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PRELIMINARY COMPARISON OF PRODUCTS FROM FOUR MICA-GRINDING TECHNIQUES

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Background of Tests

Since 1967, intermittent grinding tests have been run on the mica float concentrate produced by Foote Mineral Company at their Kings Mountain plant. The tests were run on this particular mica because it presented a problem with respect to bulk density reduction, yet had very good color characteristics. Should a grinding technique be discovered which would reduce bulk density of this mica, it might then be possible to find new markets for it. Further, such a technique could possibly be used advantageously on other micas.

It was thought that the above objective might be attained by a grinding system which would exert maximum possible lateral force, parallel to the flat surfaces of the mica, rather than crushing or impact which is random and would have more tendency to fracture along planes intersecting the flat surfaces, thus creating a blockier product of higher bulk density. Such a "lateral-force" grinding technique might effectively delaminate a mica having a more brittle crystal structure and/or stronger laminar adhesion.

R. T. Hukki* describes a mill which might maximize the desired lateral forces. Essentially, the attrition mill of Hukki is rotated at supercritical speed, that is, the shell rotates too fast for the

*Hukki, R.T. - "Fine Grinding at Supercritical Speed". Mining Engineering, vol.10, no. 5, May 1958, p.581. (or see vol.311, AIME Transactions) grinding rods or balls to cascade, and so instead they slide. Thus, the only grinding in such a case takes place in the area between mill shell and grinding media. Hukki's research disclosed some noteworthy findings, among them these:

1) Increased grinding efficiency under supercritical speed conditions is apparent at as high as 210 percent of critical rotation speed.
2) Grinding efficiency (work done vs. energy input) at optimum supercritical speed equals or excels conventional subcritical cascade grinding.
3) Supercritical attrition grinding tends to grind finer than the conventional method.

Since mica is generally ground to minus 200 mesh or finer, point (3), above, was worth noting.

In the planned MRL research, the attrition-mill concept was favored over the muller principle for two reasons:

1) The muller in operation does not exert enough lateral force in its grinding.
2) The muller is a batch device, and it was desired to perform research toward a continuous-flow unit.

The Sample

Sample No. 3417 was a recleaned muscovite froth product, essentially minus 30 mesh, produced by Foote Mineral Company. This sample was obtained wet from Kings Mountain Mica Company, which purchases it from Foote to incorporate into ground mica for color improvement.

Screen analysis and chemical assays on Sample No. 3417 are shown in Table 1.

Table 1

Screen & Chemical Analysis,
Plus Bulk Densities, Sample No. 3417

U.S. Screen	Percent Weight	Bulk Dens.	Chemical % K_2O	Chemical % Na_2O	Chemical % Li_2O	Chemical % Fe_2O_3	Chemical % Loss
+30	3.9	24.8					
-30+40	7.5	24.8					
-40+50	12.8	32.8	10.01	0.78	0.13	1.49	4.55
-50+60	9.9	36.5					
-60+70	11.5	38.3					
-70+100	18.9	40.2	10.01	0.77	0.18	1.49	4.43
-100+150	12.4	40.2	10.01	0.77	0.18	1.49	4.45
-150+200	8.9	36.7	9.84	0.77	0.18	1.49	4.40
-200+270	3.8	34.9	9.45	1.03	0.18	1.47	4.09
-270+325	3.0	31.8	8.90	1.06	0.19	1.44	4.04
-325	7.4	25.5	8.45	1.56	0.24	1.40	3.63
Total	100.0	37.1*					

*Actual bulk density of total product.

The chemical data of Table 1 show a pronounced change beginning in the 150-200 mesh range. This can be stated with reasonable certainty to be due to increasing quantities of non-micaceous minerals, mainly feldspar and quartz.

Preliminary Attrition Mill

To establish preliminary data with minimum effort and expense, a batch attrition mill was constructed as follows. An Abbe' ceramic pebble-mill shell having a cylindrical grinding chamber 7 3/4 inches in diameter and 5 1/4 inches long was loaded with five rods, each 4 7/8 inches long and close to 1 1/8 inches in diameter. These rods were made by filling ceramic cylinders with lead to increase their weight. Each weighed close to 450 grams. On all tests cited, this assembly was closed with a lid and rotated at 55 RPM. A ceramic assembly was chosen to avoid contamination by abraded metal which would be introduced by one made of steel. The rods never cascaded, but slid along the mill shell in a parallel row during mill operation. Inspection of the mill when running open indicated that the rods slid to a large degree and did not contra-rotate fully with the mill shell.

It is emphasized here that this mill assembly can be used only to give qualitative evidence regarding the basic principle of Hukki-style wet attrition-grinding of mica, but no significant parameters on variables relating to most efficient application of that principle.

Preparation of Sample

Based on preliminary tests, it was established that a short wet grind of the sample in the mill, followed by wet-screening and discarding of all minus 200 mesh material, resulted in a mica product with practically all non-micaceous minerals removed. This procedure was designated as Test 1. Procedure and results are as follows. A 100 gram batch of head sample was milled for one hour at 25 percent solids with ten pounds per ton of tetrasodium pyrophosphate, then wet-screened on 200 mesh, and the minus 200 mesh discarded. Minus 200 mesh was 29.6 percent and had an ignition loss of 3.80 percent. Plus 200 mesh was 70.4 percent, with ignition loss of 4.46 percent. Comparison of these ignition loss figures with those of Table 1 can be made.

All tests cited hereafter were carried out on a plus 200 mesh fraction of the sample, prepared as cited. About 25 percent of the mica in the head feed was thereby discarded in the minus 200 fraction, in order to be assured of a pure, or nearly pure, mica sample for experimentation.

Bulk Density of Plus 200 Fractions

Comparison can be made among three plus 200 mesh fractions:

1) That of the original head sample.

2) The plus 200 fraction after a one-hour grind.

3) The plus 200 fraction after a 16-hour grind.

All these are relatively pure mica, based on ignition loss. Table 2 gives pertinent data.

Table 2

Characteristics of Plus 200 Mica, Milled Versus Un-milled

	Screen Percent												
	+30	-30 +40	-40 +50	-50 +60	-60 +70	-70 +100	-100 +150	-150 +200	-200	Ign. Loss	Bulk Dens.	% of Head Sample	Test #
Plus 200, head sample	4.5	8.7	14.9	11.5	13.4	22.1	14.5	10.4	0.0	4.45	36.0	85.8	0
Plus 200, milled 1 hr. & re-screened (Test 1)	3.4	6.8	13.1	8.8	9.6	22.1	17.6	18.6	0.0	4.46	25.7	70.4	1
Ditto, milled 16 hours	0.7	3.2	8.1	6.4	7.7	21.2	23.4	29.3	0.0	4.46	14.3	33.5	3

It can be seen from Table 2 that the one hour and 16 hour millings have resulted in plus 200 mesh mica products having screen analyses not too different from the head feed, but having very much lower bulk density-this being principally caused by increased delamination as compared to the head sample.

Other grinds on the plus 200 mesh product from Test No. 1 are briefly set forth in Table 3.

Table 3

Effect of Grinding Time on Bulk Density of Plus 200 Mesh Product

Test #	Hrs. Grind	B.D., +200	% Yield from Head Feed, +200	Remarks
"0"	0	36.0	100.0	Also cited in Table 2
1	1	25.7	70.4	" " " " "
4	5	18.5	53.7	-
3	16	14.3	33.5	Also cited in Table 2. Started with complete hd.fd.
5	22	12.7	27.4	-

The data of Table 3 leads to the same conclusions as that of Table 2.

Tests on the Plus 200 Mesh Milled Product from Test No. 1

Test No. 1 was repeated a number of times, with close-to-identical results, in order to create a reserve of plus 200 mesh mica on which to perform subsequent tests. This plus 200 mesh material was ground for varying lengths of time, the object in each case being to create a minus 200 mesh product of lowest possible bulk density. In some cases a test series was run, taking the coarser screened or hydroclassified portion from a preceding test and milling it further.

Losses of fines during hydroclassification were often high. This was due to batch overgrind, plus a setting-aside, for the present, of the problem of working out an efficient hydroclassification system. The losses from these sources can be reduced when more refined milling techniques and equipment are developed in the future. Therefore, calculations of yield against head feed are not regarded as meaningful at this stage of research.

As in the case of the plus 200 mesh oversize remaining after each test, the minus 200 mesh product became lower in bulk density as grinding time increased. That is, the minus 200 mesh product derived from each succeeding stage grind was lower in bulk weight than the preceding. Table 4 cites a series of tests and results to illustrate this point.

Table 4

Effect of Milling Time on Bulk Density of Minus 200 Mesh Product

Test #	Conditions	B.D. of -200 Product (lbs/cu.ft.)	-200 Yield: % of Test Input feed
2	Plus 200 from Test 1,milled 3 hrs.(200g.)	12.5	14.2
4	" " " " ",milled 4 hrs.(215g.)	12.2	18.5
5	Plus 200 from Test 2,milled additional 19 hrs.(157g.)	9.3	35.3

In the above tests, the minus 200 mica product was allowed to settle 16 hours in a full six-gallon plastic bucket, then decanted. Losses ran between five percent and nine percent of input feed.

Parallel with the preceding tests, another test group was checked out with respect to a narrow screen fraction; the 200 to 325 mesh fraction only. Table 5 gives data on a series of stage grinds, with the plus 200 fraction re-introduced after each stage.

Table 5

Effect of Milling Time on Bulk Density of Minus 200 Plus 325 Mesh Product

Test #	Conditions	Total Hrs. Grind	B.D. of -200+325 Fraction (lbs/cu.ft.)
25	1 + 2 hrs. stage grind	3	18.4
26	1 + 2 + 2 hrs. stage grind	5	17.1
27	1 + 2 + 2 + 2 hrs. stage grind	7	16.5
28	1 + 2 + 2 + 2 + 4 hrs. stage grind	11	15.8
18	1 + 16 + 16 hours stage grind	33	13.4
23	1 + 22 + 22 + 8 hrs. stage grind	53	10.5

The data of Table 5 continues to demonstrate that progressively finer delamination occurs as this particular mica is milled in this device over an increasing time span.

Another series of tests was run in which a fine-milled product was separated by hydroclassification. The hydroclassification yielded a minus 400 mesh mica, size characteristics of which were difficult to determine with any equipment the MRL has on hand. However, this product was considered significant because of its very low bulk density, and because it was possibly a typical product from a milling process which is reputedly efficient at milling finer than others. The method of hydroclassification was this; the milled sample was washed out, diluted to about ten gallons in a ten gallon can, agitated, settled for ½ hour, and decanted. The decanted thin pulp was then settled for 48 hours, decanted again, and the settled solids saved as final product. Data on tests run in this manner is shown in Table 6. The three tests cited are actually three stages of one test; beginning with a plus 200 mesh product of the "purge" test group (Test No. 1, Table 2), stage-grindings of 22 hours, then 22 hours more, then finally 8 hours were carried out, with earlier stage tests repeated so as to have enough oversize for succeeding tests. Feed input in each case ranged between 144 and 163 grams per run.

Table 6.

Hydroclassified Products after Successive Milling Runs

Test #	Hours Milling Time	Fine-Milled Product % of Feed Input	Fine-Milled Product Lbs.Per Cu. Ft.	% Loss Hydro.O'flow
21	(1) + 22 = 23	29.3	7.5	18.0
22	(1 + 22) + 22 = 45	35.7	6.8	27.5
23	(1 + 22 + 22) + 8 = 53	20.4	6.9	12.5

Calculating the results of the three tests of Table 6 as one test, the following figures are arrived at for an initial four-stage feed input of 600 grams:

Total fine-milled product: 53.1% of feed input

Hydroclassification losses: 29.8%

Oversize ("Sinks"): 17.1%

Total: 100% (of o'size after "purge")

Bulk density of product: Below 7.5 lbs/cu. ft.

The hydroclassification technique, if used on quartz particles, would have yielded a product ranging between two and ten microns in average diameter. Considered here are ambient temperature, known average settling rate of quartz particles of various diameters, and the depth of the container, all of which enter as factors into calculations based on Stokes' Law. Since this mica product all passed through 400 mesh (37 microns), the major axis dimensions must be below that size.

Based on tests to date, it cannot be said whether a fine-milled product of the sort just described can be made with even lower bulk density, without additional refinements in technique. However, one test (No. 12) was run in which a portion of pre-milled (for 1 hr.) pre-screened (plus 200 mesh) head feed was roasted at 1200°F, then milled for 16 hours and hydroclassified as the tests of Table 6. This yielded a fine-milled hydroclassified product which was 32.2 percent of the input feed and had a bulk density of 5.4 pounds per cubic foot; considerably lower than the tests of Table 6.

As previously stated, the mica of which Sample No. 3417 is representative has had the reputation of being impossible to grind to low bulk density. There was some problem in obtaining actual samples of this mica ground by various commercial techniques. One commercial wet-grinder of mica stated that his fine-ground minus 200 mesh product needed to have a bulk density specification of not over 15 pounds per cubic foot, and that the Foote mica would have to be brought to that specification in order for it to be fully useful to him. He claimed the wet-milling technique he was employing did not reduce its bulk density below 19 lbs., and so he could blend in only a limited quantity of it, despite its good color. A sample of Foote mica was obtained which had been ground to minus 200 mesh in a Majac fluid energy mill. This had a bulk density of 52 pounds per cubic foot. The 200-325 mesh fraction alone had a bulk density of 70.6, or roughly 6½ times that of the same screen fraction from wet attrition-milling (Test 23, Table 5). It is of interest to note that an easily-delaminated green muscovite which is Majac-ground will yield a minus 200 product of about ten to twelve pounds per cubic foot bulk density, which emphasizes still further the special delamination problems presented by the Foote mica.

Several tests were run in the Laboratory using different milling techniques. One employed wet-pebble milling in an Abbe' laboratory mill. After grinding 128 grams of sample for four hours, 80 percent of the sample was reduced to minus 200 mesh. Bulk density of the minus 200 was 14 pounds per cubic foot. The 200-325 mesh fraction alone of this product was 17.7 pounds per cubic foot, giving indication that wet pebble-milling appeared a doubtful means for good delamination of this mica.

In another grinding test, about 300 grams of sample was attrition-scrubbed for 20 minutes at thick pulp (62 percent solids) so that the pulp reached a temperature of about 150°F. This created a new minus 200 mesh product which was 6.4 percent of input. Results were inconclusive and show need for further research along this line. The minus 200 mesh product had a bulk density of 12.6 pounds per cubic foot; the 200 to 325 mesh fraction alone, 14.3. However, the remaining plus 200 mesh fraction measured 44.1 pounds per cubic foot, and prior to this "scrub-grind" this fraction was only 25.7 pounds per cubic feet (Table 2). Thus, there is strong indication that this attrition may have separated off a small percentage of delaminated minus 200 mesh flakes which were loosely attached, but was unlikely to continue producing much more of the same.

Test on Another "Problem" Mica

Mr. Hugh Lancaster of Kings Mountain Mica Company had furnished the MRL with a sample of mica ore, the mica from which had reputedly never been reduced to a bulk density acceptable to a user. This sample, No. 3435, was a mica-quartz schist from Colorado.

By wet rod-milling, desliming, and flotation (USBM-type mica float) including cleaner float, an extremely white, minus 35 mesh mica concentrate was separated, having a bulk density of 50.6 pounds per cubic foot. Repetitions of this procedure secured 500 grams of mica concentrate. Next, "purge" grinding, as with Sample No. 3417, was employed, and the minus 200 mesh then screened out. This produced a plus 200 mesh product about 48 percent of input (primary grind was too fine), with a bulk density of 37 pounds per cubic foot. A 140-gram portion of this was further milled for 12 hours and yielded a minus 400 mesh hydroclassified product, 32.8 percent of input, with a bulk density of 5.9 pounds per cubic foot. The remaining coarser hydroclassified fraction measured 14.3 pounds per cubic foot. Time and sample quantity did not permit further research, but under essentially the same grinding conditions this mica responded in a manner closely resembling the Foote product.

Conclusions

The Hukki attrition-grinding principle, which in theory should exert a good deal of lateral force along mica flakes, appears to operate in the manner hoped for. Other grinding methods, applied to the Foote mica, did not reduce bulk density to the same degree.

Future Research

After further brief testing of a few variables using the attrition mill now in use, a small continuous-feed, closed-circuit wet attrition mill should be constructed in which the following variables can be extensively checked:

1) Relative and absolute surface speeds, mill versus grinding rod.
2) Pressure on grinding surface
3) Rate of mica feed
4) Pulp density of grind
5) Additives
6) Duration of grind
7) Method of size classification

This research should be continued confidentially with the possibility of patenting the basic technique as a novel means of improved mica grinding, which creates lower bulk density through better delamination.

One area yet to be explored is the response of easily-delaminated micas to this type of grinding. Some preliminary tests indicate that this type mica will delaminate to a certain low bulk density but not further. The question arises whether delamination will, or will not, go beyond a given point when grinding media pressure is at a certain level.

In addition to the above research on mica, there is the possibility of investigation of the effects of this grinding on such minerals as talc, pyrophyllite, and asbestos, which might acquire new or better characteristics from it, permitting exploitation of lower-grade ores or deposits.

Finally, it might be worthwhile to investigate the merits of this type grinding in the production of fine-ground feldspar, quartz, and other granular minerals. It is presently standard practice to dry-grind these below 100 mesh. However, wet attrition-grinding coupled with a good system of wet size classification might demonstrate improved efficiency, lower dust hazard, and closer size distribution.