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Attrition Mill Fine Grinding of Advanced Ceramic Powders

author

JOHN E. BECKER
Western Sales Manager
Union Process Incorporated
Akron, Ohio

abstract

The principles and applications of wet and dry grinding in the attritor, a high-energy stirred ball mill, are presented. Batch, circulation, and continuous attrition mills are described, along with their advantages and specific technical ceramic applications. Attrition mill fine grinding of technical ceramics versus conventional ceramic fine grinding methods are compared to relative cost/energy effectiveness, speed, temperature control, and particle size distribution characteristics. Available ceramic media, appropriate contamination-free linings, and accessory equipment for specific advanced ceramic applications are presented.

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INTRODUCTION

The attrition mill, more commonly known as the Attritor, is a grinding mill containing internally agitated media. It may be generically referred to as a "stirred ball mill". The Attritor is one of the most efficient fine grinding and dispersing or comminuting pieces of equipment available today for fine grinding of ceramic materials.

A key to the efficiency of Attritor grinding is that the power input is used directly for agitating media for grinding and is not used for rotating or vibrating a large, heavy vessel in addition to the media charge.

To more clearly illustrate the efficiency of the Attritor, Fig. 1 shows the relative effectiveness of the Attritor versus the vibratory ball mill and conventional ball mill for the ultra-fine grinding of Pima chalcopyrite concentrate. The data for the vibratory ball mill is represented by the top curve with the middle two curves developed from conventional ball mills. The bottom curve was derived from Attritor data. This figure clearly shows that for a specific energy input of about 100 kwh/t, the median particle size obtained through the use of Attritors is approximately 50% smaller than that obtained from conventional ball mills and about 33% smaller than that obtained from vibratory ball mills. At a specific energy input above 200 kwh/t, the Attritor continued to grind into the submicron range while the other machines can no longer efficiently produce the smaller, sub-micron particles. Therefore, the time required for grinding sub-micron particles with the Attritor is much shorter.

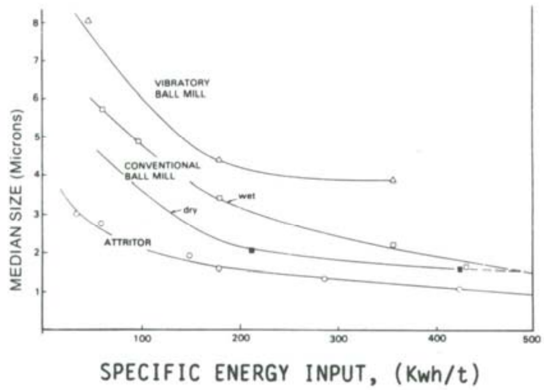

Figure 1 [(1)]

The Attritor's operation is simple and effective, and this explains the Attritor's efficiency. The material to be ground is placed in the stationary Attritor tank with the grinding media. The material and media are agitated by a rotating central shaft with arms. Both impact and shearing action result in size reduction, as well as homogeneous particle dispersion with very little wear on the tank walls. These efficient impact and shearing forces are illustrated in Figure 2, and must be present for the most effective grinding action.

The configuration of the Attritor arms result in a constant moving motion of the slurry around the tank. The area of greatest media agitation is shown in Figure 3, and is located approximately two-thirds the radius from the center shaft. In production machines, the movement is augmented by adding a pumping, circulation system. As can be seen in Figure 3, grinding does not take place against the tank walls resulting in very little wear on the walls, leading to longer service life of the vessel. The vessel walls can, therefore, be made thinner thus providing enhanced heat transfer and greater temperature control.

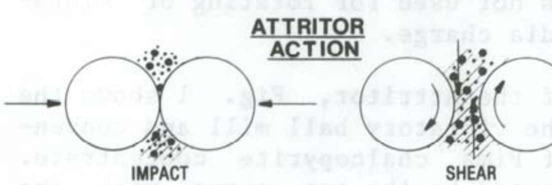

Figure 2

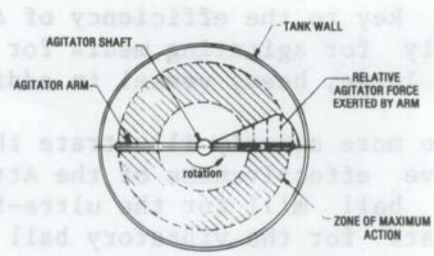

Figure 3

(1) J.A. Herbst and J.L. Sepulveda, "Fundamentals of Fine and Ultrafine Grinding in a Stirred Ball Mill", PRoc. Powder & Bulk Solids Conf., Chicago, IL, May 1978.

The following equation can be used to relate grinding time to media diameter and agitator speed:

$$T = \frac{KD^{(2)}}{\sqrt{N}}$$

T = grinding time to reach a certain median particle size
K = a constant that varies depending on material being processed, type of media, and the model of Attritor being used
D = media diameter
N = shaft rpm

This equation shows that the total grinding time is directly proportional to the media, or ball diameter, and inversely proportional to the square root of the shaft RPM. This equation also shows that increasing the media size increases the grinding time, but decreasing the media size decreases grinding time.

Grinding media selection, however, must be based upon several interrelated factors:

1. Contamination - the media wear should not adversely affect the final product, or worn media material should be able to be removed chemically, or by a magnetic separator, or in sintering.
2. Specific gravity - normally, the higher the density, the more effective and faster the grinding. Ideally, the media should be denser than the material to be ground and materials with high viscosity require media with higher density to prevent floating.
3. Feed size - the diameter of the media should be greater than the initial particle size for effective breakdown of the large particles.
4. Hardness - harder media result in less contamination, greater grinding efficiency, and longer media life.
5. Discoloration - media composition must allow white or light colored materials to retain their clean color without adverse discoloration, etc.
6. pH - Some strong acid or basic slurries may have a tendency to react with certain metallic media.
7. Final particle size - generally, smaller media are more efficient when grinding superfine particles.

For Attritor grinding, media size range is from 1/8" to 3/8", with smaller grinding media generally resulting in faster particle reduction because, for a given volume, there will be more impact and surface contact. As media become smaller than 1/8", its mass is significantly reduced, resulting in less impact force resulting in longer grinding times. When ultra fine grinding is not needed, a larger diameter media may prove to be faster and more efficient, since its mass is greater.

Attritors use many different types of media, each suitable for specific materials in various industries. These include, carbon steel, stainless steel, chrome steel, tungsten carbide, various ceramic materials, glass, and flint stones.

(2) Temple C. Patton, "Paint and Flow and Pigment Dispersion", Second Edition, Wiley - Interscience, New York, 1979.

For fine grinding applications in the ceramic industry, the types of media generally being used include steatite, consisting primarily of 64% silica, 26% magnesium oxide, and 6% alumina; high alumina with 95% alumina; mullite with 74% alumina, 21% silica, 2.5% magnesium oxide; and zirconium silicate, 69% zirconium oxide, 31% silica. The more expensive types of ceramic media currently being used include yttria stabilized high purity zirconium oxide, magnesium stabilized zirconium oxide, silicon nitride, and silicon carbide.

Three types of Attritors are:
1. Batch
2. Continuous
3. Circulation

In the batch Attritor for wet grinding, the material is charged into the top of the jacketed grinding tank and is processed until the desired particle size is achieved. Production size Attritors are equipped with a built-in pumping system which contributes toward greater product uniformity and can be used for faster discharging. No premixing is necessary, and ingredients can be added at any time during processing, while sampling and formula corrections can be made at any time without stopping the Attritor. For dry grinding, the batch Attritor can be used in either the batch or continuous mode, depending on the product initial feed size, final particle size desired, and production rate requirements.

The continuous Attritor provides a constant flow of finely ground, processed material. For wet grinding, the premixed slurry is pumped up through the bottom of the tall, narrow, jacketed tank with the fineness of the processed material determined by the "dwell time" and controlled by the pumping rate. Continuous Attritors can be arranged in series, using larger media and grid openings for the first unit. For dry grinding, the continuous Attritor can be continuously charged through the top and discharged through the bottom grid.

The circulation Attritor rapidly pumps the slurry to be ground through a confined bed of small grinding media. The media acts as a dynamic sieve - the fine particles pass through easily, and the larger particles are ground more finely - resulting in a narrow particle size distribution. The slurry passes through the Attritor until the desired particle size is achieved. Large quantities of material can be ground with a smaller investment of grinding media and Attritor equipment. Additional material can be added at any time, and the product can be continuously monitored, for quality control.

For processing of ceramic materials in the Attritor, the appropriate materials for Attritor tank linings, arms, and grid plates must be selected. Currently, several materials have been successful for lining and sleeving these machine parts to eliminate or minimize contamination of the final ceramic product. Once again, it must be remembered that, in the Attritor, over 90% of contamination will come from the grinding media, which means, by far, the grinding media selection is of the utmost importance. The remaining potential contamination will come from the tips of the agitator arms, the tank lining wall, and the grid plates.

In most ceramic applications, a high alumina tank lining with high density plastic sleeved arms, grid plates, and a plastic type pumping syste. are recommended. As we know, alumina is very abrasion resistant, and is resistant to most solvents, but for applications where alumina contamination is not desired, a tank lining made of a polymer type material, such as rubber, polyeurethane, or teflon may be appropriate. It must be remembered, however, that a polymer lining has a very poor heat transfer factor, therefore, a heat exchanger in the pumping line may be needed in some cases. However, rubber-lined Attritors have been used very successfully in grinding water-based silicon nitride and zirconium oxide slurries.

In terms of grinding silicon nitride and silicon carbide, these materials themselves can be used for tank liners, grid plates, and arm sleeves, but presently, are very expensive. In many cases, it seems to be more cost-effective to use plastic-sleeved arms and shaft, and replace them at appropriate intervals. Thus far, high alumina sleeves prove to be less cost-effective than plastic, due to the fact that they wear much faster. Silicon nitride sleeves have proved to be long wearing, but again, more expensive from an initial cost standpoint.

APPLICATIONS

The batch and circulation Attritors are preferred for advanced ceramic grinding. The batch Attritor has the advantage of grinding both dry and wet in very small batches, if necessary, while facilitating easier cleaning between different product runs. The circulation Attritor, on the other hand, can provide an even narrower particle size distribution along with greater batch size, flexibility, and lower capital investment for larger batch size requirements. The circulation Attritor, however, is limited to the processing of pumpable ceramic slurries.

The grinding and dispersing of silicon nitride powder has proved to be extremely successful using the batch Attritor, Figure 4. Figure 5 is a Microtrac readout which shows a frequency distribution of unground silicon nitride with a mean diameter of 5.62 microns ground to .98 microns in a 1-S Attritor in two hours. Here, 1/4" silicon nitride media was used along with deionized water. As is very common, this material took about 1/10th the usual time it takes to grind to the required median size in a typical ball mill.

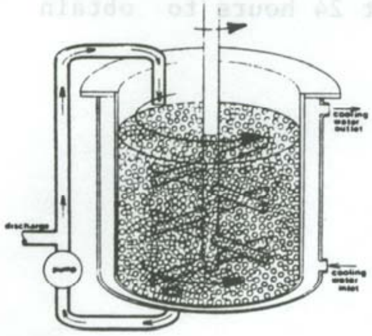

Figure 4

SILICON NITRIDE POWDER:

UNGROUND		AFTER 2 HOURS	
35.50	0	35.50	0
25.10	0	25.10	0
17.75	-----8	17.75	0
12.55	---------13	12.55	0
8.87	----7	8.87	0
6.27	--4	6.27	0
4.43	------10	4.43	0
3.13	---------14	3.13	-7
2.21	---------14	2.21	-6
1.30	-------12	1.30	-----16
0.80	----7	0.80	---------26
0.55	--4	0.55	---------25
0.39	2	0.39	---11
0.30	0	0.30	3
0.20	0	0.20	1
0.15	0	0.15	2

	UNGROUND		AFTER 2 HOURS	
(Surface Area)	CS=	3.13	CS=	9.64
(Mean)	MV=	5.62	MV=	0.98
(90% less than)	%90=	14.56	%90=	2.21
(50% less than)	%50=	3.25	%50=	0.74
(10% less than)	%10=	0.75	%10=	0.36
(Std. Deviation)	DV=	0.2586	DV=	0.1138

Figure 5

Another common advanced ceramic application for the circulation Attritor is for grinding and dispersing of alumina slurries. Typically, the material is ground in a high-alumina lined Attritor using 95% alumina media with plastic arms, grids, a plastic air diaphragm pump and nylon coated or stainless steel holding/dispersion tank, ten times the size of the Attritor. Figures 6 & 7 illustrate a typical circulating Attritor system. The high pumping rate circulates the entire contents of the holding tank through the Attritor at least once every 7½ minutes or about 8 times per hour.

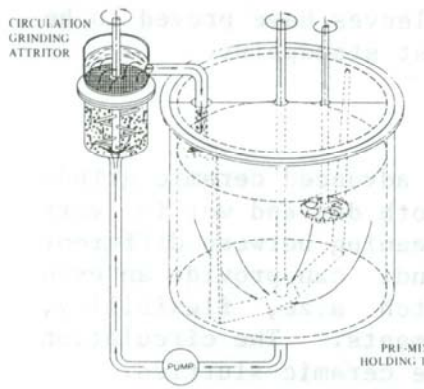

Figure 6

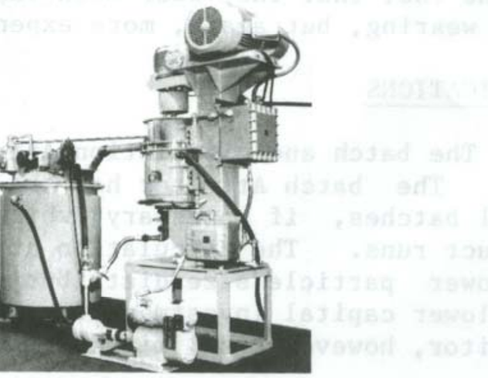

Figure 7

Figure 8 shows a frequency distribution for aluminum oxide slurry before and after grinding and dispersing in a production size Q-25 circulation Attritor. Here, 1/4" alumina media was used and the total production time was 8 hours to produce 1,600 lbs. of alumina. The initial alumina had a mean particle size diameter of 8.69 microns and after 8 hours was reduced to a mean diameter of 1.68 microns. The total residence time of the slurry grinding chamber was 40 minutes. This material is typically ground in a ball mill for at least 24 hours to obtain this particle size.

ALUMINUM OXIDE SLURRY:

FEED MATERIAL		AFTER 40 MINUTES RESIDENCE TIME	
35.50	0	35.50	0
25.10	--5	25.10	0
17.75	--------13	17.75	0
12.55	-----10	12.55	0
8.87	-------14	8.87	0
6.27	---------16	6.27	1
4.43	-------14	4.43	--9
3.13	-----10	3.13	--10
2.21	--6	2.21	---11
1.30	-3	1.30	---------27
0.80	2	0.80	-------22
0.55	1	0.55	----15
0.39	0	0.39	2
0.30	0	0.30	0
0.20	0	0.20	0
0.15	0	0.15	0

		Feed		After 40 min
(Surface Area)	CS=	1.48	CS=	5.72
(Mean)	MV=	8.69	MV=	1.68
(90% less than)	%90=	19.18	%90=	3.75
(50% less than)	%50=	6.71	%50=	1.26
(10% less than)	%10=	2.02	%10=	0.54
(Std. Deviation)	DV=	0.7276	DV=	0.4130

Figure 8

Circulation Attritors are also presently being used successfully in the grinding of barium titanate electronic materials. These materials typically start with 20 microns and are reduced in grinding in the circulation Attritor sometimes to less than 1 micron, if required.

SUMMARY

ADVANTAGES

1. Fast and efficient fine grinding
2. Low power consumption
3. Easy to operate
4. Good temperature control
5. Low maintenance
6. Smaller plant area requirements

LIMITATIONS

1. Used most efficiently for fine grinding only
2. Feed size of the material to be processed in the Attritor should typically be smaller than the Attritor media diameter
3. Wet grinding is necessary for the most efficient grinding of ceramic materials below one micron
4. The availability of the appropriate type and size of media for contamination-free grinding of a particular product