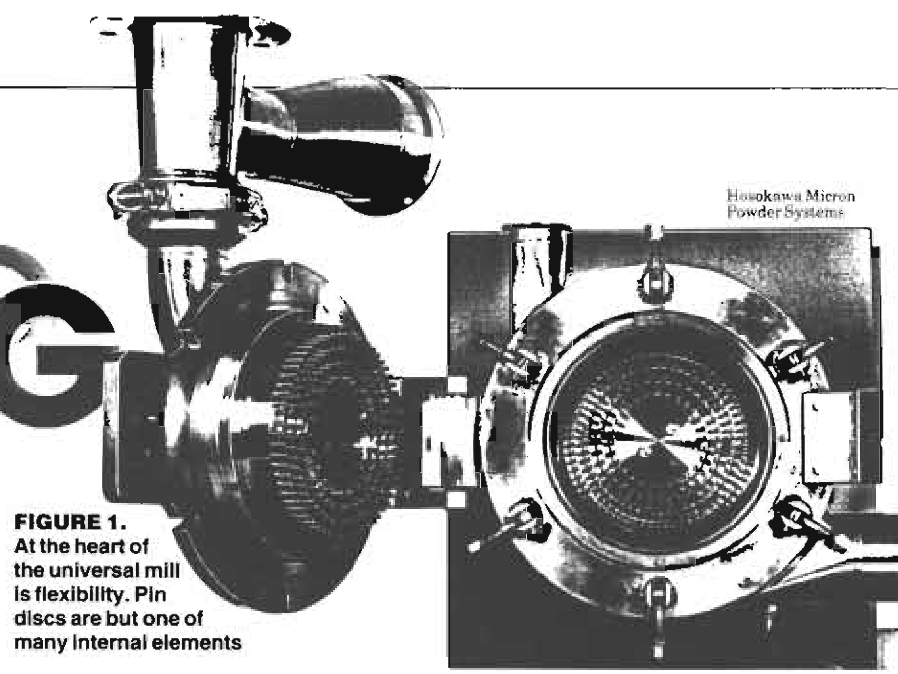
SIZING UP GRINDING MILLS



Hosokawa Micron Powder Systems

FIGURE 1. At the heart of the universal mill is flexibility. Pin discs are but one of many internal elements

TABLE 1. MILL TYPE SELECTION MATRIX

Criteria	Slurry	Fluid energy	Universal	Cone	Hammer
Particle size	less than average	very favorable	very favorable	less than average	average
Particle distribution	average	very favorable	very favorable	favorable	favorable
Cleaning	less than average	average	average	favorable	less than average
Operating cost	favorable	unfavorable	unfavorable	very favorable	favorable
Dust containment	very favorable	less than average	less than average	very favorable	favorable
Temperature	very favorable	favorable	less than average	very favorable	favorable
Flexibility	average	average	very favorable	favorable	favorable

Whether used in industrial or household applications, particle size reduction is a part of everyday life. For example, a mining company uses compression to transform mined ore and rock into easily handled lumps. At home, coffee lovers use impact and attrition to transform whole coffee beans into finely ground particles to make a fresh pot of coffee. A wooden pallet manufacturer uses cutting to transform old, broken pallets into salable wood mulch.

These example show that four main methods of accomplishing particle size reduction exist — compression, impact, attrition and cutting [*1*]. Simply put, size reduction or comminution, is the process of making large particles smaller.

The equipment used for size reduction varies with each method. The mining company uses a large crusher, whereas the coffee lover uses a small, coffee grinder and the wood pallet company, a large industrial shredder.

This article will cover only particle-size-reduction methods common to the fine chemical and pharmaceutical industries. In particular, milling, the process of reducing size by mechanical means, and its respective equipment, will be discussed.

Finding the right equipment for particle size reduction requires a case-by-case analysis

Considering all aspects

In designing a milling system, knowledge of particle size and the desired distribution is important. If a broad size distribution is desired, an open milling circuit, which does not classify size, may be acceptable. If a narrow size distribution is required, a closed milling circuit is used. Here, a classification step is used to prevent oversized material from entering the final product. The rejected material is often recycled into the mill for regrinding [*2*].

However, selecting the appropriate mill is not a simple process. Currently, the theory of milling has not developed to where the product output can be mathematically determined given a product feed. To find the right mill, pilot plant studies must be conducted with the particular product feed and mill in question (Box, p. 86). After finding the most suitable mill (Table 1), getting the desired particle size distribution is a matter of fine tuning key factors affecting individual mills.

Mulling over mills

To select the right mill, the process requirements must be matched to the capability of the mill.

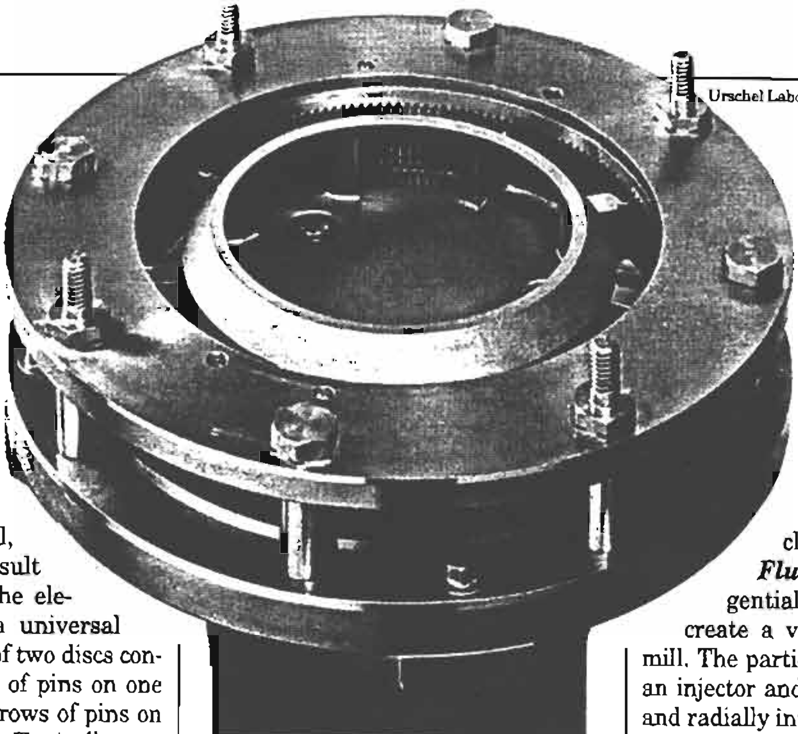

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FIGURE 2a. Using slurry as a heat sink, slurry mills are ideal for grinding heat-sensitive materials

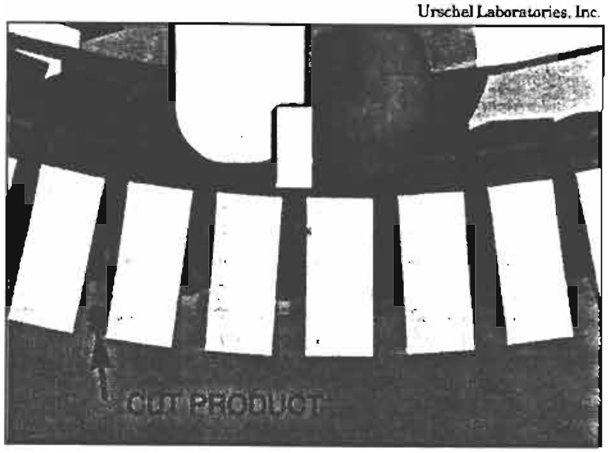

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FIGURE 2b. In slurry mills, a high-speed impeller generates centrifugal forces that push the particles outward to cutting edges

Universal mill. Flexibility is the universal mill's most attractive trait. It is capable of using a variety of internal elements, such as pins, cages, hammers, turbo discs, beater arms and perforated screens.

In a universal mill, size reduction is the result of particle impact on the elements. For instance, a universal mill with pins consists of two discs containing concentric rows of pins on one disc fitted between the rows of pins on the other disc (Figure 1). Typically, one disc rotates while the other remains stationary. Particles are fed in at the center of the stationary disc and are moved outward centrifugally through the intermeshing pins, resulting in size reduction. Other internal elements work similarly and allow the operator to tailor the universal mill to specific processing needs.

With a large selection of internal elements to choose from, the mill can handle a wide spectrum of comminution needs. This flexibility makes it a good choice for size reduction of several different materials at one facility. Also, ease of assembly and disassembly makes this mill suitable for processes that often change products.

Universal milling is also ideal when the desired particle-size distribution of a compound is narrow. For example, a universal mill equipped with pins can provide a tight size distribution of 10 μm or less.

While the universal mill is versatile, it has several disadvantages. First, a great deal of heat and noise (90-100 dB) are generated from the high-energy rotating parts, making it incapable of handling sticky and heat-sensitive materials. Second, a pneumatic conveying system may be required to transport the material through the mill. This adds to the cost of the system.

Slurry mills. This milling technique passes a liquid slurry through a high energy mill. The liquid slurry is fed to the center of a high-speed, rotating impeller (Figures 2a and 2b). Centrifugal forces then move the particles outward to the impeller tips, where the particles pass through the cutting edges of a stationary reduction head [3]. This action produces the desired reduction in particle size.

Unlike universal milling, slurry milling is ideal for size reduction of thermally sensitive materials. The liquid in the product slurry provides an excellent means for removing heat generated by the milling process.

Another good slurry milling application is size reduction of hazardous materials. Unlike the universal mill, the slurry mill does not produce dry powder. This eliminates production of hazardous or toxic dust.

However, using slurries subjects mill internals to higher wear compared with dry milling. Also, filtration of the final product from the slurry is difficult as the larger particles become smaller.

Fluid energy mill. Tangentially positioned gas jets create a vortex in a fluid energy mill. The particles are fed by means of an injector and accelerate tangentially and radially inward, where particle-on-particle impact creates the size reduction (Figure 5) [4].

With no internal moving parts, fluid energy mills can handle highly abrasive materials such as tungsten carbide, silicon carbide, boron carbide, or aluminum oxide. These materials can damage any other mill with internal moving parts.

Also, fluid energy mills are equipped with a classification unit, allowing the size of the material exiting the mill to be controlled. Therefore, the mill can be used for processes that require specific and narrow particle-size distribution [5].

However, fluid energy milling presents environmental concerns. The mill uses a large amount of carrier gas at high flows, making particulate emission difficult to control.

Cone mill. In a cone mill, the material enters the top of the apparatus and falls into the conical screen chamber (Figure 6). A rotating impeller moves the material into the action zone, the area between the impeller arms and the screen, where size reduction takes place. The impeller arms can be in close proximity to the screen (0.05 to 0.02 in.) [6], depending on the desired particle-size distribution.

With little heat generation because of low impeller speed, the cone mill is suitable for the size reduction of materials that are sticky, gummy, heat sensitive, or fatty. In addition, this mill is ideal for size reduction of moist materials that tend to clump together.

Based on work done at Eli Lilly and Co.'s Tippecanoe Laboratories (Lafayette, Ind.), the following case study demonstrates the firm's process for selecting a mill. The case history also discusses installation of the size reduction system.

Eli Lilly and Co. was developing a commercial process for the bulk manufacture of the compound substance X. During this process, improvements in the product purity and final crystallization conditions resulted in large, single crystals (Figure 3, right inset) of the bulk drug substance (BDS), rather than the small, agglomerated crystals (Figure 3, left inset) isolated from earlier synthetic routes. These larger crystals presented a problem, since they had unacceptably low tablet-dissolution profiles.

But, evaluation of the BDS physical properties showed that the particle-size distribution and the mean particle size correlated well with tablet-dissolution profiles. Hence, particle size was selected as a control strategy to ensure consistent performance of the tablet BDS. Thus, the challenge was to manufacture the BDS of similar size and distribution to that of the earlier synthetic routes.

One method was to control conditions during crystallization. Unfortunately, these attempts met with limited success, and the focus shifted to various milling techniques to reduce the particle size after crystallization.

Because slurry milling was easy to add to the process flow, it was considered as the first option. While, slurry milling provided the desired particle-size distribution, the average particle size of the early synthetic process was not achieved (results of all trials are shown in Figure 3).

Trials for multiple passes through the slurry mill were also run. Unfortunately, this process yielded no alterations in the mean particle size. Also, the tablet dissolution profile obtained from slurry milled BDS was unacceptably low (Figure 4 shows a composite of all trials). Thus, alternative milling techniques were explored.

The second milling technique tested was fluid energy milling. Fluid energy milling provided the desired particle size distribution and mean particle size. Additionally, tablet dissolution profiles obtained from the fluid-energy-milled BDS compared favorably to profiles derived from the earlier synthesis process.

While this milling method showed promise, there were concerns over the disadvantages of fluid energy milling, such as lack of control of particulate emissions. Thus, Eli Lilly further investigated other milling techniques.

As a possible alternative, a universal mill using pin discs was considered. Similar to the fluid energy mill, pin milling provided the desired particle size distribution and reduced the mean particle size to match the desired size. Tablet dissolution profiles from pin milled BDS were similar to those obtained from both fluid energy and early synthesis process.

Therefore, both pin milling and fluid energy milling appeared to satisfy the particle size reduction requirements. Since production-size pin mills were readily available for manufacturing, pin milling was selected as the method of choice.

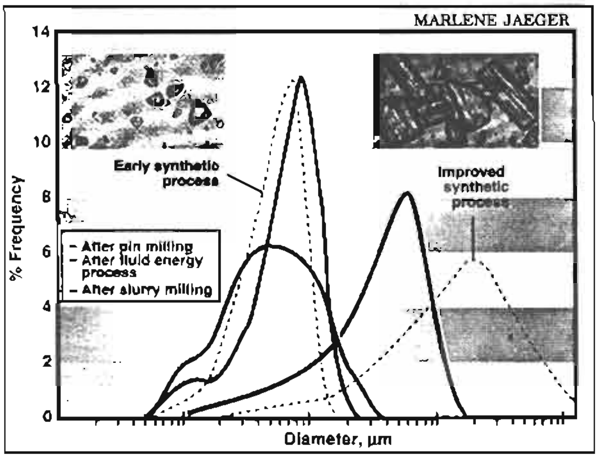

FIGURE 3. Key to the mill selection process was matching the particle size distribution of the early synthetic process. After testing three mills, the only mill that paralleled the early process was the pin mill

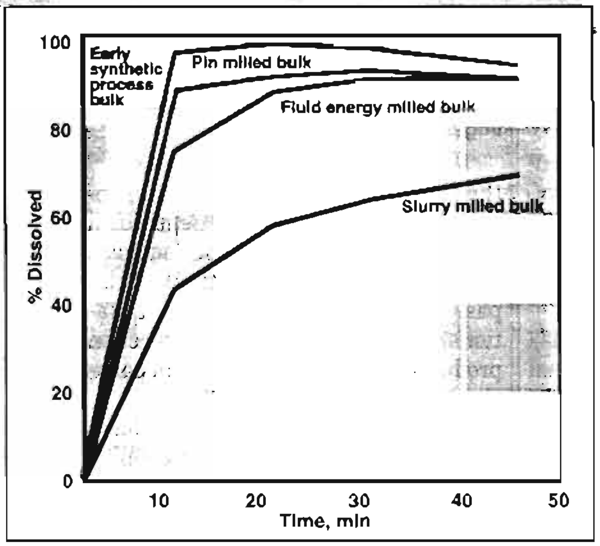

FIGURE 4. One of the objectives of the pilot test was to find a mill that could duplicate the tablet dissolution profile for the early synthetic process. According to the results, the best fit was the pin mill, with the fluid energy mill coming in a close second

Reduction in action

The full-scale size reduction system for substance X was designed around an existing universal mill. This mill was capable of disc speeds over 10,000 rpm. Additionally, a pneumatic conveying system was installed to move the material from the dryer through the pin mill to the final packaging hopper.

The BDS was gravity discharged from the dryer into a rotary valve. The valve serves as a feed control device for the size reduction and pneumatic conveying systems. Equipped with a variable frequency drive, this allowed for easy adjustment in the material feed rate.

A steady material feed rate was key to the successful operation of the system. A feed rate too high would cause the mean particle size to increase, resulting in unacceptable material. After several trials, a material feed rate of less than 120 kg/h was determined to provide the best mean particle size and particle size distribution.

Upon exiting the rotary valve, substance X was conveyed through the pin mill using nitrogen. Nitrogen was chosen as the carrier gas so the entire operation would be carried out in an inert atmosphere (oxygen content less than 5%).

Without inerting, the high volume resistivity and low minimum ignition energy of substance X made the possibility of a static spark and dust explosion likely. Thus, the additional cost of using nitrogen instead of plant air was easily outweighed by the safety afforded by operating in an inert atmosphere.

After leaving the mill, the material was conveyed into the product receiver where substance X and nitrogen were separated. The product accumulated at the bottom of the hopper, while the nitrogen passed through a series of filters, a vacuum pump, and vented harmlessly to the roof. Substance X was then gravity flowed through a second rotary valve into final product packages.

The start-up moved quickly after the size-reduction system was installed and tested. The pneumatic conveying system was started first, so operating parameters could be established. Then, the BDS was introduced into the system. Once the proper material feed rate was established, substance X was milled, and the system validated to provide material within the desired particle size distribution range and the correct mean particle size. ☐

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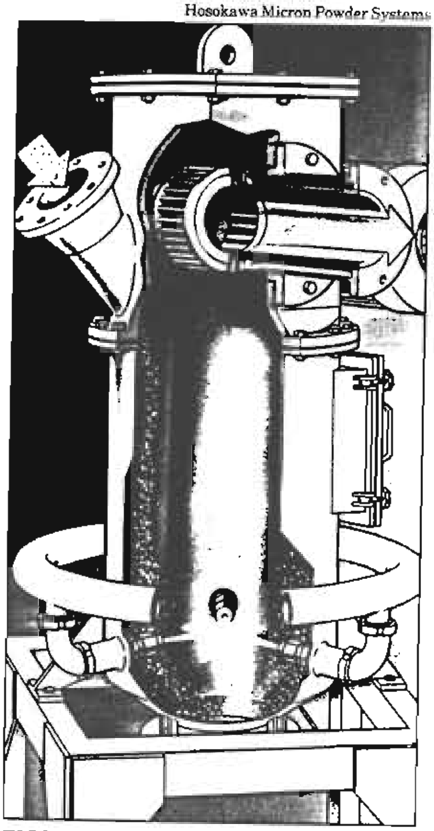
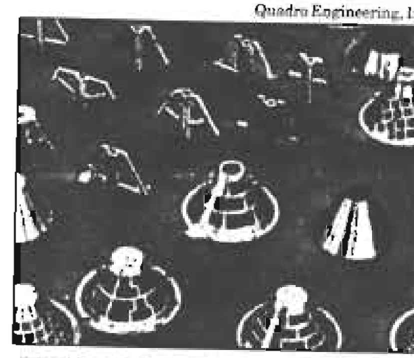


FIGURE 5. Travelling around in the vortex of this fluid energy mill, particles smash into each other, resulting in size reduction

TABLE 2. FACTORS TO CONSIDER Here are some ways to improve particle size reduction.
Mill Type
Universal mill • Decrease the material feed rate • Increase internal-element tip speed • Decrease the conveying gas flowrate
Slurry mill • Decrease the material feed rate • Decrease percent solids in the slurry • Increase the mill hp
Fluid energy mill • Increase the classifier speed • Decrease the gas flowrate • Decrease the nozzle diameter
Cone mill* • Decrease the size of the holes in the screen • Change the impeller type
Hammer mill • Increase the material feed rate • Increase the blade speed • Decrease the size of the feed throat • For particles larger than 840 μm, use sharp rotating blades • For particles smaller than 840 μm, use flat rotating blades
* To maximize performance, a cone mill manufacturer should be consulted for other factors, such as shape of the screen, impeller configuration and impeller rotational speed.

Quadro Engineering, Inc.



FIGURES 6 (above) and 7 (below). To meet the demands of various particle size distributions, cone mills and hammer mills have a wide array of screen internals to choose from

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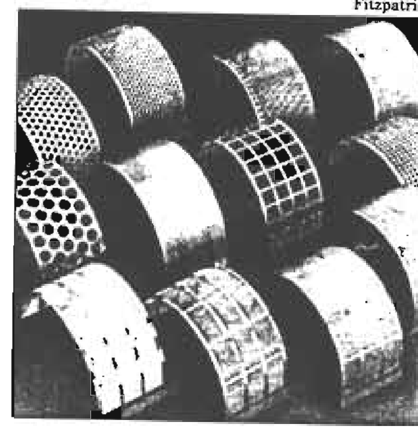

The cone mill is also effective when the particle size reduction of several different materials is required at the same facility. The cone mill screens and the impellers can be easily changed to handle specific particle-size-reduction requirements.

However, cone mills face limitations due to the screen size and mechanical energy input. Very fine screens tend to be frail and prone to breakage, making small particle sizes difficult to attain. The lower energy aspect of the cone mill also makes small particle sizes difficult to attain.

Hammer mill. Using three steps to achieve the final size reduction, materials enter the hammer mill through a feed throat and then pass into a grinding chamber. Inside the chamber the material is impacted with rotating blades (hammers) attached to a horizontal shaft. For further reduction, the material is then thrown against the inside casing of the hammer mill. The final reduction occurs when the material is forced through a screen (Figure 7) by the rotating blade [2].

As does the cone mill, the hammer mill adapts to comminution requirements through the use of different screens, making it an ideal choice for size reduction of several different materials at the same facility. The hammer mill is also simple to assemble and disassemble, making it easy to clean. This feature makes this mill suitable for processes where products change on a regular basis, and the change-over time between products must be minimized.

The hammer mill has certain disadvantages, though. For instance, the mill contains high-energy rotating parts that require high maintenance. Additionally, with the rapid movement of the impactor, the machine can act like a fan and entrain the material in the subsequent air flow [4]. This makes reduction to small particle sizes difficult. ■

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References

1. McCabe, W.L., others, "Unit Operations of Chemical Engineering," 4th ed., pp. 767-794, McGraw-Hill, New York, NY, 1985.
2. Eli Lilly and Co., Engineering Technical Guideline Milling-15127, Internal publication, January 31, 1995.
3. Urschel Laboratories, Comitrol Comminuting Machines, Bulletin #1149, 1985.
4. Lowrison, G.C., "Crushing and Grinding - The Size Reduction of Solid Materials," pp. 191-224, 261-269, CRC Press, Cleveland, OH, 1974.
5. Miranda, S.J., What is a Fluidized Bed Jet Mill?, Hosokawa Micron Powder Systems.
6. Quadro, Inc., Quadro Comil - A Truly Innovative Size Reduction Mill, Bulletin #QL/90, 1990.