (flat) up to $1\text{ }\mu$ thick and up to 50μ in diameter are used only as a pigment since any articles made from it contain cracks after pressing.

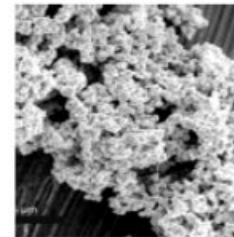
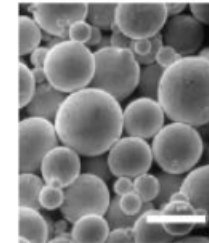
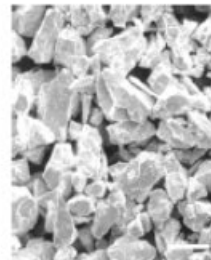
Copper powder with particles of spherical form (globules) from 100 to 700μ in diameter is pressed only at very high pressures.

A powder with irregularly shaped particles, so called dendritic, $40\text{--}45\mu$ in size is easily pressed even at low pressures.

An Introduction

Powder Metallurgy Technology

- General Classification
 - Powder Technology
 - Powder Processing
- Powder technology
 - It involves all processing operations to prepare the particles in the 100 μm range for subsequent shaping and consolidation processes.
 - Powder fabrication
 - Mechanical
 - Atomization
 - Electrolysis
 - Chemical synthesis
 - Particle classification
 - Particle handling, modification and mixing
 - Particle characterization and microstructure control are essential components of powder technology

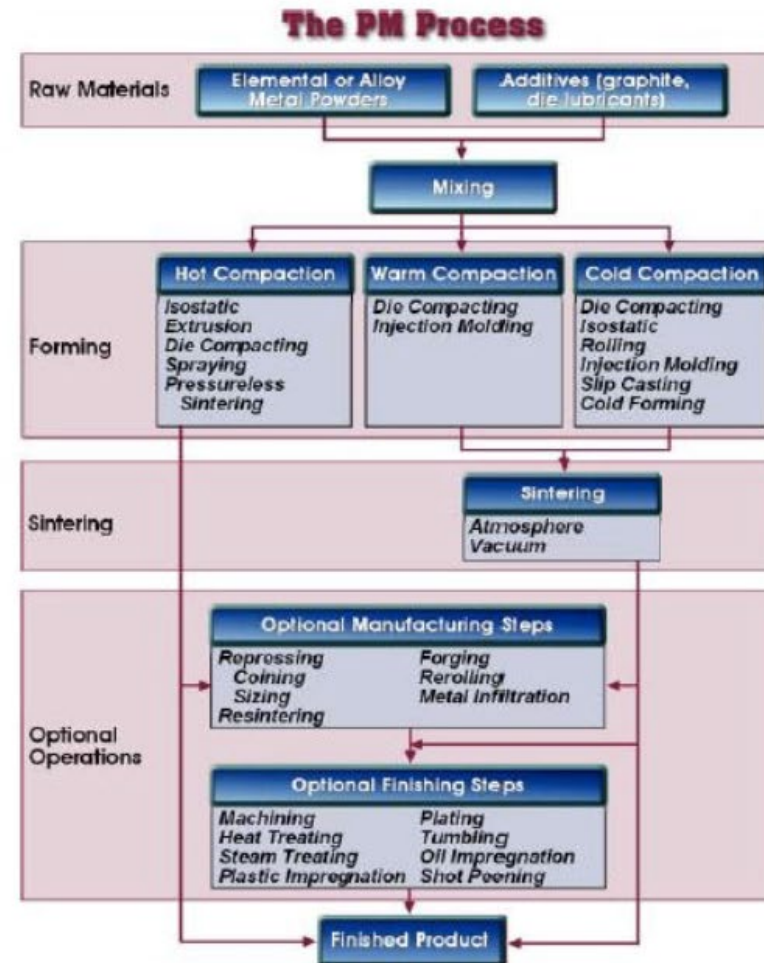


An Introduction

Powder Metallurgy Technology (cont.)

➤ Powder Processing

- It involves all operations to consolidate the particles to the final product
 - Shaping and compaction
 - Sintering
 - Densification
 - Finishing operations
 - Machining
 - Heat treatment
 - Joining
 - Coating
- Selection of processing methods depends on desired properties of final product.



An Introduction

Why Powder metallurgy?

It is the only near net-shape manufacturing technology capable of fabricating parts for all types of materials

- Metals and intermetallics
- Ceramics
- Engineered materials: Composites and porous materials



➤ Advantages

- Achieves a wide variety of alloy systems
- Facilitates manufacture of complex or unique shapes which would be impractical or impossible with other metalworking processes
- Maintains close dimensional tolerances
- Produces good surface finishes and eliminates or minimizes machining
- Provides controlled porosity for self-lubrication or filtration applications
- Suited to moderate-to-high volume component production requirements

An Introduction

Applications

Powder metallurgy is used in various industrial sectors:

- Automobile industry (motors, gear assemblies, brake pads)
- Abrasives (polishing and grinding wheels)
- Manufacturing (cutting and drilling tools)
- Electric and magnetic devices (magnets, soft magnetic cores)
- Medical and dental (implants, prostheses, amalgams)
- Aerospace (motors, heat shields, structural parts)
- Welding (solder, electrodes)
- Energy (electrodes, fuel cells)
- Other (porous filters, sporting goods)



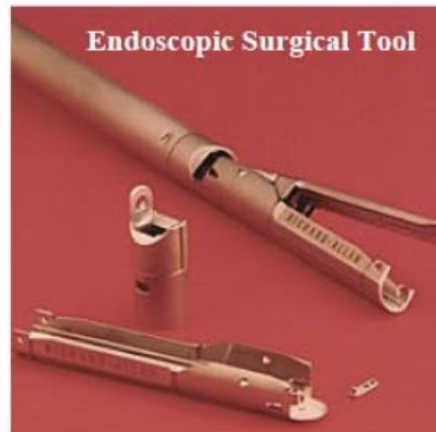
An Introduction

Example Applications

➤ Automotive



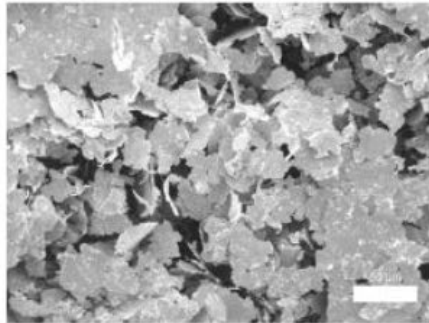
➤ Biomedical components



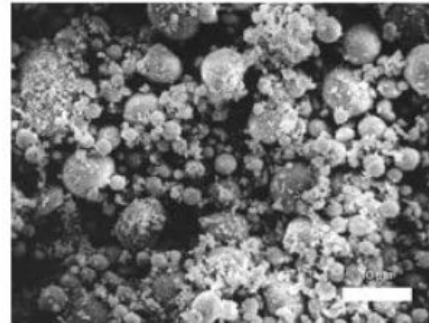
An Introduction

Powders and Particles

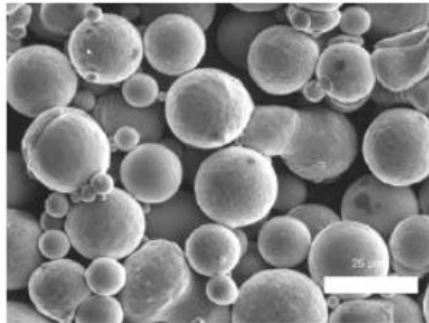
- A powder is a collection of finely dispersed solid particles smaller than 100 μm .
- The particle size of powders used in powder metallurgy ranges from 0.1 to 100 μm .
- The size and shape of the particles depends on
 - Materials chemistry
 - Powder production technique



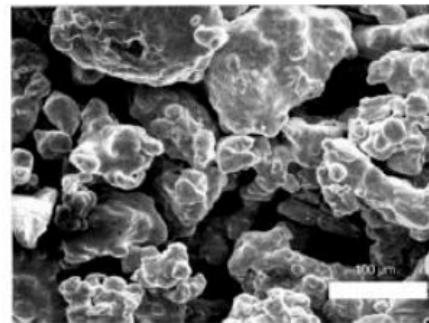
Tantalum flakes
50 μm width



Silicon nitride
Agglomerate
2 μm spheres



Stainless Steel
20 μm spheres



Iron irregular particles
100 μm long

An Introduction

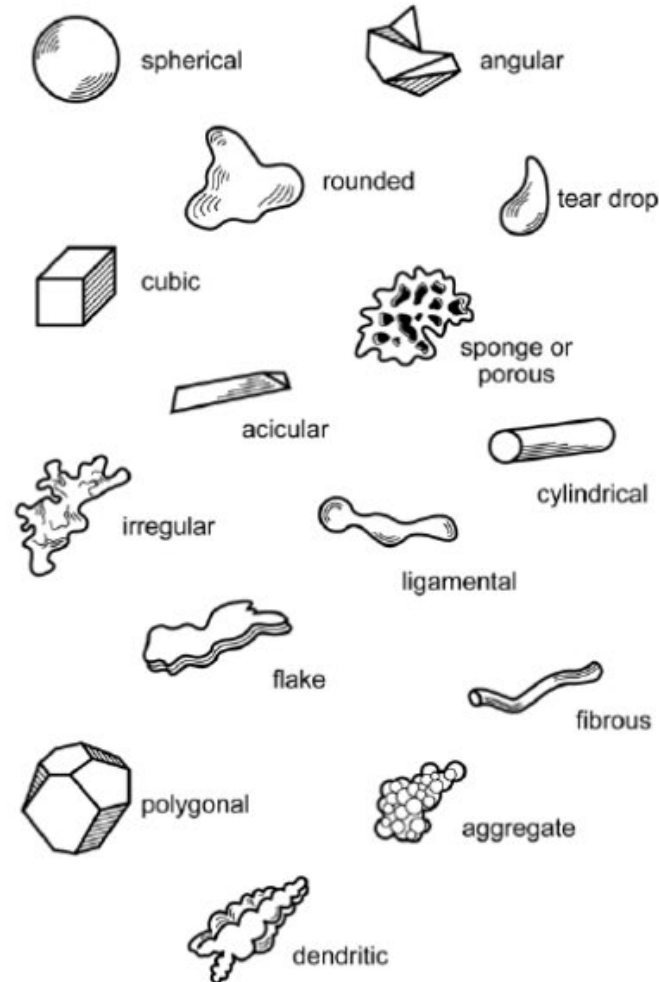
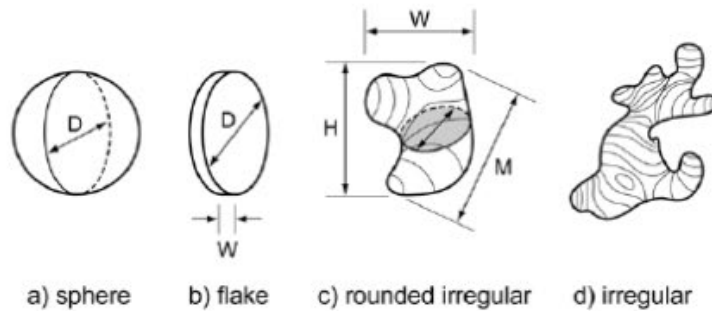
Powder Characteristics

- Powder characteristics relevant to powder processing are:
 - Particle size and its distribution
 - Particle agglomeration
 - Surface area of particles
 - Interparticle friction
 - Particle flow and packing
 - Crystal Structure of the particles
 - Composition, homogeneity and contamination
- These characteristics must be measured and quantified to
 - Optimize powder fabrication process
 - Select powder processing route
 - Specify operating conditions of compacting, sintering and finishing processes.

An Introduction

Particle Size and Shape

- Most fabricated particles have complex shapes
- Particles size and shape influence packing, flow and compressibility of the powder.
- Characteristic dimensions of the particle depends on the shape of the particle



An Introduction



While tungsten carbide refers to the specific material combining tungsten and carbon, cemented carbide is actually a broader category that includes tungsten carbide bound with a metal binder (usually cobalt)

The mid-1920s saw the emergence of two powder metallurgy products which set the patterns for the future industrial development of the technology. The first of these was a hard, wear-resisting product known as *cemented carbide*, which is produced by compacting and sintering a mixture of powders of tungsten carbide with up to 15% of cobalt. At a temperature of about 1400°C, *a molten phase is formed between the cobalt and some of the tungsten carbide* and this promotes *rapid and virtually complete densification* to give a final structure of carbide particles in a tungsten carbide/cobalt alloy matrix. Developed originally for *wire-drawing dies*, cemented carbides have found extensive application in *metal cutting, rock drilling and hot working dies*. *Many new compositions have been developed including additions of other refractory metal carbides such as tantalum and titanium.*

An Introduction



The second significant development was the *porous bronze self-lubricating bearing*, which was invented in the late 1920s, and which is made from a mixture of *90% copper, 10%tin* powders, usually with a small *graphite* addition. The powder mixture is *pressed and sintered* to form a tin-bronze alloy with little dimensional change, and containing up to *35% of fine, interconnected, porosity*. The final close dimensional tolerances required in the bearing are achieved by pressing the sintered compact in a sizing die after which it is impregnated with oil. When in use, the bronze reservoir supplies oil to the bearing interface. Other porous products today include filters for separation processes made from lightly compacted and sintered powders of, for example, bronze or stainless steel.

Powder Production Techniques

The most important metal powders are: iron and steel, copper and copper base, aluminium, nickel, molybdenum, tungsten, tungsten carbide and tin. Besides metallic powders also carbide, nitride, boride and special ceramic powders as well as whiskers and short fibers are considered.

MECHANICAL PROCESSES

The size reduction of metal powders by mechanical means is performed in the solid state as well as of melts. *Grinding and milling are the oldest processes* in powder metallurgy and ceramics for the production of particulate materials. They are *extensively applied in the field of ceramics* and in the *cement* industry for size *reduction of brittle materials*, often accompanied by *intensive mixing effects*.

Powder Production Techniques

Their use in *powder metallurgy* is more *limited*, since the *disintegration* of *metallic* materials, which mostly exhibit considerable *plasticity*, is less effective. Nevertheless, materials such as *intermetallic compounds* and *ferroalloys* can be effectively comminuted by mechanical means. Additionally, there are important mechanical disintegration processes in powder metallurgy, especially those involving *high energy milling* procedures, during which *severe embrittlement of the metal* occurs. This aspect can be turned to advantage in the process known as *mechanical alloying*.

The *general* phenomena during size reduction in the solid state are based on *fracture mechanics*: the *nucleation* of *cracks*, followed by *crack propagation* and *fracture*, by which new surfaces are formed. *A further decrease of mean particle size can take place only when these processes occur.*

Powder Production Techniques

The *kinetic energy* within the *milling* aggregate is partially transformed into *mechanical stresses* in the material to be *disintegrated*. The *forces* acting in these processes cause mainly *compression* and *shear stresses*, applied as *impact or slow-acting stresses* by the *milling balls* during vessel rotation or vibration, or by the *rotating arms* in an attritor. *In plastic materials, gross dislocation formation and movement, i.e. plastic deformation, is induced before cracking and fracturing occur.* In *ceramic* materials, *plastic deformation does not take place* in *coarse particles*, but becomes *significant for mean particle sizes in the micron range*. The main process phenomenon for all grinding and milling processes, however, is crack propagation and fracture, which is accompanied by *local, not well defined, temperature rises*. These are often referred to as *thermal 'spikes'* and can result in *anomalous structures*.

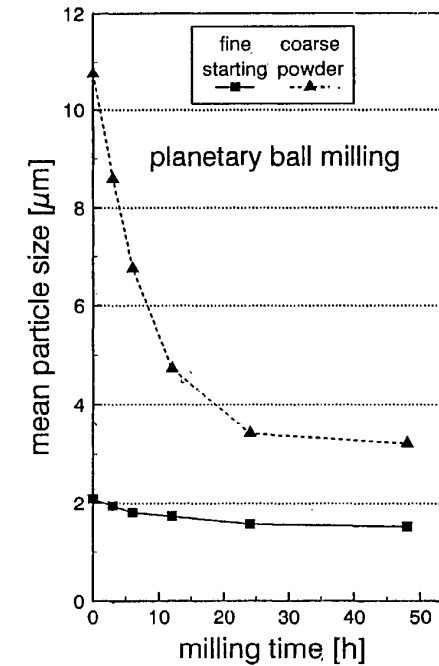
Powder Production Techniques

The *limit of the minimum obtainable particle size* depends on the *conditions* of the *mechanical process* as well as on the *material itself*. This so-called *grinding or milling equilibrium, has been investigated for many metallic and non-metallic powders*. It ranges between roughly 0.1 and 1 micron and can be explained by several independent processes as follows:

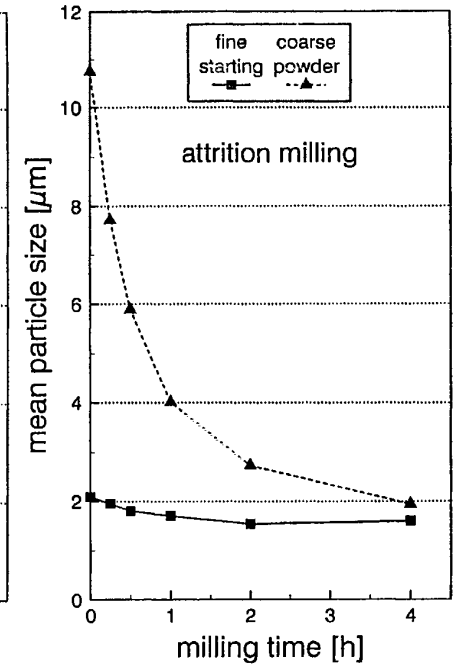
- In a solid material there is no constant binding force within the volume but only a binding spectrum, dependent on the local types of defects and defect concentration. These can be macro, micro or on an atomic scale. Only the weaker part of the binding spectrum can be destroyed by mechanical means, and a further reduction in size of small particles becomes increasingly difficult.

Powder Production Techniques

The *smaller the particle size* the more *difficult* is the application of the necessary *shear stress* on each particle in order to achieve *further particle fracture*. With small particles the elastic or elasto-plastic response to mechanical stresses becomes dominating. *Small particles exhibit higher surface activity* than larger ones and therefore, have a *higher probability* of being *re-welded*. This was previously believed to be the main reason for the '*milling equilibrium*' observed.



(a)



(b)

Powder Production Techniques

The efficiency of mechanical size-reduction processes is generally very low. Only about *0.1% of the spent energy* in the conventional ball milling process is found in the *generated new surfaces of the fine particles*. The energy E necessary for the physical process of size reduction is given by

$$E = \gamma \cdot \Delta S$$

where γ = the specific surface energy and ΔS = the increase of specific surface. *The efficiency may be somewhat higher in high energy milling processes*, but is still less than 1%. The percentage figure becomes larger when, instead of the energy defined in equation (2.1), the energy necessary for crack propagation is considered - being about one order of magnitude larger.

Powder Production Techniques

The sources of '*lost energy*' are many: they include the *elastic and plastic deformation* of the particles, the *kinetic energy of the particles in motion, impact and friction energy in the form of heat outside the powder*, etc. The main cause of lost energy, however, is the generation of *heat*. In order to *optimize milling efficiency* special conditions have to be established.

- in conventional steel ball mills, defining *the size and number of milling balls* which will take up *40-50% of the vessel volume*. The size of the *balls generally ranges between 12 and 16 mm, about 10-20 times larger than the initial size of the particles to be milled*.
- using *vibration milling* instead of *milling by vessel rotation*. The *impact intensity of the milling balls* is much *higher* in *vibration milling* than in the 'falling' process in rotational ball milling. The *noise level*, however, of *a vibration mill is very high*. Such mills were used for a long time in the fabrication of *cemented carbides*, but have now mostly been replaced by *attritors*. *For laboratory purposes discs are used successfully instead of balls in vibration milling*.

Powder Production Techniques

- other types of fine-milling equipment, such as *planetary ball milling, centrifugal milling and attritor milling* are widely used.
- *wet milling* instead of *dry milling is often advantageous* because liquids tend to *break up agglomerates* as well as *reducing re-welding of powder particles*. As milling media, hydrocarbons (hexane, heptane), ethanol and other organic liquids, which help to *avoid oxidation of the milled product*, are often used. Special effects have been achieved by using *surface active additives* (e.g. oleinic acid or other substances with a long carbon chain and a surface active group), which *reduce the interfacial energy between particles and the milling media* (Rehbinder-Effect) and, more importantly, *increase the crack propagation during milling by reducing the cohesion at crack tips*.

Powder Production Techniques

During the *milling of hard and abrasive powders the level of impurities sometimes rises considerably*, because of *severe wear loss from walls and milling media*. *Iron contamination* can be removed by subsequent *acid leaching*, when the *milled powder itself is acid-resistant*. *Rubber liners and ceramic balls (Al₂O₃)* which cause less contamination are sometimes advisable. The *best but most expensive* solution is to use *liners* and *balls* of *similar composition* to that of the material being milled (e.g., the milling of special ceramic powders, like Si₃N₄).

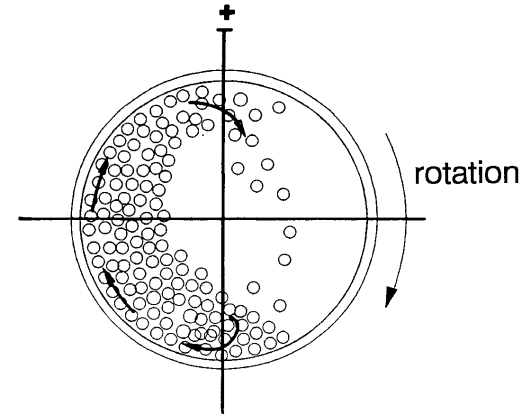
Equipment

There is a wide variety of grinding and milling equipment, but the following are selected for particular mention:

Powder Production Techniques

Ball and Vibration Milling

Figure 2.1 shows the principles of conventional *steel ball mills*, which are available from a small *laboratory size* (less than 1 litre) to *very large sizes* used mainly for the *ceramic* and *cement* industry. At *too low rotation speed* the *milling balls are mainly rolling over each other*, but at *higher speed some of the balls are in free fall at each moment* (Fig. 2.1), which is the *optimum condition for the milling process*. At *too high speed*, however, the *centrifugal forces hinder the balls in free fall*. *Ball mills are sometimes used in powder metallurgy for mixing purposes only*. In *vibration mills* the *design of the vessel* is similar to that in *rotation ball mills*, but the vessel is mounted on *special steel springs*. The *amplitude as well as the frequency of vibration can be adjusted according to the characteristics of the vibrating mechanism and the springs*. Vibration mills are often used with only *short milling times* in research and development for *size reduction of inorganic and organic products*.



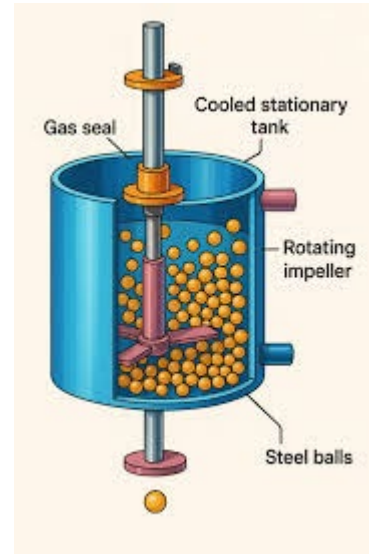
Powder Production Techniques



The *difference from the conventional ball mill* is the *superposition* of the *single vessel rotation* by *rotation of the table supporting the fixed vessels*, which *accelerates the movement of the milling balls*. Centrifugal accelerations up to 20 g (acceleration due to gravity) are possible.

Attritor milling

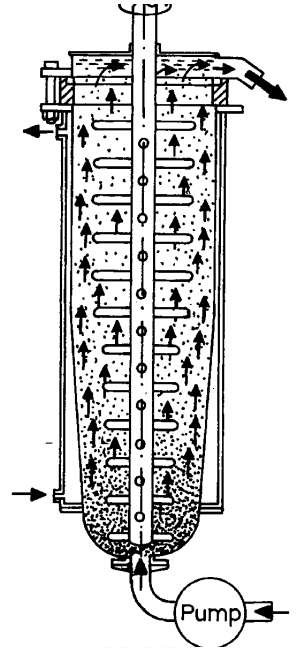
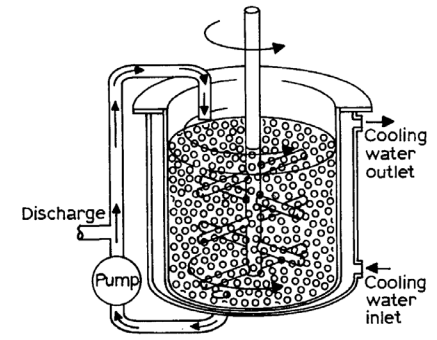
An attritor is a ball milling system in which the *balls*, together with the *material* to be milled are *set in motion by a shaft with stirring arms*, rotating at 100-300, small units up to 2000 rpm. The containing *cylindrical vessel* is usually *water cooled* because of the considerable heat generated by the process. The process may be undertaken *dry*, or *wet with water or organic liquids*. *The balls are generally smaller (0.5-2 mm) than in conventional ball mills*. For *production scales the process can be run continuously by feeding the vessel from the bottom while unloading the milled product from the top*. A closed cycle can be provided for the milling liquid, including distillation apparatus for recycling.



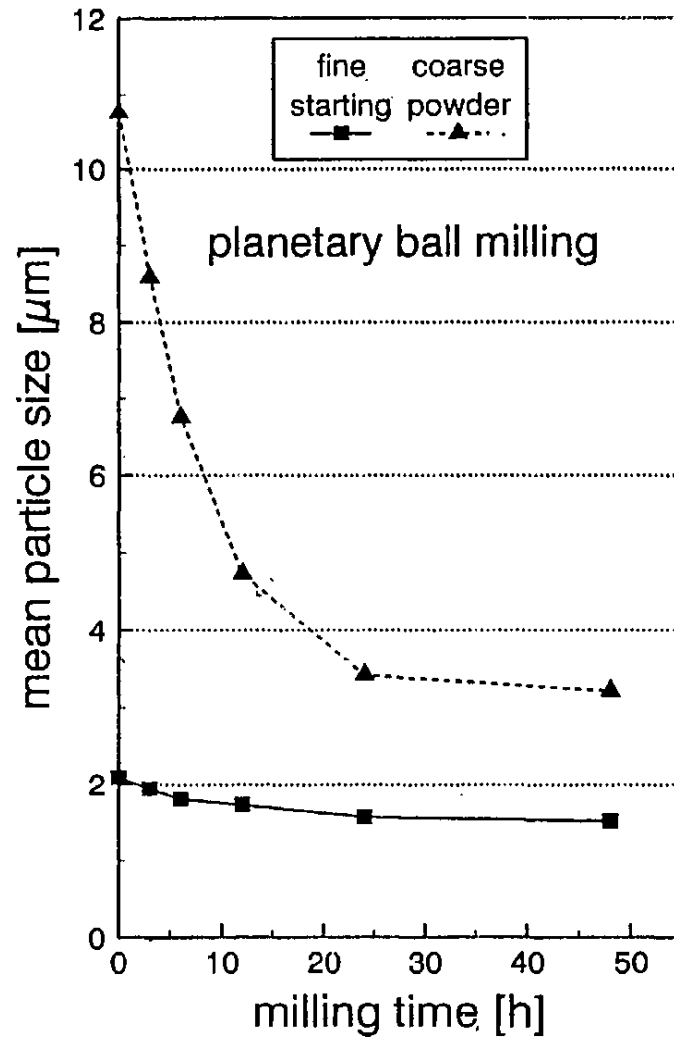
Powder Production Techniques

For processing of materials with *special oxidation sensitivity* (e.g., powders for *Co-rare-earth-magnets*) the attritor can be equipped with an *inert gas supply*, which is generally advisable in *dry milling*.

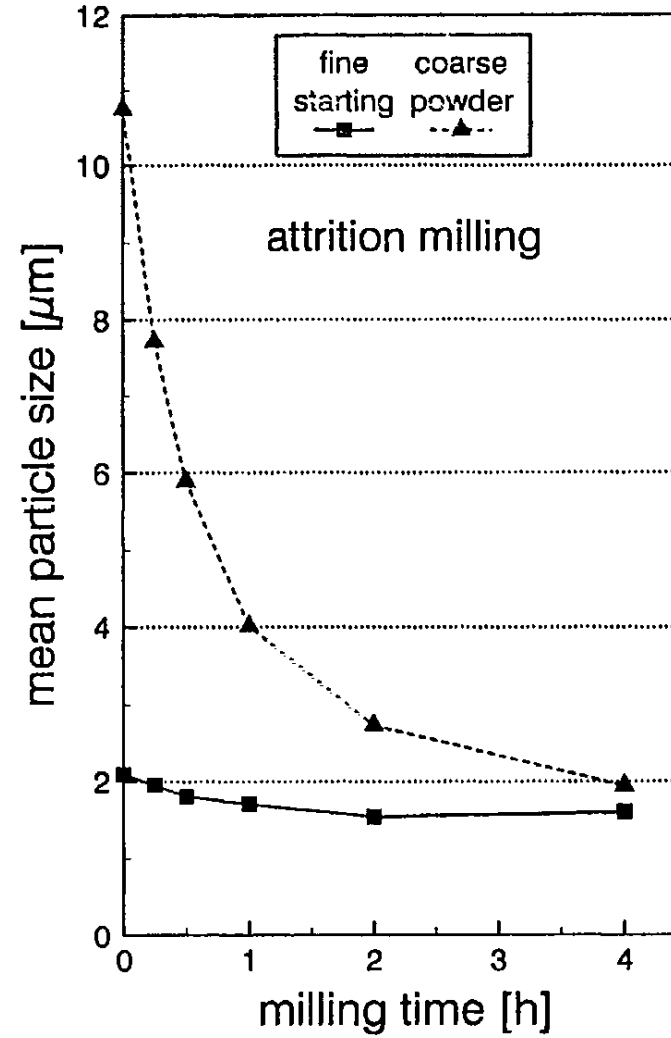
A *definite ratio of milling balls to milling material* is essential for optimum efficiency. Figures 2.3 and 2.4 show two types of attritors. *Discs with holes or profiled discs* are also used *instead of arms* as rotating units in so called *agitation ball mills*. The *milling intensity of an attritor is generally much higher than that of conventional ball mills*, the difference being at least one order of magnitude in time for achieving similar results (see Fig. 2.5). In addition to *compression stresses, high rate shear stresses* are involved leading to *lower energy losses compared with other processes*.



Powder Production Techniques

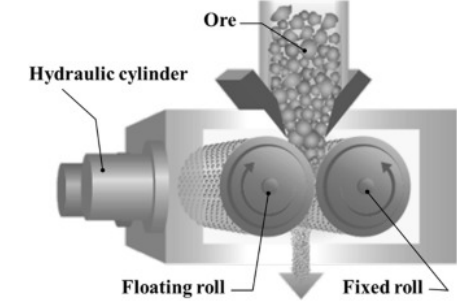


(a)



(b)

Powder Production Techniques



Dry and wet attritors are widely employed for inorganic pigment and paint production, for limestone, and oxides, and in the hard-metal industry, where they have replaced vibration mills. In these fields of application very fine particulates are required. *In the powder mixtures for cemented carbide manufacture (e.g. WC-Co), a certain degree of 'coating' of the brittle carbide by the ductile cobalt particles during milling-mixing occurs which is desirable for further processing.* Several metals and alloys may become amorphous during extended milling in an attritor. Investigations, mainly during the last decade, have resulted in the development of tailor-made amorphous alloys (glassy metals), by this method.

A metal or alloy with high hardness

Roller Milling

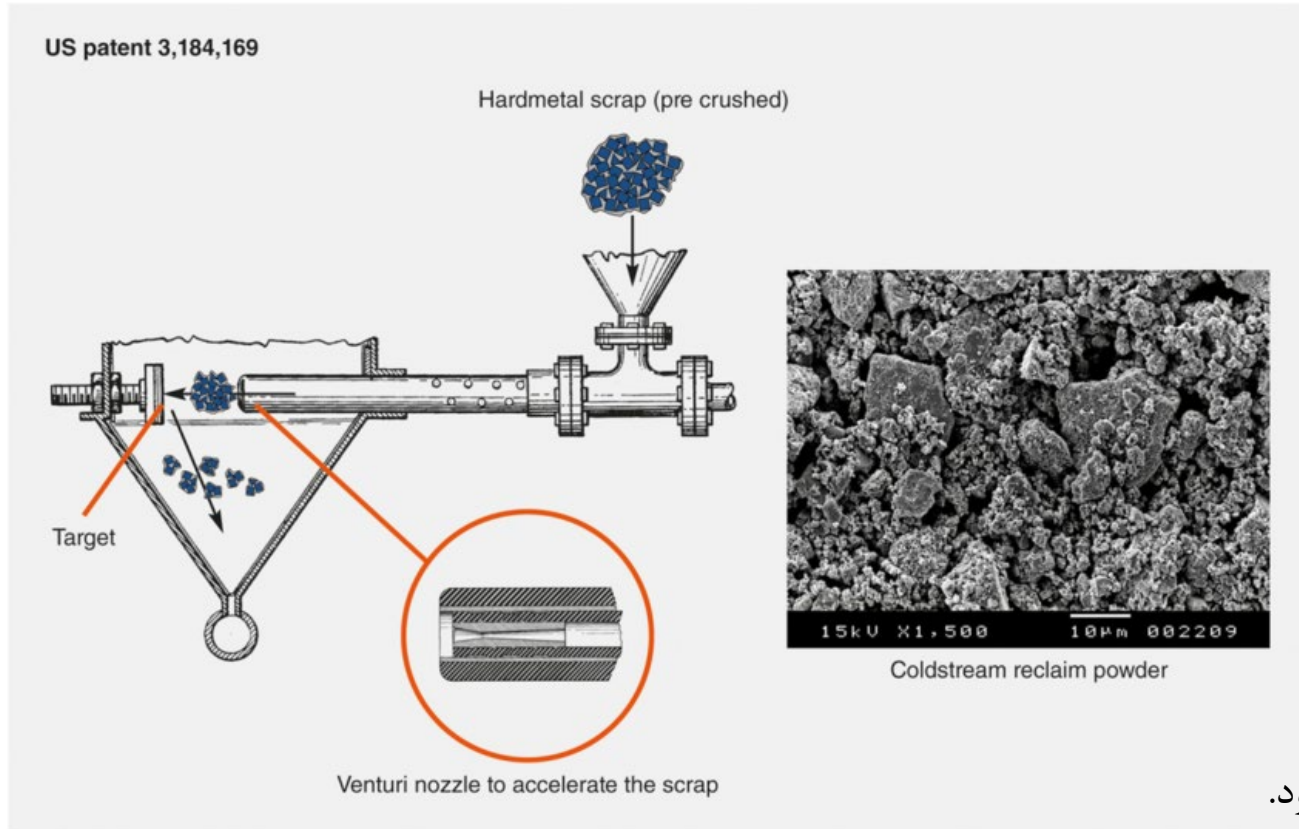
For brittle materials a new size reduction technology become important, the high-compression roller mill, Fig. 2.6, which operates with profiled rollers in the pressure range 50-500 MPa. According to the working conditions products with narrow particle size ranges, between 200 and about 5 micron are achievable. The service life is claimed to be 10 to 20 times that of ball mills.

Powder Production Techniques

The Cold Stream Process

In this process *coarse particles* (less than 2 mm) are transported in a *high pressure, high-velocity gas stream* and *shot on to a target of a highly wear resistant material*. A typical pressure of the system is 70 bar with a gas velocity up to Mach 1 or even higher. Due to the drop in pressure of the gas stream on passing through a venturi-nozzle before entering the collision chamber at lower than atmospheric pressure, a rapid cooling of the particles occurs in the nearly adiabatic system. This leads to considerable particle embrittlement and allows easy particle fracturing during collision with the target. The particle fines are extracted from the continuously repeated process and collected in special chambers, while the coarser fractions stay in the cycle. By using inert gas (Ar) the process provides fine powders (a few micron) with very low oxygen content. The surface oxides of the raw material can be spalled off and separated during the first few cycles. The cost of the process is high partly due to high energy consumption. This results in economic powder production only for materials with a high value, such as superalloys, cemented carbides, etc.

Powder Production Techniques



در این سیستم، گاز با عبور از گلوگاه نازل ونتوری، سرعتش زیاد شده و فشارش افت می‌کند. از آنجایی که این فرآیند به صورت آدیاباتیک (بدون تبادل گرما با بیرون) رخ می‌دهد، انرژی درونی گاز کاهش یافته و دمای آن به شدت پایین می‌آید، که این سرمایش سریع به ذرات معلق نیز منتقل می‌شود. این اثر برای کنترل اندازه و ساختار نانوذرات بسیار مهم است.

- افت فشار شدید در گلوگاه ونتوری باعث می‌شود که گاز با سرعت زیاد منبسط شود.
- این فرآیند در محیطی با فشار کمتر از فشار اتمسفر (خلأ نسبی) رخ می‌دهد که این شرایط انبساط را تشدید می‌کند.
- کاهش ناگهانی فشار گاز (انبساط آدیاباتیک) باعث می‌شود که انرژی درونی گاز صرف کار انبساط شود. این اتلاف انرژی باعث می‌شود دمای گاز به شدت افت کند.
- ذرات معلق در این جریان گازی، به سرعت دمای خود را با دمای گاز میزبان یکسان می‌کنند. بنابراین، ذرات به سرعت سرد می‌شوند.

Powder Production Techniques

The Hametag (Eddy-Mill) Process and Jet Milling

In this process a *ductile material*, mainly *iron* in the form of *fine scrap* (wires, sheets, etc.) is *comminuted* by rotating *propellers* in a *vessel* under a *protective gas*. *The high speed (up to 85 ms^{-1}) , generates gas streams in which the particles undergo the main part of their size reduction as self-disintegration.*

Up to 1950 much of the iron powder used for machine parts production was made by this method. The *plate-like* particles (0.05 to 0.5 mm) with a relatively *smooth surface* have only a *moderate compressibility* and the process has a *low efficiency with high energy consumption*. This type of mill is no longer in service. *New types* of jet-mills of different design are widely used, but mainly *outside the field of powder metallurgy*.

The term '*compressibility*' is defined as the ability of a *powder to decrease in volume under pressure*, and the term '*compactibility*' is defined as the ability of the powdered material to be compressed into a tablet of specified strength (i.e. radial tensile strength or deformation hardness).

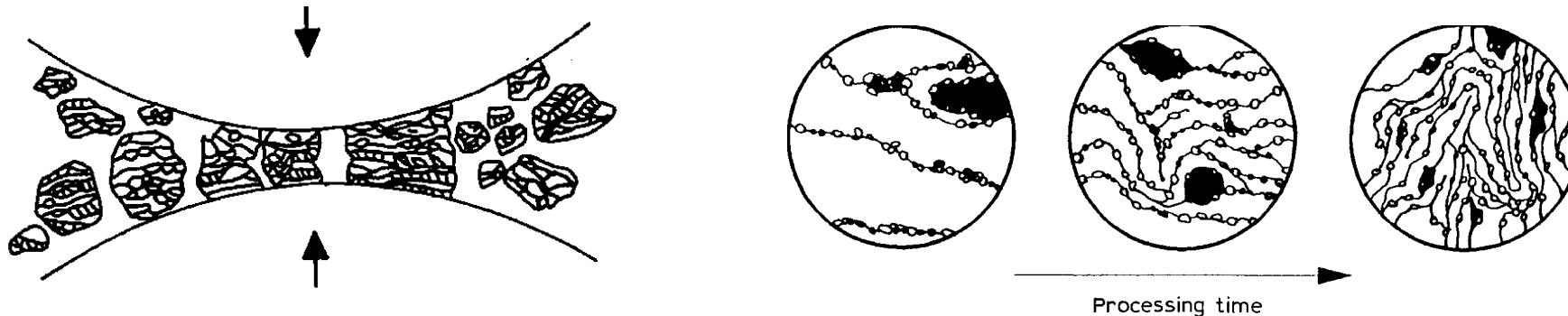
Powder Production Techniques

Mechanical Alloying

Mechanical alloying is a *high-energy ball milling process* for producing *composites* with a *controlled*, even *distribution of a second phase* in a *metallic* matrix. It was first published in 1970 and introduced in the context of developing *dispersion-strengthened alloys* (see section 11.9.3), in which strengthening by precipitations and dispersed oxides is combined. The process enables the development of special microstructures essential for achieving good high-temperature mechanical properties in multiphase powder metallurgy materials. In particular, the *distribution of a non-metallic phase in a metallic, ductile matrix can be homogenized to a degree*, which can be achieved otherwise only by chemical means. Also *chemical reactions and formation of solid solutions* can be obtained, in such cases the term '*reaction milling*' is used. *The process consists of long period milling of mixtures, in which the main component (later the matrix) is ductile.*

Powder Production Techniques

As a result of the *permanent high energy ball-powder interaction* (Fig. 2.8) the *ductile phase* undergoes a *continuous cycle of plastic deformation, fracture* and *re-welding* processes, by which the *fine dispersoids* are implanted step-by-step into the *interior* of the *ductile phase*. This can be understood as a three stage process. In the first stage *strong particle deformation ('miniforging')* and *welding* of particles are dominant, while the *dispersoid* is *forced to cover the steadily increased surface of the ductile phase*, which *embrittles continuously*. During the second stage the *large lamellar-like particles formed are fractured*, forming again *new surfaces*, which may *pick up more of the dispersoid particles, re-weld and form finer lamellae* (Fig. 2.9). An *equilibrium between deformation and welding on the one hand and fracturing* on the other is reached (more or less) during the third stage, while *homogenisation* proceeds, and the *mechanical alloying* process *terminates*.



Powder Production Techniques

A considerable *initial coarsening of the ductile phase* can take place, when the *starting particle size* of the *metal powder* is *very fine* (see Fig. 10), while *crystallite size decreases*. Very *effective mechanical alloying has been achieved by attritor milling*, but other, more economic, milling processes can also be successful. *Longer times*, however, have to be allowed with *low energy ball mills*. *Mechanical alloying is applicable to nearly all combinations of brittle phases (oxides, carbides, nitrides, carbon, intermetallics) and ductile metallic powders, which demonstrates the possibilities for extensive composite materials development*. The process has also been used for the *amorphisation of metal or alloy powders*, which is of some interest for the manufacture of *metallic glasses*. The *sintering* process, however, has to be carefully adjusted to *avoid recrystallization* and the temperature of the powders must *always be below the glass transition temperature (T_g)*. Mechanical alloying yields amorphous materials over broader composition ranges than other processes such as splat cooling.

Powder Production Techniques

Powder Production Techniques 17

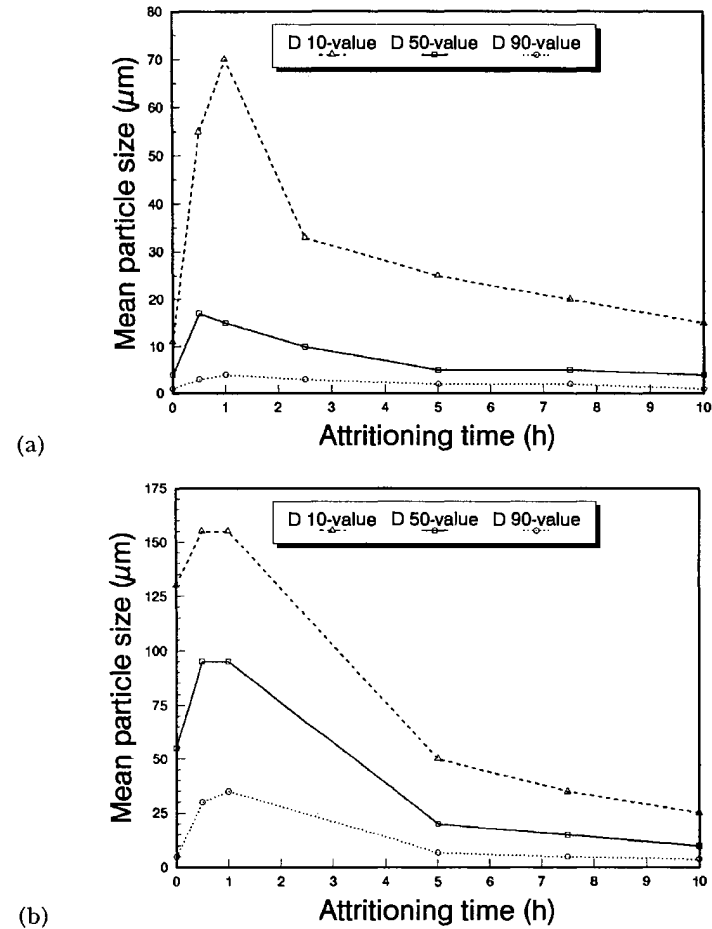


Figure 2.10 Time-dependent mean particle size in Fe-10 vol-% NbC mixture during mechanical alloying in the attritor (a) Carbonyl powder (b) Water atomised powder (Gutsfeld)

Powder Production Techniques

Processing of Metal Chips

Metal chips produced by *cutting* or *milling* during *mass production* are often treated as *scrap*, because the handling charges exceed their value as raw material. Nevertheless, attempts have been made by *mechanical processing to re-cycle ferrous and non-ferrous chips*, from which powders can be produced. This may be *economic* with *expensive non-ferrous* metals and alloys such as *copper, brass or bronze*, when the *chips can be delivered with low impurity levels*, in sufficient *quantities* and with *uniform quality*. The process of *powder manufacturing from chips* involves first *cleaning from impurities*. The following *fragmentation*, which is essential in order to get press and- sinter-grade powders, is done by *high energy (e.g. vibration) ball milling*. The *cold deformed powders have to be annealed in order to improve their compactibility*. The process may become more significant in future because of the need to conserve raw materials.

Powder Production Techniques

Principal Aspects of Atomization

Melt atomization is the *most important production method for metal powders*. The process generally consists of three stages:

- melting
- atomization (disintegration of the melt into droplets)
- solidification and cooling.

In most cases, *additional processing becomes necessary before the powders attain their desired properties*, e.g., *reduction of surface oxides, degassing, size classification*, etc. For each of the process stages different methods can be used, resulting in a large number of variants. Table 2.1 provides an overview of the present situation. *The main classification criterion is the way in which the energy for disintegration is introduced into the melt*. This can be by *capillary forces (melt drop process), mechanical impact (impact disintegration), electrostatic forces (electrodynamic atomization), liquid or gas streams or jets (liquid or gas atomization), centrifugal forces (centrifugal atomization), gas super-saturation of the melt (vacuum atomization), or ultrasonics (ultrasonic atomization)*.

Powder Production Techniques

In the *melting stage* the most *important criterion* is whether *melting and melt distribution* require a *crucible system* or not. Crucibles are one of the main sources for *contamination* of atomized powders. The *second criterion is the heating source*. Essentially all melting techniques known in metallurgy can be used, e.g., induction, arc, plasma, and electron-beam melting, but some of these may also contribute to *contamination, as for example in arc melting*.

During the solidification and cooling stage, the *cooling rate is the controlling parameter*. This depends, of course, on the *dimensions of the liquid droplets or solid powder particles*, and also on the *type of heat transfer from the particles to the surrounding medium*. The *undercooling prior to nucleation and cooling rate are the controlling factors* for determining the *microstructure of the powder particles*, as well as for the dimensions of the atomization unit, which has to provide a path, on which the droplets can solidify without touching the unit walls or structure. The various melt disintegration methods are explained in more detail below.

Powder Production Techniques

Methods Based on Capillary Drop Formation

In the *melt drop process*, the liquid metal flows vertically through the *outlet capillary of a tundish*, which controls the *melt stream diameter*, d_s . The melt stream *breaks up* into *droplets of a diameter* d_D , which depends mainly on d_s , and only slightly on the *melt viscosity*, η , and the *surface tension*, γ_s , of *the liquid metal*. Within the usual range of properties of liquid metals, the correlation between d_s and d_D can be approximated by equation (2.2).

$$d_D \approx 1.88 d_s$$

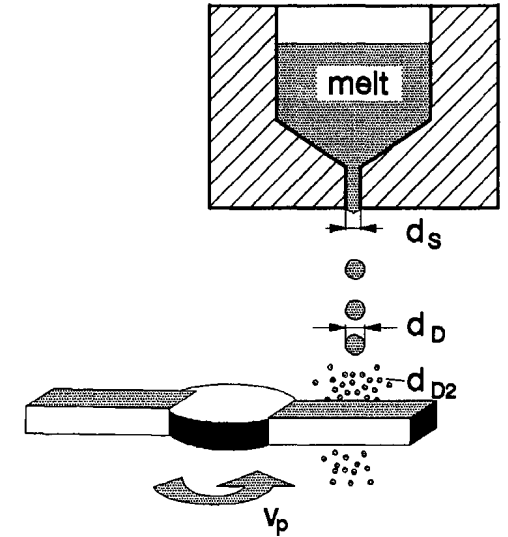
As the resulting *particle size is relatively large* and the *production capacity quite small*, the melt drop process has only been used in *laboratory scale* production.

Powder Production Techniques

A *smaller particle size* can be achieved by *impact disintegration* of the resulting droplets (Fig. 2.11). The relation between the diameter of the secondary droplets d_{D2} and d_D is given by:

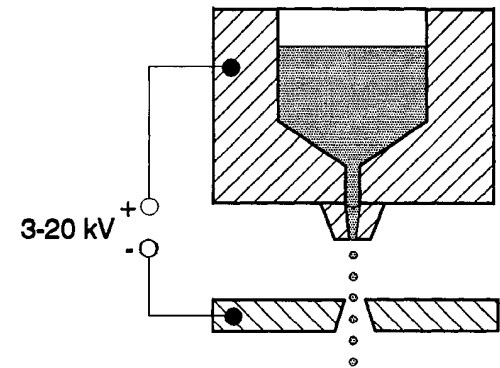
$$d_{D2} = d_D (v_c / v_p)$$

V_p being the *impact velocity* and V_c the *minimum impact velocity* at which droplets of the size d_D can be broken down. The application potential of this process is for the production of *rapidly quenched powders*. With *a cooled impact surface a cooling rate of more than 10^7 Ks^{-1} necessary in the production of amorphous powders* is said to be possible.



Powder Production Techniques

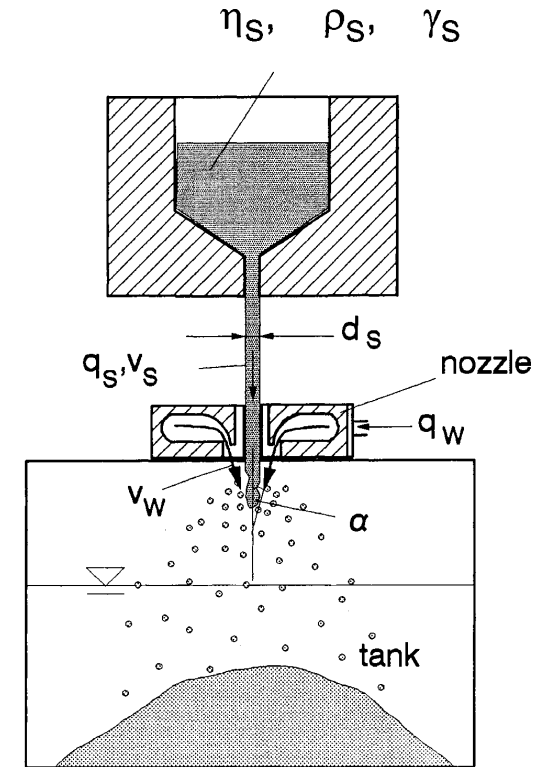
The *electrodynamic atomization* process has some similarities to the methods mentioned above, because it is also based on *droplet formation out of a capillary orifice* (Fig. 2.12). A *DC voltage* of 3-20 kV is applied between *this orifice and a perforated electrode plate* in front of it. *Melt droplets, which have a strong positive charge*, are emitted from the orifice. With a *capillary diameter of 76 micron*, a *particle size* in the range of *0.1-10 micron* has been achieved. This is a *very fine powder compared* to that from other atomization processes. The production capacity of existing units is only in the order of *grams per hour* and the process has not yet progressed beyond *laboratory scale*.



Powder Production Techniques

Atomization by Liquids

Liquid (generally *water*) and *gas atomization* are the most important process variants for *industrial powder production*. *Water* atomization is mainly used for the production of *iron base powders*. Figure 2.13 shows a scheme for a water atomization unit. *The starting material* is *melted* and *metallurgically treated in a separate furnace* and then *fed into a tundish*. The tundish provides a *uniformly flowing vertical melt stream*, which is disintegrated into droplets *in the focal area of an arrangement of several water jets*. Configurations of the water jets may *differ in the number of jets*, the *angle alpha*, between the *jets and the metal stream*, and the *focusing of the jets into a point or line*. The impact from the high pressure stream of water leads to disintegration of the flowing metal.



Powder Production Techniques

A *high energy input to the water* stream is needed. The usual pressure range is *6- 21 MPa*, resulting in a velocity of *70-250 m.s-1* of the water jets. Throughput is typically *10-100 kg.min⁻¹* of metal for *0.1-0.4 m³.min-1 water consumed*. The *overall process efficiency is much better than in the mechanical disintegration of solid materials*, but *still remains $\leq 1\%$* . Atomization occurs under an *unsteady state of turbulent flow* conditions, which makes a theoretical treatment of the process very *complicated*. From empirically derived relations it can be concluded, however, that the most important parameters which control the *average powder particle size, d_D* are the *water pressure P_w* and *velocity v_w* the *angle alpha*; the *melt stream diameter d_s* , the *melt viscosity, melt density, melt surface tension γ_s* , and the *ratio of molten metal to water flow rate (q_s/q_w)*. *The particle size decreases with increasing component of the jet velocity perpendicular to the melt stream:*

$$d_D = \text{const} / (v_w \sin \alpha)$$

Powder Production Techniques

From the empirically derived equation (2.5) it can be seen that *small melt stream diameters*, *high flow rate ratios* (q_s/q_w), *high density*, and *low viscosity and surface tension of the melt* also favour the production of *fine particles*.

$$d_D = d_S (d_S \rho_S v_S / \eta_S)^{-0.57} (d_S \rho_S v_S / \gamma_S)^{-0.22} (q_S / q_W)^{-0.043} \quad (2.5)$$

The overall range of the *mean particle size* attained by *water atomization* is about *30-1000* micron. *Quite different particle shapes* can be produced, depending on the process *parameters*. With *decreasing superheating of the melt*, *increasing jet velocity*, and *decreasing flow rate ratio the particle shape changes from nearly spherical to irregular*. *Irregular particle shapes* provide a good *green strength* of the *cold compacted powders* and are therefore preferred in the production of *iron and steel powders for structural part applications*.

Powder Production Techniques

Due to its low capital and operating costs, water atomization is, in terms of output, the most important atomization method. The *limiting factor is the reaction of the atomized metal with water*. Water atomization is therefore restricted to metals and alloys of *low oxygen affinity*, which pick up only *tolerable amounts of oxygen* or which can be *reduced easily* in a subsequent step. *Iron and low alloy steel powders* can usually be *reduced in hydrogen-containing atmospheres*. In some *special* cases, even *iron base powders containing substantial amounts of highly reactive elements* are produced by *water atomization*, e.g., *high speed tool steels*. In such processes, special measures are necessary, such as adjustment of the *carbon content* of the *melt* and *vacuum reduction annealing of the powder*.

Powder Production Techniques

The use of *high purity liquid steel* with *low concentrations of interstitial elements* ensures the production of *powders with highest compressability* ('super-compressable powders'), which are used e.g. for *low-alloy Mn-Mo-Ni-steels*. Their residual oxygen content is, depending on the alloying element, only 0.08-0.15 wt-%.

More recently, *synthetic oils have been used as the atomization liquid*, thereby reducing the *oxidation problem*. In oil atomization, however, *carbon pick-up* occurs in the powders. *Decarburization of powders containing reactive elements* is, however, *less of a problem* than is the need for *reduction in the case of water atomization*. Industrially oil atomization is used for the production of *low alloy Mn-Cr steel* powders.

Powder Production Techniques

Gas atomization

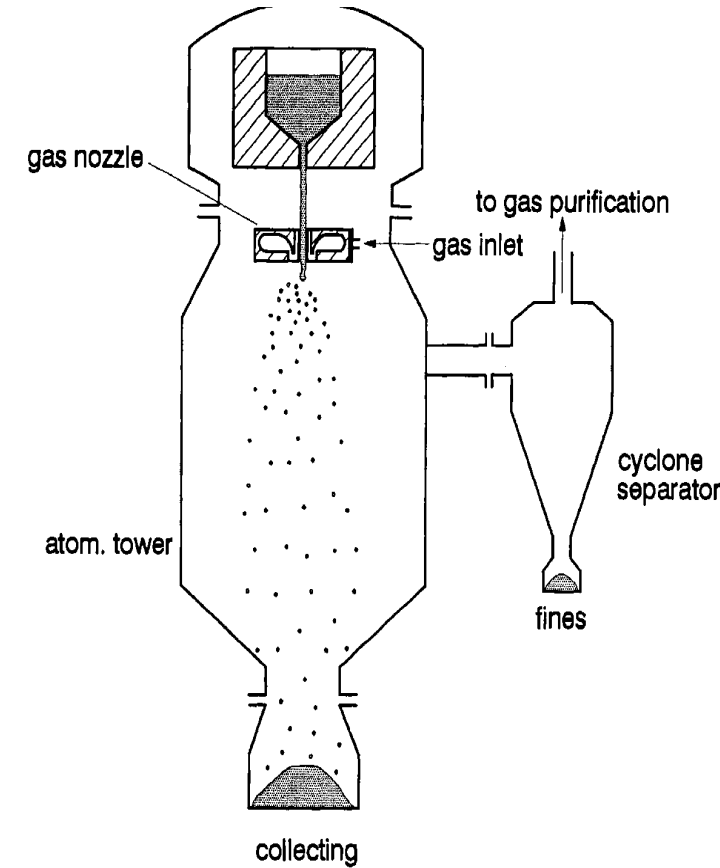
Gas atomization is the *second most important atomization* process.

Air, nitrogen, argon or helium are used, depending on the *requirements* determined by the *metal to be atomized*. Atomization

is undertaken either in *vertical or horizontal units*. In Fig. 2.14, a vertical unit for inert gas atomization is schematically represented.

The process principles are similar to those of water atomization including the problem of mathematical treatment. An empirically derived relation between the process variables and the mean particle size is given in equation (2.6).

A *cyclone separator* is an economical device for removing *particulate solids from a fluid system*. The induced *centrifugal force* is tangentially imparted on the wall of the cyclone cylinder. This force, with the *density difference* between the fluid and solid, increases the relative *settling velocity*.



$$d_D = C d_s \sqrt{\frac{v_s \gamma_s}{v_G v_G \rho_s d_s} \left(1 + \frac{q_s}{q_G}\right)}$$

Powder Production Techniques

$$d_D = C d_S \sqrt{\frac{v_S \gamma_S}{v_G v_G \rho_S d_S} \left(1 + \frac{q_S}{q_G}\right)}$$

where *V* represents the kinematic viscosity, and G is the index for the atomization gas. Again, a *high velocity of the atomizing fluid, small melt stream diameters, high density, and low viscosity and surface tension of the melt favour the production of fine particles*. The factor *C*, is dependent on the *geometrical design of the nozzle* which forms the gas stream or jets. The *nozzle design seems* to be an important *key to success* or failure of a gas atomization system. The *overall process efficiency* is similar to that of *water atomization* (i.e. ~ 1%), but the *process costs are substantially higher*. The *throughput* of *single melt stream installations is up to 50 kg/min*. *Gas pressures up to 12 MPa* are applied, resulting in supersonic *gas velocities of up to Mach 2* and a *gas flow rate of up to 40 m³. min⁻¹*. The *gas leaves the atomization chamber through a cyclone*, where the *fine powder particles are separated before the gas is recycled into the process*. Because of the relatively *low cooling rates*, the *atomization chambers are quite large*. A complete *vertical atomization* installation can be up to *20 m in height*.

Kinematic viscosity is defined as the ratio of *dynamic viscosity to fluid density*, expressed in units of *m²/s*.

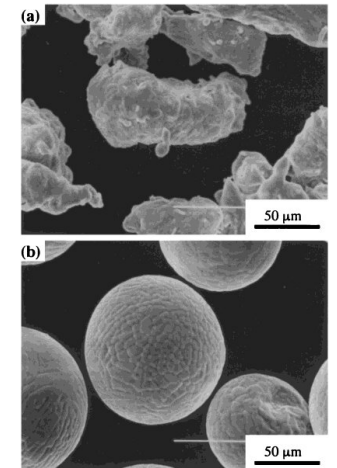
Dynamic viscosity is defined as a *measure of the resistance to flow in a fluid*, characterized for *Newtonian fluids* by the relationship $\mu = \tau / (dv/dn)$, where τ represents shear stress, v is the fluid velocity, and dv/dn is the velocity gradient perpendicular to the flow direction.

Powder Production Techniques

The *mean particle size of gas atomized* powders is in the range of *20-300 micron*. The *particle shape is spherical or close to spherical*. *Irregular particle* shapes can only be produced in systems where *reactions between the gas and the liquid metal cause the formation of solid surface layers*. This is the case, for example, in the *air atomization of aluminium*.

Melt *atomization by air* is used in the production of the so-called '*Roheisen-Zunder*' (RZ) *iron powder*. This process starts from a *cast iron melt*. The *surface oxides formed during atomization are reduced by the inherent carbon of the cast iron particles during a simple subsequent annealing treatment*.

In aluminum air atomization, a *film of oxide* is usually formed. The *spontaneous oxide formation prevents spheroidization* of the individual droplets by surface tension of the liquid metal.



Powder Production Techniques

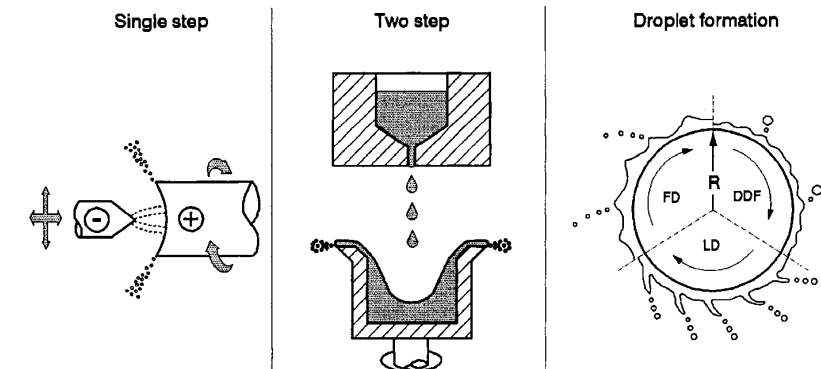
A powder *low in oxygen and carbon* is obtained only when their *concentrations within the powder* are balanced *according to equation* (2.7). Since this is normally *not the case*, a final *hydrogen reduction is advisable*. As a result of increasing quality demands, the *RZ process has now been almost entirely replaced by water atomization*. Air atomization is still used in the production of *aluminium and aluminium alloys, copper and copper alloys, precious metals, tin and lead powders*.

Inert gas atomization is applicable for *all metallic alloys which can be melted*. The main application is for *high alloy products such as stainless steel, tool steels, iron, nickel- or cobalt-base superalloy* powders, as well as *aluminium alloy* powders. *Limiting factors* are the availability of *suitable crucible and auxiliary melting process materials*. *Powders from refractory metals with high melting temperature and highly reactive materials, such as titanium alloys, are usually produced by other methods*.

Powder Production Techniques

Centrifugal Atomization

The centrifugal atomization processes use *centrifugal force* for the *melt disintegration* and involve *one or two process steps* (Fig. 2.15). In the *single step* method, *a liquid film* is produced on the *surface of a rapidly rotating consumable alloy bar* by *an arc (rotating electrode process), plasma, or electron beam source*. The *electrode or alloy bar, which is rotating at up to 15,000 rpm* has to meet stringent requirements as to *stability, uniformity and surface quality*. *The melt cannot be highly superheated, as the molten material then rapidly runs off from the electrode*. The *two-step* process overcomes these problems by the *separation of the melting and melt disintegration stages*. The latter is performed by a *rapidly revolving cooled wheel, disc or cup*. *The problem with the two-step process is to transfer high rotational velocities to the melt*.



Powder Production Techniques

At *low melting rates* the *melt disintegration* occurs by the *direct drop formation* mechanism DDF (Fig. 2.15). A *primary particle* is emitted from *liquid protuberances at the edge of the rotating disc or cup*. *This droplet is for a short time linked to the edge by a fine liquid thread*, which is also then emitted as *a smaller secondary particle*. The *particle size distribution, therefore, becomes bimodal in the DDF mode*.

At *higher melting rates*, the *protuberances stretch into ligaments*, which *break down into strings of many particles* by a ligament disintegration mechanism (LD). *The proportion of secondary particles increases steadily with the melting rate*. *At even higher melting rates, a film is formed at the edge of the disc, directly emitting the droplets by a film disintegration mechanism (FD)*.

Powder Production Techniques

$$d_D = \frac{1}{2 \pi n} \sqrt{\frac{6 \gamma_s}{\rho_s R}} \quad (2.8)$$

A mathematical treatment of centrifugal disintegration gives equation (2.8). The *liquid droplet diameter decreases* with *increasing rotational velocity*, increasing *melt density* and *decreasing melt velocity*.

The particle shape is *spherical*. One advantage of centrifugal atomization is the *narrow range of particle size distribution*. The *mean particle size*, however, is relatively *coarse*, in the range from *150-250 micron* for *single step processes*. Powders with a mean size of *80-90 micron* can only be prepared in *two-step installations*, where a crucible rotation speed up to *25,000 rpm* is feasible.

The main advantage of *single step centrifugal atomization* is that the *melt has no contact with ceramic crucibles or liners*. The process is therefore suitable for the production of *refractory powders like tungsten*, or *highly reactive powders, e.g., titanium alloys*. The *production capacity is low* (1-6 kg min⁻¹). *Two-step* centrifugal atomization is used for the production of *aluminium, titanium alloys, superalloys and refractory metal powders*.

Powder Production Techniques

Vacuum Atomization

The *vacuum atomization or melt explosion technique* utilizes the *pressure dependence of gas solubility in metal melts*. After *vacuum induction melting, the melt is saturated at an enhanced pressure (1-3 MPa) with a gas, usually hydrogen*. A ceramic nozzle system is then *immersed into the melt to connect it to the evacuated atomization chamber*. The gas is *spontaneously released and breaks the melt stream down into droplets*. The resulting *powder morphology* is similar to inert *gas atomized powders* in both particle *shape and size distribution*.

Vacuum induction melting (VIM) utilizes electric currents to melt metal within a vacuum.

Powder Production Techniques

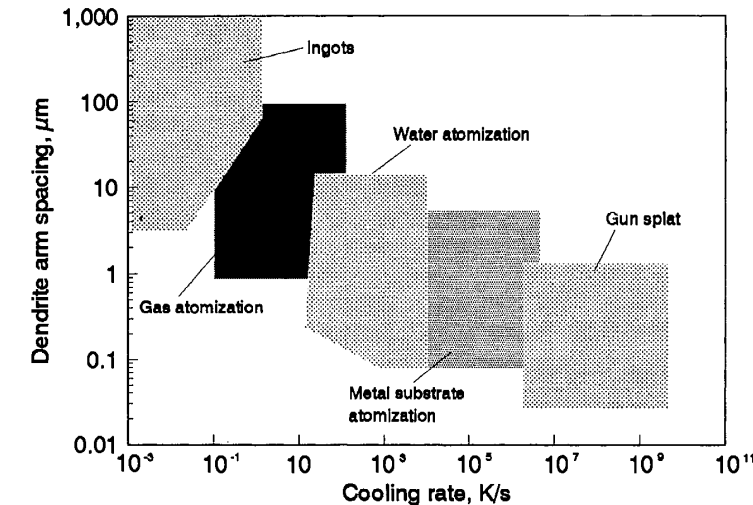
Solidification and Cooling

Depending on the *different mechanisms of heat transfer* (see Table 2.1), the processes described above lead to *different cooling rates*. The cooling rate itself determines *the powder particle shape, its microstructure and, in combination with the in-flight speed, the dimensions of the atomization chamber*. Figure 2.17 gives an order of magnitude overview of the cooling rates achieved by the different methods. Cooling occurs by heat transfer via radiation and heat flow through the medium which is in contact with the melt. *The melt volume, together with the heat flow, determines the cooling rate during solidification. Small melt volumes favour rapid cooling. Atomization under vacuum or reduced pressure conditions results in relatively low cooling rates.*

Table 2.1 Variables in the processing steps of atomization

Melting		Melt disintegration		Solidification/Cooling
crucibles/liners	heating source	melt volume	energy input	heat transfer
<ul style="list-style-type: none"> • with • without 	<ul style="list-style-type: none"> • induction • arc • plasma • electron beam 	<ul style="list-style-type: none"> ○ melt pool • melt stream • melt film ○ melt droplet 	<ul style="list-style-type: none"> • liquid/gas stream • rotational energy • gas supersaturation ○ ultrasonic vibrations ○ mechanical impact ○ hydrostatic pressure ○ electrostatic forces 	<ul style="list-style-type: none"> • radiation • convection ○ contact cooling

• commercially used; ○ experimental state



Powder Production Techniques

The *microstructure of the powder particles is determined by nucleation and growth factors*. After nucleation has occurred, the *relation of the growth rate of the solid from the liquid and the diffusion rate in the liquid determines the microstructure*. *A large undercooling increases the growth/diffusion ratio and favours uniform crystal structures*. Low growth/diffusion ratios result in dendritic microstructures as a consequence of segregation. For many common alloys the transition from dendritic to cellular microstructure occurs at a cooling rate of the order of 10^5K/s . *The secondary dendrite arm spacing is a direct measure of the degree of segregation*. It decreases with the cooling rate, as already shown in Fig. 2.17. With increasing cooling rate, the particles tend to form microcrystalline structures, combined with other non-equilibrium states such as high defect concentrations or metastable phases, which promote sintering effectively.

Powder Production Techniques

Even noncrystalline (*amorphous*) structures can be obtained. Some processes which can achieve such *rates are the droplet impact disintegration method, planar flow casting or melt extraction which uses a fine edged spinning wheel*. These utilize chill block cooling and belong to the so called *rapid solidification* methods. They are widely used for *steels, copper, magnesium and aluminium alloys*. Rapid solidification, in its different forms, is steadily growing in importance.

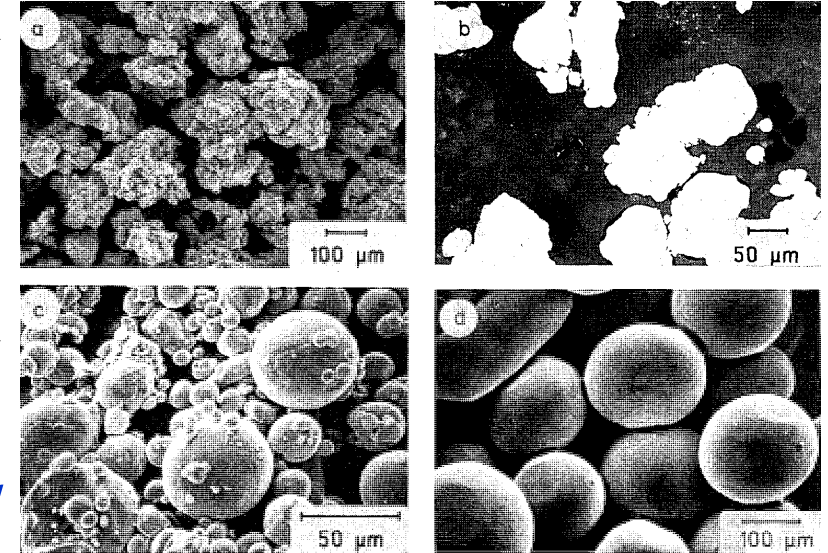
The *dimensions of the atomization chamber* are determined by the *solidification time and the in-flight velocity of the liquid droplets*. Therefore, *large units are required for vacuum processes* (e.g. centrifugal atomization and vacuum atomization) and for *gas atomization with high speed gas streams*. This problem can be reduced by deflector plates, which divert and retard the droplet flow.

Powder Production Techniques

Particle adhesion to the plates can be inhibited by *coating or by regenerating liquid films*. Small chamber dimensions are suitable for liquid atomization, due to the high cooling rates, and in ultrasonic atomization, where the droplet velocity remains moderate.

Figure 2.18 shows several powders produced by the most common atomization techniques: water, gas and centrifugal atomization.

Spherical particles result from the solidifying droplets in gas and centrifugal atomization as a result of surface tension. Water atomized powders can be produced with very irregular particle shapes for high green strength requirements, but particles close to spherical can also be produced if good flowability and tap density are required.



Powder Production Techniques

CHEMICAL PROCESSES

- *Reduction*

Hydrogen reduction

Hydrochemical reduction

Carbon reduction

Reduction by metals

- *Carbonyl Processes*

- *Hydride-Dehydride Processes*

- *Electrochemical Processes*

Electrolysis of aqueous solutions

Fused Salt Electrolysis

Powder Production Techniques

Reduction

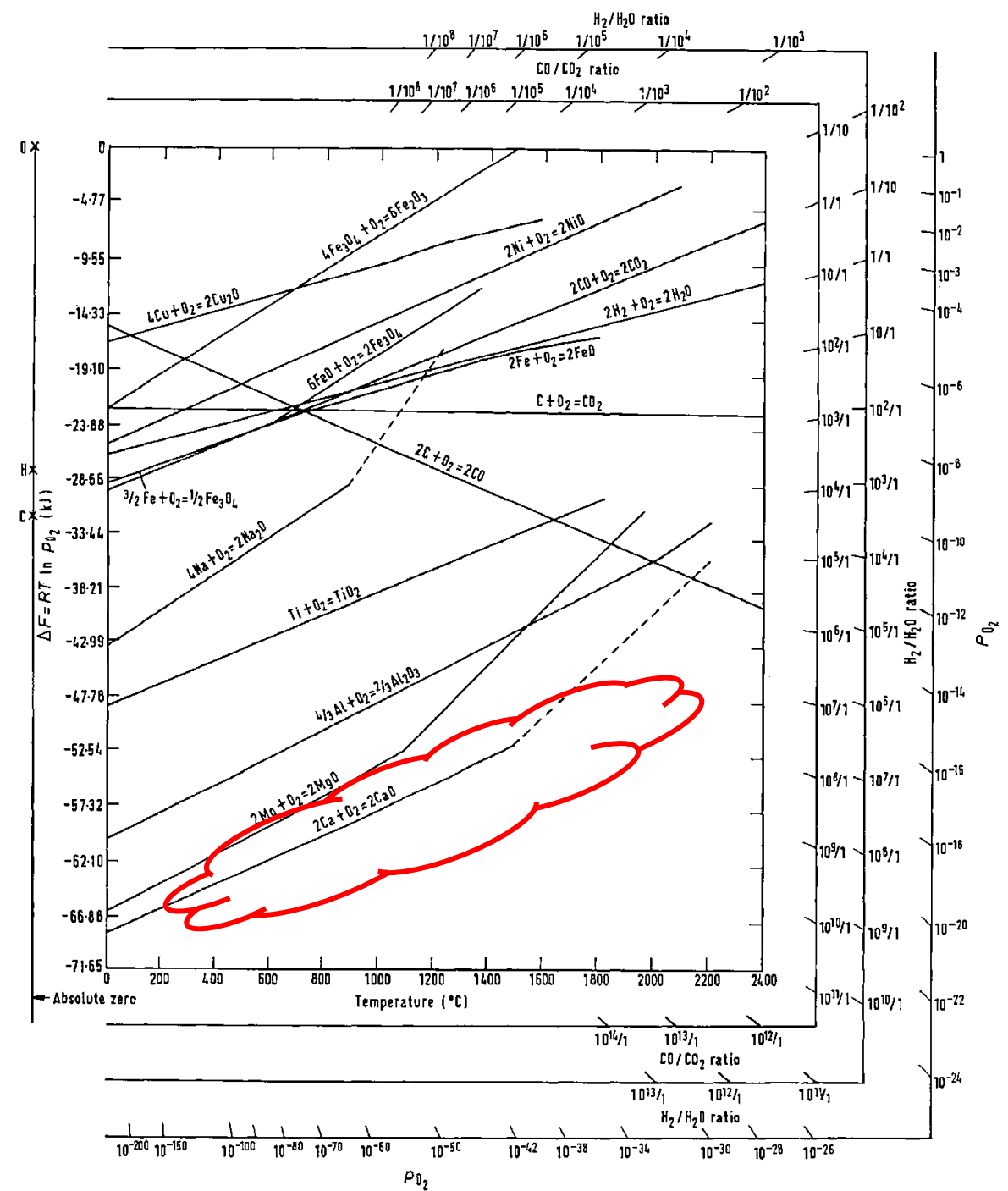
Principle aspects

The main chemical processes in powder metallurgy involve the *reduction of metal compounds* such as *oxides, carbonates, nitrates or halogenides* with *gases* (generally *hydrogen*) or *solids* (*carbon or highly reactive metals*). In *most cases the metal compounds to be reduced are in the solid state*. However, *hydrometallurgical processes* have been developed also, especially for the *reduction of nickel and cobalt solutions* by *pressurized hydrogen*. When the *reaction takes place in the solid state or by solid-gas reaction, no subsequent purification is possible*. A sufficiently *pure raw material* has, therefore, to be provided as a feedstock. For kinetic reasons, the *compound to be reduced has to have a suitably small particle size*, which should not be exceeded, otherwise the controlling diffusion processes may lead to very long reaction times which are not acceptable in practice.

Powder Production Techniques

The *process is controlled by its free energy of reaction which has to be negative*. The *more stable the compound, the stronger the reduction media* have to be. Some basic thermodynamic data are used for reduction process. *The free energies of oxidation for some of the reducing agents are included. Since alkali, and especially alkaline earth metals, form very stable oxides, they are very strong reducing agents.* Figure 2.19 also shows the extreme reducing capability of Ca, which is needed when H₂, C, CO or Na cannot be used.

Powder Production Techniques



Powder Production Techniques

Hydrogen reduction

The *reduction of oxides or other compounds* with hydrogen is generally undertaken well *below the melting temperature of the metal*. Examples of *technical importance are refractory metals such as tungsten and molybdenum, ferrous metals and copper, which form compounds with only moderate stability. Very pure and fine powders can be obtained.*

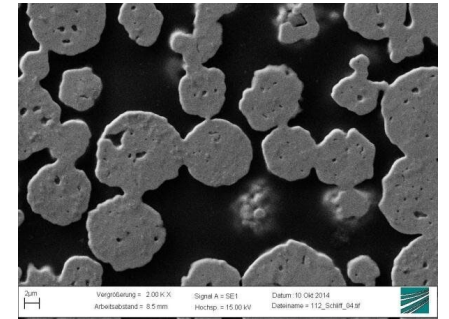
The process is often undertaken in *tube furnaces*, in which the *oxide powder is moved in flat crucibles in the opposite direction to the hydrogen stream*. The overall reaction equations such as $\text{MeO} + \text{H}_2 \rightleftharpoons \text{Me} + \text{H}_2\text{O}$ are simple.

Powder Production Techniques

In reality, several *different reaction steps* can be involved, depending on the *lower oxide compounds* that are *stable and the degree of Me-O solid solution*. Furthermore, the properties of the resulting powder and, consequently, the reproducibility of the product, are strongly dependent upon the *reducing conditions*. Some general rules can be stated as follows:

- *a higher reduction temperature and a longer time provide: larger particle size, lower specific surface, lower residual oxygen content and (possibly) formation of a 'sintered cake'.*
- *a lower reduction temperature and a shorter time provide: smaller particle size, higher specific surface, higher residual oxygen content and (possibly) pyrophoric powders* (see section 5.1).
- *a high flow rate of hydrogen* with a *low dew-point* results in a *high reduction rate*, *low residual oxygen* content and *no, or very little, re-oxidation* during the cooling period.

The *dew point* or dew point temperature of a gas is the temperature at which *the water vapor or low-boiling hydrocarbon derivatives contained in the gas is transformed into the liquid state.*

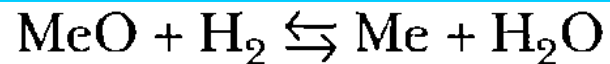


یعنی هیدروژنی که رطوبت بسیار کمی دارد.

Powder Production Techniques

چون اکتیویته مواد جامد MeO و Me تقریباً برابر ۱ در نظر گرفته می شود، عبارت ثابت تعادل فقط تابع گازهاست.

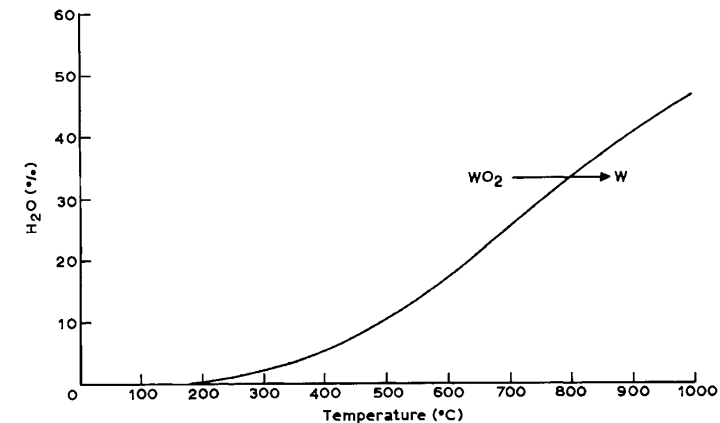
- a *low flow rate of hydrogen with a high dew-point* would produce the opposite results and would therefore be generally *impracticable*. The *danger of re-oxidation of reduced powders at low temperatures is caused by the temperature dependence of the equilibrium according to equation* (2.10) moving to the left hand side at low temperatures. This is shown for the W02 - W reduction in Fig. 2.20.



Chemical equilibria are generally defined by the equilibrium constant K in accordance with the law of mass action. *K is the ratio of the concentrations of starting materials to those of reactants*, given in this case as

$$K_{(T)} = p_{\text{H}_2\text{O}} / p_{\text{H}_2}$$

• اگر نسبت کوچک باشد → گاز احیایی تر است → واکنش به سمت راست (تشکیل فلز) پیش می رود.
• اگر این نسبت زیاد شود → تمایل به تشکیل اکسید افزایش می یابد → ممکن است پودر فلز دوباره اکسید شود.
به همین دلیل است که در دماهای پایین (همان طور که در جمله ی قبلی گفتی)، K کاهش می یابد و تعادل به سمت چپ (= تشکیل MeO) جابه جا می شود.



Powder Production Techniques

Hydrochemical reduction

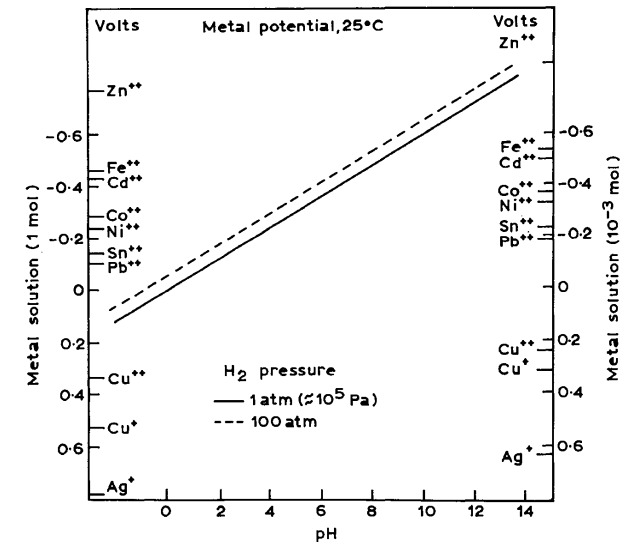
Several *metals* can be produced in *powder* form directly from *aqueous (or organic) solutions by reduction with (preferably) gaseous hydrogen*. This process has been developed by Sherrit Gordon Mines Ltd, Canada, especially for the production of *nickel and cobalt powder* from *sulphidic ores* containing (preferably) *Cu, Ni, Co, Fe* and *S.A convenient technique* is the *separation of a Cu rich and a Ni + Co rich* fraction by *flotation* of the *fine ore* with a subsequent *leaching procedure in autoclaves* with NH_4OH under air pressures of 7-9 bar. *Ni, Co (and the residual Cu) form readily soluble ammines* (e.g., $\text{Ni} [\text{NH}_3]_6 \text{SO}_4$: nickel hexammine sulphate), *Fe forms insoluble $\text{Fe}(\text{OH})_3$ and the Sulphur is transformed to sulphates. Most of the other constituents of the ore are insoluble in ammonia* and form a *solid phase together with $\text{Fe}(\text{OH})_3$* . However, a few other metals, like *Sn and Cd also enter the solution*.

Powder Production Techniques

The *clean solution* is then treated with *gaseous hydrogen under 28-35 bar* pressure at *180-220°C in an autoclave* when the following reaction occurs

(ignoring the complex ions): $\text{Me}^{++} + \text{H}_2 \rightleftharpoons \text{Me} + 2\text{H}^+$

Low [H⁺] and high pH shift the equilibrium to the right hand side and facilitate metal precipitation. The electrochemical conditions for the process are summarized in Fig. 2.21. *Gaseous hydrogen is able to precipitate metals from their ionic solution*, when *the H₂/H⁺ potential is more negative than Me/Meⁿ⁺*, i.e. in the field below the straight lines. There is a small dependence of the H₂/H potential on the H₂ partial pressure (as seen by the small distance between lines a and b), but a large dependence on pH value. Thus, most of the metals can be precipitated from alkaline solutions and only a few such as Cu and Ag from stronger acidic liquids.



Powder Production Techniques

A special application of this process is the preparation of *composite powders*, when a 'seed powder' is dispersed in the liquid, which has to be kept in continuous motion in order to achieve a homogeneous dispersion. *Each powder particle acts as a nucleus and is coated by the deposit.* Examples are *Ni-coated oxide or graphite and Co-coated diamond particles*. The uses of composite powders (see section 4.3.) include avoiding segregation of the components; facilitating the even distribution of a dispersant in a matrix (to prevent particle contacts); improving the wettability in melts etc. Composite powders can also be produced by other methods such as chemical vapour deposition (CVD), physical vapour deposition (*PVD*) , sol/gel processes, etc. Recently, WC-Co composite powders have been produced in form of nanosized (20-40 nm) particles (see section 2.4) by a spray drying process for manufacturing of special hard metals.

Powder Production Techniques

Carbon reduction

One of the most important powder production processes is the *direct reduction of magnetite ores (Fe_3O_4) by carbon*. The *final product, sponge iron powder*, is one of the starting materials for the mass production of *sintered iron and steel parts*, and more than *50% of the iron powder* used is produced by this process. The *ore is ground* to particle sizes of less than 0.5 mm and *purified by magnetic separation*, up to the iron content of about 71.5%. (the theoretical value is 72.5%.) After *drying the ore* is poured into *cylindrical ceramic capsules*, together with *mixtures of fine coke and lime in concentric layers*. The *lime is used to extract the sulphur impurities*. A number of capsules is arranged on a transportation unit which is moved through a *reduction tunnel furnace, heated by natural gas*.

Powder Production Techniques

The *furnace is divided* into a *heating zone*, a *reduction zone* with a *maximum* temperature of about *1250°C* and a *cooling zone*, with an overall length of about 270 m. The *reduction process, starting at 650-700°C*, lasts *two to three days*, works continuously and is fully automated. The reaction product is an *iron sponge formed by slight reaction sintering*, which is then *crushed*, classified and subjected to a *final treatment to adjust carbon and oxygen contents and to reduce residual stresses*.

Most of the powder is milled to particle size distributions suitable for *powder metallurgy parts* production, whilst some (particle size > 150 micron) is sold for the manufacture of *welding electrodes*. It is very important to *reduce the carbon as well as the oxygen content* (and especially the *non-metallic oxide inclusions*) to levels acceptable for sintered parts.

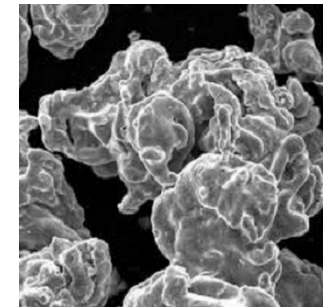
Powder Production Techniques

The *reduction* process occurs, after an *early solid state reaction*, mainly via the *gas phase*, *although no reducing gas is introduced*

$$\text{Fe}_3\text{O}_4 + 4 \text{CO} \rightleftharpoons 3\text{Fe} + 4 \text{CO}_2$$

The overall reaction is while the carbon is in equilibrium according to $\text{C} + \text{CO}_2 \rightleftharpoons 2\text{CO}$

, which is strongly dependent on *temperature*. *Side reactions, such as the formation of Fe₃C, are not important under the processing conditions*. The *sponge iron powder* has some *internal porosity*, is of *irregular shape* and has a good *compressibility*, although the *pressed density is influenced by its micro-porosity*. This is a residue of the reduction process and is in contrast to the near pore-free microstructure of water atomized particles.



Powder Production Techniques

Attempts have also been made to *reduce milling 'scale'* for the production of *iron powder*. Scale is a high *temperature oxidation product of iron and steel* and has an overall composition close to Fe_3O_4 . Reduction processing is simple in principle but technically and commercially feasible only when the *scale is available in large quantities*, i.e. from big milling plants, producing high purity and uniform quality over long periods. This *cannot be achieved*, when *different (unalloyed and alloyed) steels are milled at the same plant*, and which contain Mn, Si, Cr, Al, which form *oxides in the scale and which cannot be reduced completely by carbon* - the resulting iron powder containing uncontrollable amounts of *non-metallic inclusions*.

Powder Production Techniques

Carbonyl Processes

Several *metals react with carbon monoxide* (CO) under *certain conditions* to form metal carbonyls (*$Me(CO)_x$*). *Fine metallic powders* can be obtained by their thermal decomposition. This is especially important with iron carbonyl ($Fe(CO)_5$, boiling point = $102.7^{\circ}C$) and nickel carbonyl ($Ni(CO)_4$, boiling point = $43^{\circ}C$), from which carbonyl iron and carbonyl nickel powders are obtained. Because of the *low boiling point of the carbonyls* (which are both toxic and flammable) they *can be purified easily by distillation*. The resulting *metal powders* are therefore *very pure*, except for some *carbon and oxygen impurities*.

For *carbonyl iron production*, *spongy* iron, having a *large surface area* with *high surface activity*, is required to obtain an adequate reaction rate.

Powder Production Techniques

Hydride-Dehydride Processes

The hydride route enables *very fine and reactive powders* to be produced from metals which are capable of forming solid hydrides. The *hydrides* are obtained by the reaction of *metal with molecular hydrogen* at *elevated temperatures* under a *suitable partial pressure*. Since they are *brittle* they can be *powdered* easily and *dissociated in vacuum at similar or somewhat higher temperatures than those at which they are formed*. For powder metallurgy, the *hydrides of Ti, Zr, Hf, Ta, U, Th, Y and perhaps Mg* are of interest. *Metal powders* produced from hydrides are highly *active* and often *pyrophoric, depending on dissociation conditions*. Special *precautions*, such as *inert gas absorption on the powder surfaces*, are often necessary in order to *avoid instantaneous ignition on exposure to air*. Compacts of zirconium powder produced from the hydride can be sintered nearly to full density. *Tantalum hydride* powder is of some importance in obtaining very *pure tantalum carbide* powder by carburization, using carbon black. The hydride can be *economically obtained from Ta scrap or waste materials*. *The hydride-dehydride process does not involve any purification of the metal phase. On the contrary, an increase of oxygen and nitrogen can be expected in the final metal powder, depending on the quality of hydrogen and the vacuum system used.*

Powder Production Techniques

Electrochemical Processes

Powder production by *electrolysis* is a *reduction process* in which the *metal ions* are *neutralized by the cathodic current*. The *cathodic potential* necessary for *deposition* depends on the *position of the metal in the electromotive series* and on *special inhibiting electrode* processes, which can produce a *voltage* surge necessary for deposition. Whenever possible the process is carried out in *aqueous solutions (e.g. for Cu, Fe, Ni, Co, Zn, etc)*. *Highly reactive metals (e.g. Be, Ta, Nb, Th, etc.) which form highly stable oxides, have to be deposited from molten salt electrolytes*. While *aqueous solutions are employed at slightly elevated temperatures* (up to 60°C) the *molten salt process needs higher temperatures*, depending on the *melting point of suitable (binary or ternary) salt eutectics* which should contain a reasonable concentration of the metal to be deposited. *The powder can be obtained directly or indirectly, the latter by milling of the often hydrogen-embrittled deposits*. The main *process parameters* to be considered are: *metal ion concentration, electrolyte conductivity, temperature, voltage, current density and bath circulation*, and, in special cases, the addition of *colloids* in order to *inhibit the growth of nuclei*. *In aqueous solutions, low temperature and high current density facilitate the precipitation of powder rather than larger deposits*.

Powder Production Techniques

CERAMIC POWDERS

Since the boundaries between powder metallurgy and ceramics are no longer strictly defined, some information on ceramic powders is useful in the context of powder metallurgy. This applies mainly to advanced ceramics. While for the *traditional silicate ceramics* in general use are *natural raw materials* (*clay, kaolin*, etc.), the processing of advanced ceramics requires pure and well-defined powders normally made synthetically. Also the manufacture of ceramic parts is to some extent similar to powder metallurgy processing. It is convenient to subdivide this field into *oxide and non-oxide ceramics*. The *synthesis of ceramic powders employs a wide variety of methods*, yielding powders of very different particle size distribution and specific surface, chemical and phase composition, purity, compactibility, sinterability, etc. *Sinter grade* powders for manufacturing *dense, fine-grained parts* must be produced in the *lower micron or even sub-micron range* with *specific surfaces* between 5 and 15 m²g⁻¹ or higher. Applications of coarser powders (Al₂O₃, SiC) include abrasives, refractories (Al₂O₃, ZrO₂, Mullite), etc.

The principle methods for production are as follows:

Powder Production Techniques

Solid state reactions: *Hydroxides, carbonates, sulphates, oxalates* and other compounds can be *thermally decomposed to oxides* by well known chemical reactions. Depending on *temperature-time, loose or agglomerated powders or a sintered cake result*, which have to *be milled to the desired particle size*. *In principle the majority of the oxide powders can be produced by this method. The precursor compounds are obtained by either liquid or gas processes. Carbides are made by reaction of metals or oxides with carbon.* One of the most important industrial processes is the Acheson method for the production of a SiC from SiO₂ (see section 2.3.4), in which it is possible that part of the reaction occurs via the gas phase.

این فرآیند بر اساس **کربوترمی** (واکنش در حضور کربن در دمای بالا) استوار است و در یک کوره مقاوم الکتریکی به نام **کوره آپسون** انجام می‌گیرد.

1. مواد اولیه: مخلوطی از سیلیس (ماسه کوارتز، SiO₂) و **کک نفتی** (کربن خالص)، با درصد استوکیومتری تقریبی به دست می‌آید.

2. ساختار کوره: مواد اولیه در اطراف یک هسته مرکزی گرافیتی قرار داده می‌شوند که به عنوان **الکتروود مقاومتی** عمل می‌کند.

3. حرارت‌دهی: جریان الکتریکی بسیار قوی از طریق الکتروود مرکزی عبور داده می‌شود. این امر باعث می‌شود که دمای کوره به شدت بالا برود و به حدود **۲۵۰۰ درجه سانتی‌گراد** برسد.

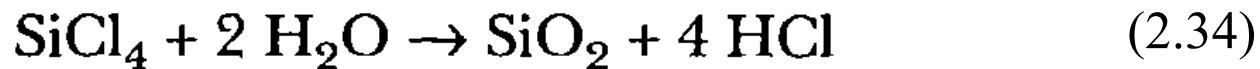
4. واکنش: در این دمای بالا، کربن با دی‌اکسید سیلیکون واکنش داده و کاربید سیلیکون تولید می‌کند و آب به عنوان محصول جانبی خارج می‌شود:



5. نتیجه: پس از سرد شدن، محصول نهایی در اطراف هسته الکتروود مرکزی، توده‌ای از کریستال‌های کاربید سیلیکون را تشکیل می‌دهد.

Powder Production Techniques

- *Solid-gas reactions.* **Oxides, carbides and nitrides** may be synthesised by the reaction of **metals with oxygen, hydrocarbons, nitrogen or ammonia** respectively. While this is **less important for oxides**, it is a technique used, for example, for the synthesis of ~ **SiC and Si₃N₄**, the latter sometimes under **increased N₂ pressure**.
- *Gas-phase reaction.* **Vapour phase decomposition or hydrolysis in a flame** is a common technique for the production of **TiO₂ and SiO₂ from halogenides TiCl₄ and SiCl₄**, according to the reaction (for SiO₂):



High purity powders with **extremely high specific surface areas** (100 m²g⁻¹ and higher) with only **very little agglomeration** of the powder particles, are obtained in this continuous process.

- *Melting processes.* Oxides such as **Al₂O₃ and ZrO₂** are manufactured on **a large scale by arc-melting** with **subsequent milling to make powders of different particle size ranges**. The process is mainly used to produce **coarse grade powders** for the manufacture of refractories.

Powder Production Techniques

POWDERS FROM SOLUTIONS

In several techniques *an aqueous or non-aqueous solution of salts* is the *precursor* for preparing powders. After *solvent removal, the solid residue is thermally converted to ceramic or in some cases metallic powders*. The *solutions* are *normally homogeneous* on an atomic scale, but the *degree of homogeneity of the residue, however, depends on the solvent removal technique*. Extremely *fine powders, powder mixtures* and *powders with uniformly distributed additives* (e.g., to improve sintering), are obtainable.

This field may be subdivided as follows:

Precipitation and filtration of single compounds or mixtures.

Hydrothermal reactions

Solution combustion.

Solvent vaporization or dehydration

Powder Production Techniques

The Sol-gel Process

In a separate sub-group are *gelation-dehydration techniques* in which *liquid droplets are transformed into a gelatinous state from which the powder is obtained*. These sol-gel techniques now play an increasing role in the preparation of *advanced ceramics*. The techniques consist in the formation of a *three dimensional network of inorganic matter* (gel) from *colloidal or molecular solutions* of the precursor (sol). This *reaction* takes place in *aqueous or nonaqueous media*, e.g. by *diminishing the water content*, by *changing the PH-value or the surface charge (zeta potential) of the sol*, or by other *means which can lead to gelation of the liquid precursor*. A wide variety of methods may be used, producing small uniform spheres or powders. The process was developed in the early 1960s for the preparation of micro-spheres of oxide nuclear fuels, consisting of highly reactive agglomerates of very fine particles, which can be sintered at low temperatures to very high densities.

Powder Production Techniques

ULTRAFINE POWDERS (NANOCRYSTALS)

Although the terms in the finest particle size range are not clearly defined it may be distinguished as follows: *submicron particles are per definition below 1 micron, preferably down to 0.5 micron*, *ultrafine particles between 0.5 micron and 0.1 micron (100 nm)* and *nano-sized (nano-crystalline) powders less than 100 nm, many of them range between 50 and 20 nm*. Such powders have become of increasing interest in the last years for both scientific and technical reasons.

Their processing, and sintering behaviour is unique due to their high surface to volume ratio. Several methods have been used in the past in order to obtain ultrafine powders, such as:

Gas phase reactions in a DC, RF or microwave plasma, Gas phase reaction powered by a laser, Hydrogen reduction of metal chloride vapours, Evaporation and condensation techniques, Processing of solutions.

Powder Production Techniques

WHISKERS AND SHORT FIBRES

Whiskers are short metallic or non-metallic, usually monocrystalline fibres, which are deposited generally by a *vapour-phase* process. Whiskers of metallic elements such as Ag and Cu and also of several compounds have been known for a very long time as naturally grown minerals. Scientific and technical studies of whiskers began in the 1940s. The *condition for whisker formation* (instead of 'normal' crystals or crystallites) is a strongly *anisotropic unidirectional growth*, which *often appears under undisturbed growth condition within a certain concentration and temperature range*. Whiskers can be easily obtained from most metals and several ceramic materials (such as Al₂O₃ and SiC). Some are manufactured industrially in greater than experimental quantities although there is considerable concern about the *safety of handling whiskers* which has held back keen exploitation. *For most whisker manufacturing processes volatile compounds are needed from which the deposition occurs on solid or liquid substrates. Metallic whiskers can be obtained by the reduction of a metal halogenide vapour with hydrogen in a carrier gas at normal reduction conditions.*

Powder Production Techniques

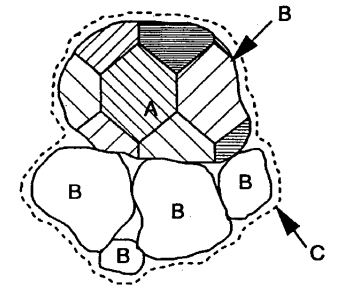
Whiskers can be obtained in a *wide range of diameters* (from 0.1 to several micron) *and lengths* (10 to several hundred micron; in some cases up to several millimetres), *depending on the material and the processing conditions*. The *length/diameter ratio ('aspect' ratio) ranges from 20 to several hundred* and is an important figure. The *cross section of whiskers* can be *hexagonal, rectangular or even irregular*, depending upon *crystal structure and growth conditions*. *Hollow whiskers, stacking faults, kinks and impurities are frequent* and determine the *quality* of the material and its efficiency for reinforcement purposes. The most outstanding property of whiskers is their *extremely high strength*, which is strongly dependent on *diameter* and *length*: the *smaller the diameter, the greater the strength*. The reason for this is that a smaller section will have *fewer or no glide dislocations*. This is important for the *strength of metallic whiskers*, while in *ceramic whiskers minimizing of the flaw size, including surface flaws*, is the decisive feature.

$$\sigma_{max} = \frac{2 \gamma E}{a}$$

! γ Surface energy, E = elastic modulus, a = atomic distance

Powder Properties and Characteristics

The common powder metallurgy terminology for the description of these elements is given in Fig. 3.1. The term *powder particle always stands for the primary particles*, which *do not develop by agglomeration* of other particles, but are directly formed as individual particles during the production process. These powder particles can be *amorphous or crystalline*. *Crystalline particles are either single crystals or consist of several crystals* or grains (polycrystalline). *Polycrystalline powders can be single- or multi-phased*. The powder particles can form secondary particles or agglomerates, as shown in Fig. 3.1. In the powder metallurgy process, agglomeration often occurs unintentionally, but can also be intentionally produced by controlled stages. *Agglomerates* are defined as *solid particles which are composed of other particles*. This definition is independent of the nature of the attractive forces which cause the primary particles to form the agglomerate. Sometimes the term *agglomerate* is *restricted to weak bonding between the primary particles*. *Hard agglomerates with strong bonding are then called aggregates*.



A= Grain
B= Powder Particle
C= Agglomerate

Powder Properties and Characteristics

3.2 PARTICLE SIZE DISTRIBUTION

3.2.1 PRINCIPAL ASPECTS

For a number of equally sized spheres or cubes the particle size characteristics are simply given by one measurement. The individual particles of practical powders usually differ in shape and dimensions. The particle shape can deviate significantly from the ideal geometries mentioned above. The *size characteristics of* powders have therefore to be treated under two aspects: first, the *real meaning of the determined size parameter*, secondly the *statistical distribution* of this parameter, which has to represent the total number of particles in the powder. *The size parameter is identical with a real linear dimension of the particles only in some exceptional cases.* Most methods for particle size measurement yield a size-dependent property of the particle. This property is then related to a linear dimension. The sieve diameter, for example, is the length of the minimum square aperture through which a particle will pass. The particle could be a sphere with the diameter of this aperture, or an *elongated platelet with only one axis corresponding to the sieve diameter, a second axis much larger, and the third axis possibly smaller than the sieve diameter.*

Powder Properties and Characteristics

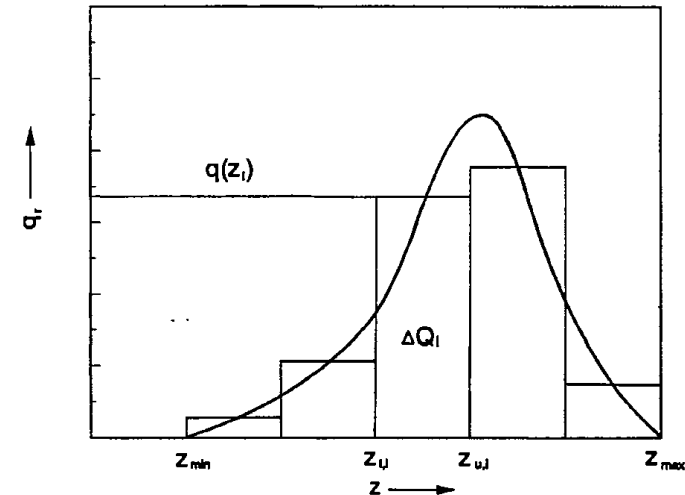
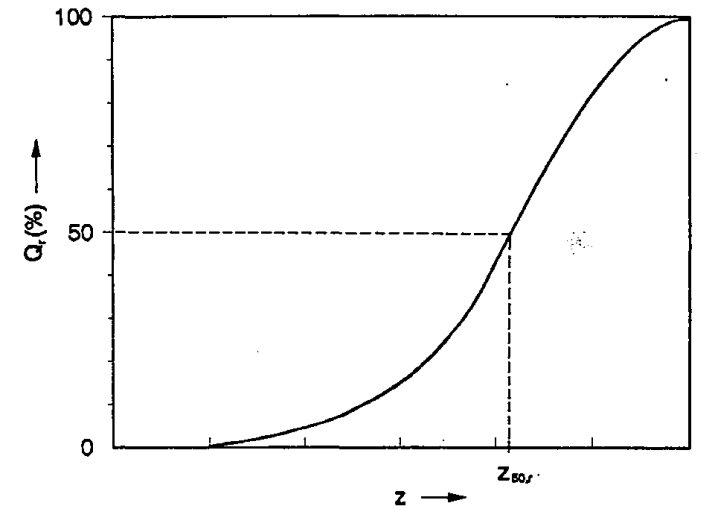
Table 3.1 Particle size parameters

Symbol	Name	Definition
d_v	Volume diameter	Diameter of sphere with same volume
d_s	Surface diameter	Diameter of sphere with same surface
d_{sv}	Surface volume diameter	Diameter of sphere with same surface to volume ratio
d_c	Perimeter diameter	Diameter of circle with perimeter or particle projection area
d_f	Free-falling diameter	Diameter of sphere with the same density and free-falling speed in a given fluid
d_{St}	Stokes' diameter	Free-falling diameter at Reynold's number < 0.2
d_A	Sieve diameter	Width of minimum square aperture through which a particle will pass

Powder Properties and Characteristics

In order to calculate the statistical distribution of the size parameter, it is necessary to characterize a statistically representative sample of the powder. The size characteristics can then be presented as distribution functions. These distribution functions represent the quantity proportion of particle belonging to a given size parameter Z_i . Quantity proportions are formulated either as cumulative distribution $Q(Z_i)$, or frequency distribution $q(Z_i)$ (Fig. 3.3). $Q(z_i)$ gives the normalized quantity proportion of particles smaller than a size parameter Z_i . The frequency distribution $q(Z_i)$ is defined as the fraction within a size class, divided by the class width.

$$q(z_{u,i} : z_{l,i}) = \frac{Q(z_{u,i}) - Q(z_{l,i})}{z_{u,i} - z_{l,i}}$$



Powder Properties and Characteristics

Table 10.1 Frequency distribution data		
Equivalent particle diameter (μm)	Number of particles in each diameter range (frequency)	Per cent particles in each diameter range (per cent frequency)
20	100	4.5
40	200	9.1
60	400	18.2
80	800	36.4
100	400	18.2
120	200	9.1
140	100	4.5

Table 10.2 Cumulative frequency distribution data			
Equivalent particle diameter (μm)	Per cent frequency (from Table 10.1)	Cumulative per cent frequency Undersize	Over size
20	4.5	4.5	100
40	9.1	13.6	95.5
60	18.2	31.8	86.4
80	36.4	68.2	68.2
100	18.2	86.4	31.8
120	9.1	95.5	13.6
140	4.5	100	4.5

Powder Properties and Characteristics

METHODS OF PARTICLE SIZE ANALYSIS

These methods can be classified into: *counting methods, sedimentation methods, separation (classification) methods, and light scattering and diffraction methods*. The *counting methods* can be subdivided into *direct and indirect methods*, depending on whether the *particles themselves are measured, or particle images or projections*. *Microscopy*, for example, is an *indirect counting method*. With direct methods, the particles directly trigger the measurement. The Coulter principle (see section 3.2.4.5) is such a method. *Sedimentation* methods utilize the *free-falling velocity of particles in a gravity or centrifugal field*. *Separation of the particles into different size classes and measurement of the fractions of every size class is realized in classical sieve analysis and in the fluid classification methods*. The *light scattering and diffraction methods* analyze *diffraction patterns of particles*. Diffraction methods have become increasingly important in powder metallurgy and ceramics in recent years because of *their versatility regarding measurable size range, and high speed of operation*.

The instrument, the *Coulter counter*, measures the *number of particles above a preset particle volume* which are present in a *suspension*. One can determine the particle-size distribution from the counts at different preset threshold volumes and the total particle number per unit volume of the suspension.

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Sieve Analysis

Sieving is an obvious method of powder classification and one of the *simplest* and most *widely used* in particle size analysis. Sieves are characterized by their *aperture size in micron or mm*, and are often also referred to by their *mesh number*, which is *the number of wires per linear inch*. A *high mesh number thus implies a small aperture*, for example 400 mesh corresponds to a 37 micron aperture with a wire thickness of 26.5 micron. The sieves are made of either woven wire, punched or electroformed plates. The *apertures of woven wire sieves are considered to be squares*, although they deviate somewhat from this shape, due to the three-dimensional structure of the weave. *Electroformed micromesh sieves are available with round and square apertures*. *Dry sieving is applicable to relatively coarse non-cohesive powders, fine and cohesive powders have to be analysed by wet sieving*. The *sieving operation* is either performed by *hand, or by various types of sieving machines*. In machine sieving, the normal practice is to use a *stack of sieves with an ascending order of aperture sizes*, placing the powder on the top sieve. The sieves are subjected to *simple vibration or more complicated patterns of movement*.

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• وقتی نمونه بسیار نازک باشد (که ذرات زیر میکرومتری آماده شده برای TEM اغلب نازک هستند)، بیشتر الکترون‌های پراورزی می‌توانند بدون برهم‌کنش شدید از نمونه عبور کنند. این خاصیت به عنوان “شفافیت” شناخته می‌شود. استفاده از “ولتاژ بالا” (مثلاً ۳۰۰ کیلوولت یا بیشتر) باعث افزایش قدرت نفوذ الکترون‌ها می‌شود.

Microscopy

Microscopy is one of the counting methods, in which *each individual particle is observed and measured*. In addition to *particle size*, microscopy can also give other information such as the *particle shape and the state of agglomeration*.

The problem in the use of microscopy is the *representativeness* of the *sample*, as microscopical measurements are carried out on *very small sample quantities* and the sampling method can *cause agglomeration*. *Optical as well as electron microscopy* are employed for particle size measurement. *Optical* microscopes can be used for particles *down to about 0.8 micron*. In this range of sizes the *particle dimensions reach the wavelength of light which causes scattering and diffraction*. The *reflection mode* is applicable down to *5 micron*; for *particles less than 5 micron the transmission* mode has to be used. Electron microscopy enables the measurement of particles *down to the nm range*. Sub-micrometre particles are often transparent to high voltage electrons, which enables additional information on the crystalline structure and structural defects of the particles. Clearly this approach is extremely time-consuming if statistically significant information is to be obtained. *Generally it is employed to gather an idea about the size distribution and the variation in particle size.*

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Scanning electron microscopy (*SEM*) utilizes *medium energy electrons* (5- 50keV) in a fine « 0.01 micron) beam scanning the observation area. *Secondary* (SE) and *backscattering electrons* (BE) are counted by suitable detectors. *Secondary electrons arise from the near surface regions* of the particles and give *high resolution images*. *Backscattered electrons arise from the somewhat larger volume of the particles near the electron beam, causing a slight reduction in resolution*. They are dependent upon the atomic number of the element emitting them, resulting in a *material contrast* for regions of *different atomic composition*. This makes it possible to distinguish between different compositions in powder mixtures as long as there is a sufficiently large difference in *atomic number* between the elements. Also wavelength and energy disperse X-ray techniques can give more quantitative compositional information. *Sample preparation is a difficult problem in all microscopical methods. Each particle to be measured has to be isolated from the neighbouring particles. Most preparation techniques therefore use suspensions with low concentrations of the particles*. These suspensions are applied to the microscope slide, electron microscope grid, or target stub and *allowed to evaporate*. There are *various methods used for the fixing of the particles*. TEM and SEM samples are *coated with thin carbon or metal films to prevent charging effects*. In many cases, analyses have to be made from the *projected images* of the particles. This of course *ignores internal porosity and concave parts* of the surface. A more precise technique is therefore to *examine sections from a random sectioning* of the particles. This can be done for *particles > 10 micron* by the normal metallographic grinding and polishing methods.

Powder Properties and Characteristics

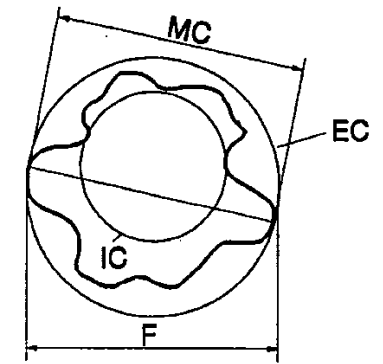
Diameters commonly used are (see Fig. 3.8):

- *Feret's* diameter, the *distance between two tangents on opposite sides of the particle*. This is a *one-dimensional projection* of the *perimeter of the particle outline*.
- *Longest dimension*, the *maximum value of Feret's* diameter.
- *Maximum chord*, the *maximum length of a line parallel to some fixed direction* and *limited by the contour of the particle*.
- Perimeter diameter, the diameter of a circle with the same circumference as the particle perimeter.
- Projected area diameter, the diameter of a circle with the same area as the described projection.
- Diameters of *inscribed* or escribed circles.

A number of definitions exist for the *particle shape factor* Ψ , e.g.:

$\Psi = \text{Perimeter} / (4\pi \cdot \text{projected area})$, or

$\Psi = \text{max. Feret's diameter} / (\text{Feret's diameter in a perpendicular direction})$



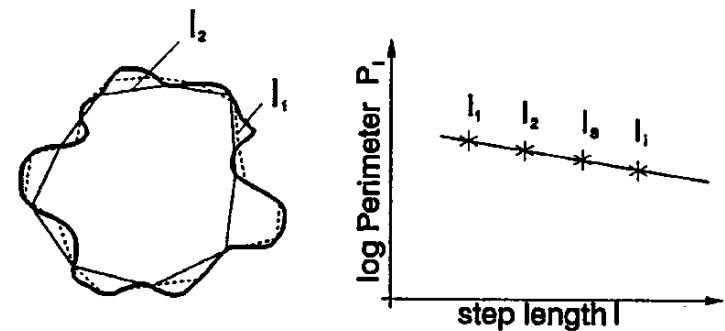
F - Feret's Diameter
MC - Maximum Chord
EC - Escribed circle
IC - Inscribed circle

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Surface roughness is another particle characteristic. Particles of the *same size and shape but different roughness* can be distinguished by their *fractal dimension*, D (see Fig. 3.8). The fractal dimension accounts for the fact that the estimated parameter P , tends to increase, as the step size l at which surface details are resolved increases. P , is given by

$$P_l = \text{const} \times l^{1-D}$$

If the *perimeter is measured by a polygonal line* arrangement with *varying step length* l , and *$\log P_l$ is plotted against l* , the results form a *straight line with the slope $1-D$* . The *steeper the line, the more rugged the outline of a particle*. An *ideally smooth surface has a fractal dimension equal to unity*, i.e. it gives a *horizontal line*.



بعد فراکتالی معیاری است که نشان می‌دهد یک شیء چقدر فضا را در ابعاد مختلف "پر" می‌کند. این بُعد معمولاً یک عدد غیر صحیح و کسری است.

Powder Properties and Characteristics

Gravitational Sedimentation Analysis

The fineness characteristic measured in sedimentation analysis is the constant free-falling or settling velocity v_f of a particle in an unbounded stationary fluid.

Three main forces act on a particle settling in a gravity field with the acceleration, g , (Fig. 3.9): the gravitational force, F_G , the drag force F_D , and the buoyancy force F_B , which are in equilibrium once the particle has reached the velocity v_f . For a sphere of diameter D and density ρ_s falling in a fluid of density ρ_f , equilibration yields

$$F_D = \frac{\pi}{6}(\rho_s - \rho_f) g D^3 \quad (3.19)$$

For Reynolds numbers $Re < 0.2$, F_D can be expressed by equation (3.20), originally derived by Stokes for the behaviour of a sphere in a laminar Newtonian fluid of the viscosity η

$$F_D = 3\pi D\eta v_{St} \quad (3.20)$$

From equations (3.19) and (3.20) the Stokes' velocity v_{St} follows as

$$v_{St} = \frac{\rho_s - \rho_f g D^2}{18\eta} \quad (3.21)$$

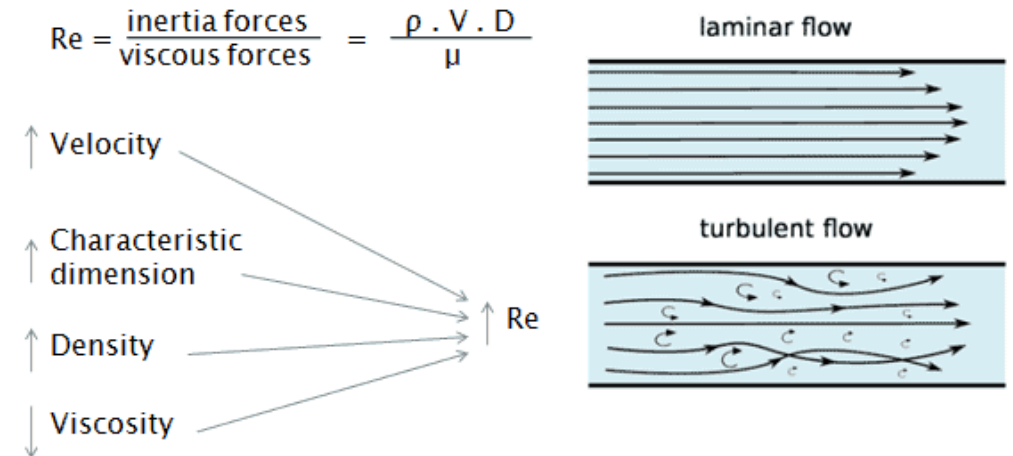
The critical diameter $D_{St,c}$ beyond which equation (3.21) should not be used is given as $Re = 0.2$. It may be expressed by:

$$D_{St,c}^3 = \frac{3.6\eta^2}{(\rho_s - \rho_f)\rho_f g} \quad (3.22)$$

The critical diameter is about $38\mu\text{m}$ for iron and $27\mu\text{m}$ for tungsten particles, respectively, settling in water. This size limitation can be extended by using more viscous liquids, e.g. glycerine/water mixtures.

عدد رینولدز (Reynolds Number)، که با Re نشان داده می‌شود، یک پارامتر بدون بُعد (بدون واحد) بسیار مهم در مکانیک سیالات است. این عدد نسبت نیروهای لختی (اینرسی) به نیروهای ویسکوز (الزجه) در یک سیال در حال جریان است.

به طور خلاصه، عدد رینولدز معیاری برای پیش‌بینی الگوی جریان سیال (آشفته‌گی یا آرامش آن) است.



جریان آرام یا laminar

$Re < 0.1$ (یا گاهی تا ۱)

• در این ناحیه، نیروهای ویسکوز غالب هستند.

• قانون استوکس (Law 'Stokes) برای محاسبه $F_d = 3\pi\mu Dv$ معتبر است.

• این ناحیه برای اندازه‌گیری ریزترین ذرات در محیط آزمایشگاهی ایده‌آل

Powder Properties and Characteristics

Light Scattering and Diffraction Methods

Sizing methods which make use of the *interaction of electromagnetic waves with the particles* have already been mentioned in connection with *x-ray and photo sedimentation*. These methods apply *radiation scattering* not directly for sizing but for the *estimation of the particle concentration in a suspension*. However, *scattered radiation also gives information on the size characteristics* and can therefore be used in direct sizing methods. Although this has been known for a long time, the practical applications of such methods, mainly based on optical radiation, has only become common during the last decade. Three modern technologies, *lasers, optic fibers and computers*, have made this development possible. The principles of these methods are based on the *interaction of an incident optical beam with a single particle*. This beam can either be *absorbed, scattered, or transmitted*. *Scattering includes reflection, refraction and diffraction*. The scattering intensity, I , in the distant field ($r \gg D$) is of the general form:

$$I = I_0 \left[\frac{\lambda}{2\pi r} \right]^2 i(\theta, \phi, \alpha, m)$$
$$\alpha = \frac{\pi D}{\lambda}$$

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$$I = I_0 \left[\frac{\lambda}{2\pi r} \right]^2 i(\theta, \phi, \alpha, m)$$

$$\alpha = \frac{\pi D}{\lambda}$$

زاویه فی (ϕ)

• زاویه‌ای است که صفحه‌ی

پراکندگی را تعیین می‌کند

• در واقع زاویه‌ای است در صفحه‌ی عمود

از دید هندسی یعنی چرخش حول

محور تابش

اهمیت فیزیکی:

در بیشتر حالت‌ها (وقتی ذرات کروی

باشند و تابش قطبیده نباشد)، شدت

پراکندگی به ϕ وابستگی ندارد

اما اگر:

• نور ورودی قطبیده باشد، یا

• ذره شکل غیرکروی داشته باشد،

آنگاه شدت ممکن است با ϕ هم تغییر

کند.

زاویه تتا (θ)

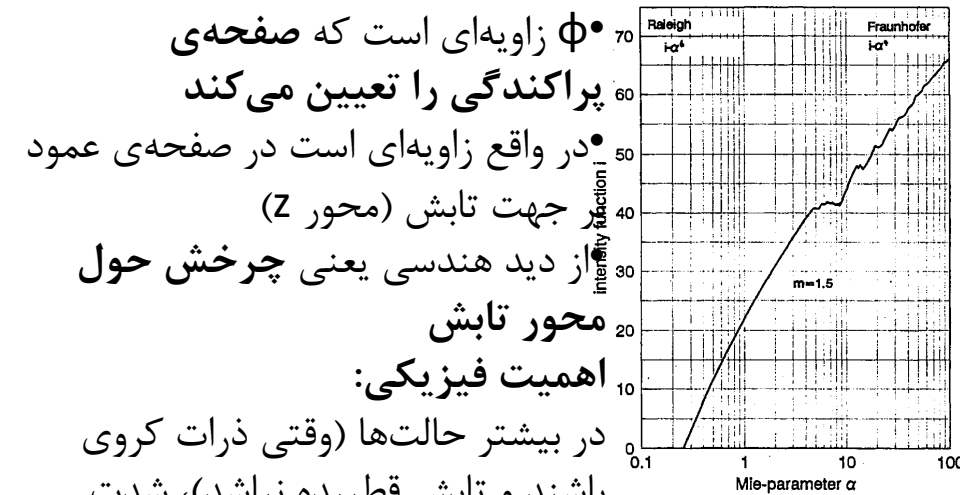
• زاویه‌ای است میان جهت تابش اولیه و جهت مشاهده‌ی پراکندگی

یعنی زاویه پراکندگی یا **Scattering Angle**

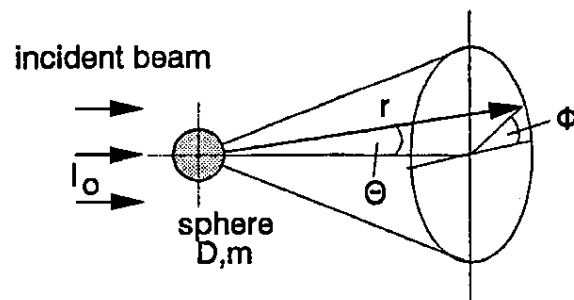
• وقتی $\theta = 0^\circ \rightarrow$ پراکندگی به جلو (Forward scattering)

• وقتی $\theta = 180^\circ \rightarrow$ پراکندگی به عقب (Backward scattering)

where I_0 is the intensity of the incident beam, λ is the wavelength of the radiation in the surrounding medium, m the index of refraction of the particle relative to the medium, θ and ϕ are the angles of observation. The so-called Mie parameter α normalizes the diameter by the wavelength. The intensity function, i , derives in a complicated manner from the variables given in equation (3.39). Figure 3.18 gives an example of the dependence of the normalized scattered intensity I/I_0 from the size parameter α for fixed observation angles ($\theta = \phi = 0$: forward direction) and a given refractive index, m . Although there is no general analytical solution relating particle size to optical measurement, special solutions exist for two limiting cases of the problem: Rayleigh scattering for smaller, and Fraunhofer diffraction for diameters essentially greater than the wavelength λ .



size range	Model	size dependency
$D \ll \lambda$	Rayleigh scattering	$i \sim \alpha^6$
$D \approx \lambda$	Lorenz-Mie theory	
$D \gg \lambda$	Fraunhofer diffraction	$i \sim \alpha^4$



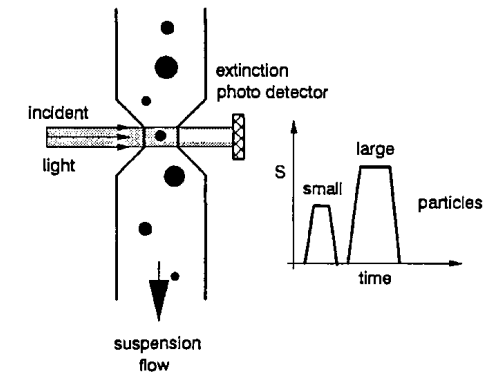
Powder Properties and Characteristics

Measurement Techniques

In powder metallurgy the most important measuring techniques based on the above principles are optical counting techniques, laser diffraction, and turbidity measurement.

The principle of optical counting methods is shown schematically in Fig. 3.22. The particles either flow individually through an illuminated sensing zone, or a measured volume of suspension is scanned by a focused laser beam. Both methods require extremely diluted suspensions. The scattered intensity or the extinction is measured by a photo-electric detector. The height of the measured impulses corresponds to the size of individual particles according to the relations in equations (3.38) to (3.41) for the single particle problem. Usually, this relation is determined empirically by calibration covering a narrow size range.

The impulse height distribution is monitored by a multi-channel analyser and converted into a size distribution on the basis of the calibration. For small particles, the refractive index is of influence and, therefore, has to be considered in the calibration, while this is not the case for large particles (see Fig. 3.21). These counting methods are applicable in the size range $> 1 \mu\text{m}$ for particles in suspensions, and $> 0.1 \mu\text{m}$ for airborne particles.



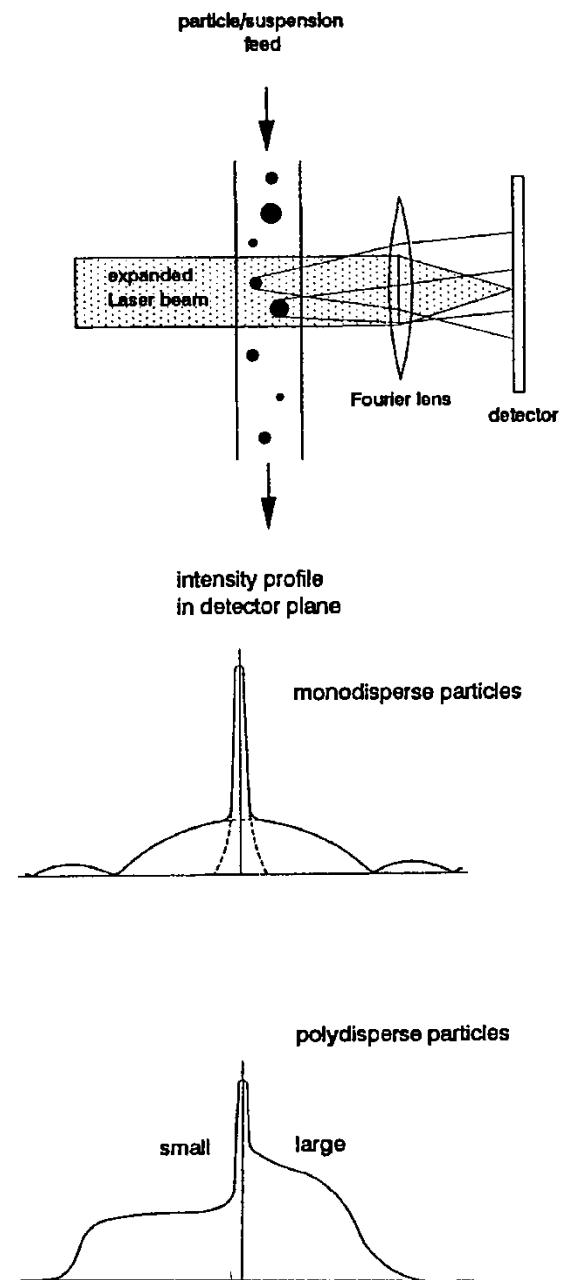
1. شدت سیگنال پراکندگی: هر ذره‌ای که از "Illuminated Sensing Zone" عبور می‌کند، یک "پالس" از نور پراکنده شده تولید می‌کند که به آشکارساز می‌رسد.

• دامنه (Height) این پالس مستقیماً متناسب با شدت نور پراکنده شده است.
• قانون پراکندگی مای (Mie): می‌دانیم که شدت نور پراکنده شده به اندازه (D) و جنس (m) ذره بستگی دارد.

2. ارتباط با اندازه ذره: در بسیاری از روش‌ها، رابطه مستقیمی بین Impulse Height (دامنه پالس) و قطر (D) ذره وجود دارد. ذرات بزرگتر معمولاً پالس‌های با ارتفاع (دامنه) بیشتری تولید می‌کنند (البته این رابطه پیچیده است و به زاویه پراکندگی هم بستگی دارد).

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Laser diffraction has become by far the most important optical sizing method for metal and ceramic powders. As diffraction involves rays external to the particle, the intensity of the diffracted light is not dependent on properties of the materials (see section 3.2.4.6.1, equation (3.44)). Therefore, mixtures of powders of different materials can be measured without difficulty — which cannot be done with many other sizing methods. A diagram of the laser diffraction method is given in Fig. 3.23. The measuring zone is illuminated by an expanded laser beam. The particles are fed through the measuring zone in a liquid or gas flow, by simple powder pouring or by stirring of a suspension in a closed measuring vessel. The diffracted and transmitted light are focused by a Fourier transformation lens on to the detector in the focal plane of the lens. By this optical arrangement, the diffraction pattern from a given particle within the detector plane becomes independent from the particle position in the measuring vessel, i.e. from particle movement.



Powder Properties and Characteristics

3.3.1 SURFACE ANALYSIS BY PERMEAMETRY

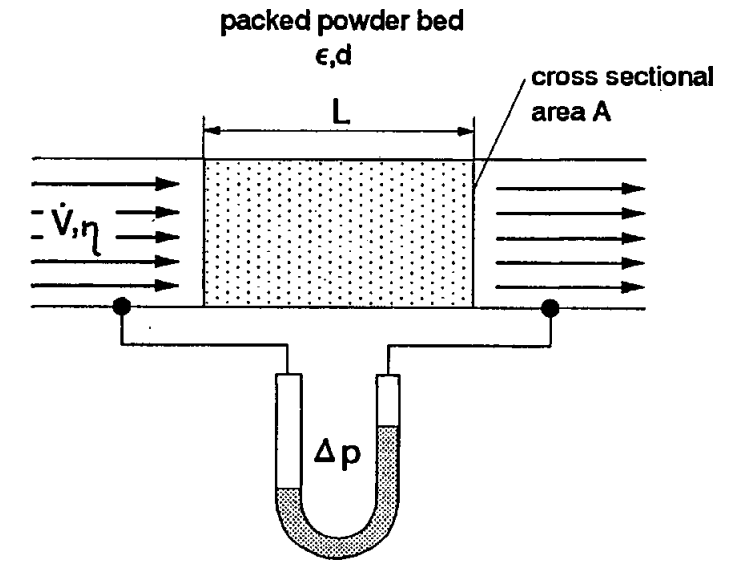
Permeametry is based on the flow of a viscous fluid through a closely packed bed of powder (Fig. 3.24). The pressure drop, Δp , increases with decreasing size of the pore channels in the powder bed. Two different types of flow have to be considered: viscous flow and molecular flow. Viscous flow is characterised by a mean free path, λ , of the fluid molecules, which is small compared with the pore size, d . The fluid-wall interaction is concentrated in a thin layer, and Δp is determined by the molecule-molecule interaction, or from this the fluid viscosity, η . The latter becomes negligible when the mean free path is large compared with pore size. This is the range of Knudsen diffusion, which is controlled by the mean molecular velocity and therefore depends on temperature, T , and molecular weight, M . With air as the measuring fluid, viscous flow is

dominant at $d \geq 10 \mu\text{m}$ at atmospheric pressure. Knudsen diffusion dominates at $d \leq 10 \mu\text{m}$ at a pressure of 100 Pa.

Assuming viscous flow, the surface/volume ratio S_v can be derived by the Carman-Kozeny equation (see Fig. 3.24):

$$S_v^2 = \frac{1}{k} \frac{\epsilon}{(1 - \epsilon)} \frac{1}{\eta} \frac{A}{L} \frac{\Delta p}{\dot{V}} \quad (3.51)$$

where ϵ is the porosity of the powder bed.



aspect factor, k , depends upon the size and shape distribution of the powder flow rate \dot{V} .

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3.3.2 SURFACE ANALYSIS BY GAS ADSORPTION

When a surface of a solid (*adsorbent*) is exposed to a gas (*adsorbate*), gas molecules are *adsorbed* for a finite time, after which they become *desorbed* and are replaced by other molecules. Depending on the interacting forces, adsorption is classified as *physisorption* (van der Waals forces), or *chemisorption* (chemical bonding). Physisorption may be compared to condensation of a vapour to a liquid and is a completely reversible process. It takes place at low temperatures and becomes significant below the critical temperature T_{crit} of the adsorbate. The equilibrium amount adsorbed (volume V) depends on the pressure, p , at which adsorption takes place, and on the nature of the adsorbate and adsorbent. Usually, this pressure is normalized by the saturated vapour pressure p_0 at the given temperature. The graph of V vs. p/p_0 can be measured and is called the *adsorption isotherm*. Adsorption theories deduce the monolayer capacity V_m from this isotherm, defined as the quantity of adsorbate needed to cover the surface with a molecular monolayer. The total surface of the adsorbent is then derived by dividing V_m by the area occupied by one molecule, σ .

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The first theoretical approach relating V and p was the Langmuir equation, based on the assumption that adsorption is completed after a monolayer is formed. This assumption holds only for chemisorption, where bonding is limited to the gas molecules directly at the solid surface. In physisorption, second and further layers can be formed before a monolayer is completed. This is expressed by the *BET theory* (Brunauer, Emmet and Teller), which is derived by applying the Langmuir equation to subsequent layers and thus ending with a system of coupled equations. The solution results in the BET equation (3.53).

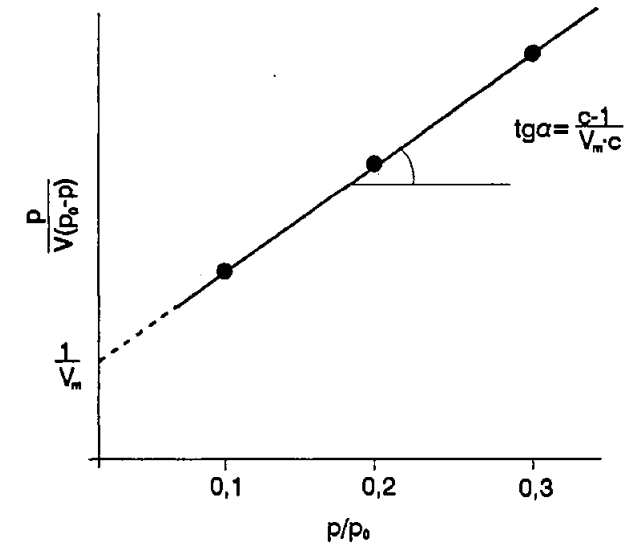
$$\frac{p}{V(p_0 - p)} = \frac{1}{V_m c} + \frac{c - 1}{V_m c} \frac{p}{p_0} \quad (3.53)$$

I II III

where c is a constant, related to the heat of adsorption. A plot of term I vs. p/p_0 should yield a straight line with an intercept and slope according to term II and term III (Fig. 3.25). The amount of an assumed complete monolayer can thus be calculated from the measured adsorption isotherm. As $c \gg 1$ in most systems, equation (3.53) can be approximated by

$$V_m = V(1 - \frac{p}{p_0}) \quad (3.54)$$

V_m thus can be deduced from only a single measured point of the adsorption isotherm.

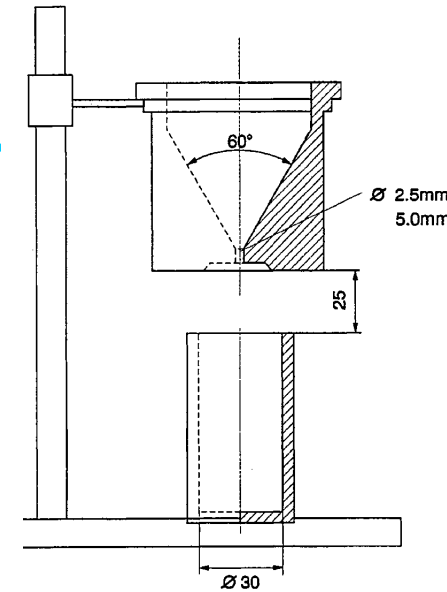


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3.4.1 POWDER FLOWABILITY

Flowability can be characterised in a simple way by the so called *Hall flowmeter test*, shown in Fig. 3.27. The measured parameter is the time required for 50 g of a powder to leave the flowmeter under the influence of gravity. The flow time depends in a complex manner on the internal friction between the powder particles, the friction between the powder and the funnel walls, the relationship between the funnel orifice and particle size, the density of the particles, and the funnel geometry. Obviously this test is only applicable for a comparative characterisation of free-flowing (non-adhesive) powders.

Cohesive powders can be treated according to the principles of flowability of bulk powders, which range between those of liquids and solids. Static liquids under gravity form a horizontal surface with hydrostatic pressure increasing linearly with the distance from the surface. Shear stresses cannot be transferred,

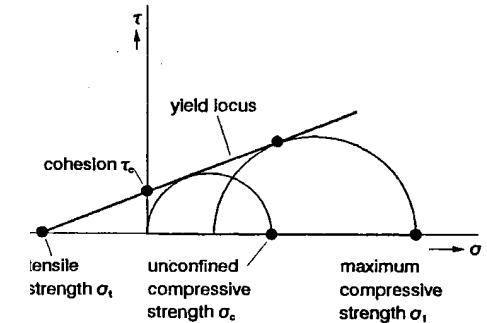


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e.g., even the lowest shear component causes movement (flow) of a liquid. The surface of bulk powders can be inclined to the horizontal to any desired amount within the so-called angle of repose. The pressure within a powder column does not increase in a linear manner with depth, but goes through a maximum. On the other hand, bulk powders can only transfer minimal stress and consequently behave quite differently from solids. A general description of the mechanical properties of a bulk powder is given by its *yield locus* (see Fig. 3.28), which provides information on the flow behaviour of a volume element of the bulk powder under a given state of stress. This is expressed in a Mohr's diagram, and shows the relation between compressed stresses σ and shear stresses τ necessary to cause the powder to flow. No flow is possible below the yield locus but a stress state at the yield locus causes flow; stress states beyond the yield locus cannot occur within the powder. Usually, a linear approximation is assumed, according to

$$\tau = \tau_c + \sigma \tan \phi \quad (3.55)$$

τ_c represents the *cohesion* and ϕ the *angle of internal friction* of the powder. With increasing compressive stress, the yield locus comes to an end point beyond which compaction of the powder begins. In Fig. 3.28, this is the case for stresses



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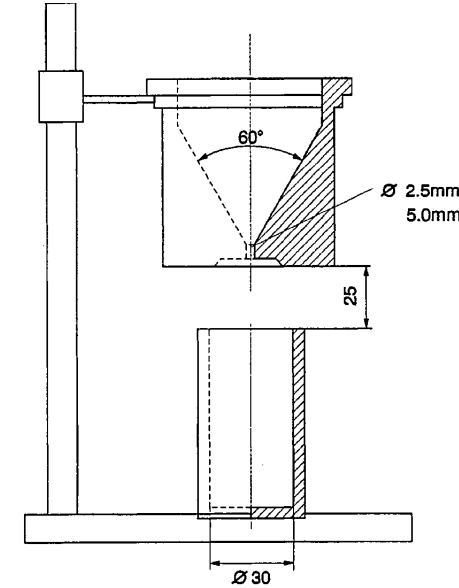
according to the Mohr's circle with the maximum compressive stress σ_1 . The smaller circle represents the unconfined compressive strength σ_c . The powder behaviour can now be expressed in terms of τ_c and ϕ . Limiting cases are given in Table 3.3.

The cohesiveness of a powder can be expressed by the parameter σ_1/σ_c which is > 4 for cohesive and > 10 for (practically) free-flowing powders. Fluidised powders behave like liquids. This state can be reached with fine powders mixed with sufficient air to eliminate interparticle friction. The yield loci can be determined experimentally by various types of *shear cell apparatus* (see Fig. 3.28) in which a sample is sheared under increasing pressure. Automatic flowability testers based on this principle are available and have been proposed for powder metallurgy applications.

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3.4.2 APPARENT AND TAP DENSITY

The apparent density of a bulk powder is defined as the powder mass divided by the bulk powder volume. The latter is related to the particle packing, which is affected by the mode of filling, the container size, vibration during packing etc. These factors have to be kept constant. Standard test procedures are based on the Hall flowmeter (funnel method), (Fig. 3.27) or the Scott volumeter. With both methods, a cylindrical container of constant volume (25 cm³) is completely filled with the powder. The measured mass of the powder is then divided by the container volume, yielding the apparent density. The funnel method is applied for powders which are free flowing through a Hall funnel with a 2.5 or 5.0 mm orifice. Cohesive powders are tested preferably with a horizontally oscillating funnel with an orifice of 7.5 mm. The Scott apparatus has a relatively large four sided orifice of 12.5 mm and is recommended for cohesive powders, which would change their properties under vibration.



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The *tap density* is the apparent density of a powder packed vertically by vibration. The powder is poured into a calibrated glass measuring cylinder, which is tapped manually against a rubber plate, or by a standard tapping apparatus at a frequency of 1.7 to 5 Hz and an amplitude of 3 mm. Tapping is continued until no further densification is visible.

The variables which control powder packing are the particle characteristics (size distribution, shape, mass, resilience (i.e. the ability to resist conversion of kinetic energy to other forms of energy on impact), interparticle friction etc.), the container (dimensions, wall friction), deposition parameters (intensity of tapping, particle velocity), and the treatment after deposition (vibration).

With monosize spherical particles, the maximum packing density theoretically achievable is 74% (by close packing with a coordination number of 12). Random arrangements of monosize spheres reach 63% in dense and 60% in loose random packing. The packing density can be increased by the addition of much smaller spheres filling the interstices between the larger ones. The maximum packing density in close packing of binary mixtures is 86%, with about 73% of coarse spheres in the mixture. A 95% packing density is the limit in a ternary mixture with an optimum volume ratio of 66:25:9 for coarse, medium and fine components respectively, if the diameter ratios are large.

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With an increasing number of different sized particles, even higher values should be possible. The limiting densities can only be reached when all the particles fall into an ideal position. The vibration time required for such an arrangement is proportional to the number of particles, which is about 1:500:70,000 in the ternary mixture considered. Thus the time for ideal mixing will increase by a factor of 140 from the binary to the ternary ideal packing. It has also to be realised, that the size relation of the smallest to the largest particle is limited in practice, thus limiting the number of component particles in multi-size systems. Up to a mixture size ratio of 10, (a typical value in powder metallurgy) a binary system allows for a higher theoretical packing density than systems with more component particles. In general, packing density increases with the range of the particle size distribution and this tendency is more pronounced in densely packed arrangements than in loosely packed ones.

The packing efficiency decreases with increasing deviation from sphericity and decreasing particle size. The latter effect is at least partially due to increasing interparticle friction. Particle mass and resilience (for definition, see above) also play a role during particle deposition. Steel particles, for example, pack

• هر پودری که در یک ظرف ریخته می‌شود، پس از تکان دادن یا لرزش (تپینگ)، در نهایت به یک چگالی ماکزیمم (یا حدی) می‌رسد که در آن، دیگر ذرات فضای کمتری برای حرکت و قرارگیری بهینه‌تر پیدا نمی‌کنند. این حالت نشان‌دهنده حداکثر فشردگی ممکن تحت شرایط ارتعاش مشخص است.

• شرط رسیدن :این چگالی حدی تنها زمانی قابل دستیابی است که همه ذرات در یک "موقعیت ایده‌آل (ideal position)" قرار گیرند. منظور از "موقعیت ایده‌آل"، آرایشی است که در آن فضاهای خالی (Pores) بین ذرات به حداقل ممکن رسیده باشد، یعنی بیشترین تعداد ذرات ممکن در کمترین حجم ممکن جای گرفته باشند.

Powder Properties and Characteristics

more closely than lead shot under the same deposition conditions, due to their higher resilience. The relevant deposition parameters are the kinetic energy of the particles and the intensity of deposition (particles/second \times area). Packing density increases with kinetic energy, but decreases when the intensity of deposition exceeds a critical value.

Near the container walls, the packing density is reduced by disorder in the particle arrangement. The critical range is within a distance of 10–50 particle diameters from the wall, as can be demonstrated from mono-size sphere experiments. This has to be taken into account, for example, in tool design for components with thin cross sections.

3.6 STANDARDS

Terminology, test procedures and evaluation of results in powder characterisation are standardized in national and international standards, including ASTM (American), BS (British), AFNOR (French), DIN (German), and ISO (International). Table 3.4 lists ISO standards, in accordance with the sequence of subjects in this chapter.

ذرات فولاد به دلیل سختی و الاستیسیته بالاتر، هنگام برخورد با ذرات دیگر یا بستر، انرژی جنبشی خود را عمدتاً به صورت ارتعاش برمی‌گردانند تا تغییر شکل دائمی (پلاستیک) یا تولید گرمای شدید. این خاصیت باعث می‌شود که ذرات در اثر ضربه “جهش” کرده و در فضاهاى خالی بهینه‌تر مستقر شوند، در حالی که ذرات سربی (که نرم‌تر هستند) انرژی ضربه را بیشتر به صورت تغییر شکل پلاستیک (له شدن یا چسبیدن) جذب کرده و در نتیجه در آرایش‌های کمتر متراکم گیر می‌کنند.

Compaction and Shaping

6.1 PRESSURE-ASSISTED SHAPING

The pressure-assisted forming operations can be subdivided into cold and hot compaction methods. From the material's point of view, cold compaction takes place in a temperature range within which high temperature deformation mechanisms like dislocation or diffusional creep can be neglected. In most practical cases, cold compaction occurs at ambient temperature, while hot compaction is carried out at enhanced temperatures. Low melting materials like lead, however, undergo hot compaction even at room temperature. Processes, where powders are dispersed in a low melting carrier medium, e.g., injection moulding, could be associated with both cold and hot compaction.

مفهوم محیط حامل با نقطه ذوب پایین

این تکنیک، که اغلب در فرآیندهایی مانند قالب‌گیری تزریقی پودر (Powder Injection Moulding - PIM) استفاده می‌شود، شامل ترکیب پودر مورد نظر (مانند فلزات، سرامیک‌ها) با یک ماده "حامل" است که:

• **نقطه ذوب پایین دارد:** این ماده حامل معمولاً یک پلیمر یا موم است که در دمای نسبتاً پایین ذوب می‌شود.

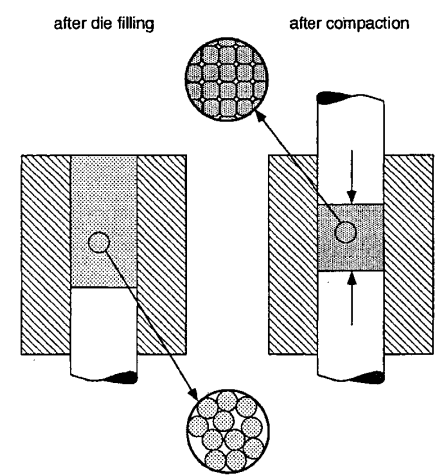
• **عملکرد:** این محیط حامل به عنوان یک "روان‌کننده" و "چسب" عمل می‌کند و به پودر اجازه می‌دهد تا به صورت یک مخلوط همگن (Slurry یا Feedstock) درآید. این کار ویسکوزیته مخلوط را برای عملیاتی مانند تزریق (Injection) تنظیم می‌کند.

Compaction and Shaping

6.1.1 COLD COMPACTION

6.1.1.1 Fundamental Aspects

In most applications, high densities of the green compacts are desired. Higher compact densities usually result in better green strength properties and smaller dimensional changes during sintering to the final density level, which exceeds 85–90% of the theoretical density of all powder metallurgy products, except products with functional porosity, such as filters and bearings. In a large variety of materials, even full density has to be achieved to meet the desired properties. This density level is well beyond the apparent density of normal bulk powders, which is controlled by the particle arrangement after feeding the powder into a cavity (Fig. 6.1). The apparent density is usually much lower than in a random, dense particle arrangement (see section 3.4.2), as packing is hindered by particle bridging. Densification is therefore principally possible solely by rearrangement of the particles without changing their shape until a random dense packing is reached. Further densification definitely requires a shape change by plastic deformation or fragmentation of the particles.



• **پل‌بندی چیست؟** این پدیده زمانی رخ می‌دهد که ذرات پودر (به ویژه ذرات با اندازه‌های متوسط یا درشت) هنگام ریختن در یک ظرف، به جای نشستن در حفره‌های کوچک موجود، بر روی یکدیگر در فضاهای بزرگتر قرار گرفته و مانند دهانه یک پل، یک ساختار قفسی موقت ایجاد می‌کنند.

• **نتیجه:** این ساختارهای پل‌بندی شده، فضاهای خالی (Voids) زیادی را در توده پودر به دام می‌اندازند و اجازه نمی‌دهند که ذرات به طور کامل در کنار یکدیگر فشرده شوند، در نتیجه چگالی ظاهری (Bulk Density) به شدت کاهش می‌یابد.

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Bridging can be overcome either by the application of a load to the particle contacts or by the reduction of the frictional forces in the particle–particle and particle–cavity wall contacts, which is possible by lubrication, e.g. through an interfacial liquid. The volume fraction of pore fluid is an important characteristic of such particle–liquid systems. It is characterised by the degree of pore saturation (DPS) which is defined as the fractional volume of pores filled by the liquid. Figure 6.2 represents the limiting cases. In pressure assisted compaction and shaping of powder metallurgy parts, the cases of $DPS \approx 0$ (bulk powders with only very small amounts of lubricants), and $DPS \geq 1$ (saturated state, e.g. injection moulding feedstocks) are of relevance.

With increasing volume fraction of the liquid, the consistency of the powder system changes from bulky over plastic and paste-like to slurry. An externally applied pressure contributes in a different manner to the state of stress in the liquid and the particle skeleton. Shear stresses can be resisted by the skeleton only. The normal stress is divided between the liquid and the skeleton, depending on their volume fractions.

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In saturated suspensions the applied pressure is carried only by the liquid. No densification is possible until the liquid is allowed to drain. The shear components of the stress result in deformation (flow) and allow for shaping of the compact and for a particle arrangement close to the random dense packing. Material flow takes place, as long as loading results in a shear stress component.

Bulk powders exhibit a compressible behaviour. When a pressure is applied to the loose powder filling, some of the bridging particles enter a different state of aggregation. With increasing pressure, the fraction of large voids or packing defects is reduced by such restacking events. However, the strength of remaining particle bridges increases, as the particle contacts become flattened by elastic and plastic deformation, and the frictional forces are increased by cold welding and interlocking of rough particle surfaces. Thus, rearrangement and shape change of particles occur simultaneously and are interacting mechanisms. The dominance of rearrangement is limited to the beginning of densification, depending on the apparent density, particle shape, size, and deformability. Its contribution to total densification increases with increasing rigidity of the particles. It is in the order of 10% for typical metal powders, while it makes the major contribution in compaction of fine ceramic powders. The remaining interstices between the particles can be filled by plastic flow of material into the voids, or by fragmentation of the particles, allowing void filling by a secondary type of rearrangement. The required compaction pressure increases steadily with density, resulting in a state of equilibrium between external loading and the compact compliance.

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6.1.1.2 Cold Pressing

6.1.1.2.1 Principal Aspects

Cold pressing is the most important compaction method in powder metallurgy. It starts from bulk powders containing very small amounts and sometimes even no lubricant or binder additions. One usually distinguishes between axial (die) and isostatic pressing (Fig. 6.3). In axial pressing, the powder is compacted in rigid dies by axially loaded punches. The axial compaction pressure P_{ax} which is defined as the punch load divided by the punch face area is the main process variable. In isostatic pressing, the powder is sealed in an elastic mould and exerted to the hydrostatic pressure P of a liquid pressure medium.

The compaction behaviour of powders, expressed by their overall pressure-density relations is shown in Fig. 6.4. The controlling parameters are mainly the particle size and the ability for plastic deformation. Densification starts from the apparent density, which is similar for the coarse iron and alumina powder, and which is not too far away from random dense packing for both of them. The fine powders exhibit a significantly lower starting density, due to hindered packing. With increasing compaction pressure, the average density of the compacts increases. The slope of the curves differs significantly for the ductile metal powder and the non ductile alumina. The density increase for a given pressure increment is always higher for the ductile material. In all cases, the curves asymptotically approach a final density level, which is below the theoretical density of the materials. The density difference between the fine and the

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corresponding coarse powder is not reduced with increasing pressure. Much higher pressures are necessary for the fine powders to reach the same density as the coarse ones. Figure 6.4 exactly represents the microscopic situation: the brittle alumina powder can be compacted only to a density relative to that of its random dense packing, while the maximum densities of the ductile iron powder are much higher, which can only be explained by extensive filling of interparticle voids through plastic deformation. Interparticle friction and bridging effects increase with decreasing particle size. With increasing density, the resistance to densification increases rapidly.

It is common, to correlate this behaviour with a three-stage compaction process with different controlling mechanisms. These are: particle rearrangement in the first, elastic-plastic deformation in the second, and work hardening (ductile powders) or fragmentation (brittle powders) in the final stage. As already explained, these stages are overlapping with strong interactions between the different mechanisms.