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Autogenous Attrition Microgrinding of Calcium Carbonate Minerals

By J. P. Hansen, E. G. Davis, and G. V. Sullivan

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To help assure an adequate domestic supply of minerals essential to the Nation's economy and security by developing more efficient minerals extraction technologies, the Bureau of Mines' Tuscaloosa Research Center examined the application of autogenous microgrinding to calcium carbonate minerals. Treatment of minus 8-mesh Alabama marble resulted in a product 80 pct minus 5 micrometers (μm) at a power consumption of 70 to 80 kwhr/ton. This study shows that coarse particles of calcium carbonate minerals are effective as the grinding media for the autogenous attrition grinding of these minerals. The minus 8- plus 30-mesh fraction is an effective grinding medium (sand) for particles smaller than 30 mesh. A 40-pct-solids slurry and a sand-marble ratio of 2.5 appeared to be near optimum. For residence times above 13 minutes, products were greater than 80 pct minus 5 μm and greater than 54 pct minus 2 μm. Power consumption was uniformly low, ranging from 50 to 100 kwhr/ton of product depending upon the feed rate. One marble sample from Georgia was shown to be the most persistent grinding medium, with losses of less than half those of the other materials tested.

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ERRATA

Page 11, line 6: Sentence starting on this line should read as follows: The power consumption increases linearly with the residence time, as expected, since the power to the grinder is nearly constant with only minor variations in the measured torque and motor speed.

Page 13, line 5: Sentence starting on this line should read as follows: Increased solids content and/or increased sand:marble ratio in runs 8, 9, and 10 gave grinding results that were not significantly different from the results using 40 percent solids in a 2.5 sand:marble ratio, shown in figure 7.

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AUTOGENOUS ATTRITION MICROGRINDING OF CALCIUM CARBONATE MINERALS

by

J. P. Hansen,[1] E. G. Davis,[2] and G. V. Sullivan[3]

ABSTRACT

In order to help assure an adequate domestic supply of minerals essential to the Nation's economy and and security by developing more efficient minerals extraction technologies, the Bureau of Mines Tuscaloosa Research Center examined the application of autogenous microgrinding to calcium carbonate minerals. Treatment of minus 8-mesh Alabama marble resulted in a product 80 pct minus 5 micrometers (µm) at a power consumption of 70 to 80 kwhr/ton.

This study shows that coarse particles of calcium carbonate minerals are effective as the grinding media for the autogenous attrition grinding of these minerals. The minus 8- plus 30-mesh fraction is an effective grinding medium (sand) for particles smaller than 30 mesh. A 40-pct-solids slurry and a sand:marble ratio of 2.5 appeared to be near optimum. For residence times above 13 minutes, products were greater than 80 pct minus 5 µm and greater than 54 pct minus 2 µm. Power consumption was uniformly low, ranging from 50 to 100 kwhr/ton of product depending upon the feed rate. One marble sample from Georgia was shown to be the most persistent grinding medium, with losses of less than half those of the other materials tested.

INTRODUCTION

The use of the attrition grinding process to produce very fine particles exemplifies the Bureau of Mines' efforts to help assure an adequate domestic supply of minerals essential to the Nation's economy and security by developing more efficient minerals extraction technologies. Research on utilization of a coarse waste product from kaolin processing led to development and patenting of an attrition grinding process. This process has permitted the grinding of coarse waste kaolin into the fine sizes required and thereby has

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[1] Metallurgist (Faculty).
[2] Metallurgist.
[3] Supervisory metallurgist.
All authors are associated with the Tuscaloosa Research Center, Bureau of Mines, Tuscaloosa, Ala.

maximized the recovery of kaolin from domestic resources. It has also been effective in reducing the energy requirement for producing finely ground material. The process has also been used industrially for grinding TiO_2 pigment.

The Tuscaloosa Research Center has made several studies on the application of this patented attrition grinding process to a wide variety of products (4-5, 7-9).[4] Large-scale continuous tests have been conducted on attrition grinding and sizing of kaolin for use as paper-coating clays (2, 10-11). Autogenous attrition grinding has been applied to a North Carolina dunite to produce an olivine foundry sand (1, 6).

Recently there has been increasing interest in the grinding of calcium carbonate minerals because of their expanded use as paint pigments, extenders, and fillers, and also in plasterboard joint cement. Previous research on grinding of calcium carbonate minerals with Ottawa sand and some pioneering autogenous grinding studies with olivine have indicated that autogenous grinding of calcium carbonate minerals may produce a higher grade ground product at lower energy requirements than present practices. Autogenous grinding would improve the grade of the ground material by preventing its contamination with quartz abraded from the Ottawa sand grinding media (3). Rapid abrasion from the calcite grinding media should increase the amount of product and thereby make grinding more energy efficient.

The present investigation of the autogenous attrition grinding of calcium carbonate minerals was to determine: (1) if the coarse calcium carbonate particles would persist long enough at the coarse size to be effective grinding media; (2) if particles in the minus 50- plus 325-mesh range would grind fast enough so that they did not continuously accumulate in the mill; (3) the product size attainable at reasonable residence times and flow rates; and (4) the energy consumption for various sized products.

EQUIPMENT AND MATERIAL

The basic 5-inch grinder used in this investigation was described in an earlier publication (8). The coarse calcium carbonate particles which are required for autogenous grinding and hereafter referred to as "sand" were difficult to feed into the grinder. Therefore, to facilitate feeding these coarse particles, the grinder was modified to the design shown in figures 1 and 2. The grinder impeller recirculates the sand and slurry through the feed section rapidly and permits the addition of the feed material in that stream.

The feed used for most of the test work was a minus 1/4-inch calcium carbonate from Alabama, which was stage-crushed to minus 8-mesh in a cone-and-ring laboratory crusher. This was necessary because the plus 8-mesh material was caught and crushed between the impeller and stator of the grinder. Screen

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[4] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

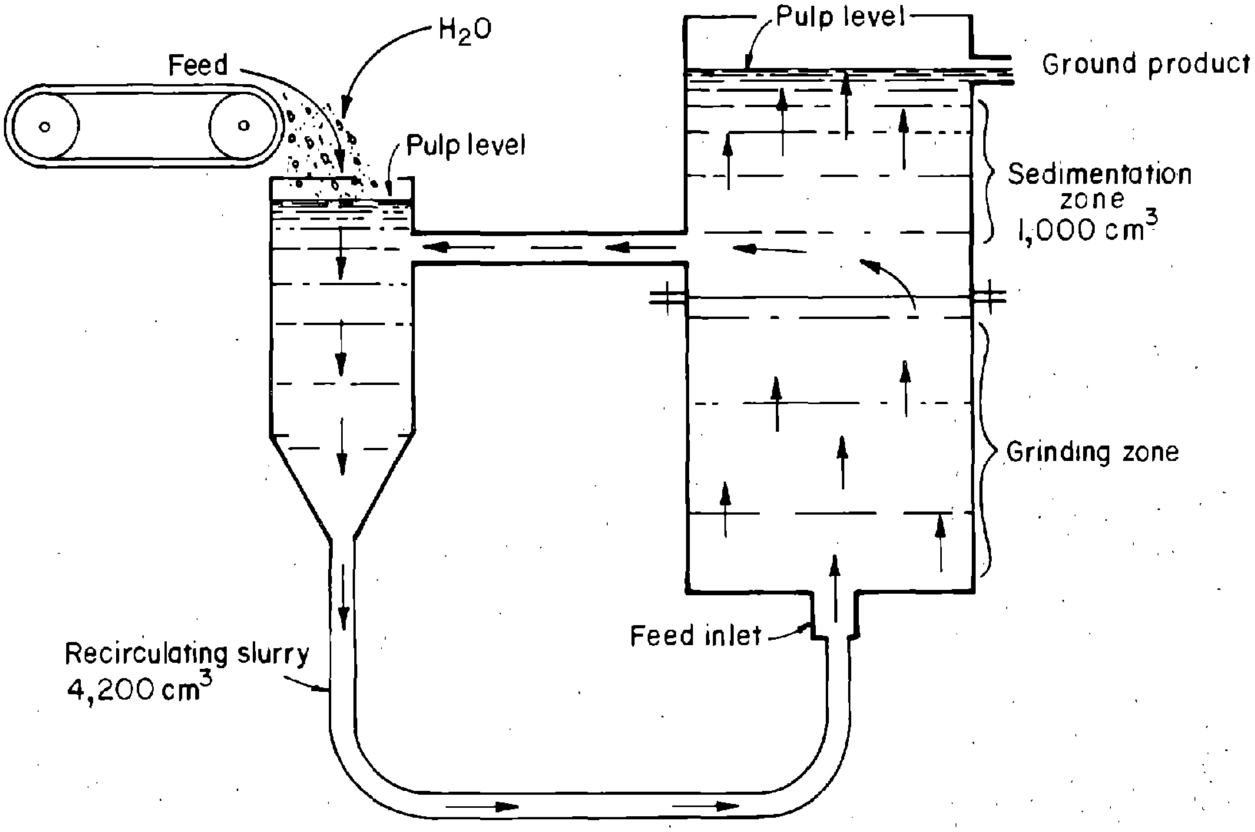

FIGURE 1. - Schematic of grinder modification.

analysis of the feed is shown in table 1. The grinding media (sand) are provided by the plus 30-mesh fraction of the feed. The fine material to be ground (mineral) is provided by the minus 30-mesh material in both the feed and the abraded grinding media.

TABLE 1. - Screen analysis of feed material

Screen size, U.S. No.	Weight, pct retained on	Cumulative weight, pct coarser than
16.........	47.4	47.4
20.........	9.5	56.9
30.........	6.2	63.1
40.........	4.7	67.8
50.........	4.9	72.7
70.........	4.9	77.6
100.........	6.2	83.8
200.........	8.1	91.9
325.........	5.4	97.3
Pan.........	2.7	100.0
Total.....	100.0	NAp

NAp Not applicable.

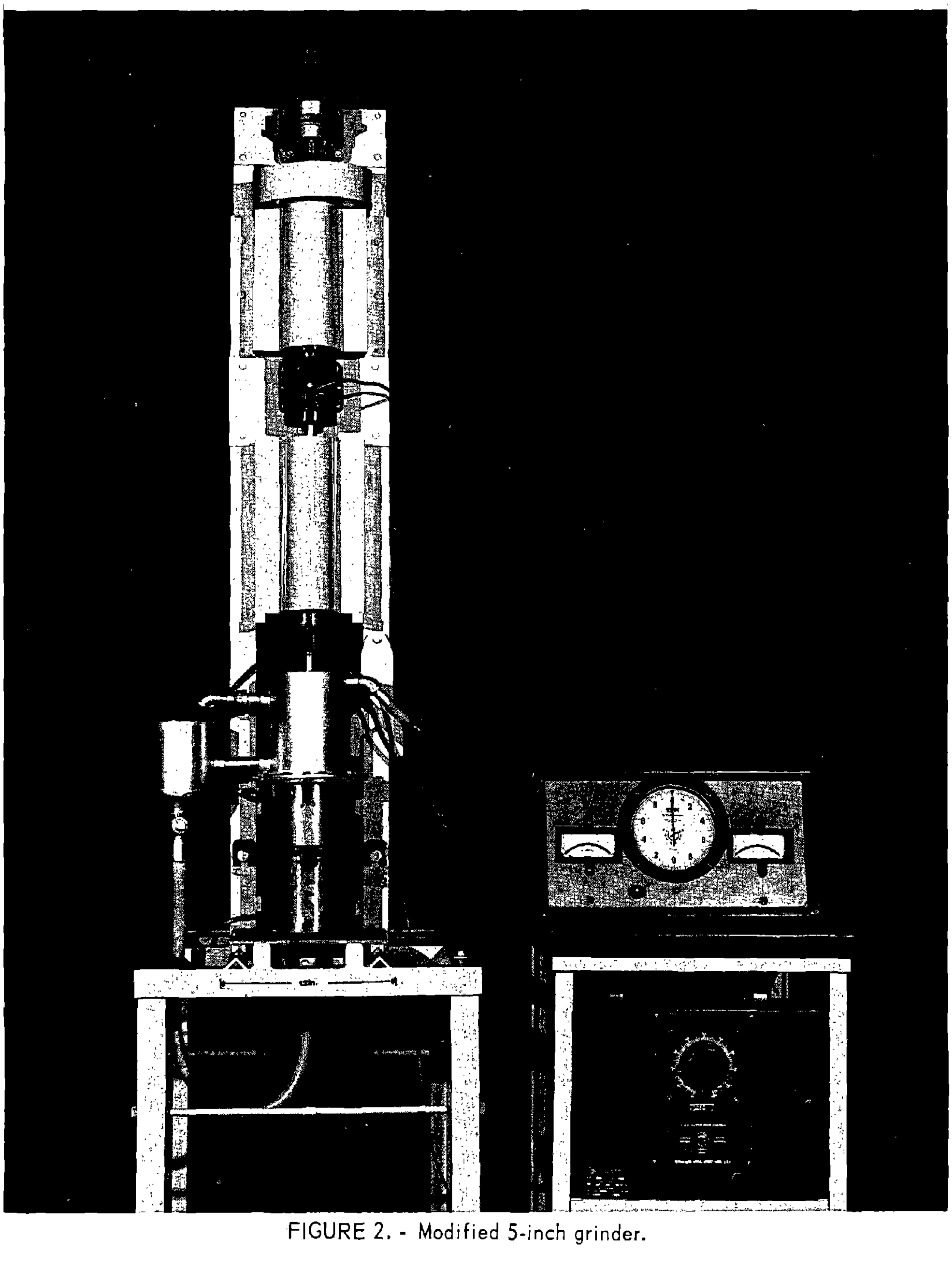

FIGURE 2. - Modified 5-inch grinder.

EXPERIMENTAL WORK

Prior Attrition Grinding of Calcium Carbonate Minerals

Stanczyk and Feld (9) studied the effects of sand size, solids content, and the sand:marble ratio for the grinding of marble using Ottawa sand grinding media. It was expected that the effects of these variables would be comparable for the grinding of fine marble with coarse marble sand. Therefore, these results are summarized in figures 3 and 4. The nomenclature used in the earlier publications and continued here is that, for example, a slurry containing 40 pct marble and a sand:marble ratio of 2.5 would contain the ratio of 40 g minus 30-mesh marble, 60 g water, and 100 g sand (plus 30-mesh marble) for an overall 70 pct solid:liquid ratio. Figure 3 shows that a high percentage of minus 2 µm material is produced when the grinding media are finer than 16 mesh, when the marble being ground is 40 pct solids or more, and when the sand:marble ratio is greater than 2.5. Figure 4 shows that the power required per ton of minus 2 µm product produced is most dependent on the marble content; the power consumption drops rapidly as the marble content is increased to 40 and 50 pct. The sand size and sand:marble ratio have little effect on the power requirements. The results indicate that for autogenous grinding the best results will probably be attained with grinding media (coarse marble) in the minus 16- plus 30-mesh range, solids content of the mineral being ground (minus 30-mesh marble) of between 40 and 50 pct, and a sand (plus 30-mesh marble):marble (minus 30-mesh marble) ratio between 2.5 and 3.0.

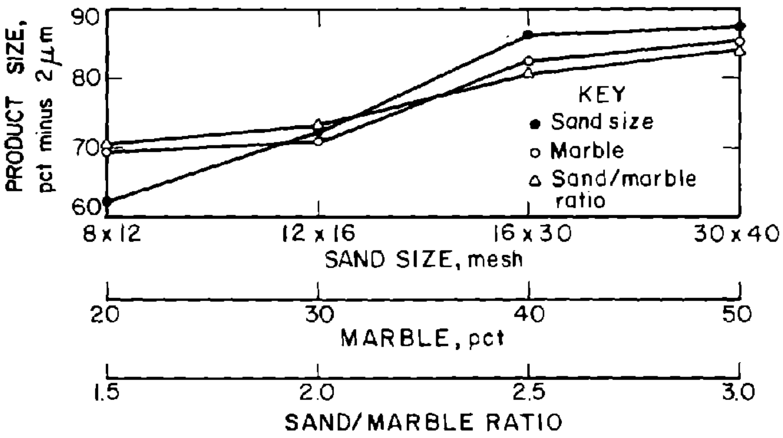

FIGURE 3. - Effect of sand size, marble content, and sand:marble ratio on the product size.

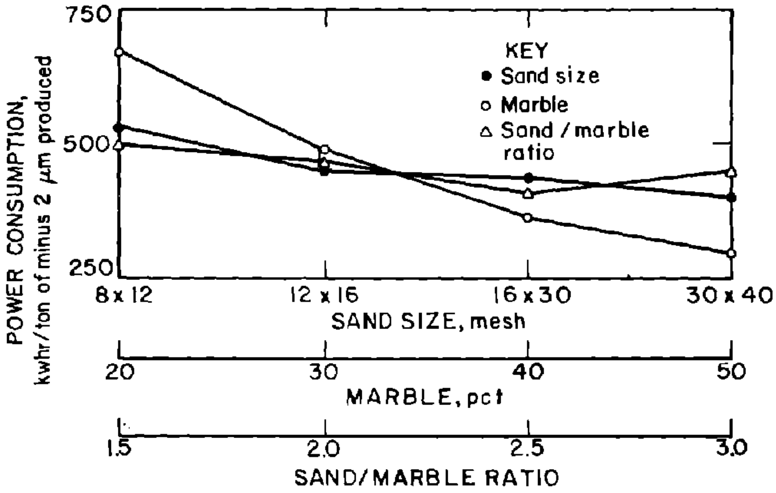

FIGURE 4. - Effect of sand size, marble content, and sand:marble ratio on the power consumption.

Batch Testing

Batch tests were run to establish a mathematical model that would predict the rate at which the coarse calcium carbonate would be

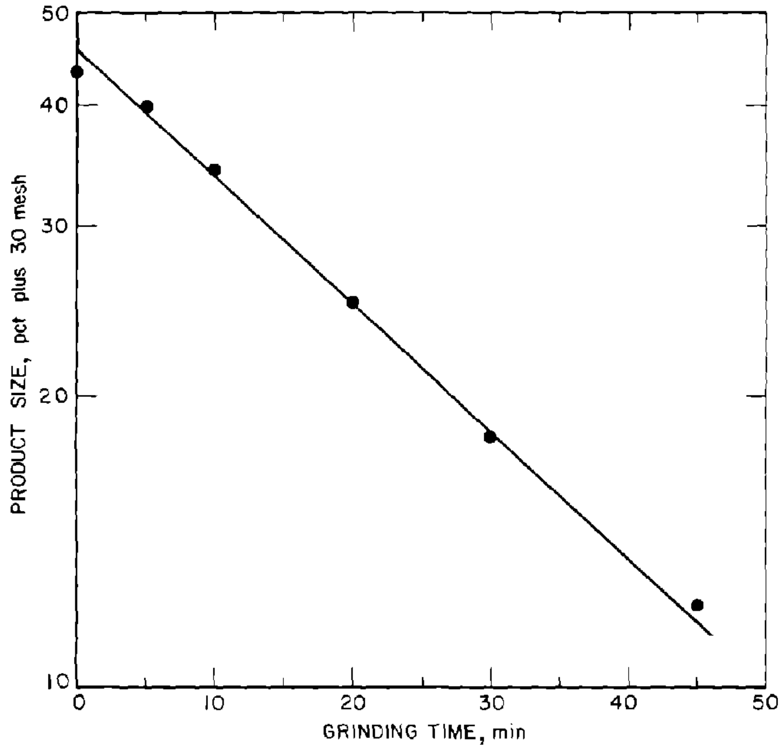

FIGURE 5. - Loss of plus 30-mesh particles with grinding time.

ground. This rate must be known in order to maintain a steady state in the continuous runs, since coarse material must be fed at the same rate as it is lost by grinding.

Batch tests were run by putting 3,060 cm^3 of water in the grinder, starting the grinder, and slowly adding 5,810 grams of the minus 8-mesh feed. After runs of 0, 5, 10, 20, 30, and 45 minutes, the slurry was drained from the grinder. The sedimentation size analyses of the minus 325-mesh ground products from the batch tests are shown in table 2. Since the loss of plus 30-mesh particles was expected to be first order, the log of percent coarser than 30-mesh material is plotted against time in figure 5. The linear relation between the log of percent coarser than 30-mesh material against time indicates that the loss of plus 30-mesh particles is a first order reaction. That is, $\frac{dC}{dt} = - KC$ and $\frac{dC}{C} = Kdt$, so that upon integration $\ln C = - Kt + I$ where C is the percent coarser than 30-mesh material, t is time (min), and K and I are constants. From these data the rate of loss of the percent plus 30-mesh material (dC/dt) can be calculated:

$$\frac{dC}{dt} = -0.03\ C \text{ (where C is percent plus 30-mesh)},$$

or weight loss of plus 30-mesh particles:

$$\frac{dw}{dt} = -0.03\ (58.10)C \text{ g of plus 30-mesh material ground per minute.}$$

TABLE 2. - Product size after batch grinding

	Time of grind, min											
	[1]0		5		10		20		30		45	
	Wt, g	Pct	Wt, g	Pct	Wt, g	Pct	Wt, g	Pct	Wt, g	Pct	Wt, g	Pct
Plus 30-mesh.......	2,523	43.4	2,318	39.9	1,983	34.1	1,445	24.9	1,061	18.2	703	12.1
Minus 30- plus 325-mesh..........	1,172	20.2	762	13.1	358	6.2	152	2.6	126	2.2	37	.6
Minus 325-mesh[2]....	2,115	36.4	2,730	47.0	3,469	59.7	4,213	72.5	4,623	79.6	5,070	87.3
Total............	5,810	100.0	5,810	100.0	5,810	100.0	5,810	100.0	5,810	100.0	5,810	100.0
Minus 5 µm[3]........	813	14.0	1,145	19.7	1,575	27.1	1,935	33.3	2,359	40.6	2,789	48.0
Minus 2 µm[3]........	331	5.7	476	8.2	697	12.0	819	14.1	1,052	18.1	1,267	21.8
Minus 5 µm[3] as pct of minus 325-mesh fraction..........	NAp	38.4	NAp	41.9	NAp	45.4	NAp	45.9	NAp	51.0	NAp	55.0

NAp Not applicable.

[1] Material charged to grinder and immediately discharged.

[2] By difference.

[3] By sedimentation sizing of minus 325-mesh fraction.

Photographs of the feed, the plus 30-mesh material remaining in the grinder after conclusion of the 45-minute grind, and the ground product are shown in figure 6. The photograph of the remaining coarse material shows the ground rounding of the coarse material remaining in the grinder. The photograph of the product shows that a redundant preponderance of the particles are smaller than 5 µm.

Continuous Grinds

Continuous tests were run to determine the effect of residence time, solids content, and the sand:marble ratio.

Procedure for the continuous test was to place the water in the grinder, start the grinder, add the initial charge of solids to grinder, and start feeder and water flow at established feed rates. A few minutes were required to fill the grinder to the overflow port, at which time the product overflowed. Feed rates and the overflow products were checked periodically during the run. Size analyses were made of the overflow product and the material in the mill at the conclusion of the test.

To calculate the initial charge and the residence times, the following. mathematical model was used (see fig. 1):

A. Recirculating volume is 4,200 cm^3 and contains all of the sand in addition to slurry.

B. The volume of the sedimentation zone is 1,000 cm^3 and contains only slurry.

For example, the initial charge and feed rate for a continuous autogenous run similar to a batch media-ground test at 40 pct solids and a 2.5 sand: marble ratio would be as follows:

(1) Sedimentation section:

Volume	= 1,000 cm^3
Solids (plus 30-mesh)	= 0 wt-pct
Solids (minus 30-mesh)	= 40 wt-pct
Water	= 60 wt-pct
S.G. calcite	= 2.72
Solids (plus 30-mesh)	= 0 vol-pct
Solids (minus 30-mesh)	= 19.7 vol-pct
	= 197 cm^3
	= 536 g
Water	= 80.3 vol-pct
	= 803 cm^3

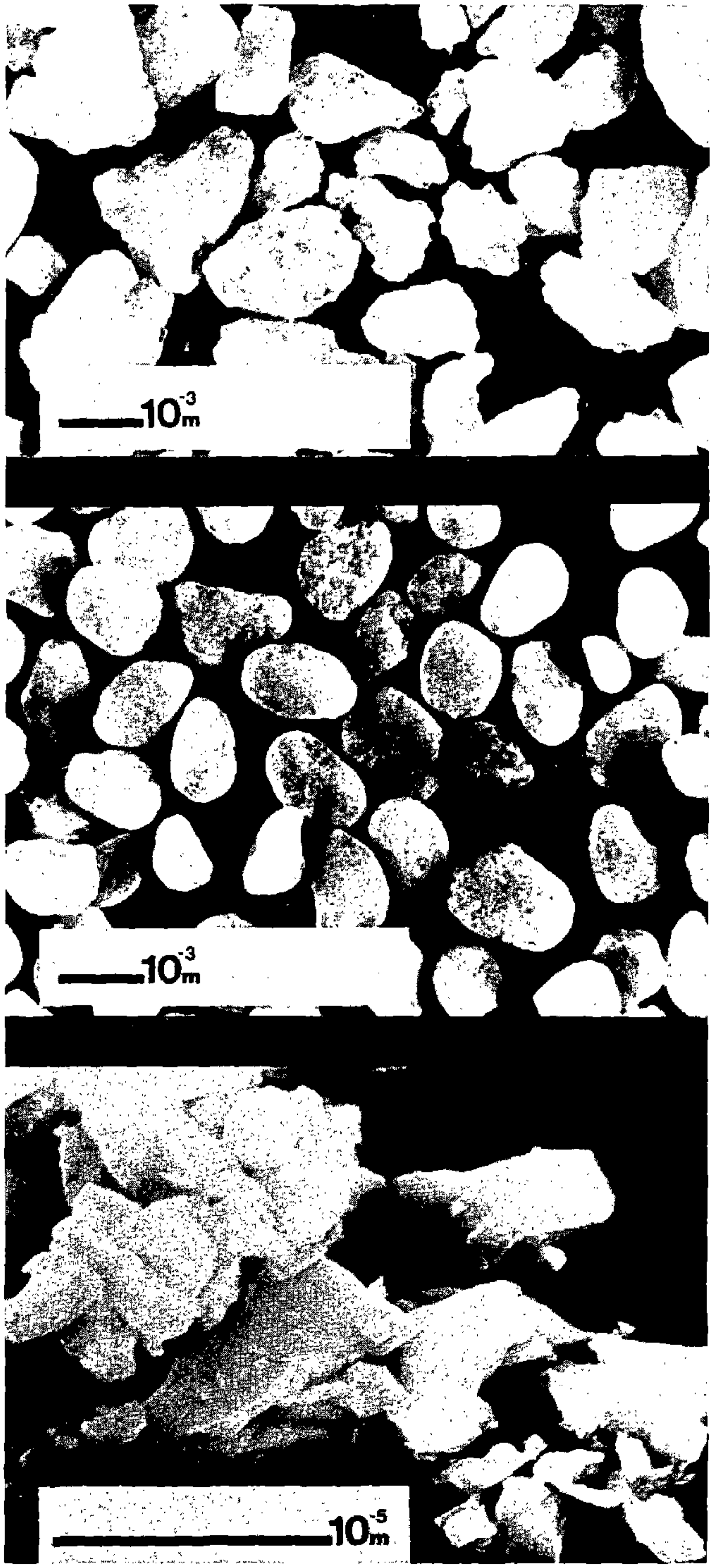

FIGURE 6. - Feed, coarse particles remaining in the mill (sand), and product (top to bottom).

(2) Recirculating section:
Volume = 4,200 cm^3
Solids (plus 30-mesh) = 50 wt-pct of sand plus slurry
Solids (minus 30-mesh) = 20 wt-pct of sand plus slurry
Water = 30 wt-pct of sand plus slurry
S.G. calcite = 2.72
Solids (plus 30-mesh) = 33.0 vol-pct
= 1,386 cm^3
= 3,770 g
Solids (minus 30-mesh) = 13.2 vol-pct
= 554 cm^3
= 1,507 g
Water = 53.8 vol-pct
= 2,260 cm^3

(3) Total charge:
Volume = 5,200 cm^3
Solids (plus 30-mesh) = 3,770 g
Solids (minus 30-mesh) = 2,040 g
Water = 3,060 cm^3

The plus 30-mesh material in the starting charge was recycled from the previous run. The plus 30-mesh material was screened from the material remaining in the grinder at the conclusion of the previous test so that the coarse material in the grinder would be close to the steady state condition. The minus 30-mesh material was obtained from the feed sample.

The feed rate of plus 30-mesh particles was calculated to maintain the 3,770 g charge in the grinder. From the batch testing it was determined that

$\frac{dC}{dt}$ = loss of plus 30-mesh particles = -KC where C is the percent plus 30-mesh in the entire grinder and K = 0.03.

$$\frac{dC}{dt} = 0.03\ (3,770/5,510)\ (100) = 1.95 \text{ pct/min.}$$
$$= 0.195\ (5,810) = 113 \text{ g plus 30-mesh/min.}$$

The solid feed rate must be calculated using the amount of plus 30-mesh material in the feed to supply 113 g/min of the coarse calcium carbonate particles, which serve as the grinding media to maintain the 2.5 sand:marble ratio. Enough water is fed to maintain 40 pct solids in feed and overflow products, based upon solid feed rate.

The average residence times (θ) can then be calculated from the definition: $\theta = \frac{V}{v}$, where V is the quantity in the system and v is the flow rate. The average residence time used was the mean of:

θ water = 3,060 cm^3/(feed rate of water in cm^3/min), and
θ minus 30-mesh = 2,040 g/feed rate of solids in g/min.

These residence times should be equal, but vary slightly because of variations in the feed rates.

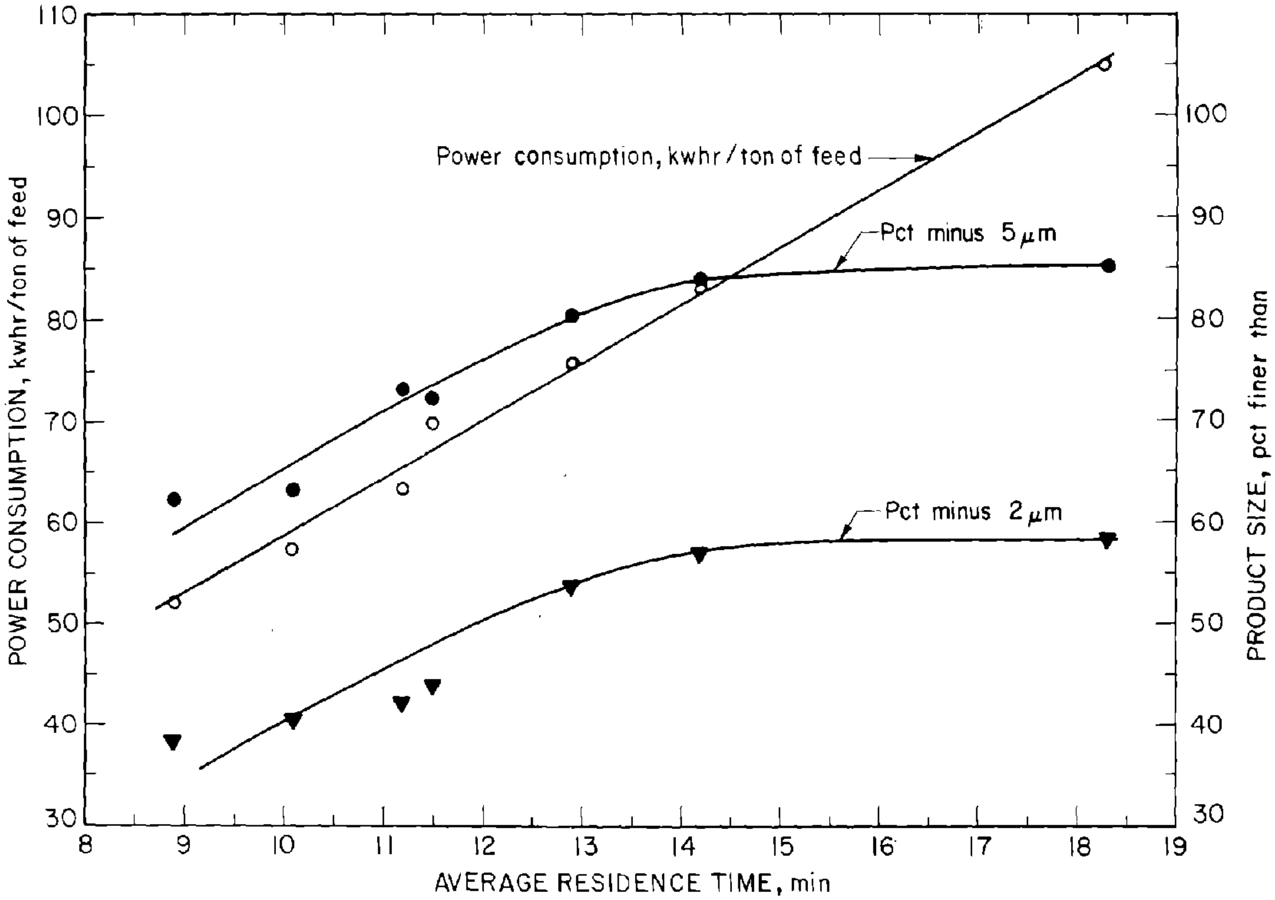

FIGURE 7. - Variation of product size and power consumption with average residence time.

Seven continuous grinds were made at 40 pct solids and a sand:marble ratio of 2.5. The feed rate was varied to change the average residence time. The amount of plus 30-mesh feed was held constant at 113 g/min, but the total feed was varied by additions of minus 30-mesh material or screening out the minus 30-mesh fraction from the coarse material. The results are tabulated in table 3 and shown graphically in figure 7. The power consumption increases linearly with the residence time, as expected. Since the power to the grinder is nearly constant with only minor variations in the measured torque and motor speed. The percent minus 5 µm and 2 µm in the product increases with the residence time until about 13 minutes, at which time the product is 80 pct minus 5 µm and 54 pct minus 2 µm. Increasing the residence time above 13 minutes has only a small effect on the size of the product. After 18.3 minutes, which is the maximum residence time used for this material, the product is 85 pct minus 5 µm and 58 pct minus 2 µm. Producing a finer sized product will require an additional media grinding stage or some better in-circuit sizing in which the oversized fraction is returned to the grinder.

TABLE 3. - Summary of continuous grinding results

Grind	Pct solids	Sand: marble	Feed[1], pct plus 30-mesh	Feed rate, g/min		Av. res. time, min	Product: pct finer		Power used, kwh/ton of feed
				Solid	Liquid		2 µm	5 µm	
1..	40	2.5	70	180	278	11.2	42.0	73.0	65.7
2..	40	2.5	63	175	270	11.5	44.0	72.0	69.7
3..	40	2.5	70	157	240	12.9	53.7	79.9	75.4
4..	40	2.5	56	208	297	10.1	40.5	63.0	56.9
5..	40	2.5	49	231	341	8.9	38.1	62.0	52.1
6..	40	2.5	80	145	215	14.2	56.8	83.4	83.0
7..	40	2.5	100	112	167	18.3	58.0	85.0	105.0
8..	45	2.5	70	169	208	13.58	52.5	83.4	70.0
9..	50	2.5	70	185	184	13.90	54.0	80.0	68.0
10..	40	3	70	175	267	11.03	42.0	76.0	72.0
11..	45	3	70	190	232	11.54	(2)	(2)	(2)
[3]12..	40	2.5	83	165	248	12.35	44.0	72.0	66.0
[3]13..	40	2.5	83	150	225	13.60	47.0	80.0	84.0
[4]14..	40	2.5	70	163	243	(5)	73.0	90.6	(5)
[4]15..	40	2.5	40	162	243	(5)	76.0	90.0	(5)
[4]16..	40	2.5	20	150	225	(5)	80.0	94.0	(5)

[1] Obtained by screening on 30-mesh screen and removing undersized fraction.

[2] 5-inch grinder could not move the material past physical limits of grinder.

[3] Marble from Vermont.

[4] Marble from Georgia.

[5] Run did not reach steady state.

Four additional continuous grinds were made with increased solids content in the slurry and/or sand:marble ratio. Two other calcium carbonate samples were ground to determine if the coarse material from the more crystalline varieties is more resistant to abrasion in the attrition grinder. The results of these runs are also tabulated in table 3. Increased solids content and/or increased sand:marble ratio, shown in figure 7. The power consumptions are not directly comparable to those from figure 7 in that the average residence time depends on both the amount of charge in the grinder and the feed rate, while the power consumption is expected to vary inversely with the solid feed rate. The power consumption versus the reciprocal feed rate for all grinds, plotted in figure 8, shows that the power consumption follows the inverse relation. There is some variation of power consumption because of slight differences in the viscosity of the charge, torque, speed of the grinder, and/or measuring errors.

In grind 11, the solids content was increased to 45 pct and the sand:marble ratio was increased to 3. The grinder could not pump the slurry because the physical limits of the grinder had been exceeded.

Grinds 12 and 13 were run on a sample of Vermont marble. The grinding results show that the Vermont marble does not grind quite as well when compared with the ore from Alabama in figure 7. The minus 5 µm is about 2 pct less and the minus 2 µm is about 8 pct less than for comparable grinds.

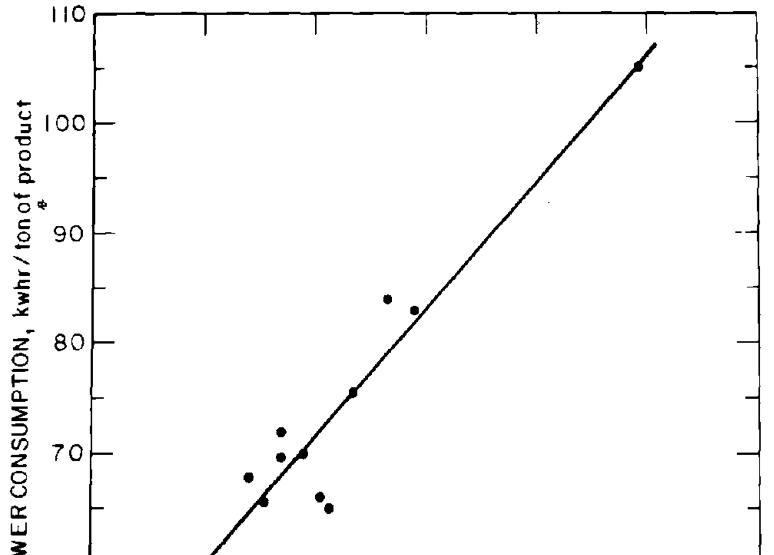

FIGURE 8. - Power consumption versus the reciprocal feed rate.

Grinds 14, 15, and 16 were made with a Georgia marble. In grind 14, feeding 113 g/min of plus 30-mesh material, the amount of plus 30-mesh material increased from an initial charge of 3,770 g to 7,374 g at the conclusion of the 70-minute run. The plus 30-mesh grinding media abraded at only about half the rate of other marble samples, indicating that it would be more persistent as a grinding medium. The ground product was 90.6 pct minus 5 µm and 73 pct minus 2 µm. In grind 15 the feed rate of plus 30-mesh material was reduced to 65 g/min, but the plus 30-mesh fraction increased from 3,770 g to 6,780 g during the 80-minute grind. In run 16 the feed rate of plus 30-mesh material was further reduced to 30 g/min, and the coarse fraction still increased from 3,500 to 4,800 g during the 60-minute grind. The experimental work using the Georgia marble was terminated without having achieved steady state because the sample was exhausted. In any case, the coarse fraction of this particular sample appears to be a very much more persistent grinding medium than any of the other materials autogenously ground.

CONCLUSIONS

Plus 30-mesh calcite mineral particles were effective as the grinding media for autogenous attrition grinding of marble. The optimum particle size, sand:marble ratio, and solids content in the slurry are essentially the same as when using Ottawa sand as the grinding media.

The percent minus 5 µm and minus 2 µm in the ground product depends upon the residence time in the mill. When the residence time was above 13 minutes, the ground product was more than 80 pct minus 5 µm and more than 54 pct minus 2 µm.

Power consumption was uniformly low, ranging between 50 and 100 kwhr/ton of products, depending on the feed rate.

The marble sample from Georgia was an especially persistent grinding medium, with grinding losses of less than half those of the others tested.

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