

► The most efficient fine grinding takes place when both impact action and shearing force are present.

Media Milling Advances

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Particle size reduction has been around almost since the beginning of time and has undergone many changes. We've all seen the picture of the ox turning the millstone to grind grain, a method that is still used in many parts of the world today. In 1922, Dr. Szegvari hit upon the idea of using pebbles, a gallon can, a drill press, and a special agitator he designed for the drill bit to achieve a fine dispersion. In less than an hour, he invented an apparatus that would produce exactly what he needed. The device was his first attritor, which is defined as a grinding mill with internally agitated media.

A useful and simple equation that describes the grinding momentum is $M \times V$ (mass x velocity), which enables us to see how the attritor fits into the family of mills. For example, ball mills use large media, normally $\frac{1}{2}$ in. or larger, and run at a low rpm (10-50). Other mills, such as sand, bead and horizontal, use smaller media, from 0.3 to 2 mm, but run at a very high rpm (roughly 800-1200). Table 1 (p. 14) offers a comparison of grinding mills.

Attritor Basics

The main feature that makes the attritor more efficient than other mills is that its power input is used directly for agitating the media to achieve grinding, not for rotating or vibrating a large, heavy tank in addition to the media. The most efficient fine grinding takes place when both impact action and shearing force are present, as illustrated in Figure 1.

Wet Grinding

When wet grinding, impact action is created by the constant

impinging of the grinding media due to its irregular movement. Shearing action is present as the balls (media) in their random movement are spinning in different rotation and, therefore, exerting shearing forces on the adjacent slurry. As a result, both liquid shearing force and media impact force are present. Such combined shearing and impact results in size reduction as well as good dispersion.

The operation of the **batch attritor** is very simple. All the material can be loaded directly into the grinding tank; no pre-mixing or pre-dispersing is needed. Since the top-open grinding tank is stationary, the process can be visually observed and corrections and additional ingredients can be introduced at any time. The maximum feed material size can be up to 10 mm, provided the material is friable; otherwise, any 10 mesh down material is feasible.

Batch attritors are used to process hard-to-grind materials, such as tungsten carbide, silicon carbide, ceramics, glass frits, ink, chocolate, metal oxides and various metals. High-viscosity slurries up to 30,000 cps can also be processed easily in batch attritors.

A **circulation attritor** system is a combination of an attritor and a holding tank that is generally 10 times the size of the attritor. One of the essential requirements of the system is the high circulation (or pumping) rate. The entire contents of the holding tank are passed through the attritor at least once every 7-8 minutes.

At this rapid speed, the premixed slurry is pumped through a confined media bed. The media act as a dynamic sieve, allowing the fines to pass through quickly, while the coarser particles follow a more tortuous path and are ground finer. The slurry can be continuously monitored, additional ingredients can be

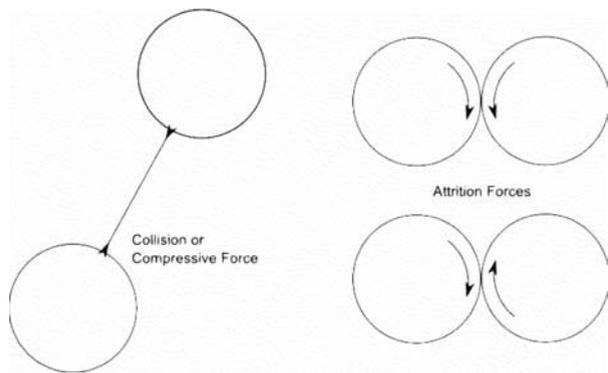


Figure 1. The most efficient fine grinding takes place when both impact action and shearing force are present.

added to the premix tank at any time during the grinding, and the processing can be terminated precisely.

One advantage of the circulation system is that large quantities of material can be handled with a smaller investment in grinding media and attritor equipment. Another advantage is better temperature control, which is achievable because the holding tank is jacketed for cooling or heating and acts as a heat sink. In addition, the slurry passes through the grinding chamber very quickly (20-30 seconds per pass), so it has less time to heat up. These advantages are very important when the grinding chamber is lined with plastic or rubber for metal-contamination-free processing.

Continuous attritors are best suited for the continuous production of large quantities of material. Well-premixed slurry is needed to be able to use this type of process. The slurry is pumped up through the bottom of a tall, narrow grinding tank and discharged out the top of the tank. The residence time required for certain fineness is controlled by the pumping rate. Continuous attritors can be set up in a series, using larger media and grid openings for the coarser feed, then subsequent units with smaller media to achieve a finer grind.

Dry Grinding

An expanded moving bed of media achieves the principle of attritor dry grind processing (a condition described as kinematic porosity). The dry particles are subjected to various forces such as impact, rotational, tumbling and shear; therefore, micron-range fine powders can be easily achieved. In addition, combinations of these forces create a more spherical particle than other impact-type milling equipment.

Dry milling can provide reduced transportation costs compared to wet grinding, since 50% of the gross weight is liquid in many wet slurry processes. Because the removal of the liquid from a wet grinding process involves not only another process step but also requires large amounts of energy, dry grinding can provide reduced energy costs. Another advantage is the elimination of waste liquid disposal. Following stricter environmental regulations, the disposal of any waste liquid (whether water or solvent) can be costly.

Dry grind attritors use grinding balls from 5 to 13 mm, and the shaft RPM generally runs from 75 to 500. These types of attritors are suitable for harder-to-grind materials such as metal powder, metal carbides and glass chunks. The feed material size for these machines can be quite coarse, but smaller than the grinding media chosen.

Small Media Milling

About 20 years ago, the industry trend began to move in the direction of smaller and smaller particles. Most people involved with milling agree that small media mills of one type or another are the preferred method to produce these particles. Small media is generally defined as 3 down to .25 mm, but mills are now available that utilize media as small as 0.1 mm. Small media milling provides several advantages over larger sizes, including putting more energy into the process and allowing more media per volume, and it results in a large surface area-to-weight ratio as well.

Small media mills have undergone a transition over the years as manufacturers continually look for increased efficiencies. The first generation of small media mills utilized a cantilevered chamber with a wedge wire screening mechanism. At that time, customers were looking for media as small as .25 mm. Mill manufacturers had trouble with mills packing out, however, and the industry moved back to media of about .75 mm.

Second-generation mills were designed with a shorter chamber and a rotor to agitate the media and material. The screening mechanism was changed from the wedge wire screen to rotating caps, rotating screens or a series of rings to provide more reliable operation.

One mill in particular was developed to combine the advantages of circulation grinding with small media (see Figure 2, p. 14).^{*} The mill is designed to accommodate media from 0.3 to 1.0 mm, and a screening mechanism was developed that allows for high product flow without media plugging. This is accomplished by having a screen of rings separated by appropriately sized spacers between them. Since the rings are the full diameter of the mill, the slurry can pass through a maximum open area, thus eliminating any plugging of the screen.

By using different-sized spacers, users can maximize the open area for the size of media being used. The mill utilizes specially designed delta discs that are indexed to provide directed and uniform media distribution throughout the mill chamber while simultaneously providing greater random media motion and improved milling. The mill parts that come in contact with the material being milled (chamber, rings and discs) can be made out of harder metals for longer life or out of ceramics, such as MgO-stabilized zirconium oxide, for applications where contamination is an issue (see Figure 3, p. 15).

Grinding Media

The grinding media is another important factor to consider for optimizing the milling process, since media cost can be a significant part of the expense. The selection of the grinding media depends on several factors, some of which are interrelated. In

*DMQ Small Media Mill developed by Union Process.

Table 1. Comparison of grinding mills.

Mill Type	Media Size	RPM	Tip Speed (fpm)
Ball Mill	1/2 in. and larger	10-50	n/a
Attritor	1/8-3/8 in.	60-350	600-1000
High-Speed Attritor	0.5-3.00 mm	320-1700	2500-3000
Sand Mill/Horizontal Mill/DMQ	0.25-2.00 mm	800-3800	2000-3000
Rotor Stator	n/a	1000-3600	2000-4200

general, high-density media give better results, and the media should be denser than the material to be ground. Also, highly viscous materials require media with higher density to prevent floating. Smaller media cannot easily break up large particles, but are more efficient when ultrafine particles are desired. The harder the media, the lesser the contamination, and, consequently, the longer the wear.

Contamination is another important consideration. The material resulting from the wear of the media should not affect the product or should be removed through the use of a magnetic separator, chemically, or in a sintering process. Cost can also be a factor. Media that may

be 2-3 times more expensive may wear better (sometimes 5-6 times longer), and therefore could be worth the extra cost in the long run. Additional factors include pH (some strong acid or basic materials may react with certain metallic media) and discoloration (e.g., white material should remain white).

Cryomilling

Cryomilling is a variation of mechanical milling in which powders are milled in a cryogen slurry (usually liquid nitrogen) or at a cryogen temperature under processing parameters such that a nano-structured microstructure is attained.¹ Figure 4 shows a research attritor being used with a cryogen at Advanced Ceramics Research

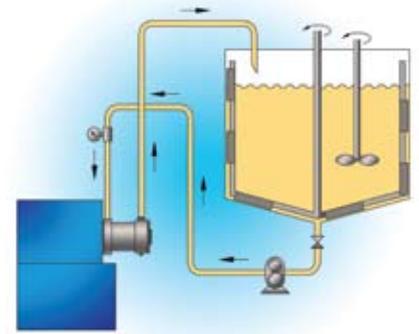


Figure 2. This mill was developed to combine advantages of circulation grinding with small media.

in Tucson, Ariz. One can readily see the effects of the lower temperatures on the equipment, which illustrates the point that special precautions need to be taken for personnel protection.

Figure 5 shows some of the special equipment that must be used with cryomilling. This photo, supplied by Advanced Ceramics Research, shows the vapor recovery system and special mill covers that are used with this type of milling. Cryomilling takes advantage of both the extremely low cryogen temperature and the benefits that are provided with conventional mechanical milling. The extremely low milling temperature in cryomilling suppresses the material's recovery and recrystallization, leading to finer grain structures and more rapid grain refinement.

A lot of work is being done with this technique to produce a much stronger aluminum metal. Cryomilling, which yields a grain size as small as 30 nm, encourages the formation of nano-scale oxides and nitride particles. This results in a metal that is 2-3 times stronger than the starting metal. The Marine Corps is working to develop a 30- to 40-ton tank that will offer the same level of protection as a 70-ton tank.²

Studies are also underway to develop cryomilled ceramics for the Navy. James Murday, Ph.D., head of the chemistry division of the National Research Laboratory in Washington, D.C., has reported on a ceramic nanocomposite coating that

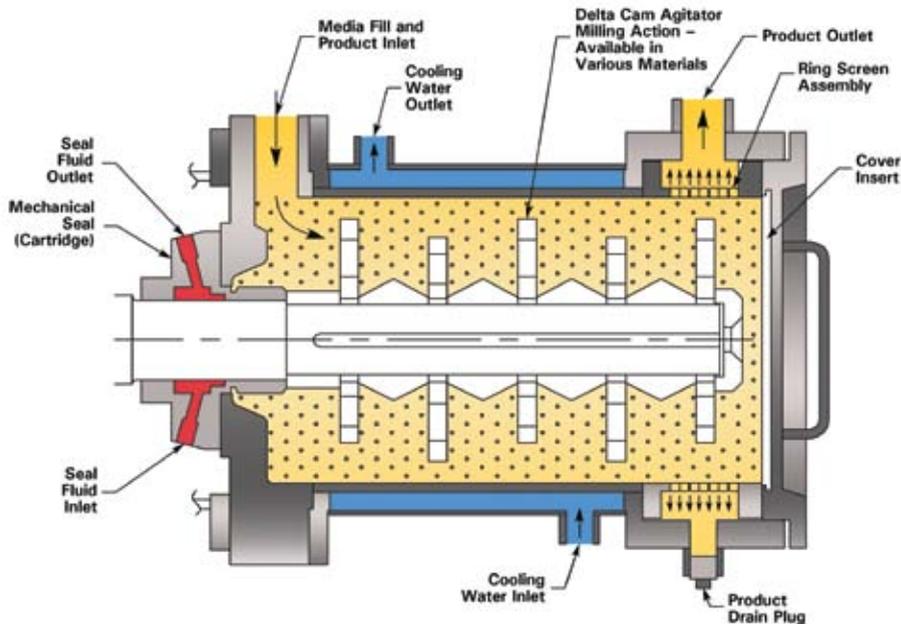


Figure 3. The mill parts that will be in contact with the material being milled (chamber, rings and discs) can be made out of special harder metals or ceramics.

has been used to coat the drive shaft on four ships.³ After four years in service, inspections showed no visible damage. Technical cost modeling done by Schoenung, He, Tang and Witkin at the University of California in Davis and Irving has shown that cryomilling has the potential to be commercialized to fabricate nanostructured material economically.

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Ceramic nanoparticles have enabled the economical manufacturing of ceramic parts. According to R. W. Siegel of Rensselaer Polytechnic Institute, "The ability to net shape form ceramics into final parts has become a reality in recent years, owing to advancements made in the scaled-up production and processing of ceramic nanoparticles. With the availability now of tonnage quantities of nanophase ceramic powders with their unique rheological and mechanical

properties, it is possible to directly form ceramic parts in a sinter-forging mold under sufficient pressure and temperature to yield final parts with all of the definition and precision of the original mold. Nanophase ceramics such as titania and alumina, made from the consolidation of ceramic nanoparticles, have been shown in the laboratory to be readily formable into small samples, and studies of their mechanical behavior indicate that a significant degree of ductile behavior in compression is exhibited in these ultrafine grain size materials."⁴

Ensuring High-Quality Results

With increasingly higher standards required by the ceramic industry, fine grinding/particle size reduction has become one of the most important factors for success. Over the years, attritors have proven to be an excellent and reliable means to achieve these milling tasks. 🌐

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Figure 4. A research attritor being used with a cryogen.



Figure 5. Vapor recovery system and special mill covers must be used with cryogenic milling.

Editor's note: This article is based on a paper given at the Ceramic Manufacturers Association (CerMA) conference held May 2008 in Columbus, Ohio.

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