Retsch®

The Art of Milling

An expert guide to neutral-to-analysis size reduction
and homogenization in the laboratory

part of VERDER scientific

with detailed information on materials

The Art of Milling

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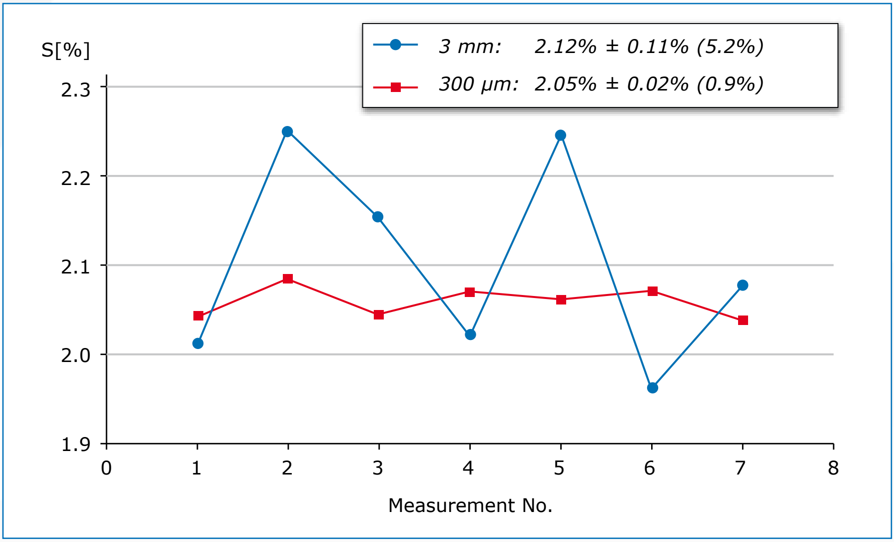
1. Why Size Reduction?

A reliable and accurate analysis can only be guaranteed by reproducible sample preparation. The "Art of Milling" describes the process of turning a laboratory sample into a representative part sample with homogeneous analytical fineness.

For this task RETSCH offers a comprehensive range of the most modern laboratory mills and crushers for coarse, fine and ultra-fine size reduction of almost any material. The wide selection of grinding tools and accessories not only ensures contamination-free sample preparation but also adaptation to the specific requirements of such different areas of application as construction materials, metallurgy, foodstuffs, pharmaceuticals or environment.
Particle size reduction of solids or bulk materials is required when the particles are too coarse or the sample is too inhomogeneous for subsequent processes such as analysis, division, mixing or further processing. The standard deviation of any subsequent analysis can be minimized drastically by particle size reduction and homogenization of the analysis sample (see diagram below).



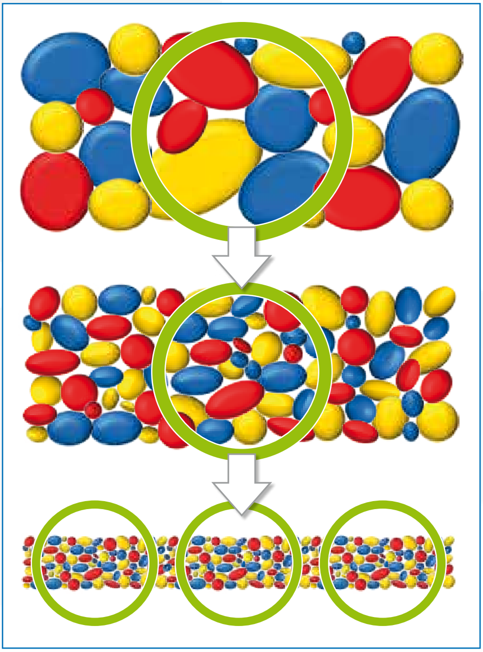
A rubber sample was ground to different particle sizes. The degree of homogenization increased with decreasing particle size. The subsequent measurement of the sulfur content (measured with an ELTRA CS-580 analyzer) showed that the more homogeneous a sample was (red) the lower was the standard deviation, i. e. the more reliable were the analysis results.

Required Fineness

A frequent requirement is to "grind the sample to fine powder". The term "powder", however, is not precise. Washing powder, coffee powder or baking powder are bulk materials which are all characterized as "powders", although they have very different particle size distributions.
Another typical request is to have the sample ground "as fine as possible". This involves a high input of energy and time which in turn increases costs. A much more effective approach is **not to grind as fine as possible but only as fine as necessary.** This parameter is typically determined by the subsequent analysis.

Homogenization

Usually, only a few gram or milligram of a laboratory sample are required for an analytical method. If the sample is inhomogeneous, this small amount does not represent the entire sample as some ingredients may be over-representative or missing altogether. Therefore, the sample must be homogeneous to ensure reliable and representative analytical results.



Only a homogenized analytical sample can represent the total initial sample and lead to reliable and reproducible results.

Analytical fineness

The required analytical fineness of the sample material depends on the analytical method or further processing and can vary greatly. Most methods require a fineness in the size range from 20 µm to 2 mm.

As product properties (e.g. extraction, filtration or absorption capacity) are often influenced by the particle size, size reduction on a laboratory scale is also essential for the development of new products or production processes.

Aspects which influence the selection of a suitable mill

When searching for a suitable mill and grinding tools, one has to keep in mind that the sample properties to be determined (such as moisture or heavy metal content) must not be altered in any way during the process.
To make the right choice not only precise knowledge of the instruments is required but also profound experience in the preparation of different materials.

To find the best suited mill for a specific application, the following aspects should be considered in advance:

- Quality/characteristics of sample
  (e.g. dry, tough, abrasive, fibrous, brittle, hard, soft, temperature-sensitive etc.)
- Feed size
- Required final fineness
- Sample volume
- Sample throughput
- Subsequent analysis
  (which type of contamination by abrasion of grinding tools is acceptable?)
- May the sample be dried or embrittled before grinding?
- Not to forget: Is the laboratory sample representative?

Depending on the quality of the material different size reduction principles are applied to obtain the required fineness. Large particles cannot always be ground to analytical fineness in one step. In some cases it is possible to carry out coarse and fine grinding in the same mill with different settings; in other cases two mills or crushers are required.

2. The Size Reduction Process

To produce a size reduction effect, the comminution principle of the mill should be matched to the breaking behavior of the respective material (see chapter 2.2).

When selecting a suitable instrument and before beginning the preparation process, a thorough evaluation of the material is necessary. Properties such as density, hardness, consistency, residual moisture or fat contents have to be examined. The success of the grinding process can also be influenced by temperature stability, tendency to agglomerate or surface reactions.

In any case, the requirements of subsequent analysis should always be taken into account when carrying out a particular grinding task.

2.1. Sample Preparation

Before starting the actual grinding process it must be ascertained that the sample can be processed without further treatment. Moisture, agglomerations, segregations or impurities could disturb the process and falsify the grinding result.

The sample quantity is also an important factor. How much is needed for analysis? How big is the total sample amount in comparison to that and what is the grain size? These parameters determine the minimum amount which is needed for the part sample to be representative. Representative means that the composition of the part sample is exactly the same as that of the total sample.

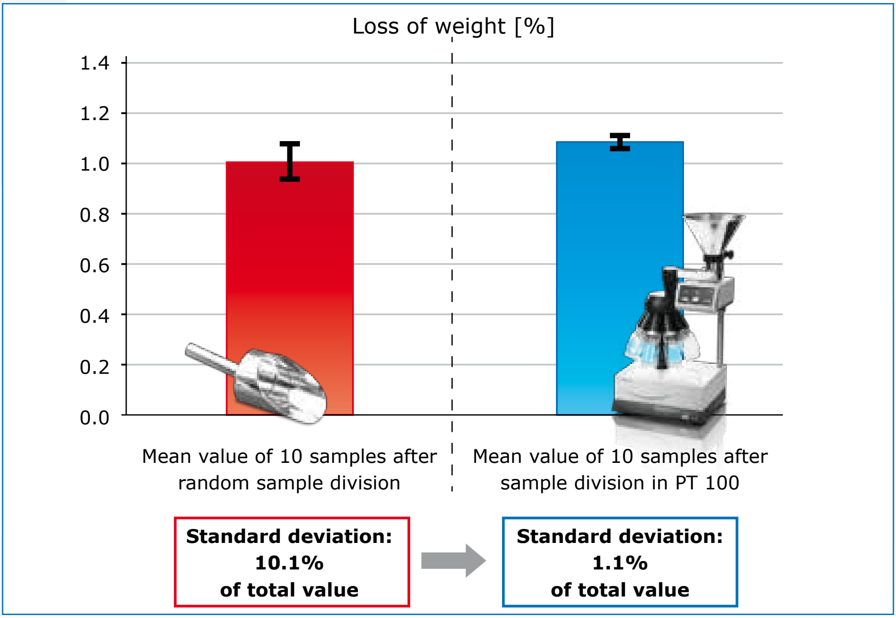
Different models of sample dividers

Sample Division

Most laboratory samples consist of an inhomogeneous mixture. Different particle sizes can lead to segregation during transportation of the sample. If the sample is not ground completely, a part sample should be extracted.

If the initial grain size is too big, preliminary size reduction must be carried out before the sample can be divided. The selection of the division method and the instrument depends on the sample material and quantity. Dry, free flowing samples can be fed via vibratory feeders to rotary tube dividers and sample dividers with a rotating dividing head whereas sample splitters are used for materials with a low flowability.

Manual random sampling is only acceptable if the sample is homogeneous with regards to material and grain size. However, without preliminary examination, this is difficult to ascertain.



The standard deviation e.g. in a plastic sample analyzed for its moisture content can be decreased drastically by correct sample division using a sample divider.

Drying

To grind moist or even wet sample materials is not possible without bothersome side effects, especially in jaw crushers, rotor or disc mills. Moist materials tend to block the ring and bottom sieves which can lead to a blockage of the machine. As a consequence, material is lost and much time and effort has to be spent on cleaning the mill.

Fluid Bed Dryer TG 200

There are a few exceptions: colloidal grindings can only be carried out in ball mills by adding a liquid. Fresh fruit and vegetables can be homogenized in knife mills without material loss.

However, in most cases, moist samples have to be dried (e.g. leaves) before they can be subjected to size reduction. When choosing the drying method and temperature, care must be taken that the properties of the sample to be determined are not altered in any way. That is especially important with regards to volatile components such as furans, polychlorinated biphenyls (e.g. PCBs) and dioxins. Usually, these types of sample can only be air-dried at room temperature.

RETSCH's TG 200 is suitable for gentle and quick drying. It uses the fluidized bed drying method, similar to that of industrial dryers. For many products the drying time is as little as 5 to 20 minutes.

Further methods include vacuum and freeze drying as well as drying in drying ovens.

Metal Separation

Samples such as industrial waste, recyclable waste and secondary fuels often contain metallic components which cannot be pulverized with laboratory mills. On the contrary, metallic objects such as steel nails or iron screws can damage the grinding tools which can lead to a considerable deterioration of the mill's performance. Therefore, it is necessary to separate the metal components before grinding (e.g. with magnetic forces). If required, they have to be evaluated separately.

Embrittlement (with liquid nitrogen or dry ice)

Cooling a jar in liquid nitrogen

Cooling the sample material often improves its breaking behaviour. Therefore, temperature-sensitive materials, such as some types of plastics, have to be cooled directly before they can be subjected to preliminary or fine size reduction. One way is to embrittle the sample in liquid nitrogen (N_2, LN) before grinding. At a temperature of -196 °C even soft rubber becomes so hard and brittle that it can be ground without problems. Another possibility is to mix the sample with dry ice (CO_2). For indirect cooling, the grinding jar is placed in liquid nitrogen.

If the sample contains volatile substances which must be preserved during grinding, cryogenic grinding is also the method of choice.

However, materials which must not become moist should not directly be treated with cooling agents. The reason for this is that the steam in the air is frozen and is precipitated as water when it unfreezes.
Cooling agents should not be used in closed grinding tools as evaporation causes overpressure in the jar.

2.2. Size Reduction Principles

Laboratory mills work with different size reduction principles. Which type of mill is used for a particular size reduction task always depends on the breaking properties of the sample material. Hard-brittle materials are best pulverized with impact, pressure and friction whereas soft and elastic substances require cutting and shearing effects to be successfully comminuted.

Size reduction machines for large particle sizes above 40 mm are known as crushers or shredders while particle sizes below this are processed with mills.

The next pages show the most common mechanisms for the size reduction of solids.

Size Reduction of hard / brittle materials

Pressure
Force is applied between two solid surfaces that either represent the grinding tool surfaces directly or may be the surfaces of adjacent particles. Pressure is exerted by the grinding tools.
Examples:
jaw crushers, toggle crushers.

Impact effects
Force 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.
Examples:
mixer mills, planetary mills, impact mills, jet impact mills, drum mills.

Friction
Force between two solid surfaces. Caused by the vertical pressure of one surface and the simultaneous movement of the other surface.
Examples:
mortar grinders, disc mills,
hand mortars, rod mills.

Size Reduction of soft, elastic, fibrous materials

Shearing

Force between two or more solid surfaces moving in opposing directions which results in a shearing effect. At least one fixed and one moving surface.

Examples:

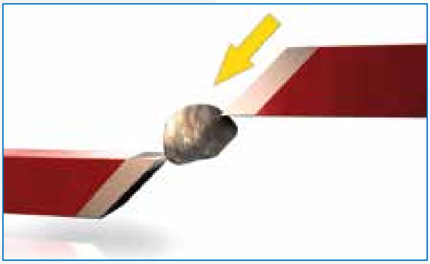
rotor beater mills, cross beater mills, ultra centrifugal mills.

Cutting

Force between two or more sharp-edged surfaces. At least one fixed and one moving cutting edge.

Examples:

shredders, cutting mills, knife mills.



Usually, various size reduction principles are combined in one mill, such as impact and friction in planetary ball mills or shearing and impact in rotor mills.

2.3. Grinding Tools

Grinding jars "comfort" and grinding balls for Planetary Ball Mills

Each RETSCH mill comes with grinding tools that are optimized with regards to their functionality and handling. However, due to the wide range of applications, the requirements may differ greatly. Therefore, RETSCH offers a great variety of accessories. For ball and disc mills, for example, the grinding sets are available in various sizes. By using distance sieves and rotors it is possible to process temperature-sensitive materials in rotor mills.

All grinding tools are available in different materials.

Materials

The materials used for RETSCH grinding tools can be grouped as follows:

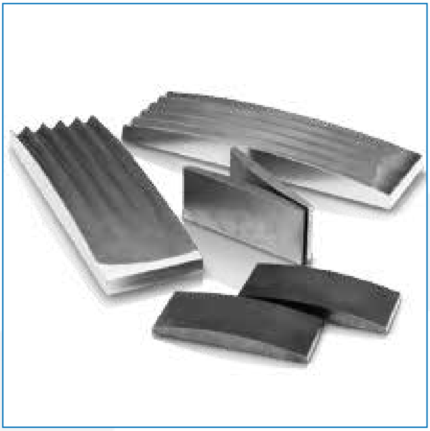
- Metal (steel, cast iron, titanium)
- Ceramics (tungsten carbide, zirconium oxide, sintered aluminium oxide, hard porcelain, silicon nitride)
- Natural stone (agate)
- Plastics (PTFE)

The chemical and physical properties of a material determine whether it is available for a particular type of mill. Grinding tools made of steel are available for all mills.

The table below gives a survey of parameters such as hardness, energy input, wear resistance and possible contamination through abrasion:

Materials					
	Hardness	**Density**	**Energy input***	**Wear resistance***	**Possible contamination through abrasion**
Stainless Steel	48 - 52 HRC (approx. 550 HV)	7.8 g/cm³	very high	good (to certain extent)	Fe, Cr
Hardened Steel	58 - 63 HRC (approx. 750 HV)	7.85 g/cm³	very high	good	Fe, Cr, C (less than SS)
Tungsten Carbide	approx. 1250 HV	14.8 g/cm³	extremely high	very good	W, C, Co (marginal)
Agate	hard and brittle 6.5 - 7 Mohs (approx. 1000 HV)	2.65 g/cm³	very low	good (to certain extent)	SiO_2
Sintered Aluminum Oxide	hard and brittle 8 - 8.5 Mohs (approx. 1750 HV)	3.9 g/cm³	low	good	Al_2O_3, SiO_2 (low), no contamination with Fe, Cr, Ni or Co
Zirconium Oxide	hard and brittle, tougher than agate 7.5 Mohs (approx. 1200 HV)	5.9 g/cm³	high	very good	ZrO_2 and Y_2O_3 (marginal), insignificant for analyses
Silicon Nitride	approx. 1500 HV	3.2 g/cm³	low	excellent	Si_3N_4, Y_2O_3, Al_2O_3
PTFE	elastic Shore Hardness D 56	2.1 g/cm³	very low	poor	contamination with F, C

*e.g. for ball mills



Breaking jaws BB (left)
Rotors and sieves ZM (right)

When choosing a suitable grinding set, several factors have to be considered such as the **hardness** of the sample material and its breaking properties. The material of the grinding set should be harder than the sample to avoid excessive wear. For example, silica sand should not be ground with agate tools but with zirconium oxide or silicon nitride as these are much harder.

Abrasion resistance is also an important parameter. Tungsten carbide, zirconium oxide and silicon nitride are highly resistant against abrasion. However, the amount of abrasion also depends on the properties of the sample and the size reduction principle of the mill.
In mechanical size reduction processes, abrasion cannot be completely avoided. Therefore, when choosing a material it should be taken into account if possible contamination will have a negative influence on the product or the subsequent analysis (e.g. abrasion of chrome or nickel influences subsequent heavy metal analysis). Thus, neutral-to-analysis materials should be chosen.

Another important feature of ball mills and vibratory disc mills is the **energy input** generated by the different materials. Grinding balls of tungsten carbide, for example, generate a much higher energy input, and thereby a better size reduction effect, due to the higher density of the material than balls of the same size of other materials.

Application examples:

- If soil samples are to be analyzed for iron, chrome or cobalt, grinding tools of stainless or hardened steel are not suitable as they contain the elements which are to be determined.
- If, however, calcium or silicon dioxide are to be determined in cement clinker, grinding jars of steel are suitable.
- PTFE, zirconium oxide, silicon nitride and glass can be sterilized; therefore, they are often used for preparing food or microbiological samples.
- Abrasion is also an issue for the further processing of samples, even if there is no subsequent analysis. Homeopathic products, active agents in lactose and pharmaceuticals, for example, should only be ground in ceramic or agate mortars in order to avoid contamination of the sample.

Please refer to the annex for detailed information on the materials of the grinding tools.

2.4. Grinding Aids

Many grinding tasks which are known from the field of mechanical process engineering can be solved by using one of the various mill types with a suitable size reduction principle. However, some applications cannot be carried out successfully with common laboratory mills despite the wide range of accessories. Difficult samples are, for example, moist materials which must not be dried or soft and elastic materials and those which contain fat or oil. To produce ultra-fine powders by mechanical energy input, it is often necessary to add a liquid.

In the above cases, the use of a grinding aid can be helpful. Grinding aids are additives which activate, accelerate and also improve chemical or physical processes. Before using a grinding aid for the preparation of solids it must be ensured that the additive does not influence the subsequent analysis or further processing of the sample in any way.

Grinding aids are differentiated according to their state of aggregation:

- Solid (powder, granulate, pellets) to bind fat and/or moisture (e.g. talcum)
- Liquid (water, alcohol, benzine) to avoid agglomeration
- Gaseous (inert gas, cooled air, e.g. argon)

Another grinding aid is:

- Tempering (cooling with liquid nitrogen or dry ice, heating)

Solid Additives

When preparing samples for XRF analysis, neutral-to-analysis aids, such as Spectromelt pellets (based on cellulose) are often added to the sample material during grinding in planetary ball mills or vibratory disc mills. If they are mixed in the correct ratio, they support the size reduction effect and help to avoid caking of the material inside the grinding jar. When pelletizing the sample material afterwards, this grinding aid also serves as a binding agent.

The addition of sodium sulfate is a common method to bind fat or moisture that is to be determined afterwards (e.g. when grinding insects, small marine animals or moist soils). Trituration is carried out in mortar grinders which guarantees 100 % sample recovery.

Liquid Additives

To homogenize oil seeds such as rape seed, soy beans or mustard seeds in ball mills or mortar grinders, it is helpful to add benzine. Benzine is used as extraction liquid for the determination of oil contents.

The production of ultra-fine powders, e.g. in the ceramics industry, powder metallurgy or mineralogy, can often only be realized by wet grinding. Usually, water or isopropanol are used as dispersants. Ball mills and mortar grinders are especially suitable for wet grinding.

Air or Inert Gas

If a size reduction system is sufficiently ventilated, e.g. through a cyclone or a filter system, the frictional heat is continuously discharged. This helps to reduce the heating up of the sample material and to increase the throughput.

Gassing with inert gas during grinding prevents the reaction of surface active particles with oxygen (= oxidation).

3. RETSCH's Product Range

The RETSCH product range covers applications from the preliminary size reduction of particles of several centimeters to fine grinding down to the nano range. The following criteria are decisive when selecting a suitable laboratory mill:

- **Material properties of the sample (e.g. breaking behavior)**
- **Feed size of the sample**
- **Required final fineness**
- **Feed quantity**

If the initial particle size of the sample is coarse, it might be necessary to use two mills, one for preliminary size reduction and one for fine grinding, to achieve analytical fineness. To choose suitable grinding tools is also part of the selection process (see chapter 2.3). Here the important criteria are hardness, abrasion resistance, possible contamination and, for ball mills, the energy input.

Interdependence of instrument, sample material and working principle

Device	Hard and brittle materials	Soft, elastic and fibrous materials	Working principle
Jaw Crusher	■		Pressure
Ultra Centrifugal Mill		●	Impact, shearing
Cyclone Mill		●	Friction, shearing
Cross Beater Mill	■		Impact, shearing
Rotor Beater Mill	●	● ■	Impact, shearing
Cutting Mill		■	Shearing, cutting
Knife Mill		●	Cutting, impact
Mortar Grinder	●		Pressure, friction
Disc Mill	●		Pressure, friction
Mixer Mill, CryoMill	●	●	Impact, friction
Planetary Ball Mill, E_{max}	●	●	Impact, friction
Drum Mill	●	●	Impact, friction
Rod Mill	●	●	Friction

■ Preliminary size reduction

● Fine grinding

3.1. Jaw Crushers

Jaw crushers are used for the preliminary size reduction of hard-brittle, dry materials, such as ores, minerals, slag and coal.

RETSCH offers eight sizes: the floor models **BB 100, BB 200, BB250 XL, BB 300, BB 400 XL, BB 500 XL, BB 600 XL** and the benchtop model **BB 50.**
The BB 600 XL crushes particles of up to 35 cm while the maximum feed size for the BB 50 is approx. 4 cm. The final fineness achievable with a BB 600 XL is approx. 6 mm, the BB 50 produces particles of 0.5 mm and smaller. The final fineness is determined by the (adjustable) gap width of the breaking jaws.

Jaw Crusher BB 50

With increasing size of the jaw crusher, the potential throughput of sample material also increases as the BB 200 and the BB 300 achieve a high throughput of more than 100 kg/h and the BB 600 XL even a throughput of up to 3.5 t/h (depending on the sample material).
Four models can be integrated in process lines: BB 200, BB 300, BB 500 XL and BB 600 XL.
The jaw crushers of the XL line can be equipped with an automated sorting machine.

Jaw Crusher BB 600 XL

The breaking jaws and the wearing plates are available in five different materials (depending on the model):
Manganese steel, stainless steel, steel for heavy metal-free grinding, tungsten carbide and zirconium oxide.

3.2. Rotor Mills

Ultra Centrifugal Mill ZM 200

The **Ultra Centrifugal Mill ZM 200** is used for the rapid fine size reduction of soft, medium-hard, brittle and fibrous materials such as fertilizer, plastics or feed pellets. For the size reduction of elastic plastic materials it may be necessary to embrittle the sample in liquid nitrogen before grinding.

Size reduction in the ZM 200 is effected through impact and shearing forces between ring sieve and rotor. The maximum feed size is up to 10 mm. Depending on the material, a final fineness down to 40 µm and below can be achieved. The final fineness is determined by the exchangeable ring sieves. The speed of the ZM 200 ranges from 6000 to 18000 min^{-1}. The patented cassette principle guarantees 100% sample recovery and easy cleaning.

For an automatic and uniform feed of larger amounts of free flowing materials the Vibratory Feeder DR 100 is recommended. A cyclone with a 3 liter or 5 liter collector can also be used for larger amounts as well as for temperature-sensitive materials. The frictional heat that is generated during the grinding process is partly discharged through the cyclone. The use of distance sieves instead of standard ring sieves also helps to reduce frictional heat due to the greater gap between sieve plate and rotor.

Accessories for the ZM 200 include ring sieves and rotors of titanium for heavy-metal-free size reduction. If hard and abrasive materials are to be ground, a rotor with abrasion resistant coating is required. For processing very small amounts of sample, e.g. of pharmaceuticals, RETSCH offers a mini-cassette with matching 316 L stainless steel rotor and various ring sieves.

The **Cyclone Mill TWISTER** is specially designed for the processing of foods and feeds for subsequent NIR analysis.

It processes fibrous and soft products quickly and gently to the required analytical fineness. The mill is ideally suited for grinding feeds, forage and cereals as well as various types of food.

Cyclone Mill TWISTER

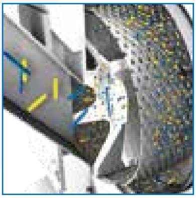
The TWISTER is equipped with a rotor and grinding ring with sieve insert. The high speed of up to 14000 min^{-1} and the optimized grinding geometry of rotor and grinding chamber generate an air stream which carries the sample through the integrated cyclone into the sample bottle. The cyclone provides additional cooling of the sample and the grinding tools. This prevents loss of moisture and thermal degradation ensuring preservation of the sample properties to be determined. The ground material is separated in the cyclone and collected in a sample bottle for complete recovery.

The provided sieves guarantee an optimum particle size distribution so that it is not necessary to recalibrate the NIR spectrometer. The rotor speed can be adjusted in 3 steps allowing for perfect adaptation to the sample requirements. For most products the air stream effects a complete discharge of the material from the grinding chamber, particularly if a vacuum cleaner is connected, so that hardly any cleaning is required. This helps to avoid cross contaminations and is especially convenient when processing a series of samples.

Rotor Beater Mill SR 300 with cyclone attached

The **Rotor Beater Mill SR 300** is used for the preliminary and fine size reduction of soft, medium-hard and brittle materials with a maximum feed size up to 25 mm. The final fineness is determined by the aperture size of the exchangeable ring sieves. The SR 300 can achieve a fineness down to 50 µm and below, depending on the properties of the sample material. Typical applications include the grinding of grain, coal, chemicals and soils.

Size reduction in the rotor beater mill is effected by impact and shearing forces between rotor and ring sieve. Rotor beater mills are usually equipped with ring sieves. To achieve an additional size reduction effect through impact, a 180° grinding insert can be used for medium-hard materials.



The speed of the SR 300 is adjustable between 3000 and 10000 min^{-1}. A higher speed does not only generate a higher throughput but also more frictional heat which can affect the sample material. For temperature-sensitive materials the use of a distance rotor is recommended. The larger grinding gap ensures a reduction in frictional heat. Moreover, a cyclone is available which also reduces generation of heat by dragging ground particles faster out of the grinding chamber and generating a cooling air flow.

The **Cross Beater Mill SK 300** is suitable for the preliminary and fine size reduction of medium-hard/brittle materials such as ores, minerals and cement clinker with feed sizes up to 25 mm. The final fineness is determined by the exchangeable bottom sieves with various aperture sizes. Size reduction is effected through impact and shearing forces between the baffle plates of the rotor and the toothed grinding insert.

Particles which are ground to the desired fineness pass the bottom sieve. The SK 300 is available in cast iron, stainless steel, hardened steel and steel 1.1740 (for heavy-metal-free size reduction). An optional cyclone is also available.
The speed of the SK 300 is adjustable in steps of 200 min^{-1} between 2000 and 4000 min^{-1}.

Cross Beater Mill SK 300

3.3. Cutting and Knife Mills

The **Cutting Mill SM 100, SM 200** and the **Heavy-Duty Cutting Mills SM 300** and **SM 400 XL** are used for preliminary size reduction of bulky, soft and fibrous materials such as branches, straw and plastics. Due to an additional flywheel mass on the drive shaft the SM 300's performance equals that of motors with twice the power rating. Therefore, it is also suitable for the size reduction of solid and tough materials such as leather, thick pieces of rubber and secondary fuels or electronic scrap. The speed of the SM 300 is freely adjustable between 700 and 3000 min^{-1}.

Cutting Mill SM 300

The final fineness which can be achieved with the cutting mills depends on the aperture size of the exchangeable bottom sieve and the breaking properties of the sample material. For all members of the Cutting Mills family three types of rotors are available: a parallel section rotor which is espe-

cially suitable for soft, elastic and fibrous materials. And a 6-disc rotor with replaceable and reversible tungsten carbide cutting plates. For the SM 300 the V-rotor is available which is especially suitable for tough, soft and fibrous material. Further accessories include bottom sieves and rotors made of steels which are suitable for heavy-metal-free size reduction.

Knife Mills Grindomix GM 200 and GM 300

The **Knife Mills Grindomix GM 200** and **GM 300** are suitable for the size reduction and homogenization of samples with a high fat, oil or water content. The mills are frequently used in food control laboratories.

Whereas the GM 200 can process sample volumes from 300 - 700 ml, the GM 300 homogenizes sample amounts of up to 4 500 ml.

A wide range of accessories is available for the knife mills. The standard container of the GM 200 is made of polypropylene. Additionally containers of stainless steel, polycarbonate and borosilicate glass are available which can be sterilized and autoclaved. The GM 300 features containers from polycarbonate and stainless steel which can also be autoclaved.

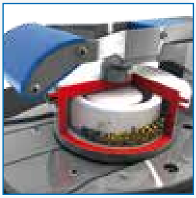
When using a gravity lid the volume of the container is reduced and automatically adapted to the sample amount. For samples with a high liquid content gravity lids with overflow channels are best suited, as the liquid contents of the sample ascend the container walls and are returned to the center of the container.

For heavy-metal-free grinding and homogenization processes neutral-to-analysis knives are available.

3.4. Disc Mills

Vibratory Disc Mill RS 200 (left)
Disc Mill DM 200 (right)

The **Vibratory Disc Mills RS 200** and **RS 300 XL** are frequently used for the sample preparation to XRF analysis. They are suitable for the fine size reduction of medium-hard and hard materials such as slag, ore, cement clinker and minerals with a maximum feed size up to 20 mm. Depending on the material and the selected parameters a final fineness down to 20 µm can be achieved. A fineness of <100 µm can usually be obtained after only 1 - 2 minutes of grinding. Grinding sets are available in the sizes 50, 100, 250, 800, 1000 and 2000 ml in up to six different materials (depending on model).



Size reduction in the RS 200 is effected by pressure and friction between the grinding jar wall, disc and ring which move in horizontal circles. The speed can be selected in a range from 700 to 1 500 min^{-1}.

The preliminary and fine size reduction of hard-brittle materials is effected in the **Disc Mills DM 200** and **DM 400** through pressure and friction between two vertical grinding discs, one of which is fixed and the other one rotates. The maximum feed size is up to 20 mm. The final fineness is determined by the gap width setting of the grinding discs and is around 0.1 mm. The grinding discs are available in 4 different materials.

3.5. Mortar Grinder

Mortar grinders are used for grinding, mixing and triturating soft, hard and brittle materials. Mortar grinders are dust-tight and therefore ideally suited for wet grindings and trituration of pastes.

Size reduction in mortar grinders is effected by pressure and friction between mortar and pestle. The pestle pressure can be set manually and the position of scraper and pestle is adjustable.

Mortar Grinder RM 200

The RETSCH **Mortar Grinder RM 200** features a useable volume of 10 to 190 ml. The maximum feed size is up to 8 mm. Depending on the properties of the sample material, a final fineness down to 10 µm can be achieved. Grinding tools are available in seven different materials.

3.6. Ball Mills

Ball mills are frequently used for the fine size reduction of hard-brittle materials. A crucial advantage of ball mills is their great versatility. Grinding jars and balls are available in various sizes and materials, e.g. agate and ceramics such as zirconium oxide. This is important if the sample is analyzed for heavy metals.

The grinding tools for ball mills always consist of a grinding jar and grinding balls made of the same material (exception: XRD mill). The following rule of thumb can be applied for the jar filling: 1/3 is filled with balls, 1/3 with sample material. When choosing the ball size, the feed size of the sample and the desired final fineness have to be taken into account. 30 mm grinding balls, for example, are suitable to reduce particle sizes of approx. 10 mm while for colloidal grindings which produce a fineness down to the submicron range grinding balls of only 2 or 3 mm are used. Due to the dust-tight closure of the grinding jars, ball mills are also suitable for wet grindings.

Planetary Ball Mill PM 100

RETSCH offers 4 models of **Planetary Ball Mills**: the **PM 100** and **PM 100 CM** with one grinding station, the **PM 200** with two grinding stations and the floor model **PM 400** with four grinding stations. Grinding jars are available in six different materials with volumes from 12 to 500 ml. The maximum feed size is 10 mm. Depending on the material properties a final fineness down to 100 µm can be achieved with dry grinding.

A final fineness below 10 µm down to the nano range can only be achieved with wet grinding. Usually, grinding balls with small diameters (e.g. 3 mm) are used for this. Approximately 60 % of the jar volume is filled with grinding balls. A dispersing agent such as water or alcohol is added to the sample. Care must be taken that the dispersing agent does not dissolve the sample or change its chemical properties. For wet grinding it is recommended to use a safety closure device, because the long grinding times (of several hours) increase the temperature inside the jar which can lead to the buildup of overpressure.

The grinding jars are arranged eccentrically on the so-called sun wheel. The rotational movement of the sun wheel is opposite to that of the grinding jars in a ratio of 1:-2. Size reduction is effected through impact and friction.

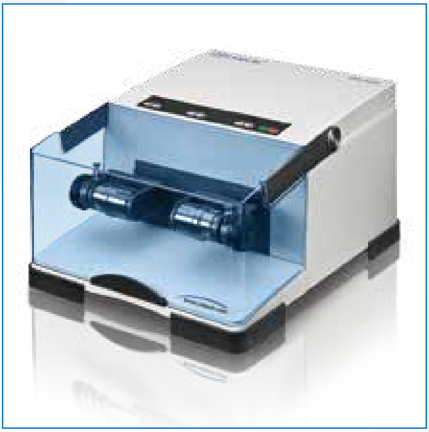
Planetary Ball Mill PM 400

Measurement System PM GrindControl

The Planetary Ball Mill PM 100 CM operates in centrifugal mode, i.e. the speed ratio of sun wheel to grinding jar is 1:-1. Size reduction with the PM 100 CM is effected through friction and pressure, which leads to a more gentle size reduction process with less abrasion.

RETSCH's planetary ball mills can also be used for mechanical alloying where mixtures of metals and metal oxide powders are ground for several hours to form new materials with new properties. A speed ratio of 1:-2 is usually sufficient for ductile metals whereas for hard-brittle materials, such as covalent-bonded semiconductors, a higher energy input is required. For this type of material the **PM 400 MA** with a speed ratio of 1:-2.5 or 1:-3 is the most suitable.

To monitor the processes which occur during grinding, RETSCH offers the measurement system **PM GrindControl** which is used to document pressure and temperature inside the grinding jar.



Mixer Mill MM 400

The **Mixer Mills MM 200** and **MM 400** are suitable for grinding small sample quantities. The grinding jars perform radial oscillations in a horizontal position. Size reduction is effected through impact forces. When used with a great number of small grinding balls (for an increased inner friction), mixer mills are also suitable for the disruption of biological cells.

For the preparation of pellets for XRF analysis, the previously ground sample can be mixed and homogenized with wax (binding agent) using polyamide or steatite balls.

The MM 400 has a greater oscillation radius than the MM 200 which results in an approx. 30 % higher energy input. This means that a greater fineness can be achieved with shorter grinding times. Grinding jars for the MM 400 have a size range from 1.5 to 50 ml. They have a screw-top lid which makes them suitable for wet grinding. Another option is the use of different adapters which hold up to 20 x 2 ml or 6 x 5 ml reaction vials or 8 x 50 ml centrifugation tubes; those single use vials can all be used for efficient cell disruption (bead beating) or homogenization of biological tissue material like liver.

The MM 400 can also be used for cryogenic grinding. The stainless steel grinding jars which are filled with balls and sample material are cooled for 2-3 minutes in liquid nitrogen and are then fastened in the quick-clamping device.

The **CryoMill** has been specially designed for cryogenic grinding. The grinding jar is continually cooled with liquid nitrogen from the integrated cooling system before and during the grinding process. Thus the sample is embrittled and volatile components are preserved. The liquid nitrogen circulates through the system and is continually replenished from an autofill system in the exact amount which is required to keep the temperature at −196 °C. This results in reduced consumption and guarantees reproducible grinding results.

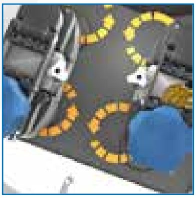
CryoMill

Various grinding parameters can be stored which helps to simplify routine tasks. LEDs in the display indicate the current state of operation, e.g. cooling or grinding.

The size reduction principle is the same as that of the MM 400. With a vibrational frequency of 30 Hz the CryoMill grinds most materials very effectively in a few minutes. It is equipped with one grinding station for grinding jar volumes of 25 ml, 35 ml and 50 ml. Another option is the use of an adapter which holds up to six 2 ml grinding jars.

The CryoMill can also be operated without cooling which makes it suitable for a vast range of applications.

High Energy Ball Mill E_{max}



The **High Energy Ball Mill E_{max}** is an entirely new type of ball mill for high energy milling down to the nanometer range.

The novel size reduction mechanism of the E_{max} unites the advantages of different mill types: high-frequency impact (mixer mill), intensive friction (vibratory disc mill) and controlled circular jar movement (planetary ball mill) allow for unrivalled grinding performance. This unique combination is generated by the oval shape and the movement of the grinding jars.

The grinding jar brackets are mounted on two discs each which turn in the same direction. As a result, the jars (sizes 50 or 125 ml) move on a circular course without changing their orientation. The interplay of jar geometry and movement causes strong friction between grinding balls, sample material and jar walls as well as rapid acceleration which lets the balls impact with great force on the sample at the rounded ends of the jars. This significantly improves the mixing of the particles resulting in smaller grind sizes and a narrower particle size distribution than achieved in ball mills.

Grind sizes on a nanoscale can only be achieved by wet grinding. For this method a large number of grinding balls with diameters of 0.1 mm to 3 mm is used to create as much friction as possible. The resulting grinding energy is extended even further by the high speed of 2000 min^{-1} in the E_{max}. The high energy input is fully exploited as the unique liquid cooling system quickly discharges the frictional heat.
Depending on the sample characteristics and grinding mode, cooling breaks of approx. 60% of the total grinding time are recommended for conventional planetary ball mills to prevent overheating. The E_{max}, on the other hand, is suitable for continuous grinding without breaks thanks to its efficient liquid cooling system.

The **XRD-Mill McCrone** was specially developed for the preparation of samples for subsequent X-ray diffraction (XRD).

The XRD-Mill McCrone carries out size reduction mainly by friction. 48 cylindrical grinding elements are placed into the grinding jar in eight rows of six elements each.
During operation, the jar's circular motion causes the elements to grind the sample from < 0.5 mm to the low µm-range (typically < 10 µm).
Thanks to the very gentle size reduction process, the crystal lattice of the sample is preserved. This makes the XRD-Mill McCrone the instrument of choice for the sample preparation for subsequent X-ray diffraction analysis

XRD-Mill McCrone

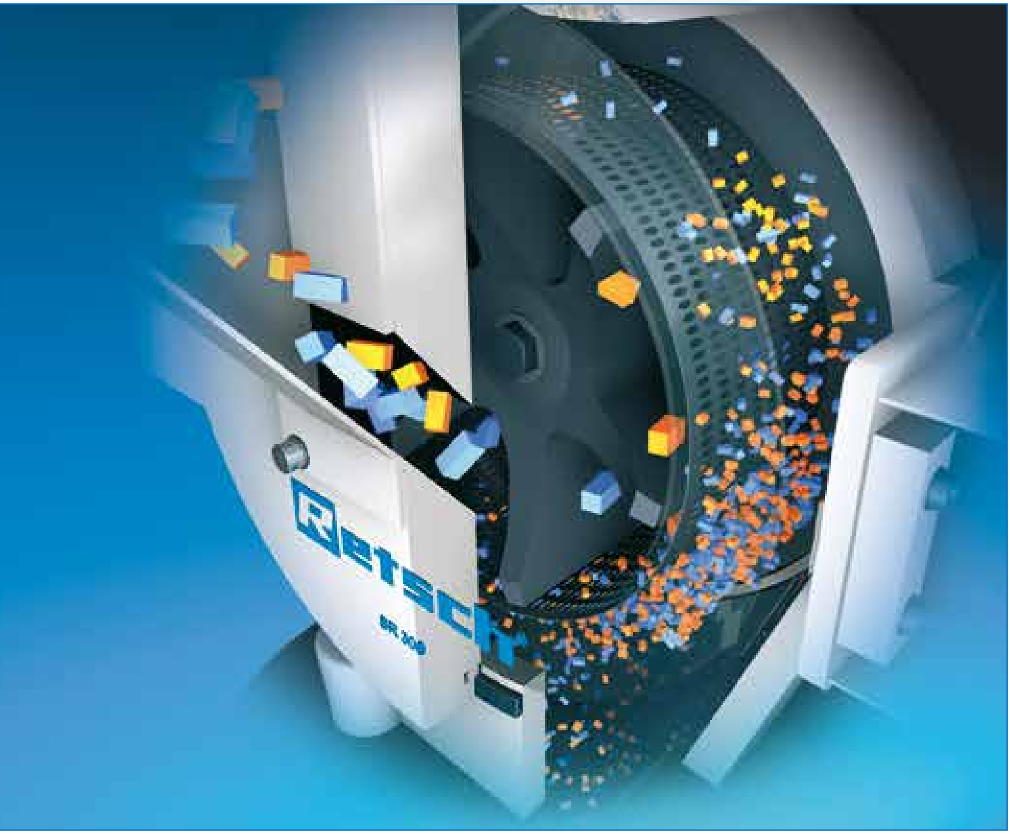
The **Drum Mill TM 300 XL** is used for the preparation of granules and powders.
The grinding process is performed either in dry or wet conditions with variable speed up to 80 min^{-1}. The drum mill can be operated either as a Ball or as a Rod Mill by using the corresponding module. A sufficient number of balls or rods is required for an effective grinding process. Typically, a final fineness below 20 µm is obtained.

Drum Mill TM 300 XL

4. Conclusion

For many applications size reduction is an important step in the process of sample preparation. In this expert guide we have presented the different size reduction principles and have discussed which type of mill is best suited for which type of sample.

New challenges in research and applications technology as well as increasing precision of instrumental analysis have lead to increasing requirements and a continuous optimization of size reduction instruments and grinding tools.



Annex: Tips & Tricks for Ball Milling

How to find the correct grinding ball size?

When choosing the ball size, the initial feed size and the required final fineness of the sample have to be taken into account. Some rules of thumb help to find the adequate ball sizes:

- The grinding balls should be 3 x bigger than the biggest particles of the sample to provide enough energy to crush the particles. Consequently, particles with an initial size of e.g. 10 mm are crushed most effectively with grinding balls of 30 mm in diameter.

- Note: If very hard sample material is to be pulverized, it may be advantageous to use bigger balls which generate a higher energy input. If, however, the sample tends to cake, less energy input is required which means smaller grinding balls.

- Generally speaking, the final fineness which can be obtained with grinding balls of a certain diameter is approximately 1/1000 of that size. This means that 30 mm grinding balls are required when the initial particle size is 10 mm, and a final fineness of approximately 30 µm can be obtained. To achieve smaller particle sizes, two or more grinding steps with different ball sizes may be necessary.

Important: Never mix different ball sizes in one grinding step. The bigger ones would crush the smaller grinding balls, leading to contamination of the sample. Always use grinding jars and grinding balls of the same material (e.g. stainless steel) to minimize wear of the grinding tools.

Initial particle size

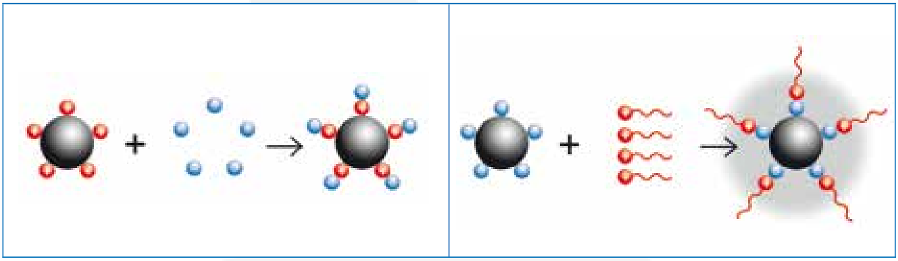
Grinding balls 3 times bigger
than the biggest particle

Required particle size

3 mm balls ➡ down to 3 µm particles
...
0.1 mm balls ➡ down to 0.1 µm particles

Maybe two or more grinding steps are needed to obtain very fine particles , including intermediate change(s) of the ball size.

For final sizes <10 µm wet grinding is the only option. Due to their significantly enlarged surface in relation to their volume, small particles are attracted to each other by their electrostatic charges. With the addition of dispersants such as water, buffer or alcohol the charges on the surfaces can be neutralized.



Neutralization of charged particles by adding a buffer (electrostatic stabilization, left) or by adding long-chained molecules (steric stabilization, right)

Recommendations for dry and wet grinding (colloidal grinding)

Dry grinding - The following rule of thumb should be obeyed when filling the jar for dry grinding: 1/3 is filled with balls, 1/3 with sample material, 1/3 remains empty to leave enough room for the ball movement. Mostly, balls >3 mm are used. The impact forces needed to crush larger particles increase with the size of the grinding balls.

Wet grinding - A final fineness below 10 µm can only be obtained by wet grinding for which small grinding balls of 0.1 - 3 mm (colloidal grinding) are used. Approximately 60 % of the jar volume is filled with grinding balls to increase the friction surface required for producing very fine particles. Grinding balls made of an abrasion-resistant material like zirconium oxide are recommended for wet grinding to minimize wear and abrasion. 30 % of the grinding jar volume is filled with sample material.
A dispersing agent like water or alcohol is added to the sample until the mixture shows a consistency like engine oil. It is important to choose a dispersing agent that does not dissolve the sample or change its chemical properties. If the sample has a tendency to swell, add more dispersant right from the beginning or after 5 or 10 minutes of grinding.

Removal of the grinding jar

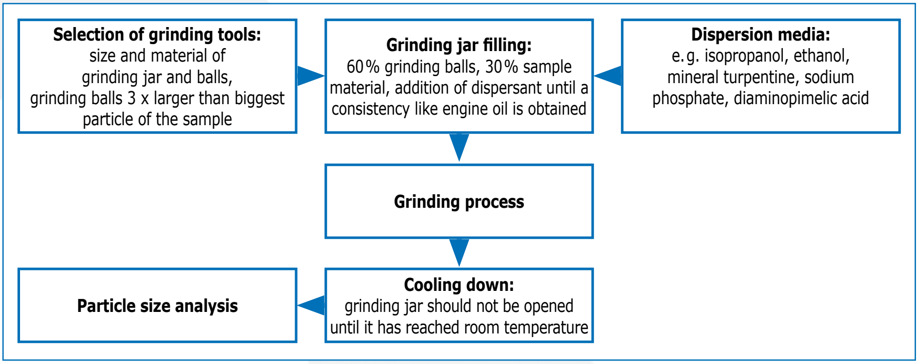
Care should be taken when removing grinding jars from planetary ball mills after grinding as they may be very hot (~ 150 °C). Heat is generated during the grinding process and pressure inside the grinding jar increases. Therefore, it is advisable to use the optional safety closure device for the "comfort" grinding jars of the PM series which allows for safe removal of the jar. After finishing the grinding process the jar should cool down for a while.

The E_{max} jar is already equipped with an integrated safety closure. Moreover, the effective cooling system of the E_{max} prevents the jars from heating up too much. The jars for the Planetary Ball Mills and the E_{max} can be equipped with optional aeration covers which allow working under inert atmosphere.

Attached safety closure device and aeration lid of PM jars (left) and E_{max} jar with integrated safety closure (right).

How to separate the grinding balls from the sample after wet grinding

Pour the content of the jar on a test sieve (with aperture sizes 20 % to 50 % smaller than the balls) with collecting pan. The vibratory movement of the sieve shaker helps to separate the sample material from the balls. Wet material tends to stick to the balls due to capillary forces. Flushing with more dispersant to wash away the sample helps to increase sample recovery.



Grinding process and parameters for wet grinding.

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The percentages of the chemical components of the materials are rounded. Only the major or characteristic components have been considered. Therefore, the percentage values do not necessarily add up to 100 %.
More detailed information, also listing the minor components, can be found in the pdf-document "Material Analyses of Grinding Tools" on www.retsch.com.

A-1. Metals

A-1.1. Steel

What is steel?

Steel is an alloy whose major component is iron with a carbon content that is usually less than 2 %.

Varying the amount of alloying elements (such as chrome or manganese) and their distribution in the steel controls qualities such as the hardness, elasticity, ductility and tensile strength of the resulting steel. There are various procedures for manufacturing steel which, however, will not be discussed here.

RETSCH use the following types of steel for their instruments and accessories:

- Stainless steel
- Hardened steel (“chrome steel”)
- Steel for grinding without heavy metal contamination
- Manganese steel (“precision-cast manganese steel”)

Stainless steel

The term “stainless steel” refers to corrosion-resistant steel. The corrosion resistance of this type of steel is due to a very thin, not visible oxidic protective film which is formed when the chrome content is >12 %. The corrosion resistance increases with the chrome content. However, if the chrome content is more than 20 %, the steel loses its hardenability and thus its wear resistance.

Hardness

48 to 52 HRC (Rockwell)

Density

7.8 g/cm³

Composition

Fe: 62 – 84.5 %
Cr: 13 – 19.5 %
Ni: <14 %

Constant subjection to mechanical stress, as is the case with grinding tools, can lead to the destruction of the protective film and can also roughen the surface. This can result in cross contamination as well as the forming of corrosive spots on the material. These are not a sign of low quality but are caused by wear.

To remove corrosion stains, dirt or rust from the grinding tools standard brushes or cleaning agents for metals can be used. After wet cleaning it is recommendable to clean the parts with isopropanol or acetone and to dry them thoroughly.

Hardened steel ("chrome steel")

Hardened steel, just like stainless steel, belongs to the group of chrome steels. Its chrome content however is not more than 12%. Consequently, corrosion resistance is not the strong point of this type of steel but its great hardness.

Hardness

58 – 63 HRC (Rockwell)

Density

7.85 g/cm³

Composition

Fe: 84 – 85%
Cr: 12%

Steel for grinding without heavy metal contamination (e. g. steel 1.1750, St 1203)

These steels are chromium- and nickel-free and can be used for sample preparation for analysis of heavy metals, provided that possible contamination with iron is acceptable.

Heavy-metal-free steel has a high carbon content which allows for a hardness up to 62 HRC (Rockwell). Due to the high iron content of 97 - 99% it is not resistant to corrosion and should therefore be cleaned and stored with great care. This is especially important after the tools have come into contact with wet sample materials to avoid the formation of

Hardness

up to 62 HRC (Rockwell)

Density

7.85 g/cm³

Composition

Fe: 98 – 99%

rust. Grinding tools which have not been used for a long time can be protected with a thin oil or fat film against corrosion.

Hardness

up to 55 HRC (Rockwell)

Density

7.2 g/cm^3

Composition

Fe: 85%
Mn: 13%

Manganese steel ("precision-cast manganese steel")

Manganese steel is also called Hadfield steel after its inventor. The manganese content lies between 12 - 14%, the carbon content between 1 - 1.2%.

After the 1100°C metallic melt is quenched, the material still shows a very ductile structure. Subjected to impulsive pressure, e.g. in jaw crushers, a part of the structure could be modified. Thus manganese steel can obtain a hardness of 600 HV (approx. 55 HRC). The technical term for this process is called strain hardening.

Applications:
e.g.
- *Mineralogy*
- *Construction materials*
- *Plastic*
- *Fabrics*
- *Secondary fuels etc.*

Sample materials:
- *Minerals*
- *Ores*
- *Cement*
- *Polymers*
- *Circuit boards*
- *Paper*
- *Feed pellets*

Application areas of steel grinding tools

Stainless steel

Grinding tools of stainless steel are frequently used for mineral and metallurgical samples. Further suitable materials are coal, coke and slag as well as all raw materials and intermediate products for the production of cement and lime.

As chrome and nickel are substantial parts of the alloy, stainless steel is not suitable for the preparation of samples for the subsequent determination of heavy metals.

When used with hard and abrasive samples, grinding tools of stainless steel may not have the necessary degree of wear resistance, especially when the main stress mechanism is friction.

Hardened steel ("chrome steel")

Hardened steel grinding tools are used for applications where stainless steel would wear too quickly, e.g. for the cutting plates of cutting mills or, generally, when hard, abrasive sample materials have to be pulverized. They are also highly suitable for mills which pulverize with friction.

Steel for grinding without heavy metal contamination (e. g. steel 1.1750, St 1203)

Crushers and grinders whose stress mechanism does not allow for ceramic materials – such as jaw crushers, cutting mills or cross beater mills – are usually equipped with heavy-metal-free grinding tools.

Heavy-metal-free steel can be used for the preparation of samples which will be analyzed for their heavy metal contents provided that possible contamination with iron is acceptable. Examples: Size reduction of secondary fuels in cutting mills or of soils in jaw crushers/cross beater mills.

Manganese steel ("Precision-cast manganese steel")

Manganese steel is used for the breaking jaws in jaw crushers as it is ideal for wear-resistant tools which are subjected to pressure.

Advantages

- Wide range of applications due to different types of steel with different properties
- Very high energy input
- Low price

Disadvantages

- Not absolutely wear-resistant (contamination with iron and other components is possible)

Available grinding tools made of steel:

Stainless steel
- Jaw Crushers BB 50, BB 100, BB 200, BB 300
- Ultra Centrifugal Mill ZM 200
- Rotor Beater Mills SR 200, SR 300
- Cross Beater Mill SK 100
- Cutting and Knife Mills GM 200, GM 300, SM 100, SM 200, SM 300
- Mortar Grinder RM 200
- Planetary Ball Mills PM 100, PM 200, PM 400
- High Energy Ball Mill E_{max}
- Mixer Mills MM 200, MM 400, CryoMill

Hardened steel ("chrome steel")
- Cross Beater Mill SK 100 (baffle plates, grinding inserts)
- Cutting Mills SM 100, SM 200, SM 300 (knife for parallel section rotor)
- Disc Mills RS 200, RS 300 XL, DM 200, DM 400
- Mortar grinder RM 200
- Planetary Ball Mills PM 100, PM 200, PM 400
- Mixer Mills MM 200, MM 400, CryoMill

Steel for grinding without heavy metal contamination (e. g. steel 1.1750, St 1203)
- Jaw Crushers BB 50, BB 100, BB 200, BB 250 XL, BB 300, BB 400 XL
- Cross Beater Mill SK 100
- Cutting Mills SM 100, SM 200, SM 300

Manganese steel ("precision-cast manganese steel")
- Jaw Crushers BB 50, BB 100, BB 200, BB 250 XL, BB 300, BB 400 XL, BB 500 XL, BB 600 XL
- Disc Mills DM 200, DM 400

A-1.2. Cast Iron / Malleable Iron

What is cast iron / malleable iron?

Cast iron is a ferrous alloy with a carbon content of >2 % and a silicon content of >1.5 %. In the so-called grey cast iron carbon is present in the form of fine irregularly distributed graphite flakes. The term "cast iron" refers to the production process of this material which involves casting a metal melt into forms.
As opposed to cast iron malleable iron is tempered for several days at a temperature of approx. 1000 °C in oxidizing atmosphere.

Application areas of cast iron/malleable iron grinding tools

Cast iron is an inexpensive alternative to steel. However, due to its low hardness it is not used frequently. Malleable iron is more ductile than cast iron and allows for minor plastic deformations without breaking. Therefore, it is mainly used for tools which have to bear hammering effects such as the cross beater in the cross beater mill SK 100. The baffle plates which are attached to the cross beater are made of steel because stress and wear are especially strong there.

Advantages
- Insensitive to hammering effects
- Very high energy input
- Low price

Disadvantages
- Limited wear resistance (contamination with Fe and other components possible)
- Lower hardness than steel

Cast iron / malleable iron grinding tools are available for

- Rotor Beater Mill SR 200 (housing, inlet, outlet)
- Cross Beater Mill SK 100 (grinding insert, cross beater)

Tensile strength

Cast Iron:
200 – 300 N/mm²
Malleable Iron:
approx. 370 N/mm²

Density

Cast Iron:
8.25 g/cm³
Malleable Iron:
7.2 g/cm³

Composition

Cast Iron:
Fe: 94 %
C: 4 %
Malleable Iron:
Fe: 95.5 %
C: 3 %

Applications:
e.g.
- *Mineralogy*
- *Construction*
- *Agriculture*

Sample materials:
- *Minerals*
- *Ores*
- *Cement*
- *Feed stuffs*

A-1.3. Titanium

Hardness

6 Mohs

Density

4.5 g/cm^3

Composition

Ti: 99.5%

Applications:
e.g.
- *Pharmaceutics*
- *Food*
- *Heavy metal analysis*

Sample materials:
- *Vegetables*
- *Fruit*
- *Meat*
- *Drugs*
- *Cables etc.*

What is titanium?

Titanium (Ti) is a chemical element with the atomic number 22. It belongs to the group of transition metals and is very strong despite its relatively low density. Titanium forms a protective oxide coating which makes it resistant to corrosion. In its purest form it is ductile. In order to improve its mechanical properties (embrittlement), other elements such as oxygen and nitrogen can be added.

Titanium is one of the most abundant elements in the Earth's crust but it mostly occurs in low concentrations in the form of oxides. It is mainly extracted from the minerals rutile (TiO_2) and ilmenite ($FeTiO_3$).

Application areas of titanium grinding tools

RETSCH supplies titanium grinding tools for the Ultra Centrifugal Mill ZM 200 and for the Knife Mill Grindomix GM 200 for grinding without heavy metal contamination. Ceramic materials are not suitable for these mills as they could break easily.

Advantages
– No heavy metal

Disadvantages
– Expensive
– Less hard than steel

Titanium grinding tools are available for

- Ultra Centrifugal Mill ZM 200 (rotor, sieve, cassette)
- Knife Mills Grindomix GM 200, GM 300

A-2. Ceramics

A-2.1. Tungsten Carbide

What is tungsten carbide?

Tungsten carbide is a hard metal. Pure tungsten carbide, a mixed crystal of tungsten and carbon, is very brittle. Therefore, 6 - 10 % of cobalt is added which increases the toughness of the material and reduces abrasion. The appropriate material composition of this hard metal depends on the technical requirements of the application.

Tungsten carbide is produced by sintering tungsten and carbon. The components are heated under high pressure and carbon is incorporated into the crystal structure of the tungsten. The resulting carbide increases the melting point and the hardness of the metal.

The extreme hardness (even at high temperatures) and wear resistance are characteristic for hard metals. Depending on the composition, hard metal can be as hard as diamond. Therefore, it is frequently used for chipping tools and non-cutting moulding tools. It is also used for tools which are subjected to frictional abrasion. These include grinding tools for mechanical size reduction as used in RETSCH mills and crushers.

Hardness

approx. 1250 HV (Vickers)

Density

14.8 g/cm³

Composition

WC: 90 – 94 %
Co: 6 – 10 %

Application areas of tungsten carbide grinding tools

Due to their wear-resistance and hardness, hard metals are brittle and therefore sensitive to impact effects. Also, sudden temperature changes, e.g. during cryogenic grinding, can cause the material to break. Therefore, not all our mills can be equipped with grinding tools of hard metal (e.g. Rotor Beater Mills).

The composition of the hard metal as well as the production process determine to a certain extent properties such as pressure resistance, resistance to bending, toughness, high

Applications:
e.g.
- *Mining and metallurgy*
- *Chemistry*
- *Mineralogy*

Sample materials:
- *Ores*
- *Ferro alloys*
- *Metal oxides*
- *Cement clinker*
- *Charcoal*
- *Minerals*

density and hardness. As a result, the percentage of tungsten carbide and cobalt in our grinding tools can vary. However, it is always ideally adapted to the stress mechanism of each mill or crusher. Grinding tools for planetary ball mills, for example, have to withstand friction and impact effects and therefore have a higher tungsten carbide content. Breaking jaws in jaw crushers, however, have to withstand high pressures and therefore contain more cobalt to increase their pressure resistance.

Advantages
- High wear resistance and elevated-temperature hardness
- Excellent grinding results due to high material density (especially in grinding processes with free-to-move grinding balls)
- High energy input

Disadvantages
- Cannot be used, if possible contamination with tungsten or cobalt (metal) influences the subsequent analysis
- If the material is soft, the sample may cake during grinding
- Warming of the sample material

Grinding sets made of hard metal are available for

- Jaw Crushers BB 50, BB 100, BB 200, BB 250 XL, BB 300, BB 400 XL, BB 500 XL, BB 600 XL
- Cutting Mills SM 100, SM 200, SM 300 (reversible cutting plates for 6-disc-rotor)
- Disc Mills RS 200, DM 200, DM 400
- Mortar Grinder RM 200
- Planetary Ball Mills PM 100, PM 200, PM 400
- Mixer Mills MM 200, MM 400
- High Energy Ball Mill E_{max}

A-2.2. Zirconium Oxide

What is zirconium oxide?

The main component for the production of zirconium oxide (ZrO_2) is the mineral zircon ($ZrSiO_4$). By melting it with coke and lime (reduction of SiO_4) ZrO_2 is won on an industrial level. The resulting powder is mixed with aggregates and dry pressed into the desired form. The formed part is transferred to the raw product by sintering and is then abraded and polished according to its intended purpose.
The sintering process can be carried out under atmospheric pressure as well as under high pressure. The formed parts receive their actual properties during the sintering.

Zirconium oxide occurs in different crystal modifications, depending on the temperature. These have different volumes. By adding yttrium oxide, zirconium oxide is prevented from turning into the modification which is more stable at room temperature – it is thus kept in the partially stabilized form. If there's a micro crack, the zirconium oxide changes at that specific point by volume extension into the more stable modification. This effect causes the crack to close immediately.

Zirconium oxide is traditionally used as a refractory ceramic. Due to its properties and high bio-compatibility zirconium oxide is increasingly used as a ceramic for implants and dental prostheses. In addition, there are many technical applications for partially stabilized zirconium oxide. It is highly resistant to thermal, chemical and mechanical influences which makes it very suitable for grinding tools.

Hardness

approx. 7.5 Mohs
(approx. 1200 HV)

Density

5.9 g/cm^3

Composition

ZrO_2: 94.5%
Y_2O_3: 5%

Applications:
e.g.
- *Biotechnology, medicine*
- *Analysis of trace elements and heavy metals*

Sample materials:
- *Soils*
- *Sewage sludge*
- *Compost*
- *Plants*
- *Bones*
- *Teeth*
- *Tissue*

Application areas of zirconium oxide grinding tools

Zirconium oxide has a high density and mechanical wear resistance. It is free of heavy metals and can therefore be used for sample preparation to heavy metal analysis. Further areas of application are biology, the preparation of plant and animal tissue, as well as human medicine, where any kind of contamination can be hazardous. Zirconium oxide only leaves neutral contamination, if at all, for this type of application.

RETSCH uses zirconium oxide for ball mill and disc mill grinding tools. The high density of the material allows for a high energy input. As zirconium oxide is a rather brittle ceramic, it is important to follow the recommendations for sample amount and maximum feed size to avoid damages to the material. It is also used in the jaw crusher BB 50 for heavy-metal-free grinding. However, brittleness and low resistance to impact are the reasons why zirconium oxide is not used in the bigger jaw crushers or in rotor mills.

Advantages
- No heavy metal
- High density
- High wear resistance

Disadvantages
- Expensive
- Limited suitability for mills which comminute by impact and hammering

Zirconium oxide grinding tools are available for

- Jaw Crusher BB 50
- Disc Mills RS 200, DM 200, DM 400
- Mortar Grinder RM 200
- Planetary Ball Mills PM 100, PM 200, PM 400
- Mixer Mills MM 200, MM 400
- High Energy Ball Mill E_{max}
- XRD-Mill McCrone

A-2.3. Sintered Aluminium Oxide

What is sintered aluminium oxide?

Sintered aluminium oxide (Al_2O_3) is a synthetic ceramic material. In nature Al_2O_3 occurs in form of corundum, the second hardest mineral after diamond. Due to its hardness Al_2O_3 often is used as abrading medium.

When producing sintered aluminium oxide, pressed alumina powder is fired to 1300 °C. The powder is an intermediate product in the process of winning aluminium from bauxite. The main reasons for using sintered aluminium oxide in grinding tools are its considerable hardness and purity but also the reasonable price. Due to the relatively low density of sintered aluminium oxide and the low energy input which results from this, the sample material is hardly subjected to thermal stress during grinding. This can be an advantage with sensitive materials such as plants.

Hardness

approx. 8 - 8.5 Mohs
1750 HV (Vickers)

Density

3.9 g/cm³

Composition

Al_2O_3: 99.7%

Application areas of sintered aluminium oxide

Ceramics

The abrasion which is to be expected from sintered aluminium oxide can be considered as "neutral" as it is pure Al_2O_3. That is why one of the main areas of application for these grinding tools is the preparation of ceramic materials and glazes as found in sanitary and household ceramics. Refractory ceramics are also often prepared with grinding tools made of sintered aluminium oxide.

Homeopathic substances

As sintered aluminium oxide has a greater wear resistance than hard porcelain, it sometimes replaces this material in mortar grinders when homeopathic ingredients are prepared.

Applications:
e.g.
- *Biology*
- *Microbiology*
- *Ceramics*
- *Homeopathy*

Sample materials:
- *Ceramics*
- *Soils*
- *Grass*
- *Conifers*
- *Compost*

Heavy metal analysis

The relevant standards for heavy metal analysis in soils often recommend the use of ball mills with grinding sets made of zirconium oxide as suitable tools for the sample preparation. Due to the lower costs, grinding sets made of sintered aluminium oxide are also frequently accepted from an analytical point of view. Al_2O_3 has a hardness of 9 - 9.5 on Mohs' scale but less wear resistance than zirconium oxide. Therefore, it is not recommended to use them in planetary ball mills at high speeds.

Advantages

- No heavy metal
- Low heat build-up / sample doesn't cake
- good wear resistance
- Reasonable price

Disadvantages

- Susceptible to shock by impact and temperature
- Low energy input

Grinding sets made of sintered aluminium oxide are available for

- Mortar Grinder RM 200
- Planetary Ball Mills PM 100, PM 200, PM 400
- Mixer Mills MM 200, MM 400
- XRD-Mill McCrone

A-2.4. Hard Porcelain

What is hard porcelain?

Hard porcelain is a silicate ceramic which is composed of the raw materials kaolin (non-ferrous clay), potash, feldspar and quartz. It consists of 25 - 70 % Al_2O_3 and 30 - 75 % SiO_2. Hard porcelain is fired to a temperature of approx. 1400 °C.
In the middle of the 19th century, porcelain was first used as engineering ceramics, mainly in the electronic industry where it remains an important material until today (e.g. as insulators).

Hardness

approx. 6 – 7 Mohs (approx. 1200 HV)

Density

2.4 g/cm^3

Composition

SiO_2: 68.5 %
Al_2O_3: 26 %

Application areas of hard porcelain grinding tools

A major field of application is the trituration of substances used in homeopathy and medicine with the help of mortars. Therefore, RETSCH only uses hard porcelain for the grinding tools in the mortar grinder RM 200.

Advantages
- Reasonable price
- Heavy-metal-free

Disadvantages
- Low wear resistance against abrasive sample materials -> rough surface

Applications:
e.g.
- *Pharmaceutical industry*
- *Medicine*
- *Biology*

Sample materials:
- *Plants*
- *Pharmaceuticals*
- *Oil seeds*
- *Pastes*

Hard porcelain grinding tools are available for

- Mortar Grinder RM 200

A-2.5. Silicon Nitride

Hardness

1500 HV

Density

3.2 - 3.4 g/cm^3

Composition

Si_3N_4 *92.5%*
Al_2O_3 *4.5%*
Y_2O_3 *2.5%*

What is silicon nitride?

Silicon nitride is a non-oxidic ceramic which stands out by extreme fracture toughness and wear resistance. The material does not occur naturally and is produced in different ways. One of the most popular manufacturing methods is reaction bonding: a compact of silicon powder is heated in a nitrogen atmosphere. The reaction in which the silicon powder is forming nitrides starts at 1200°C. Since silicon nitride decomposes at standard pressures and temperatures above 1700°C the powder has to be sintered under very high pressures. Silicon nitrides are characterized by a homogeneous grey colour.
By adding sinter additives (AL_2O_3 and Y_2O_3) the material hardness can be even increased whilst tensile strength remains. These ceramics are then termed SiAlONs.
Because of its high abrasion resistance silicon nitride is particularly well suited for the use in bearing technology and as cutting tool.

Applications:
e.g.
- *Biotechnology, medicine*
- *Analysis of trace elements and heavy metals*

Sample materials:
- *Soils*
- *Sewage sludge*
- *Ceramics*
- *Compost*
- *Plants*
- *Bones*
- *Teeth*
- *Tissue*

Application areas of silicon nitride grinding tools

The incredibly high abrasion resistance of silicon nitride qualifies it for heavy metal free fine grinding.This makes it perfectly suitable for the use in RETSCH Ball Mills.
Despite the high stability of the material, grinding jars should be filled and handled according to RETSCH recommendations in order to avoid unnecessarily high wear or abrasion.

Advantages
- No heavy metal
- high abrasion resistance
- Very good impact and shock resistance
- Excellent resistance to temperature changes

Disadvantages
- Low density
- More expensive than steel

Silicon nitride grinding tools are available for

- Planetary Ball Mills PM 100, PM 200, PM 400

A-3. Other Materials

A-3.1. Agate (Natural Stone)

Hardness

6.5 – 7 Mohs
(approx. 1000 HV)

Density

2.65 g/cm³

Composition

SiO_2: 99.9%

What is agate?

Agate is a quartz-mineral (silicon dioxide SiO_2) and belongs to the group of semi-precious stones. It is formed in cavities of volcanic rocks through precipitation of SiO_2 from solutions. Agate was named from the river "Achates" (today "Drillo") in Sicily where it was found for the first time. Today's habitats include Bohemia, Brazil, Sicily and Uruguay.

At the beginning of the 20th century agate was first used as a technical material. Pharmacists and homeopaths soon recognized that agate hand mortars offered considerable advantages for the trituration of powders and pastes due to their pure SiO_2 composition and their wear resistance. Agate does not cause abrasion and is easy to clean due to its smooth surface which also helps to avoid cross contamination. These properties distinguish agate from brass or hard porcelain mortars.

The procedures for processing agate have improved through the years by using diamond tools which finally allowed for its use in a variety of grinding instruments. As a consequence, hard porcelain is gradually losing its significance as a material for mechanical size reduction.

Application areas of agate grinding tools

As mentioned before, agate consists mostly of silicon dioxide. This makes it ideal for grinding instruments which are used for neutral-to-analysis sample preparation as is required in fields such as food, biology, medicine and pharmaceutics. Further areas of application are sample preparation for heavy metal determination, trituration of pigments (metallic abrasion can lead to changes in the colour quality) or trituration of pasty substances and pharmaceutic ointments.Agate is resistant to pressure and friction but is susceptible to impact. Therefore, it is mainly used for mortar and pestle in mortar grinders. It can also be suitable for ball mills as, due to its relatively low density, it is resistant to the impingement effect which occurs in ball mills. The vibratory disc mill features a sensor which recognizes agate grinding sets and automatically reduces the speed to 700 min^{-1} so as to avoid premature damage of the grinding tools.

Agate is a natural product, therefore its quality can vary. Before processing it further, the raw material is checked for hollows and impurities. However, no uniform and homogeneous structure – like that of artificially produced materials – can be guaranteed. High mechanical and thermal stress over a long period of time can lead to premature material fatigue and even breaking of the grinding set. That is the reason why planetary ball mills should only run at 60-70 % of the maximum speed when used with agate grinding jars.

Thermal shocks which may occur when grinding with liquid nitrogen in mortar and mixer mills should also be avoided as this could lead to stress cracks in the material.

Advantages
- No heavy metal
- Traditional natural product (widely accepted)
- Hardly any thermal stress, samples don't cake

Disadvantages
- Susceptible to shock by impact and temperature
- Can be inhomogeneous
- Very low energy input

Agate grinding tools are available for

- Vibratory Disc Mill RS 200
- Mortar Grinder RM 200 and hand mortar
- Planetary Ball Mills PM 100, PM 200, PM 400
- Mixer Mills MM 200, MM 400
- XRD-Mill McCrone

Applications:
e.g.
- *Pharmaceutics*
- *Food*
- *Medicine*
- *Biology*

Sample materials:
- *Plants*
- *Spices*
- *Cocoa*
- *Chocolate*
- *Drugs*
- *Pastes*
- *Oil seeds*
- *Biological tissue and spores*

A-3.2. Glass

Hardness

approx. 6 Mohs

Density

2.2 g/cm³

Composition

SiO_2 : 80 %
B_2O_3 : 13 %

What is glass?

Glass is an amorphous, i.e. not crystalline, substance. It is usually produced from a melt which cools down very rapidly without sufficient time for a regular crystal lattice to form. Glass mainly consists of SiO_2. The properties of glass can be modified with the help of various additives.

Laboratories mostly use borosilicate glass due to its high chemical and temperature resistance. The chemical resistance is determined by the contents of boron oxide which amounts to approx. 13 %. Borosilicate glass is also known as "Jenaer Glass" or "Duran Glass".

Applications:
e.g.
- *Food*
- *Pharmaceutics*
- *Medicine*
- *Biology*

Sample materials:
- *Vegetables*
- *Fruit*
- *Fish*
- *Pharmaceuticals*
- *Biological tissues*
- *Cell disruption*

Application areas of glass grinding tools

Containers of borosilicate glass are available as an alternative to the standard polypropylene container for the knife mill Grindomix GM 200. They are suitable for products which are soft or have a high water or oil content. One of the crucial benefits is that glass containers can be easily cleaned, sterilized and autoclaved which makes them ideal for preparing food samples. Moreover, the grinding progress can be observed through the glass.
Another application area of glass are grinding beads which are used for cell disruption in the mixer mills MM 200 and MM 400. As these glass beads are economically priced, they can be used as disposable items together with the polypropylene reaction vials. Thus, cleaning of the grinding tools is no longer necessary.

Advantages
- Can be sterilized/ autoclaved
- Good chemical resistance
- Transparent

Disadvantages
- May break when subjected to impact

Glass grinding tools are available for

- Knife Mill Grindomix GM 200 (container)
- Glass beads for ball mills

A-3.3. PTFE (Plastic)

Hardness

D 56 (Shore)

Density

2.1 g/cm³

Composition

PTFE: 100 %

What is PTFE?

PTFE or *Teflon* is a thermoplastic (polymer). The abbreviation PTFE stands for polytetrafluoroethylene, a fluorine-carbon compound. The term "Teflon" is a trade mark of the company DuPont.

PTFE is practically inert which means that it is resistant to many chemicals such as acids, bases, alcohol and benzine. Therefore, PTFE coatings are often used as anticorrosive against aggressive substances. Further applications of PTFE are non-stick coatings (for pots and pans), high performance fabrics (Gore-Tex), sealing materials and medical technology (implants).

PTFE was developed in the 1930s while searching for a new cooling agent for refrigerators.

Applications:
e.g.
- *Pharmaceutics*
- *Medicine*
- *Biology*

Sample materials:
- *Plants*
- *Drugs*
- *Biological tissue*
- *Cell disruption*

Application areas of PTFE

Compared to other materials PTFE is less hard and should therefore only be used for grinding soft sample materials such as cell tissue. To generate a higher energy input, PTFE grinding balls are equipped with an iron core.

PTFE grinding tools are often used for biological and pharmaceutical samples because the material is inert and, due to its non-stick properties, PTFE can be easily cleaned. RETSCH's PTFE grinding jars are not protected with an iron jacket and, as PTFE is temperature-resistant, are therefore suitable for cryogenic grinding.

Advantages

- Inert (resistant to many chemicals, also aggressive ones)
- Temperature-resistant from -200 °C to +260 °C, suitable for cryogenic grinding
- No heavy metal
- Non-stick properties

Disadvantages

- Low hardness
- Low energy input
- Possible contamination with fluorine and carbon
- Low wear resistance

PTFE grinding sets are available for

- Mixer Mills MM 200, MM 400

A-3.4. Polypropylene (Plastic)

Density

0.89 - 0.92 g/cm³

Composition

PP: 100 %

What is Polypropylene?

Polypropylene (PP) is a thermoplastic resin (polymer) which is produced by polymerization from the hydrocarbon propene.

Polypropylene is frequently used in the laboratory sector. It is resistant to almost any organic solvent and fat as well as acids and bases.

Applications:
e.g.
- *Food*
- *Biology*
- *Medicine*

Sample materials:
- *Fruit*
- *Vegetables*
- *Cheese*
- *Sausage*
- *Tissue*
- *Cell disruption*

Application areas of polypropylene grinding tools

The standard container as well as the various lids for the knife mill Grindomix GM 200 are made of polypropylene. The material is suitable for products which are soft and soft-elastic or which have a high content of water or oil. The polypropylene container is an economically priced and break-proof alternative to the glass container.
Reaction vials for cell disruption are also made of polypropylene. These are available in volumes of 0.2 ml, 1.5 ml and 2 ml thus allowing the processing of very small sample quantities. The reaction vials are disposable items and therefore don't need any cleaning.

Advantages
- Inert (resistant to many [aggressive] chemicals)
- Economically priced

Disadvantages
- Low hardness

Polypropylene grinding tools are available for

- Knife Mill Grindomix GM 200
- Mixer Mills MM 200, MM 400 (safe-lock reaction vials)

A-3.5. Polycarbonate (Plastic)

What is Polycarbonate?

Polycarbonate is a thermoplast (polymer), which is produced by the esterification of carbonic acids with diols.

Due to its scratch and shock resistance polycarbonate is used e. g. as a protective film for data layers on CDs and DVDs. It is resistant to mineralic acids and most unpolar solvents, but not to acetone.

Density

1.2 g/cm^3

Composition

PC: 100%

Application areas of polycarbonate grinding tools

The standard container for the knife mill Grindomix GM 300 is made of polycarbonate. A polycarbonate container is also optionally available for the GM 200. In contrast to polypropylene polycarbonate offers the advantage that it is transparent and therefore allows observation of the grinding process. Additionally, polycarbonate can be autoclaved.

Advantages
- Transparent
- Can be autoclaved
- Reasonable price

Disadvantages
- Unstable against certain solvents like acetone

Applications:
e.g.
- *Food*
- *Feed stuffs*

Sample materials:
- *Fruit*
- *Vegetables*
- *Cheese*
- *Sausage*
- *Meat*
- *Cereals*

Polycarbonate grinding tools are available for

- Knife Mills Grindomix GM 200, GM 300

A-4. Annotations

A-4.1. Hardness

The hardness of a particular material can be given with different values, depending on the hardness scale to which this value refers (e.g. Mohs or Brinell). The different hardness scales have different origins. The Mohs' scale, for example, classifies the scratch hardness of minerals on the basis of a 10-step scale. The scales of Brinell (HB), Rockwell (HRA / HRB / HRC) and Vickers (HV) originate from the metallurgical sector. The Shore hardness is frequently used in the plastics industry.
Hardness is the mechanical resistance of a material against the penetration of a foreign material. In materials testing the hardness of a material is ascertained by determining the penetration depth of a defined body under a given pressure.

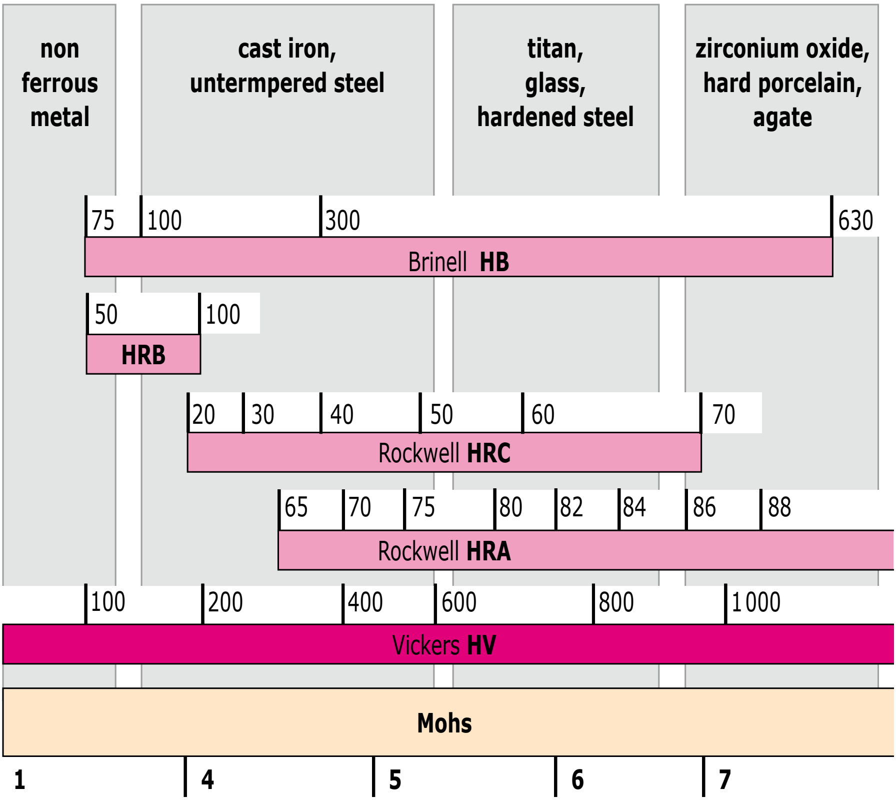
To convert the hardness values to another scale is not always possible. To facilitate a comparison of the scales of Mohs, Vickers, Rockwell (HRA / HRB / HRC) and Brinell, we have included the table on page 62/63.

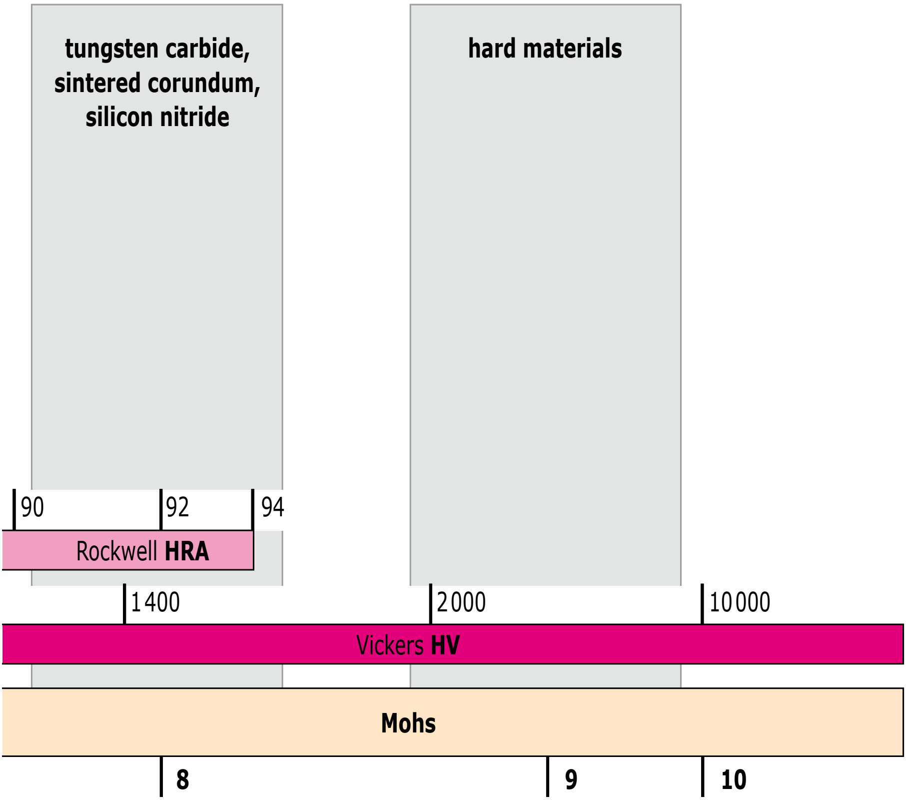
Materials are also characterized by their tensile strength which is sometimes given instead of the hardness, usually in N/mm^2. To determine the tensile strength, tensile force is applied to the material until it breaks. Elastic/plastic materials tend to deform (elongate) before breaking.

A-4.2. Chemical components

The percentages of the chemical components of the materials are rounded. Only the major or characteristic components have been considered. Therefore, the percentage values do not necessarily add up to 100 %. More detailed information, also listing the minor components, can be found in the pdf-document **"Material Analyses of Grinding Tools"** on www.retsch.com.

A-4.3. Hardness table (approximate values)




tungsten carbide, sintered corundum, silicon nitride
hard materials
90
92
94
Rockwell HRA
1400
2000
10000
Vickers HV
Mohs
8
9
10

Notes

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