

Comminution

The process of physically dividing solids into fine pieces by grinding in a mill is the best-known method for the manufacture of powders. However, there are problems associated with grinding, such as the contamination of the particle surface by atmospheric gases and by materials used in the construction of the mill. The smallest particle that can be produced by grinding is also limited by a tendency of the ground product to re-aggregate. The size, shape, size distribution and grinding limit of fine particles produced by mechanical reduction techniques are very dependent on the conditions of grinding and the type of mill used.

Mechanisms of Grinding

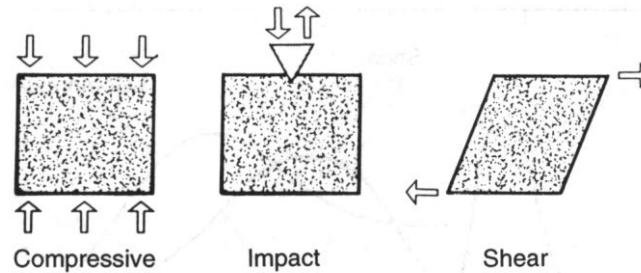


Figure 4.1 The most common forces in grinding.

It is possible to imagine many types of force (such as tension, bending, compression, torsion, impact and shear) occurring when solids are ground, but the most common forces to which particles would be subjected are compression, impact and shear, as shown in Fig. 4.1. The properties of ground substances, such as the size, shape and size distribution of the particles, depend on the nature of the material being ground. These properties are also sensitive to the action of the mill, which determines the kinds of stresses being applied, even when the same substance is being ground. The general effects of the three main types of stress that occur during grinding are shown in Fig. 4.2.

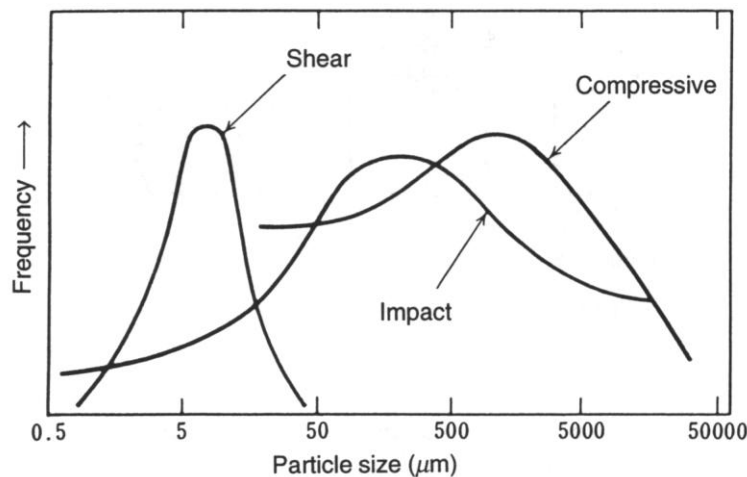
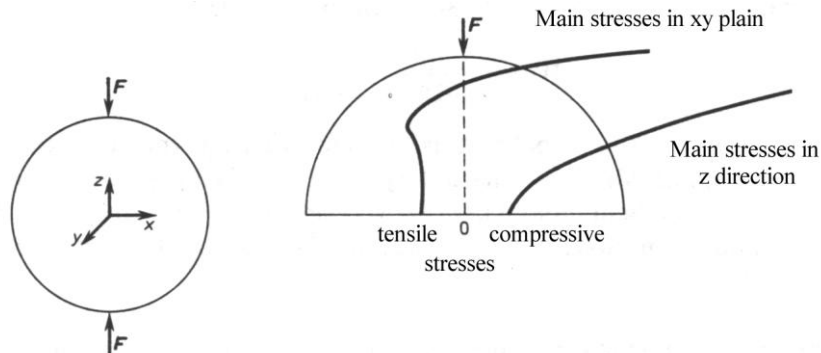


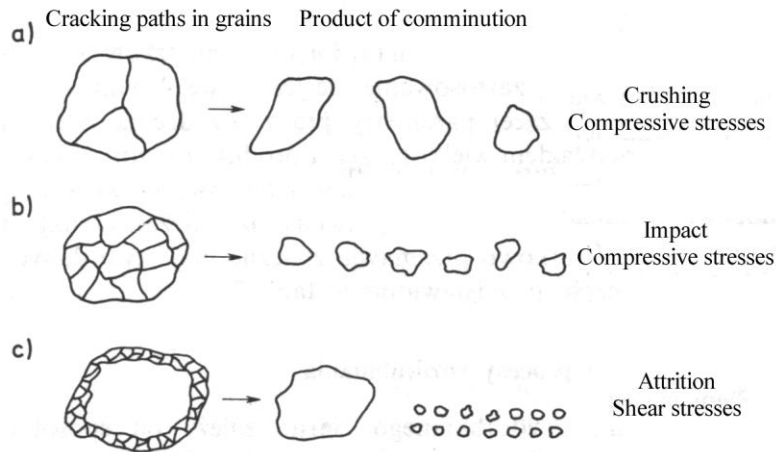
Figure 4.2 Size distribution of ground particles due to different grinding actions.

Finely ground particles with a narrow size distribution are produced when the applied stresses are mainly due to shear. It is difficult to produce fine particles by grinding when only compressive stresses are used, and the width of the size distribution obtained tends to be broadened. Impact grinding gives results intermediate in character between those produced by the application of shear and compressive stresses. The application of shear

stress by grinding is the most effective way of producing fine particles and commonly causes the physicochemical properties of the ground particles to be changed due to the effects of shear on the crystalline lattice structure.



Rys. 3.29. Main stresses distribution in the spherical grain subjected to a load F .



Rys. 3.30. Elementary processes of comminution

Comminution Equipment

Schematic diagrams of typical industrial crushers and grinding mills are illustrated in Fig. 4.3. Jaw and gyratory crushers mainly apply compressive stresses. These machines are generally used for the rough crushing of minerals or other materials that tend to cleave easily. Roll crushers and edge runners are widely used for both the grinding and mixing of clays and ferrites. Ball mills, which apply impact forces between the moving grinding media, and hammer mills, which apply impact forces by the use of moving hammers, are other kinds of mill. The mortar is typical of a mill using mainly shear stresses. However, no one kind of mill can apply only one type of stress because practical mills are designed for the effective grinding of solids by the use of a combination of stresses. The grinding mechanisms of mills and crushers are shown in Fig. 4.3, and they are classified according to the stresses involved in Table 4.1. Types (a) and (b) are used to reduce solids from sizes as large as 100 mm to sizes below 40 mm; feed sizes up to 2–3 m can be handled in large-scale equipment with corresponding increase of the product size. Types (f) and (g) are used to reduce solids of 3–10 mm size to below 150 μm . Types (c), (d), (e) and (h) are used as intermediate mills to grind solids from 6–50 mm down to 3–10 mm. The ratio of the feed/product particle sizes is respectively 3–4 in crushers, 5–10 in intermediate grinders and 20–50 in fine grinders.

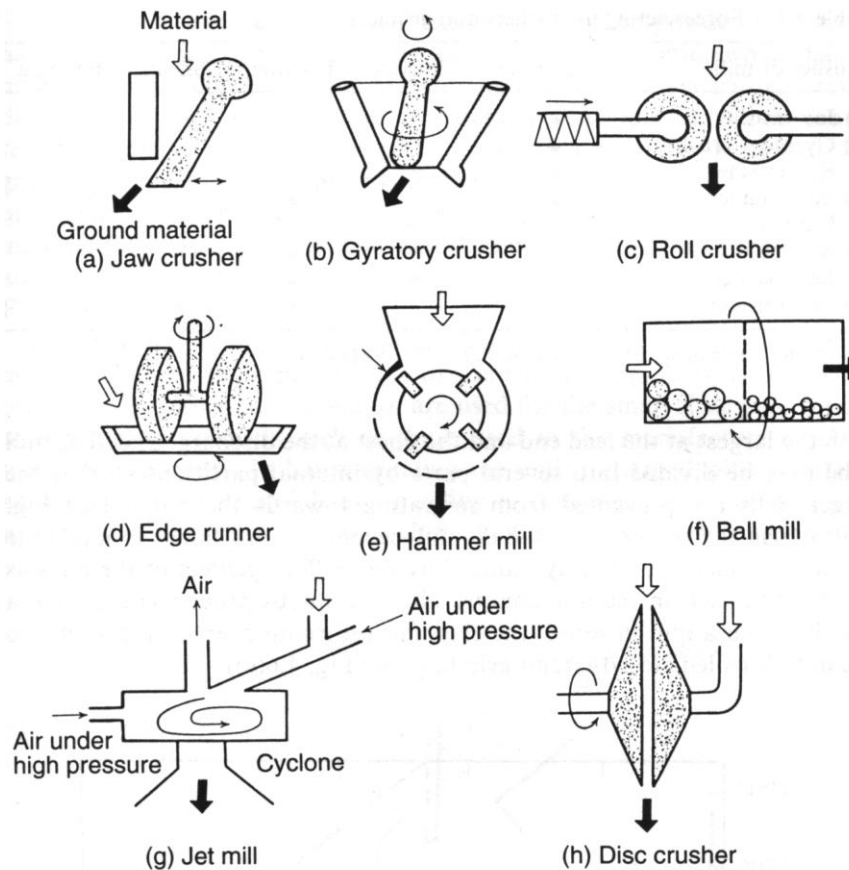


Figure 4.3 Typical crushers and grinders.

Table 4.1 Forces acting in crushers and grinders

Crusher or mill	Compressive	Impact	Friction	Shear	Bending
(a) Jaw crusher	•				
(b) Gyratory crusher	•				•
(c) Roll crusher	•			•	
(d) Edge runner	•		•	•	
(e) Hammer mill		•			
(f) Ball mill		•	•		
(g) Jet mill		•	•		
(h) Disc crusher			•	•	

The fine powdered materials needed for the manufacture of ceramics are mainly produced by grinding the raw materials, and many types of mill have been designed to meet this need. Examples are the vibratory mill, which is a vibrating ball mill, and jet mills (micronizers or fluid energy mills), which grind by using jets of high-pressure air or steam to cause collisions between the particles being ground. The jet mill (Fig. 4.3(g)), which mainly creates impact forces, is very effective in making the ultra-fine, needle-like, 0.5–1 μm long ferrite powders that are used in the manufacture of magnetic tapes. The more common types of mill have a tendency to produce spherical, aggregated products. The disc crusher (Fig. 4.3(h)), colloid mill and sand grinder, which apply a frictional force between two rolling discs, are also used for grinding ferrite.

Tube mills are used widely in cement manufacture and consist of long slightly conical cylinders, like an elongated ball mill, with a charge of balls that are graduated in size. During grinding, these balls arrange themselves with the largest at the feed end and the finest at the discharge end. The mill tube may be divided into several parts by internal partitions so that the larger balls are prevented from migrating towards the outlet (see Fig. 4.3(f)). As the material in a ball mill becomes more finely ground, the grinding efficiency is rapidly reduced by the buffering effect of the fines as the balls impact on the mill charge. The continuous process comprising a classifier and a mill in series, the classifier returning over-sized product to the mill, is called closed-circuit grinding (see Fig. 4.6(c)).

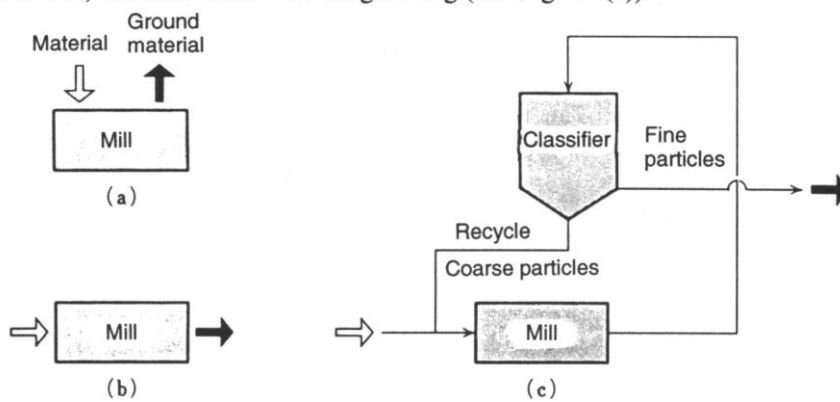


Figure 4.6 Grinding systems for a ball mill: (a) batch; (b) continuous; (c) continuous, closed circuit.

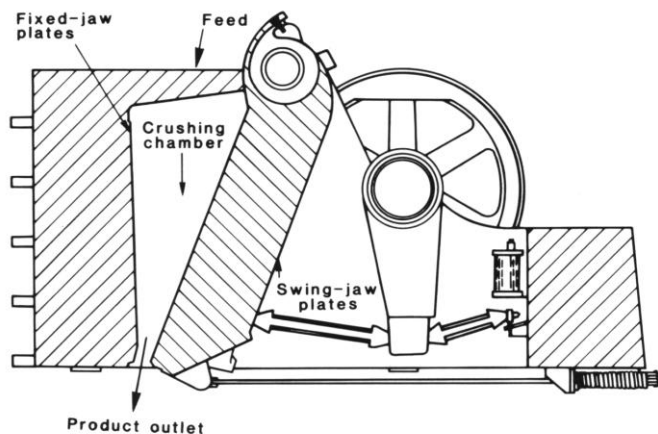
Continuous grinding, as shown in Figs 4.6(b) and (c), is needed in mass-production environments like the cement industry, whilst batch-wise processes, as shown in Fig. 4.6(a), are used for the small-scale production of high-purity products such as those used in the manufacture of electronics components. In batch grinding the mill is charged with a certain amount of a raw material and grinding is continued until the required product size is reached. Although there remains the problem of separating unground feed from product, batch grinding is a better method of producing ultra-fine powders without an undue loss of grinding efficiency.

Principles of Operation

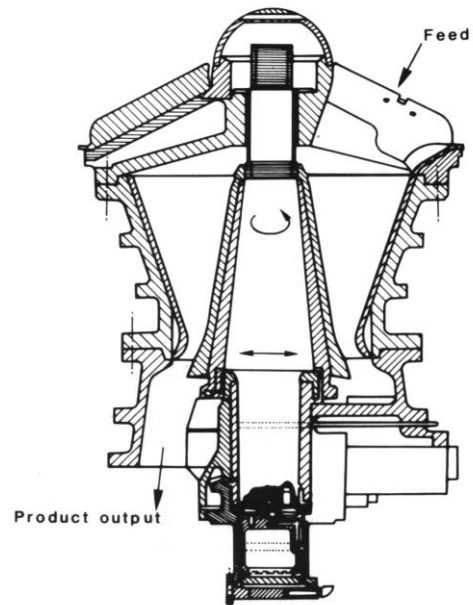
TABLE 4.1 Types of Size-Reduction Equipment

Jaw crushers (continuous)	Stirred media mills (batch and continuous)
Gyratory crushers (continuous)	Stirred ball mills
Heavy-duty impact mills (continuous)	Stirred sand mills
Rotor breakers	Vibratory media mills (batch and continuous)
Hammer mills	Fluid shear mills (batch and continuous)
Cage impactors	Colloid mills
Roll crushers and shredders (continuous)	Microatomizer
Tumbling media mills (batch and continuous)	Fluid impact mills
Ball mills	Opposed jet
Rod mills	Jet with anvil
Autogeneous	Centrifugal jet

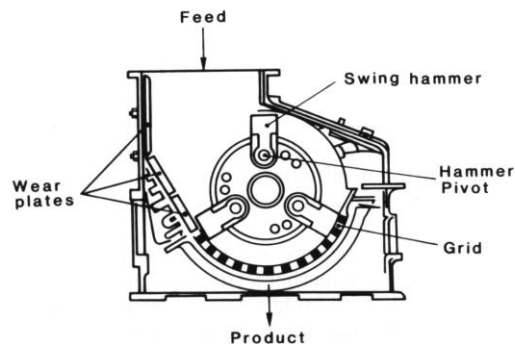
Crushing Equipment



Jaw crusher



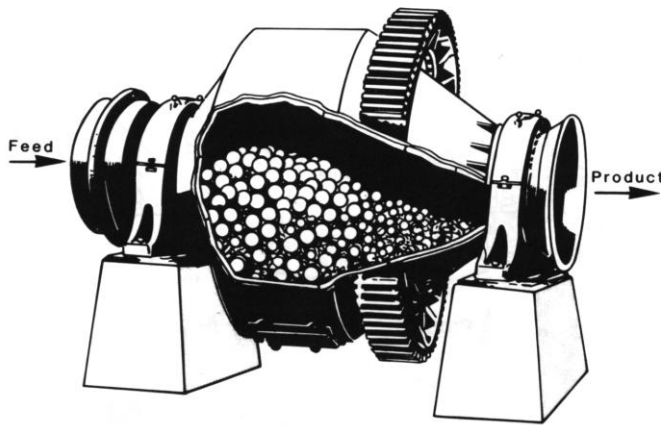
Gyratory crusher



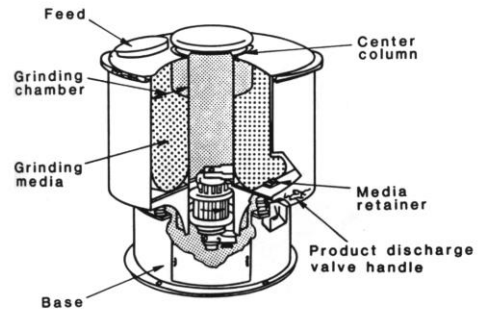
Hammer mill

The jaw crusher has an articulating arm that moves the jaws back and forth, crushing material to a size small enough to fall from the bottom of the jaws. This type of crusher can be as large as 3 m across, producing powders of 5 cm at the outlet, or as small as 10 cm across, producing powders of 1 mm at the outlet. It is commonly used in large-scale mining operations. A more efficient grinding mill for the same duty is that of a gyratory crusher. The gyratory crusher can also be as large as 3 m in diameter, producing material of 5 cm in diameter at the outlet. It consists of a cone-shaped pedestal, oscillating within a larger cone-shaped bowl. The angles of the cone are set such that the width of the passage decreases toward the bottom of the working faces. The pedestal consists of a mandrel that is free to turn on its spindle. The spindle is driven from an eccentric bearing below. Differential motion causing attrition can occur only when pieces are caught simultaneously at the top and bottom of the passage due to the different radii at these points. Gyratory crushers generally require about one-fourth of the energy input per ton of material for grinding similar to that of the jaw crusher [4]. For smaller particle sizes, the hammer mill is often used. Here the material is impacted with a heavy duty hammer mounted on a horizontal shaft. Material is forced at high speed into the breaker plate and then falls through a grating at the bottom of the device. Hammer mills often clog and as a result are designed so as the motor can be reversed to unclog them.

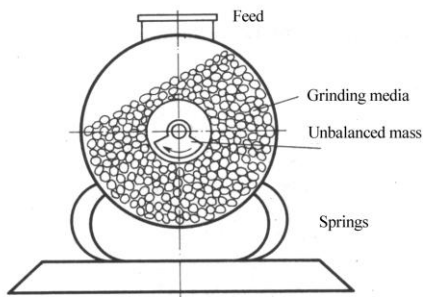
Grinding Equipment



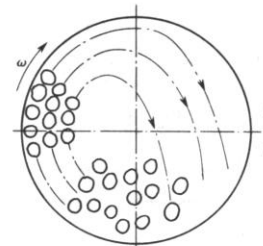
Ball mill



Vibratory mill

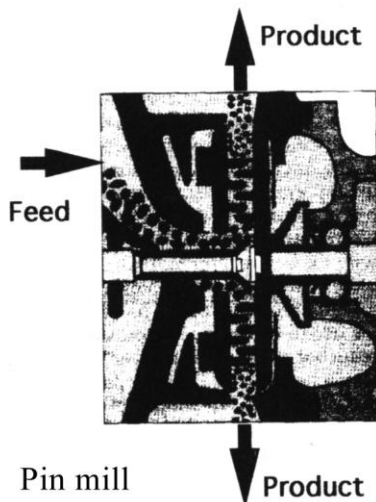


Vibratory tumbling mill

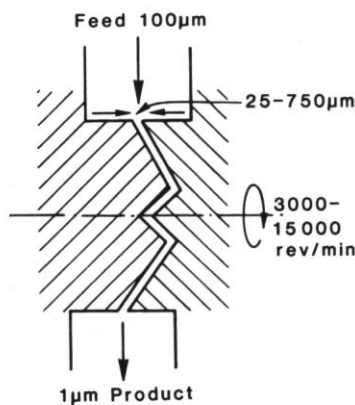


Movement of grinding media in a rotary-vibratory mill

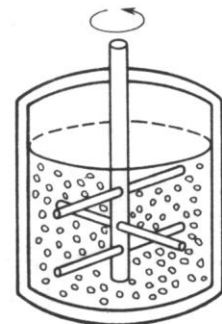
By far the most common mill for fine particle sizes is a ball mill. Here material is continuously fed to grinding media that are in constant motion, tumbling one over another. The impact of balls (media) against each other grinds the material trapped between them. After the material has been in the mill for a certain period of time, it is flushed out of the system by a flowing over a central part of the cone at the exit of the mill. The balls, which are generally larger, are retained inside the mill by a grate. Other media mills use the same comminution concept as the ball mill (i.e., collision of media with material trapped between) but apply the energy in the form of vibrational energy (e.g., vibro-energy mill) or mechanical stirring energy (e.g., a colloid—or attrition—mill).



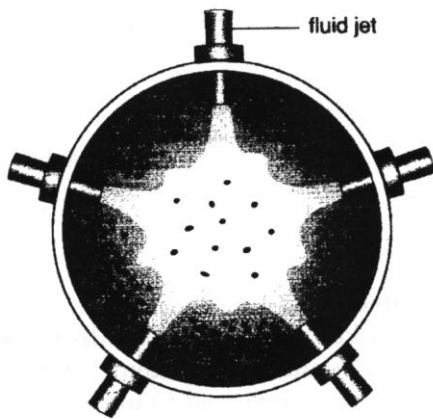
Pin mill



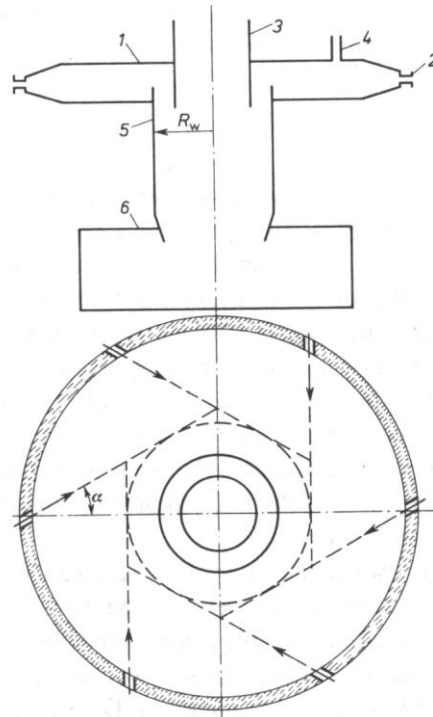
Fluid shear mill



Stirred ball mill
Attrition mill



Fluid jet mill



The fluid energy mill consists of a high-speed jet of particle laden gas that impinges on either another jet directed in the opposite direction or a hard wall. Particles are broken by the impact of high-speed collision. Fluid energy mills are able to produce the very finest powders, because with this method all the energy is absorbed by the particles to be comminuted. None is lost to other grinding media.

Energy Required for Size Reduction

If force is applied to the external surface of a particle, tensile and shear stresses are likely to be created within that particle. If the stresses exceed a critical value for an individual particle, that particle will suddenly fracture. The critical breaking stress σ is related to the Young's modulus of the solid by

$$\sigma = \sqrt{(E\gamma/r)} \quad (4.1)$$

The breaking stress σ is closely related to the surface energy γ , Young's modulus E and interatomic distance r between atoms in the crystal lattice. Equation (4.1) explains how a higher atomic density and strong chemical bonding will result in a greater strength.

Calculations show that σ is about $E/10$ for a perfect single crystal whilst $E/1000$ is typical of polycrystalline-like ceramics. The markedly lower values of σ in polycrystalline substances are attributed to the presence of intergrain boundaries and flaws. Even for single crystals, σ is markedly decreased by very fine cracks on the crystal surface. This effect of structure on strength is called 'structural sensitivity'. Equation (4.2), developed from equation (4.1), illustrates effects due to the presence of flaws and cracks:

$$\sigma = \sqrt{[E(\gamma + P)/L]} \quad (4.2)$$

where P is the work of surface plasticity and L is the depth of surface cracking. P is particularly significant in metals whilst L and γ are more important for ceramics.

The energy required to grind a metric ton of material with the different types of mills is given in Figure 4.3. For the very smallest grind, between 10 and 1 μm typically used for the production of ceramic powders, ball mills, pin mills, vibratory ball mills, fluid energy mills and attrition mills are the most frequently used. The energy required for these types of mills can vary drastically from 10 kWhr/ton to 1000 kWhr/ton. The largest values are associated with the fluid energy and attrition mills. This energy is a sum of the energy required (1) to move the machine, its kinetic energy, and friction; (2) to move the material, its kinetic energy, plastic and elastic deformation, and internal friction; and (3) to break the material into smaller particles. In almost all cases, the energy required by the mills shown in Figure 4.3 is very poorly utilized. The energy required to break the material is often less than 1% of the total energy needed to run the mill [4,5].

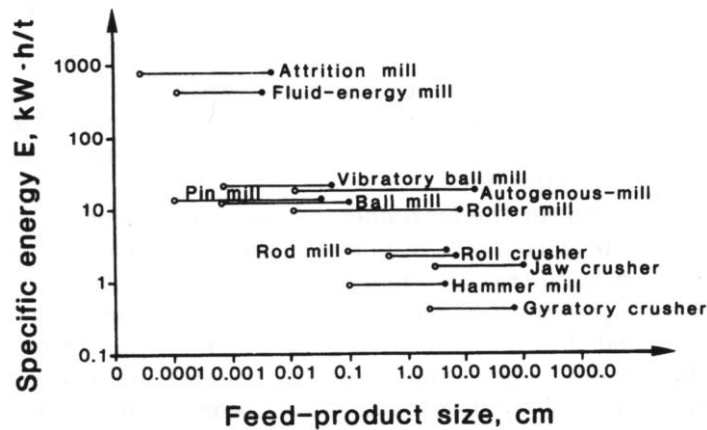


FIGURE 4.3 Average energy required for size-reduction equipment, ○ is typical product size, ● is typical feed size.

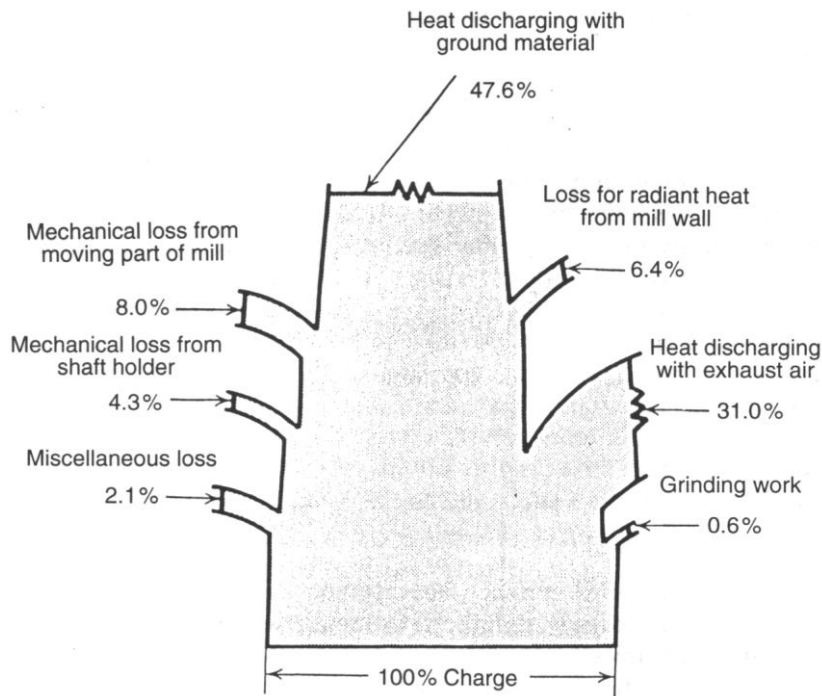


Figure 4.7 Heat balance of grinding by a vibratory ball mill. (From Y. Arai and T. Yasue, *Mater. Tech.*, 2, 43 (1984).)

The energy, E , required to grind material into a smaller particle size, L , from size, L_0 , can be described by [6]:

$$E = - \int_{L_0}^L \frac{C}{L^n} dL \quad (4.1)$$

where C is a constant. This equation has an exponent, n , which is given by the following “laws” [7]:

n	“laws”
1	Kick’s
2	Ritenger’s
1.5	Bond’s

Ritenger’s law suggests that the energy required to fracture material is simply the energy required to generate new surface area. Kick’s law on the contrary suggests that the energy required to fracture a particle is related to the energy stored within the volume of the particle. Bond developed a theory where $n = 1.5$, corresponding to a best fit of the data from many ball mill runs. Peterson [7] has suggested that there is a critical diameter above which the energy required to comminute a particle is related to the Kick’s law and below which the energy required to fracture a particle is related to the Ritenger’s law. The energy required to fracture material given in equation (4.1) corresponds to less than 1% of the energy required to run the mill, as shown in Figure 4.3. In comparing dry grinding with wet grinding, the value of constant C in equation (4.1) for dry grinding is 30% higher than that for wet grinding the same material [1]. For this reason, the energy utilization is 30% better for wet grinding than for dry grinding.

Grinding Energy from Bond

Consider the particle size defined as d_{80} (μm), i.e. when 80% of the particles will pass through a sieve of aperture size d_{80} . The grinding energy E_g required for a grinding operation is

$$E_g = E_i(1 - 1/\sqrt{r})\sqrt{(100/d_{80})} \quad (\text{kWh t}^{-1}) \quad (4.3)$$

where r is the grinding ratio (the ratio of initial size to final size) and E_i is a characteristic coefficient called the ‘work coefficient’, which is the work (kWh t^{-1}) required for grinding from infinite size to $100 \mu\text{m}$. E_i is also a measure of the difficulty of grinding and depends to some extent on both the mechanism of grinding and the design of the grinding machine. Some examples of E_i values that have been quoted are 56.70 for diamond, 43.56 for graphite, 13.57 for quartz, 12.74 for calcite, 9.92 for phosphate rock and 6.73 for gypsum. For all these materials, except graphite, the E_i values rank in the same order as the corresponding Mohs hardness values.

Comminution Efficiency

Grinding material to smaller and smaller sizes is a process that requires more and more energy. Part of the reason is that smaller particles have more strength than larger particles, because the smaller material has fewer defects than the larger particles. An example of this phenomenon is shown in Figure 4.4 [8], where the strength of quartz glass spheres is measured at different diameters. At 80 μm diameter, the strength is on the order of 10^3 N/mm^2 but at 10 μm the strength has increased threefold simply as a result of their being fewer volume defects in the smaller particles. The Griffith law [9] of failure applies to both large and small particles.

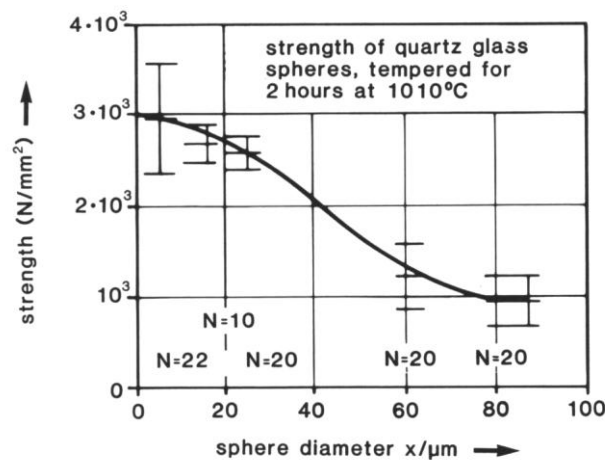


FIGURE 4.4 Strength of glass spheres. Taken from Leschonski [8].

A reduction of grain sizes during grinding is accompanied by decreasing of a width of the grain size distribution with grinding time.

Tanaka's Equation

$$S = S_{\infty}(1 - e^{-KE})$$

This equation shows that the change in surface area during grinding is an exponential function of the energy E . Here, K is the grinding coefficient, which is dependent on the nature of the material being ground and on the conditions of grinding. As $S \rightarrow S_{\infty}$ the effect of continuing to add energy by grinding becomes less. The aggregation of particles due to van der Waals or electrostatic forces and the relaxation of stresses by lattice distortion of the surface are possible causes of the existence of S_{∞} .

The efficiency of grinding rapidly decreases as the limit S_{∞} is reached. It is sometimes found that materials exist where the specific surface area S decreases again after S_{∞} is reached. This is due to the fresh surfaces produced by the grinding recombining by van der Waals or electrostatic forces. To improve grinding efficiency in these cases, it is necessary to avoid adhesion between particles due to recombination of their surfaces. Grinding aids are often added to the mill contents for this purpose. Liquids such as ether, alcohol, triethanolamine, lignosulphonic acid and stearic acid that adsorb onto the fresh surface and prevent adhesion of the particles are commonly used as grinding aids. If the material being ground is insoluble, does not react with water and is not markedly hydrophobic, the dispersibility of the particles would clearly be increased by the addition of water.

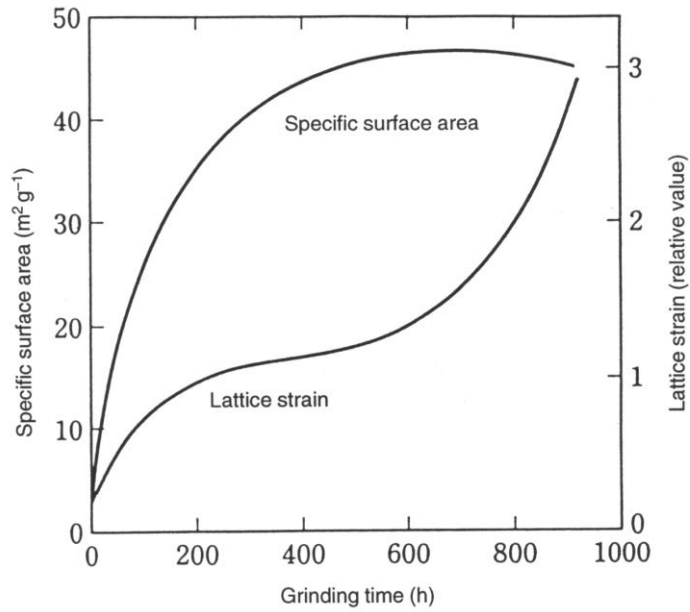


Figure 4.11 Changes in specific surface area S and lattice strain ϵ of ground dolomite with grinding time t . (From Y. Arai and T. Yasue, *J. Ind. Chem. Jpn.*, **72**, 1980 (1969).)

This can be seen as an acceleration of grinding by allowing water to adsorb on the surface of the particles. When the water content is very low, i.e. the system is merely moist, the efficiency of grinding is often reduced by the adverse effect of moisture causing the material being ground to adhere to the walls of the mill chamber. If the particles are well dispersed in a suitable amount of water, grinding will progress smoothly without adhesion of the particles occurring, the size distribution will be narrow and the change in size distribution will occur progressively. A necessary requirement of wet grinding is that, when the water is eventually removed from the wet product by drying it, this must be done without re-adhesion occurring. As an example, the size distributions of α -alumina ground by both the wet and dry processes are shown in Fig. 4.12.

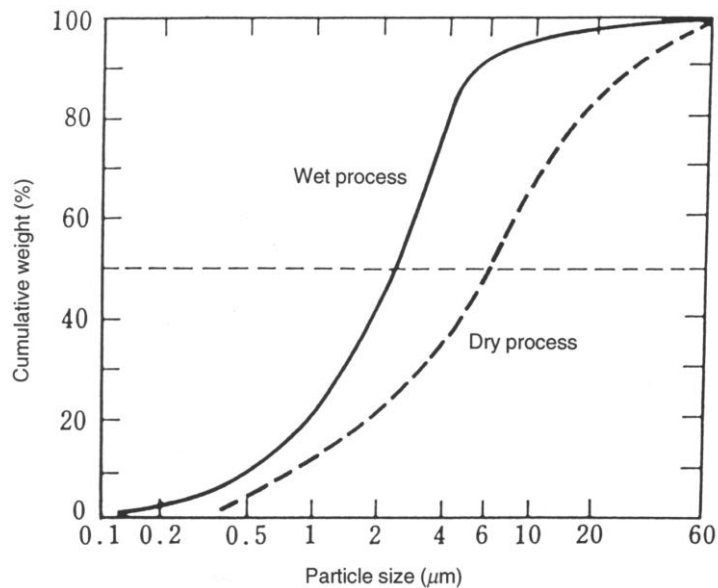


Figure 4.12 Size distribution of α -alumina powder. (From Y. Arai, *Materials Chemistry of Ceramics*, 2nd edn, Dainihon-Tosho, p. 192 (1980).)

Classification of Ceramic Powders

Classification is the separation of particulates into a coarse and fine fractions. Classification should be distinguished from solid–fluid separation, although the two unit operations overlap. Classification is usually by size, but may also depend on other properties of the particles: density, particle shape, electric, magnetic, and surface properties. Classification of particulates usually takes place in a conveying fluid either liquid or gas. Classification equipment generally operates in the 1000–0.1 μm range by the selective application of any of the following forces: gravity, drag, centrifugal, and collision. Table 4.2 gives a listing of various classification equipment.

TABLE 4.2 Classification Equipment

Classification	Size range
Wet	
Screens	1 m–44 μm
Sedimentation Classifiers	1 mm–10 μm
Hydrocyclones	500 μm –0.1 μm
Elbow Classifier	100 μm –0.1 μm
Centrifuge	50 μm –0.1 μm
Dry	
Screens	1 m–44 μm
Expansion chamber	100 μm –10 μm
Air Classifier	1000 μm –0.1 μm
Gas Cyclone	500 μm –0.1 μm

Dry Classification Equipment

Dry classification equipment uses a gas stream to convey the solids. The gas used most often is air, and for that reason the term *air classifiers* is often used to describe this type of equipment (see Fig. 4.10). Air classifiers evolved from two sources, the original simple expansion chamber and the Mumford and Moodie separator, patented in 1885. In the former, coarser particles drop out of a gas stream as its velocity is decreased upon expanding to a larger space. Baffles, vanes, or other directional and impact devices were later incorporated into the expansion chamber to change the flow direction and provide collision surfaces to knock out coarser particles, as in the Mumford and Moodie separator. In the Mumford and Moodie separator, solids are fed into a rising gas stream with a rotating distributor plate that imparts a centrifugal force to them. While the coarser particles drop into an inner cone, the fines are swept upward by the action of an internal fan, move with the gas between the vanes in the expansion section of the outer cone, and are collected at its bottom. The gas is then recirculated up toward the distributor. Many types of classification equipment are commercially available. Klumpar *et al.* [25] discussed the major designs in a review article. Basically, there are classifiers with and without rotors that collide with the particles, those with an updraft, those with a side draft, and other miscellaneous equipment. Equipment designed for classification takes advantage of a number of different phenomena: small particles settle more slowly in a fluid than large particles; small particles have less inertia and can change their direction with gas flow more easily than large particles; larger particles require a higher conveying velocity; larger particles have a greater centrifugal force in cyclonic flow than small particles; and large particles have a larger probability of collision with a rotating blade. A classifier is designed to minimize particle–particle interactions in the classification zone allowing the fluid–particle interactions to facilitate classification.

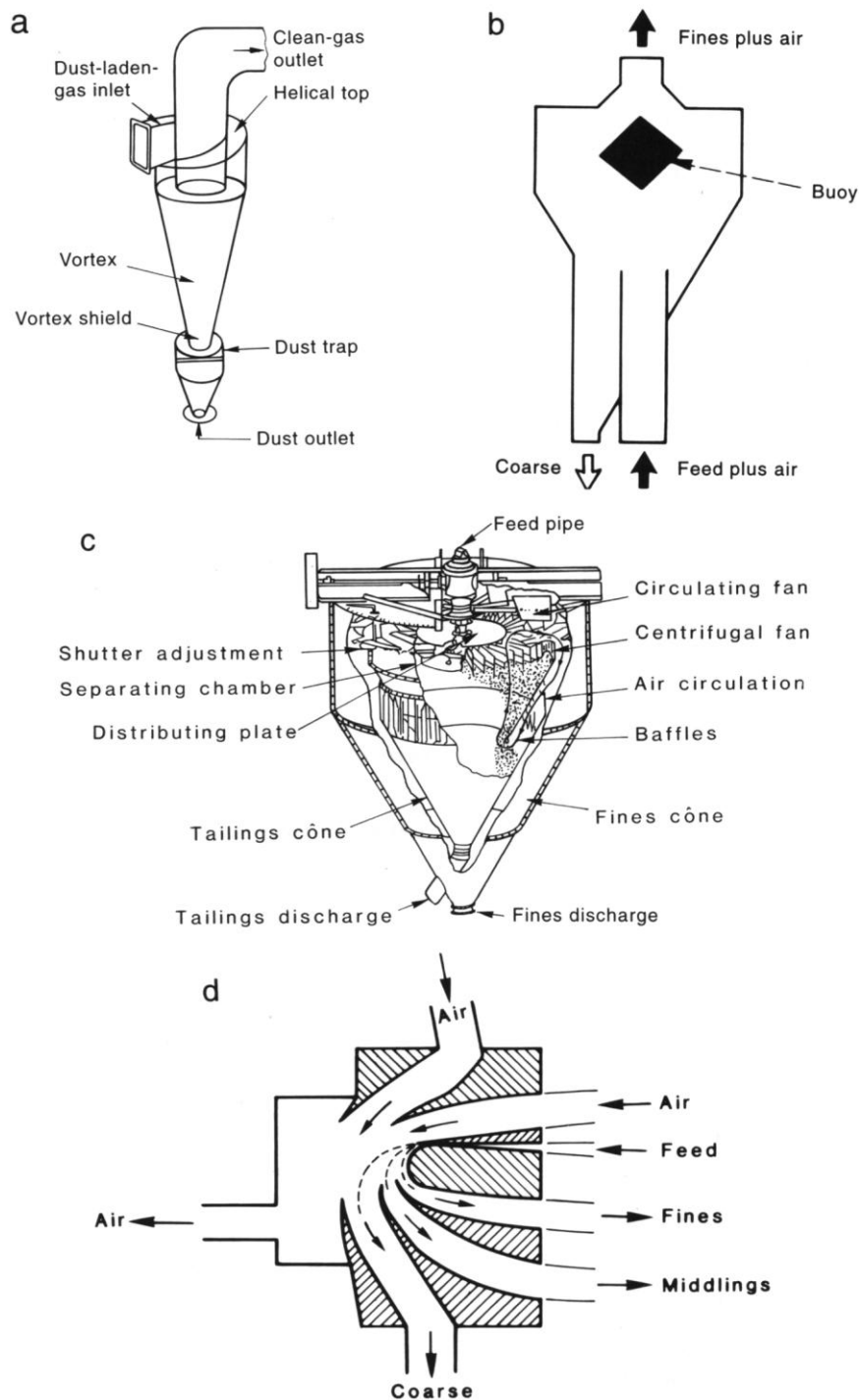


FIGURE 4.10 Air classification equipment: (a) cyclone, (b) expansion chamber, (c) modern complex air classifier, and (d) classifier based on particle inertia.

Wet Classification Equipment

Wet classification is performed by filtration, settling, centrifugation, and hydrocyclones. When operated in conjunction with grinding equipment, the wet classification equipment must operate continuously and give a pumpable fluid. This is often accomplished in practice with hydrocyclones because the other methods are unsuitable (e.g., settlers and centrifuges are used for dilute suspensions, filters and screens produce a nonpumpable cake.)