

Size reduction

within the context of sample preparation

In general "size reduction" is taken to mean the disintegration of solid substances by mechanical forces without altering their state. This also includes the division of liquids into drops or gases into bubbles. However, the physical and chemical condition of the disintegrated material may alter, particularly when inhomogeneous substances are present. The preparation for separation according to material components, e.g. dressing ores or grinding grain, is therefore one of the classical tasks of size reduction techniques.

Different substances such as minerals, ores and coal, grain and cellulose, fertilizers, drugs, plastics and color pigments, to name but a few, must be processed for a wide range of purposes. The state of dispersion of a collective, i.e. the particle size or particle distribution, in many respects determines its properties and behavior. For example, the internal friction, agglomeration behavior, solubility and miscibility, transportability as well as color and taste are all functions of the mean fineness and particle distribution.

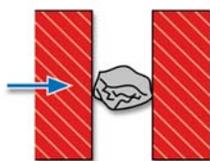
Size reduction systems

To carry out a size reduction process smoothly and effectively the comminution principle of a size reduction machine must be matched to the breaking behavior of the particular material. In practice materials from a wide range of industrial sectors and manufacturing processes are encountered. This obviously means that their properties also have to be evaluated differently. The density, hardness, consistency and not least the geometric particle shape of the sample therefore require different size reduction systems. The extraordinary variety of size reduction tasks has resulted in the development of a correspondingly large range of size reduction tools. Size reduction machines for large particle sizes, i.e. particles above 40 mm, are known as **crushers or shredders**, while particle sizes below this are processed by **mills**. In general these are known as coarse and fine crushers and fine and ultrafine mills.

Based on the comminution principles presented and described below, size reduction instruments have been developed for meeting the different demands placed on the mechanical preparation of solid substances. These comminution principles always stand in close relationship to the breaking properties of the sample, however, each case has to be considered individually.

Mechanic comminution principles

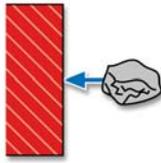
1. Strain by pressure.



Strain is applied between two solid surfaces that either represent the grinding tool surfaces directly or may be the surfaces of adjacent particles. The surfaces may move toward each other frontally or tangentially to exert the required pressure.

Examples: jaw crushers, toggle crushers.

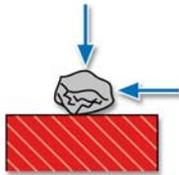
2. Strain by impact effects



Strain at a solid surface. This could either be that of a grinding tool, or be represented by other particles. Strain by impact is mainly caused by one-sided and opposing particle acceleration as a result of the kinetic energy of the relative movement.

Examples: impact mills, jet impact mills.

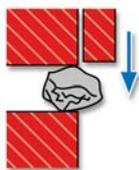
3. Strain by pressure and friction



Strain between two solid surfaces. Caused by the vertical pressure of one surface and the simultaneous horizontal, centric or eccentric rotary movement of the other surface.

Examples: hand mortars, mortar mills.

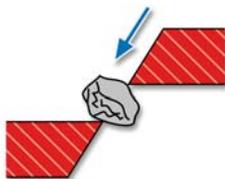
4. Strain by shearing action



Strain between two or more solid surfaces as a result of shearing action. Size reduction is triggered by two surfaces moving in opposing directions or one moving and one stationary surface. Additional impact action is possible.

Examples: shearing action between ring sieve and rotor in rotor beater mills, cross beater mills, ultra-centrifugal mills, etc.

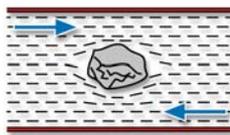
5. Strain by cutting action



Strain between two or more sharp-edged surfaces. The cutters are opposed vertically to one another. In many cases there is one fixed and one moving cutting edge.

Examples: shredders, cutting mills, etc.

6. Strain by a surrounding medium



Strain by the surrounding medium, gas or liquid, is only effective with high shearing gradients and low material strengths, e.g. with agglomerations or materials with a hardness < 3 on Mohs' scale. Deagglomeration in highly viscous media is a typical field of application.

Examples: high-speed stirrers such as blenders, Ultra-Turrax, etc.

Selection criteria

In addition to product-specific properties, when selecting size reduction apparatus the technical aspects of the machine to be used must also be taken into account. The aim of the preparation must be defined by consideration of the following points:

Sample material (grinding product)

What physical and chemical material properties could primarily affect the size reduction process (e.g. degree of hardness, toughness, thermal sensitivity, abrasiveness or aggressiveness)?

Feed size

What is the maximum particle size of the sample and how could the particle shape influence the grinding process (e.g. round, needle or flat shapes)?

Ultimate fineness and grinding fineness

What degree of analytical fineness must be achieved? What particle size distribution is required? In this case the primary concern is for the subsequent analysis.

Abrasion

What abrasion of the grinding tools could affect and falsify the subsequent analysis? What "contamination" from abrasion can still be accepted?

Grinding duration

How much time is available for the grinding process? How much time is required for preparing and setting up the mills?

Grinding aids

Is the use of grinding aids necessary and permissible? Is it necessary to thermostat the sample before or during the grinding process? Must the sample be prepared under protective gas or vacuum?

Only when all these questions have been answered and an unambiguous definition of the analytical targets has been set down is it possible to select a suitable product-specific and application-specific size reduction system from the vast range of mills that is available.

Preliminary size reduction

Daily work in the laboratory: an initial laboratory sample has to be prepared as an analytical sample which is then analyzed for particular characteristic features. Because of the initial particle size and amount it is not usually possible to achieve the necessary analytical fineness in a single working step. Preliminary size reduction, sample division and then the pulverization of a representative part sample is the most appropriate working procedure for sample preparation. Care must be taken that the characteristic features to be determined do not alter during the whole of the sample preparation process.

Classical preliminary size reduction systems for hard-brittle, dry products (examples: ores, minerals, slag) are all roller mills and jaw crushers as well as rotary crushers, cone crushers and hammer mills. The size of these machines depends on the required throughput and maximum feed size. For particles sizes up to 100 mm a fineness down to < 3 mm can be achieved in a single working step. The ultimate fineness can be influenced by the adjustable gap width of the grinding tools or by exchangeable insert sieves (base sieves). The breaking tools, i.e. the force-applying surfaces, are usually made from a range of different materials including manganese steel, hardened special steel as well as tungsten carbide.

For the preliminary size reduction of soft, tough and ductile substances high-performance cutting mills are increasingly in demand. The low speed of the cutting rotor reduces any frictional heat that may be generated and could affect thermally sensitive samples. High rotary masses at the rotor ensure effective size reduction even with heterogeneous samples with different breaking properties (examples: domestic waste, electronic scrap, auto-shredders, etc.). The achievable ultimate fineness depends on the exchangeable base sieves that are used.



Fig.: Retsch Jaw Crusher BB 200



Fig.: Retsch Cutting Mill SM 2000

Fine grinding / Pulverization

In principle it is necessary to finely grind the sample before each analysis. The degree of fineness influences not only the homogeneity of the sample, but also affects product properties such as extraction behavior, absorption and filtration, solubility and suspension. These properties are used in many analytical methods such as those for the detection of chemical elements or for biological and physical reaction behavior. The required analytical fineness differs and can lie anywhere in the range from $< 20 \mu\text{m}$ - $2000 \mu\text{m}$. The required degree of fineness always depends on the particular analytical method.

When selecting the correct mill and its grinding tools the breaking properties and material condition are again of crucial importance. **Ball mills and mortar mills** are the most frequently used size reduction machines for this purpose. Among other things, they have the decisive advantage that **grinding tools in different sizes and made from different materials** – also non-metallic – can be used. This is particularly important for the analysis of heavy metals, where grinding tools made of heavy metals cannot be used for sample preparation. Preferred grinding tools for this purpose are made from agate or zirconium oxide. Ball mills and mortar mills work with grinding jars that are sealed dust-tight; these also allow wet grinding to be carried out. The grinding tools for a ball mill always consist of the grinding jar and a charge of grinding balls that depends on the sample substance and its volume. The mortar mill grinding tools consist of a pestle and a grinding jar made from the same material.



Fig.: Retsch Planetary Ball Mill PM 100

For the fine grinding of soft to medium-hard products it is frequently only possible to achieve the required degree of fineness by using **rotor mills**, such as **ultra-centrifugal mills or knife mills**. Only the application of shearing and cutting forces is effective when processing plastics and rubber. By the effects of impact and shearing, such as are produced in the ultra-centrifugal mill, the sample undergoes homogeneous pulverization between the rotor and ring sieve. The defined mesh sizes of the exchangeable ring sieves are primarily responsible for the achievement of the expected ultimate fineness. Heavy-metal-free titanium rotors are available for applications in which grinding tools made

from stainless steel are not acceptable. In contrast, abrasion-resistant tungsten-carbide-coated grinding tools must be used for processing fertilizers or feed pellets.



Fig.: Retsch Ultra-Centrifugal Mill ZM 200



Fig.: Retsch Knife Mill GRINDOMIX GM 200

With a knife mill such as the RETSCH Grindomix GM 200, whose comminution principle is cutting action, it is normally possible to pulverize and homogenize all samples containing fats, oils and water.

Grinding tools made from different materials are available for all Retsch mills.

When choosing the material please remember:

- The aim of sample preparation, i.e. the subsequent analysis or further processing of the sample
- The material properties of the sample, primarily its degree of hardness and abrasiveness
- The type of machine and the corresponding comminution principle

Guidelines for material selection

	Hardness	Density	Energy input e.g. in ball mills	Abrasion resistance	Possible contamination
Stainless Steel	48 - 52 HRC (ca. 550 HV)	7,8 g/cm ³	Very high	Moderately high	Fe, Cr
Chrome Steel	62 - 63 HRC (ca. 750 HV)	7,8 g/cm ³	Very high	High	Fe, Cr, C (less than in st. steel)
Tungsten Carbide	approx. 1200 HV	14,8 g/cm ³	Extremely high	Very high	WC, Co (marginal)
Agate	hard and brittle 6,5 - 7 Mohs (approx. 1000 HV)	2,6 g/cm ³	Very low	Moderately high	SiO ₂
Sintered Aluminium Oxide	Hard and brittle 8 - 8,5 Mohs (approx. 1500 HV)	3,8 g/cm ³	Low	High	Al ₂ O ₃ , SiO ₂ ; (marginal) no contamination with Fe, Cr, Ni oder Co
Zirconium Oxide	Hard and brittle, tougher than agate 7,5 Mohs (approx. 1200 HV)	5,9 g/cm ³	High	Very high	ZrO ₂ and Y ₂ O ₃ (marginal)
Teflon	elastic Shore Hardness D 56	2,1 g/cm ³	Very low	Low	Contamination with F, C

Closing remarks

The mechanical size reduction sector in process engineering is so complex and extensive that only a brief overview of the most important size reduction methods can be given here. Ever-new applications from the research and development divisions of different target markets lead to the continual further development of size reduction machines and grinding tools. It is true in principle that different analytical requirements demand different size reduction techniques, so that one application cannot simply be transferred to a different one. This is why it is not possible to do without the help of a "mill builder" when searching for a user-specific solution. RETSCH has a specialized application laboratory and, in addition to practically oriented tests, can also offer a comprehensive database together with the corresponding advisory service; these are free of charge.

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