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WET ATTRITION-GRINDING OF MICA: CONTINUATION OF RESEARCH TOWARD OPTIMUM TECHNIQUES AND A PRODUCTION PROTOTYPE

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by
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BACKGROUND OF TESTS

In Progress Report No. 69-42 of January 1970, preliminary tests employing the principle of attrition-grinding of wet mica pulp gave superior results in terms of lower bulk density of fine-ground mica product. Further, the mica sample used (No. 3417) was of a type which hitherto had been impossible to both fine-grind and delaminate properly. This demonstrated the probable superiority of the wet attrition technique with micas having unusually strong laminar bonds: such micas tend to fracture into "booky" pieces rather than to delaminate.

PURPOSES OF TESTS

Further development of the wet attrition technique was needed in order to bring certain variables closer to optimum conditions, and also to determine, if possible, what techniques could be used to impart to the final product the qualities generally known as slip and sheen, which characteristics will be frequently referred to during this report. The products of tests reported up to this point had been observed to have a generally undesirable chalky appearance and texture similar to dry-ground mica, even though low in bulk density like water-ground mica. In addition to establishing techniques imparting slip and sheen, it was desired to find a means of objectively measuring these characteristics. No such measuring techniques appear to be known in the mica industry, although the paper industry has standard tests involving some measuring techniques which could possibly be borrowed. A still further purpose of this current research was to establish data tending to encourage or discourage the design and construction of a small continuous-production prototype attrition mill. If constructed, this might usefully turn out quantities of product for further variables testing and for samples to potential users, and produce firmer data regarding production performance of the basic mechanical setup.

THE SAMPLES

Principal sample used (No. 3569) was one which was nearly identical to Sample No. 3417 of the previous report. Both these samples were recleaned muscovite froth products, essentially minus 20 mesh, produced by Foote Mineral Company. They were obtained wet from Kings Mountin Mica Company, which uses this very white mica in limited quantites for color improvement of their ground mica products. The quantity used by Kings Mountain Mica Company must now be limited because the Foote mica does not delaminate to low bulk density.

Sample No. 3569 of this report was prepared for grinding in a somewhat different manner than No. 3417, and all tests on it were run, not on the total head sample, but on the head sample without most of the minus 200 mesh fraction. Table 1 shows screen size data on Samples No. 3417 (total) and No. 3569 (screened dry on 140 mesh with a Hum-mer).

The screening was carried out in order to minimize any non-micaceous grit in Sample No. 3569. It had been observed that, in the case of No. 3417, the small amount of grit present was principally at or below the 200 mesh size. It was further noted that, under attrition grinding conditions, the grit tended to report rather quickly as superfines which could then be hydroclassified out. Preparation of Sample No. 3569 by the simple expedient of screening supplied a large quantity of sample quickly and eliminated the "grit purge" grinding carried out on No. 3417.

Table 1

Screen Data on Samples 3417 and 3569

U. S. Screen Size	No. 3417, Total % Weight	No. 3569, Screened % Weight
+100	64.5	67.2
-100+140	12.4	21.9
-140+200	8.9	7.2
-200	14.2	3.7
Total	100.0%	100.0%

A second sample used briefly was No. 3593. This was the recleaned mica froth product concentrate from the feldspar flotation plant of Kings Mountain Mica Company. It was used for a quick comparison of delaminating characteristics. As a sample, it appeared characteristic of a muscovite having normal delamination characteristics during fine-grinding. Since there was less familiarity with its level of grit content, a portion of No. 3593 was prepared for a single grinding test by first dry-screening it on 100 mesh and using only the plus 100. This removed 23 percent of the head sample as fines. The remaining sample ranged from 16 to 100 mesh. It had a more usual gray color rather than white, as No. 3569.

GRINDING APPARATUS

During this period of research, the Abbé mill previously employed was used exclusively. Various weights of media were used. Except during several early tests (Table 3), the media consisted of two ceramic tubes with cores of lead,or wet sand plugged in with stoppers. The media were never allowed to cascade or cataract, but slid or rolled along the mill shell as it rotated. Table 2 gives certain data on this unit, repeated from the preceding report cited.

Table 2

Characteristics of Attrition Mill

Dimensions of Abbé ceramic shell -	Inside length (media raceway)	- 5¼"
	Inside diameter	- 7 3/4"
Speed of rotation -		- 55 rpm
Length of rod media used. (Two rods in all cases)		- 4 7/8"

Other data involved variables and will be cited as needed.

VARIABLES STUDIED

Earlier testing on Sample No. 3417 had yielded results indicating that attrition grinding at thin pulp (20 to 25 percent solids) with tetrasodium pyrophosphate (TSP) could create a fine-ground product of low bulk density (6 to 7 lbs/cu ft). Two comparative tests on Foote mica had been run earlier with the single variable of grinding pulp density. After an 18-hour grind with standard hydroclassification techniques, the product from a grind at 25 percent solids was 56.6 percent of head feed and measured 7.3 pounds per cubic foot bulk density. Under the same conditions but at

40 percent solids grind, product equalled 56.1 percent and had a bulk density of 9.3 pounds per cubic foot. Current tests were accordingly begun under those general conditions, without repeating grinding tests at higher pulp density. The variables investigated are described below.

Hydro-Settling Time

A group of three tests explored variable settling time to separate oversize, product and superfines. In this group, run at an earlier time, the mill was run with five free ceramic, lead-cored rods close to 1 1/8 inches diameter and weighing close to 450 grams each. Five pounds per ton of TSP was used; pulp density was 25 percent solids; grinding time was 18 hours. Milled feed (200 g. solids) from each test was then agitated and diluted to 20 liters, settled 10 minutes and decanted with a siphon tube; the oversize (sinks) set aside; and the thin pulp with suspended solids again settled, for a variable time, prior to decanting off the fines and drying and evaluating the product. Table 3 gives data on these three tests.

Table 3

Variable Hydroclassification Technique

Test No.	Variable Condition	% O'Size	% Product	% Fines
3569-3N	Second decant after 4-hr settling time	42.7	26.7	30.6
4N	" " " 8 " " "	39.0	32.6	28.4
5N	" " " 24 " " "	27.3	49.0	23.7

Products from the above tests were all 96 to 99 percent minus 325 mesh, with bulk densities 8.3 to 8.5 pounds per cubic foot. An unanticipated happening created an unwanted effect: apparently some rods, which became flat on one side, rotated less and less and slid increasingly on a flat side. This seemed to be more productive in terms of oversize milled down (which should have been constant), but made the tests less valid for demonstrating ratio of fines vs. product. It can be seen, however, that a 4-hour settle (Test 3N) creates a roughly 1:1 split of product vs. fines, whereas the 24-hour settling time changes it to about 2:1, product vs. fines. (Test 5N). A decision was made, based on this test group, to standardize on a second settling time of 4 hours' duration, as Test 3N: increased settling time did not create a product of lower bulk density, and the magnitude of difference in product/fines ratio as between the different settling times was not considered great enough to warrant a longer one of 8 to 24 hours.

Weight and Rotation of Rods, and Grinding Time

During preliminary tests, certain data pecularities emerged indicating that these variables were important. A group of four tests was run under these constant conditions: 200 gram sample, 18-hour grind at 20 percent solids with ten pounds per ton of TSP, followed by two hydroclassifications involving dilution/agitation to 20 liters, and then separatory settling times of ten minutes and four hours, as previously described; followed by siphon decantation to remove fines. There were two variables: rod weight, and rod rotation. Rotation was either nil (the rods being fastened in a frame) or else it occurred as the rods happened to rotate, which would thus be partial, variable, and not controllable within the mill. Thus, comparison was between no rotation, and a certain amount which was not measurable. Table 4 gives resulting data from these tests.

Table 4

Variables, Weight of Rods; also Rotation vs. Non-Rotation

Test No.	Conditions	% O'Size	% Product	lb/cu ft Product	Fines (Loss)
3569-31N	2 free rods, total 2300 g.	76.9	16.3	13.5	6.8
3569-34N	2 free rods, total 1007 g.	79.8	13.3	13.8	6.9
3569-32N*	2 rods, total 2300 g., non-rotating	71.6	15.8	12.7	12.6
3569-35N	2 rods, total 1007 g., non-rotating	71.9	16.8	9.0	11.3

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* See discussion of Table 4, following.

Regarding the third test of Table 4 (No. 32N), it should be noted that an accident occurred which broke one of the rods at a time when the mill was unattended. Apparently the rods did not slide during some instant, and consequently moved up the mill and then pitched over, causing one alumina rod shell to break. An attempt to repeat the test with new rods resulted in an identical mishap, and so the attempt to grind with the heavy, joined rods was abandoned, since there was no way to prevent the tumbling within a closed batch mill. The data which did emerge from this imperfect test tended to suggest that there might be an optimum linear pressure for achieving low bulk density of product, and that the heavy rods might be above that optimum.

To gather more information regarding media pressure, two additional tests were run. One of them was identical to Test No. 35N of Table 4 except for being run for twice as long (i.e, for 36 hours instead of 18). As a direct comparison with this test, another one of 36 hours' duration was run with a pair of rods weighing, in toto, 614 grams instead of 1007 grams as in Test 34N. Reason for changing duration of grind in this instance was the possibility that the lightweight rod test might not yield enough product in 18 hours to properly evaluate, but at the same time it was necessary to have a test with the intermediate-weight rods which reflected constant grind time. Table 5 shows the two 36-hour grind tests cited, and also repeats the data from Table 4 on Test 35N (18-hour grind).

Table 5

Data on Tests with Differing Rod Weights. Also Variable Grinding Time (All Tests with Non-Rotating Rods)

Test No.	Conditions	% O'Size	% Product	lb/cu ft Product	Fines (Loss)
3569-35N	18-hr grind, 2 rods weighing 1007 g.	71.9	16.8	9.0	11.3
3569-39N	36-hr grind, 2 rods, 1007 g.	56.9	24.1	7.9	19.0
3569-47N	36-hr grind, 2 rods, 614 g.	57.7	23.9	5.9	18.4

In comparing the first two tests of Table 5, it can be seen that the grinding energy of the extra 18 hours went, at least to some degree, into greater delamination of the fine mica - as shown by the lower bulk density figure. Comparison of the second and third tests indicates that the lighter rods were considerably more effective in delaminating fine mica, while at the same time reducing screen size. Creation of superfines (last column of Table 5) was about the same. The data of the last two tests tend to reinforce a theory that an optimum linear rod pressure is needed for maximum delamination in the 325 mesh size range, with greater pressure being detrimental. It cannot be claimed at this point that the optimum condition in this respect is known.

Flat Vs. Linear Grinding Surface Contact

The assumption can readily be made that more grinding should take place in a given mill if there is more media contact with the grinding surface, especially in the case of an attrition mill such as the one discussed here. Data from two comparable tests is submitted to check out the effect of this variable. For one test, No. 3569-39N (Table 5) will again be cited. The second test was identical except for the use of two non-rotating rods totalling 1530 grams in weight, and being flat on the bottom (contact) side. The flat area on each rod was roughly 3/8 inches wide, and 4 7/8 inches long. Unlike the hard alumina rods used in other tests, these rods were of softer porcelain, which accounts for their wearing flat. Table 6 gives data.

Table 6

Linear Vs. Flat Grinding Surface

Test No.	Conditions	% O'Size	% Product	lb/cu ft Product	Fines (Loss)
3569-39N	2 round rods, 1007 g., 36-hr grind	56.9	24.1	7.9	19.0
3569-43N	2 flat-sided rods, 1530 g., 36-hr grind	53.8	20.2	8.0	26.0

Data of Table 6 indicates that linear, rather than flat, media contact on the attrition surface is preferable: more product of equal or lower bulk density is created, along with fewer fines. However, further research is indicated.

Simulation of Continuous Production

In order to gain some idea of the quality of a product made under the best conditions known currently, but on a continuous-production basis, one test was carried out wherein a grinding and hydroclassification cycle was followed by replacement of feed removed as product or fines. Non-rotating rods were used to grind a 200 gram portion for 18 hours; the milled feed was hydroclassified as described earlier, and the oversize dried and weighed. The next cycle was begun with more fresh feed added to the previous oversize to bring total feed weight to 200 grams. Then another 18-hour grind was made, followed by repetition of the previous steps. Five successive runs were made by this procedure. Table 7 furnishes data on results.

Table 7

Stage Grinding plus Hydroclassification
to Simulate Continuous Grind, Test No. 3569-41N

	Run 1	Run 2	Run 3	Run 4	Run 5
% Oversize	70.6	74.7	73.4	71.9	73.4
% Product	18.8	16.4	16.8	17.8	16.7
% Fines +Loss	10.6	8.9	9.7	10.3	9.9
Total	100.0%	100.0%	100.0%	100.0%	100.0%
Product:					
% +325	0.3	0.6	0.3	1.1	0.9
% -325	99.7	99.4	99.7	98.9	99.1
lb/cu ft, -325	9.1	7.1	6.6	6.6	6.6

Discussion of Results, Table 7

It can be seen from Table 7 that the system apparently became stabilized at the third run, producing a fairly constant quantity of product and fines, as well as a constant quality (bulk density) of product.

Under the conditions of this test, loss through overgrind appears to be about 36.4 percent of mill feed. This figure, in a commercial flowsheet, would probably be considered too high. Several considerations, however, improve the picture. First, under conditions of a true closed circuit grind with short residence time and efficient hydroclassification, that overgrind loss would undoubtedly be lowered. Second, the fines of this test may contain some mica which could have been included in the product; however, an empirical hydroclassification procedure had to be set up and adhered to. Finally, the portion of the fines which is actually too fine as product in one application may be usable in another, especially if it is free of other minerals.

It should be recalled that Sample No. 3569 had been prepared for the tests of this report by prior dry-screening which removed close to ten percent of head feed. That fraction might need to be either discarded or else put through a special "purge" grinding circuit to differentially grind and classify out the non-micaceous grit: a procedure shown possible in earlier tests. The de-gritted remainder could then enter the principal grinding circuit. The data of this report does not deal with any processing of that initially-removed fraction.

At the time the data of Table 7 was developed, it was not known that still lighter grinding rods would create a mica product of even lower bulk density. It appears likely that, by using the rods weighing 614 grams instead of 1007 grams, an even lower product bulk density could have been attained. (Refer to Table 5).

A Different Mica

Principal efforts during tests to date had been directed toward production of a fine-ground mica of low bulk density using a head feed having a reputation for being impossible to fine-grind to low bulk density (Foote flotation mica). Since this had now been proved possible, it was desired to use the best grind techniques discovered to date on a mica feed which was currently being fine-ground to an acceptable bulk density by a commercial process. The sample selected (No. 3593) was a recleaned 20 mesh mica float concentrate from the feldspar flotation plant of Kings Mountain Mica Company. This concentrate was being fine-ground by its producer by wet process to a minus 200 mesh product whose minus 325 fraction had a bulk density of 9.5 to 10 pounds per cubic foot. This mica was regarded as having no particular problems of delamination.

To insure total grit removal from the head sample, an aliquot was dry-screened on 100 mesh. Two hundred grams of the plus 100 were used in a 36-hour grinding test duplicating Test No. 3569-39N, previously cited (Table 5). The set of non-rotating rods weighing 1007 grams was used. Table 8 gives data.

Table 8

Grinding Characteristics of Two Different Micas

Test No.	Sample	% O'Size	% Product	lb/cu ft Product	Fines (Loss)
3569-39N	Foote mica	56.9	24.1	7.9	19.0
3593-4N	Kings Mountain Mica Co. mica	59.5	22.4	7.5	18.1

The data of Table 8 tends to indicate that the technique used on the Foote sample, considered impossible to delaminate, works at least as well on another, more typical mica, reducing it to a bulk density figure which, if not lower than the Foote, is at least lower than when it is commercially ground under present process. What needs yet to be ascertained is whether special refinements in technique will reduce the bulk density of "usual" micas proportionately lower than the Foote mica; i.e. to a bulk density of 4 to 5 pounds per cubic foot, since it is now possible to fine-grind the difficult Foote mica to 5.9 pounds per cubic foot. On different micas, variables in pulp density, media pressure, hydroclassification, etc. may need to be called into play.

SLIP AND SHEEN OF PRODUCT

Problems and Needs

Up to this point, research efforts had been concentrated on grinding procedures to reduce bulk density. When a product of low bulk density was obtained from the Foote mica, however, it had a chalky appearance and also gave the impression of high frictional drag when rubbed between the fingers. This was also true of the product from the Kings Mountain Mica Company mica. Contrasted to this, a well-known water-ground mica (ground in a muller by Franklin Minerals Products) had a glistening appearance, plus a "soapy" or slippery feel when rubbed between the fingers. The latter type mica is regarded as being desirable for most consumer demand, whereas lack of slip and sheen is a detriment to the product: this is the general statement made by producers in the field.

The difficulty inherent in evaluating these characteristics lies in the fact that there are presently no known objective measuring instruments or criteria for measurement of mica "slip" or "sheen". Specimens having, or not having, these attributes to an extreme degree are easily distinguished, but intermediate degrees are often hard to appraise relatively. Presence of moisture, or type of light, can influence judgment of a specimen, as well as manner of rubbing it - and quite probably other variables as well. Therefore, a means of objectively measuring "slip" and "sheen" was seen as a pressing need.

To measure "slip", it might be assumed that instrumentation able to determine relative sliding friction would be applicable. "Sheen" implies a characteristic related to light reflection.

The paper industry employs a standard ASTM procedure, with a machine and other special apparatus, to measure frictional resistance between two pieces of paper. This is done by placing a standard paper-covered weight upon a paper-covered plane and then inclining the latter increasingly until, at a given angle, the weight slides, thus denoting a relative frictional characteristic of the paper. This technique appeared interesting as a basis for design of a standard test for mica "slip".

For directly measuring whiteness or reflectance of a given surface, both the Photovolt and G. E. reflectance meters are well-known, and have standard test procedures built around them. These procedures, however, involve reflectance of a light beam impinging at a precise right angle upon a planar surface of the tested material. This procedure, with a Photovolt instrument, did not measure any significant differences of two products from Sample No. 3865, one of which was observably chalky and lacking in "sheen", and one identical to it except for having been given an observable nacreous appearance through some additional and different mill processing, which will presently be discussed. It appeared likely that the chalky, contrasted to the nacreous, appearance would need to be differentiated by measuring some type of oblique reflectance, wherein a light source would impinge a beam at a given angle or angles upon the sample surface, and a properly placed sensor would receive whatever light was thus reflected and activate a meter. Again, an instrument is available which can carry out this function: it is known as a glossmeter and is used by the paint and paper industries, among others.

In the case of the "chalky" mica product milled from Test No. 41N (Table 7), it was desired to increase its characteristics of slip and sheen. Preliminary tests had indicated that milling at thicker pulp, and milling with rolling (not sliding) rods tended to impart a "soapier" texture and increase nacreous appearance. It had also been observed that use of these variables increased bulk density. With this as background, a test using a single 1¼-inch rod covered with soft rubber (weight 1150 grams) was tried. One hundred fifty grams of product from Test 41N was milled (polished) with this single rod at close to 50 percent solids for a total of 72 hours. A 4-hour hydroclassification followed this (as 41N and others), resulting in discard of 24.5 percent of the mill feed as fines. The remaining mica was dried and evaluated. It was observably less chalky and had a more glossy appearance. Table 9 gives characteristics of this product, before and after the "polishing" test.

Results as shown in Table 9 indicate apparent change of the fine-ground mica in terms of both shape and surface characteristics. Further discussion will take place later in this report. At this point it is desired to give only sufficient data as background for a new, hopefully objective, test for mica "slip", discussed immediately following.

Despite instruments to measure both frictional slip and gloss or sheen of other materials, the mica grinding industry apparently has no instrumentation or test procedures of universal recognition to carry out tests in these areas on fine-ground mica. The opinion is expressed here that the principal reason for this lack is the extreme difficulty of establishing a good standard procedure for preparing and maintaining a constant and standard sample surface upon which to conduct reproducible measurements. A paper or paint sample surface does not present such problems.

Solution of the problem of the angular-beam measurement of gloss or sheen of mica is still being worked upon, with emphasis on standard preparation of sample surface. While little progress has been made to date on this, a test for frictional slip has been devised and is now reported.

Table 9

Characteristics of Test No. 41N Mica Product Before & After "Polishing"

		Product Characteristics			
		lb/cu ft	Tristimulus Reflectance		
Test No.	Description	Bulk Density	Green	Amber	Blue
3569-41N	Milled -325 mica	6.6	89.5	90.0	84.0
3569-45N	41N Prod. polished 72 hrs	9.3	89.0	85.0	88.0

Slip Testing of Mica: A Test Designed at the MRL

Starting with the concept of a block sliding on an inclined plane, a hand-operated instrument was designed and built which can be described as follows:

On a rectangular base, a small vertical windlass mechanism is mounted, operated through a worm gear drive with a hand crank. This windlass, by means of a flat tape winding around a small capstan, slowly inclines a narrow, pivoted stainless steel tray (having side walls) to any angle from 0° to 50°. The tray's inner bottom surface is permanently coated with grit. Within this tray the mica sample is placed by a procedure to be described presently.

The rectangular base has a mounted dial indicating angles from 0° to 50°. The tray has an extension on its moving end to register, against the dial, its inclination in degrees. Within the tray a short weighted slider with a smooth, polished, rectangular under-surface is placed upon the mica sample surface, for the purpose of sliding at an observed angle inclination.

To compress the mica sample bed before the test, another (long) weighted slider with a polished bottom is used. It is slightly longer than the tray and of a width to fit closely inside it.

A small screed-like device is used to slide along the tray walls to level the mica sample bed prior to the test. A funnel of given volume, with a wide bottom opening, is used as a means of initially distributing the proper quantity of mica along the tray.

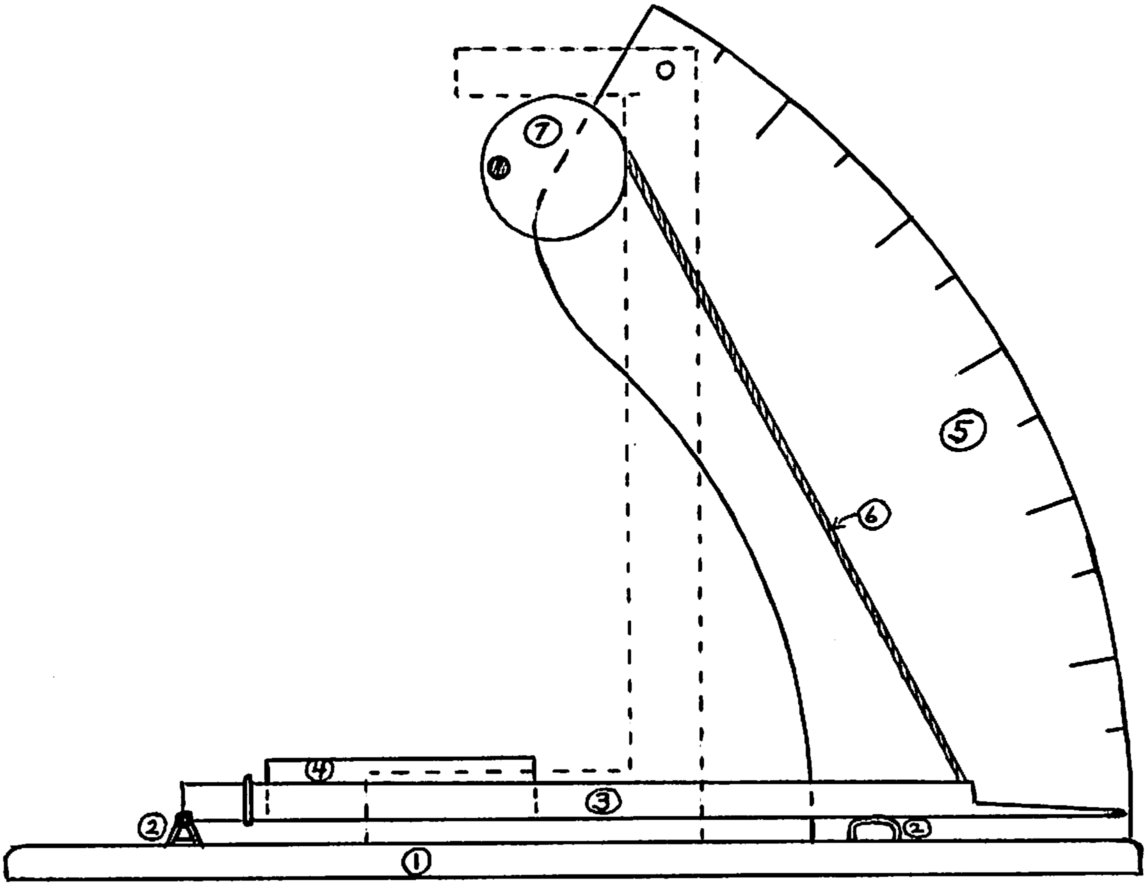
Figure 1 is a diagrammatic sketch of this instrument. It should be stated at this point that description of the instrument, the diagram, and the account of the standard test procedure are all somewhat generalized. Details regarding the apparatus, the procedure, and preliminary test results are to be presented in a separate technical report, MRL No. 71-17-P. The main purpose of presenting this information here is to give background for certain interesting data which developed from the test.

In operating this test, this is the general procedure as developed:

1) The funnel is filled with mica sample and used to spread the sample fairly evenly along the horizontal tray.

FIGURE 1

Simplified Drawing, Principal Components, Mica Slip Tester
(Side View)



LEGEND:

1. Base
2. Tray supports: pivot on left
3. Mica tray with side walls and rubber band. Pivot on left, pointer on right.
4. Slider, placed within tray.
5. Inclination scale, 0°-55°
6. Elevating tape connecting windlass and tray
7. Windlass; supporting frame shown in broken lines.

2) The screed is moved back and forth along the tray to form a flat, even sample bed of constant depth.

3) The long weighted compression slider is laid on the sample bed in the tray, gently reciprocated for a given number of strokes to compress the bed, and then carefully removed.

4) The tray is mounted on the base and attached to the windlass.

5) The short slider is carefully placed within the tray at a point near the pivot end.

6) The tray is slowly inclined until the slider moves all the way to the bottom of the tray. Moving angle is recorded.

7) The tray is lowered slightly, and the slider is slid back to a position slightly further back than its first placement. The tray is lowered to horizontal.

8) A rubber band is stretched between the walls of the tray to restrict the slider to a short traverse.

9) The tray is inclined again to the point of slider movement, and the angle recorded.

10) The tray is again lowered a few degrees, and the slider again pushed manually back to a point slightly beyond its previous positioning. The tray is leveled and the rubber band advanced slightly more.

11) The cycle is repeated, and angle of slip recorded. A total of 16 runs are made and recorded, with the slider moved back slightly each time; also the rubber band.

12) The four highest angular slip readings are averaged and the average reported as the slip angle for that sample.

Theories Behind the Test Procedure

Some of the theories behind the procedure in this test are as follows:

1) A certain standard non-resiliency and particle orientation of sample bed is necessary, which is the reason for preliminary compression.

2) A given portion of the sample bed, if used repeatedly, can deteriorate: therefore it is gradually renewed by adding a small amount of new area on each run.

3) If the slider gains very much momentum, it tends to dig into the bed and make it out-of-level.

4) The sample bed is re-conditioned each time by gentle pressure during careful manual return of the slider.

5) Despite careful handling, the human factor causes undesirable variation in slide angle readings. It is theorized that, when a high reading is obtained, this represents a run on a flat, well-constituted sample bed having maximum frictional purchase on the slider, and therefore representing most truly the particle contact characteristics of that particular sample. A high number of runs (16) is made in order to secure at least four readings which are likely to be close to the highest possible for that sample.

Based on performance data of several individuals operating this test on the same sample, there is no doubt that test results are subject to variations in experience and aptitude of the operator. On a given sample, one operator secured an average slip of 35°, and another 37.5°, which is often greater than the difference between two samples when one experienced operator tests them. However, the first operator was inexperienced. The second (experienced) operator secured reproducible results from a series of tests on the same sample. The degree of fluctuation which might be brought about by variations in temperature, humidity, and electrostatic charges is not known. All tests were run in a dry, heated room at about 70° F. The presence of non-micaceous grit was not measured, although it is believed all samples tested for this report were 99 percent pure mica or higher. Presence of grit would undoubtedly affect slide angle.

Tests on a Group of Samples

Using the test apparatus and procedure just described, a group of fine-ground mica products was evaluated for slip. All were first screened on 325 mesh and only the minus 325 fraction given the slip test. Reason for this was the probability that variation in particle coarsness would affect slip angle.

The principal screened fine-ground mica products (minus 325 fraction only) tested were as follows:

1) Milled product from Test No. 3569-41N, referred to in Table 7 (Foote mica).

2) Milled and "polished" product from Test No. 3569-45N, referred to in Table 9 (Foote mica).

3) Sample No. 3836: water-ground (mulled) commercial product made from "jig" mica (Franklin Minerals Products).

4) Sample No. 3839: fluid-energy (Majac) - ground flotation mica (Diamond Mica Company).

5) Sample No. 3840: water-ground commercial product, described by producer as "high sheen" (Diamond Mica Company).

6) Sample No. 3841: As 3840, but described as "good sheen" - presumably of slightly lower quality than 3840.

7) Sample No. 3079: A batch product wet pebble-milled at the MRL from a float concentrate from Haywood County mica schist, and having a general appearance and texture similar to dry-ground mica.

8) Sample No. 3871: Majac-ground mica of Foote Mineral Co.

9) An un-numbered sample of pure dry-ground feldspar, minus 325 mesh.

Table 10 gives results of the slip test applied to the above samples, plus bulk densities. All data is on the minus 325 fractions.

Table 10

Slip Test and Bulk Density Data on Eight Fine-Ground Micas and a Ground Feldspar

Sample	Description of Sample	Slip Angle	lb/cu ft Bulk Density
3569-41N Prod.	Wet-milled to low bulk density	39.5°	6.6
3569-45N Prod.	41N product, polished 72 hrs	43.3°	9.3
3836 (Prod.)	Water-ground jig mica (Franklin)	41.1°	9.5
3839 (Prod.)	Majac-ground float mica (Diamond)	36.2°	12.5
3840 (Prod.)	Water-ground, "high sheen" (Diamond)	41.3°	9.7
3841 (Prod.)	Water-ground, "good sheen" (Diamond)	40.0°	9.8
3079 (Prod.)	Wet pebble-milled mica schist float concentrate	38.3°	13.8
3871 (Prod.)	Majac-ground float mica (Foote)	37.5°	15.2
--	Dry-ground feldspar	31.6°	38.7

Discussion of Data, Table 10

Four wet-ground micas tested had a slip angle of 40° or over. Two additional wet-ground samples registered 38.3° and 39.5° respectively. These two had been milled under conditions at variance with usual commercial wet-grinding techniques, and had a physical appearance resembling dry-ground mica. The two Majac-ground samples had 36.2° and 37.5° slip angles.

Bulk densities for the wet-ground mica samples ranged between 6.6 and 9.8 pounds per cubic foot, except for No. 3079 (ball-milled), at 13.8 pounds. The two (dry-ground) Majac products measured 12.5 and 15.2 pounds per cubic foot. All bulk density determinations were made by means of a Scott Volumeter.

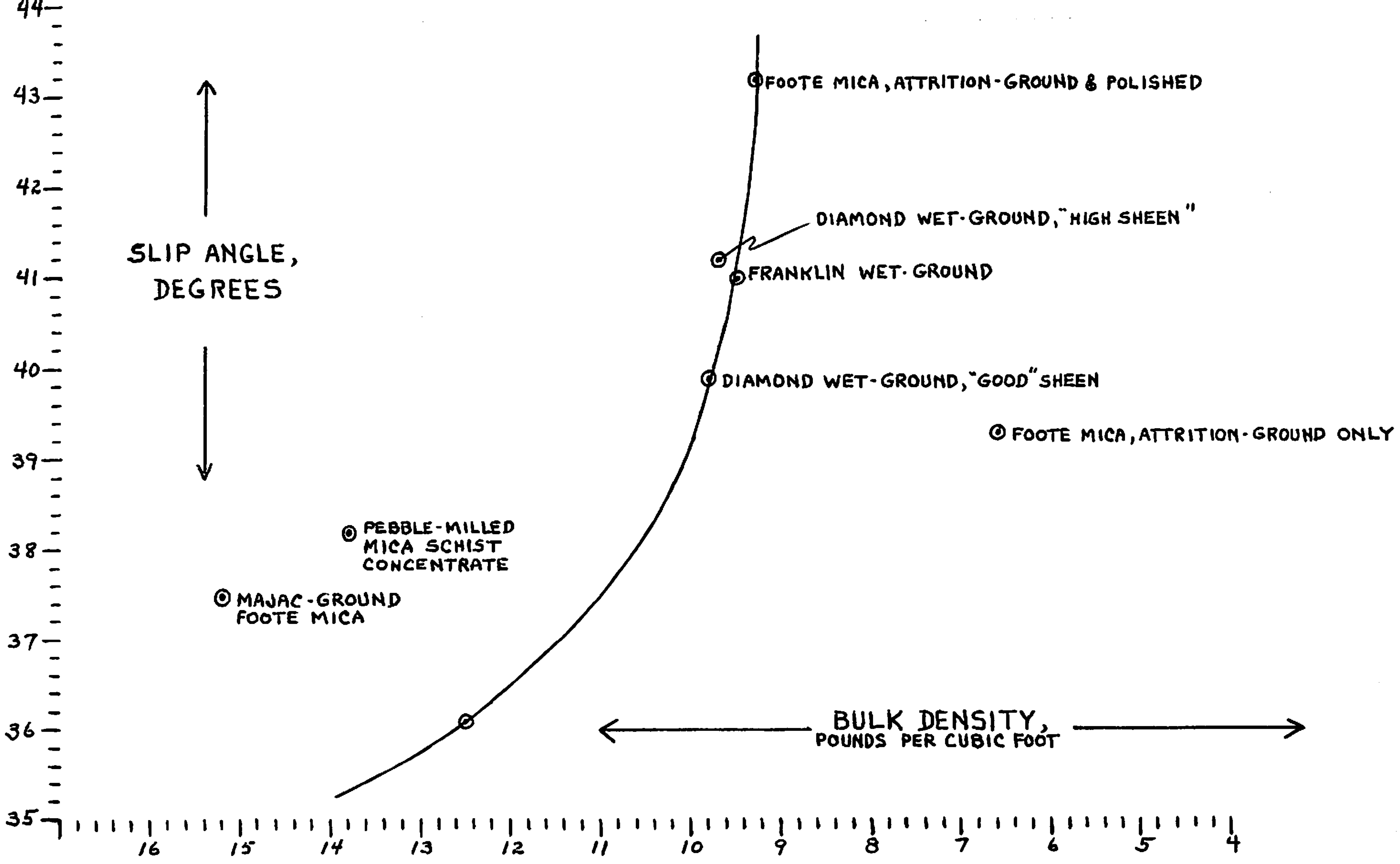
In most cases there was an inverse relationship between bulk density and slip angle: micas higher in the one tended to be lower in the other. The single major exception to this is the product of Test 41N: Foote mica wet-ground by attrition. The Majac-ground Foote mica (3871) and the ball-milled mica schist (3079) tended to conform less to an arbitrary curve. (See Figure 2). The Foote mica, as previously stated, is difficult to grind to low bulk density: thus its variant character. The mica schist is regarded as having been ground under poor conditions to produce delamination; other conditions might have done better.

To obtain further data, a pure dry-ground feldspar was also tested. This would be expected to be granular and high in superfine particles, based on microphotographs of other specimens of the same origin. It tended to correlate with the other data in having a still lower slip angle plus still higher bulk density. It was not included in Figure 2.

FIGURE 2

Inverse Relationship of Slip Angle and Bulk Density, Eight Mica Samples*

*Refer to Table 10



Relation of Ground Mica Particle Characteristics to Slip

Usual concepts of the characteristic of slip could lead one to a prior opinion that, the greater the observable slip of the mica, the lower should be its slip angle when measured by a test such as described. The opposite in fact appears true, based on the data of the tests just cited, with the exception of the product from Test No. 41N (Foote mica, wet attrition-milled). This last apparently represents a special case (to be discussed). Choosing two more representative instances, one of Majac-ground mica (No. 3839) and one of wet-ground (mulled) mica (No. 3836), it can be stated with certainty that these are observably quite opposite in texture. The former is chalky to the feel; the latter has a slippery texture when rubbed between the fingers. The former has the lowest slip angle of the group, while the latter has one of the highest. (Table 10).

When a mica is ground by fluid energy (Majac mill or other), the grinding occurs from a relatively straight-line impact of particle against particle. The directional impact against any given particle is random with relation to particle orientation, as particles move at high velocity against others moving oppositely. Thus, while in some instances the inter-particle impact may lead to laminar splitting, in other cases there is more likely to be a fracturing or shattering along planes not parallel to the laminae, resulting overall in a high percentage of jagged, multiplanar, blocky particles having many frayed and broken edges - plus copious small superfine particles which cling to larger pieces as debris. It is this type mica which presents an appearance which is not glossy, and, which "drags" when rubbed between the fingers. The particle characteristics described above can be observed visually if a sample is photographed under a scanning electron microscope (SEM). A visitor from E. I. du Pont de Nemours and Company, Mr. Roland Wetzel, allowed the Minerals Research Laboratory staff to inspect such photographs in April 1970.

The milling (or mulling) of a mica such as Sample No. 3836 is under quite different conditions. A batch of coarse mica, in wet pulp, is placed in a circular mortar-like vessel, and a weighted roller, or rollers, traverses the circular bottom, moving horizontally continually around. State of the art controls variables such as size of charge, pressure, pulp density, and milling time. According to several people versed in this grinding method, the action of the rollers causes the mica particles to compress into a bed of flakes which are mainly oriented horizontally. The action of the rollers then flexes and splits the flakes somewhat in the manner of a deck of playing cards being cut and shuffled. The energy thus exerted upon the mica goes principally into delamination, with the onset of smaller particle-size perhaps mainly due to incidental fracturing as the laminae become thinner and more fragile.

In contrast to a fluid-energy ground mica, the individual particles of the wet-ground product tend to be more single-planed on each side, and the edges lack the fraying and splitting characteristic of the other. There is also less ultrafine debris. These characteristics were readily observable in another SEM photograph of wet-ground mica shown at the Minerals Research Laboratory by Mr. Wetzel of du Pont. They can also be seen in a similar photograph taken at North Carolina State University as part of this project.

Returning, then, to the slip characteristics of these two fine-ground micas: the product having frayed, multi-planar, blocky particles, including fine debris, has a definitely lower slip angle than the one with particles which are flat, thin, un-frayed, and relatively free of debris. (No. 3836 vs. 3839, Table 10). The slider of the experimental instrument described apparently is more impeded by an oriented bed of flat particles which presumably are bearing upon a greater percentage of its under-surface, thus causing a higher degree of friction. The jagged, blocky, debris-laden, Majac-ground mica cannot be oriented into a bed having an equal area of actual surface contact with the slider; therefore, overall coefficient of friction is apparently less, as is the slide angle.

It can be theorized that, to the practiced touch, dry-ground mica feels more granular or "chalky" for the same reason as cited: the particles cannot be made to slide flatly across the fingers or across one another because of shape, and due to much interfering debris. Instead, many of them roll. The wet-ground mica, having more smooth, flat, clean laminar surfaces, readily orients so that many more particles can slide to impart the "soapy" texture, with relatively less of the rolling of rough pieces which causes the tactile sensation of chalkiness.

Statements of the two preceding paragraphs are further strengthened by the data on the sample of fine-ground feldspar, Table 10.

Evidence to date, then, indicates that one desirable characteristic of a fine-ground mica can be measured as a steep, not shallow, angle of slip, using the measuring technique described.

Delamination Related to Mineralogy

In 1966, A. F. Alsobrook, a mineralogist with the Minerals Research Laboratory staff at that time, examined a group of muscovite samples, among them some Foote flotation mica identical to No. 3569 of previous reference, and also some which was quite similar to No. 3840 from Diamond.(Table 10) These two micas were stated to be different mineralogically and geologically, in these respects:

1) The Foote mica is unweathered, and was formed at a relatively low temperature of crystallization but with a strong alkali bond. This resulted in a somewhat soft mica, nevertheless hard to delaminate.

2) The Diamond mica is somewhat weathered so that the alkali bond is weaker, permitting easier delamination. However, having crystallized at an intermediate temperature, it is harder and thus less likely to overgrind.

The Diamond mica, and others like it, is obtainable in larger pieces (jig or spiral concentrate). This lends itself well to the flexing action which is presumably the basis for delamination in the muller. Flotation mica does not presently appear generally popular as a feed for muller grinding. Reason may be relative inability to process it into a bed where flexing (and thus delamination) readily occurs. Past commercial wet-grinding attempts on Foote flotation mica have yielded a fine-ground product weighing between 15 and 19 pounds per cubic foot, compared to the desired 9 to 10 pounds. The Foote mica is not avilable in jig product size (plus 1/8-inch) to try out in a muller.

GRINDING THEORY: MRL ATTRITION MILL ON FOOTE MICA

As was seen (Table 10), a minus 325 mesh product was obtained at the Minerals Research Laboratory from Foote flotation mica weighing 6.6 pounds per cubic foot. To obtain this, it was necessary to direct energy upon the mica flakes so as to shear them along laminar planes (in spite of the theoretically-strong K-bond) without undue fracturing of the relatively soft mica crystals.

The following theory is offered regarding the mechanism of this shearing: in the most successful tests, the instant of relatively linear pressure occurring between the cylindrical rod and the mill shell acted upon the small mica flakes caught between, in a manner somewhat similar to muller wheels in the conventional situation. Possibly also, in addition to flexing the flakes like a muller, the non-rotating rods at point of contact may exert a lateral drag of high friction in relation to the mill shell, thus expediting delamination while minimizing any vertical fracturing force. This combination of forces, unlike any previous known technique, apparently was sufficient to delaminate the Foote mica to a previously unattainable degree.

Within the parameters of the grinding research performed to date, there are obviously certain optimum conditions which minimize fracturing and carry out a maximum of shearing. One appears to relate to rod media rotation. Data of Table 4 indicates that stationary (sliding) rods create lower bulk density product than those which rotate to some degree. Apparently media rotation increases fracturing. Data of Table 5 points toward an optimum linear rod pressure per inch, with the optimum not yet established. Table 5 also indicates that lowest bulk density of minus 325 mesh product depends upon optimum conditions of mill residence time and/or closed milling circuit including screen, hydroclassifier, or both.

While the effects of variations in pulp density; pressure, weight, and motion of media; and residence time are known to be of significance, it is not yet known what effect some other variables will have - such as speed differential between media and shell; absolute rate of motion of either; size of feed charge and residence time; factors to change pH or alter coefficient of friction relative to feed and media. There are, of course, other variables. Still another variable seen as needing further research is the use of linear (tangential) contact between media and mill (with some mica between , of course); and a grinding situation wherein the media have a flat contact with the mill shell, measurable in square inches or centimeters. Data of Table 6 indicates that linear contact may be superior, but further confirmation is needed.

SHORTCOMINGS OF ATTRITION-GRIND PRODUCT

Table 10 cites Test No. 3569-41N, in which Foote mica was ground to a bulk density of only 6.6 pounds per cubic foot. Its slip angle was 39.5°, which was superior to dry (Majac)-ground micas, or one wet ball-milled mica, but somewhat less than the water-ground samples. With its very low bulk density but also relatively low slip angle, it was thus unique in the group. In addition, this mica had a chalky appearance and feel characteristic of dry-ground mica.

To ascertain the basis for this combination of characteristics, a sample was inspected and photographed at various powers (300 X to 6000 X) beneath the NCSU scanning electron microscope (February 1971). In this instance, a 325-400 mesh fraction only was scanned in order to obtain a clear view of individual particles. The particles were delaminated in the same flat manner as commercial water-ground (mulled) mica (i.e. particles were not jagged or multi-laminar), but, unlike the latter, there was a high incidence of curled, frayed edges. This is apparently the factor contributing to chalky appearance and texture.

From the above observations it can be theorized that the curled edges of this laboratory product also tend to inhibit flat particle alignment in the slip test, and so cannot give as high a reading in slip angle. However, despite this defect, the mica product is still cleanly delaminated and gives a better slip reading than dry-ground mica. Removal of the frayed edges should improve quality still further in terms of "slip" and "sheen".

IMPROVEMENT OF ATTRITION-GROUND MICA QUALITY

As a means of removing frayed edges from the mica product, the following test was undertaken: A single ceramic rod weighing 1150 grams was covered with a piece of soft rubber hose, and used as media in the same mill shell as previously described (Table 2). Feed was 150 grams of Foote mica, fine-ground product, made by the same technique as Test No. 41N. This procedure was discussed in connection with data of Table 9, preceding. The test was No. 3569-45N.

Table 9 data shows an increase in bulk density (6.6 to 9.3 pounds per cubic foot) plus a change in reflectance characteristics from this treatment. Table 10 data shows an increase in slip angle: from 39.5^{o} to 43.3^{o}, which latter is the highest slip angle recorded of any sample tested - by two degrees. Thus, a two-stage grinding and polishing treatment created a product from the Foote mica which substantially equalled typical commercial high-quality wet-ground products in low bulk density, and at the same time definitely surpassed them in the characteristic of slip angle.

Theory Regarding Changed Characteristics after Polishing

SEM photographs taken of this 45N product, as of several other samples cited, indicated that the second (polishing) single-rod treatment, followed by another hydroclassification - described in connection with Table 9 - removed to a large extent the curled, frayed edges from individual mica particles (typical of the product from Test 41N). However, visual evidence remained that many particle edges had been previously frayed, and there was still a certain amount of laminar splitting visible in single particles, which was not evident in the particles of commercial water-ground mica. A second visual difference was also observed as between these: two comparative photographs at 3000 X showed that the planar surface of a commercial water-ground mica particle was mainly quite smooth in appearance, whereas this surface presented a spalled appearance in the case of the 45N product - even though it was, on a larger scale, quite single-planed. This is seen as evidence of the strong interlaminar potassium bond of the Foote mica, mentioned earlier. Further SEM photographic studies are indicated along with continued research.

As measured, the bulk density of product from Test 45N went up to 9.3 pounds per cubic foot, compared to a previous 6.6 pounds. This is regarded as the result of removing the curled, frayed edges of mica, allowing the flakes to lie closer together when heaped. The question remains whether, under even better conditions of grinding, the bulk density of the product might not be brought down lower, for example to seven pounds per cubic foot even after a polishing operation.

Purely subjective observation of the gloss or sheen of the product of Test 45N is inconclusive: it appears to have less sheen than those commercial water-ground micas cited in Table 9. The question arises, however, whether this is due to the inherent whiteness of the mica, or to the observed remnants of frayed edges and spalled particle surfaces. One objectively-measured characteristic of the 45N product (slip) indicates possibility of superiority.

FUTURE RESEARCH

It is not considered that the processes cited have yet been optimized so as to make the best possible product from Foote mica, or from others of like or differing mineralogy. There is evidence that the Foote mica will require a 2-stage process in order to yield a product having low bulk density combined with high slip and sheen. Future research should be planned to encompass two areas: attrition-grind to lower bulk density, and polishing to achieve maximum slip and sheen. A third area of research should probably also be initiated, connected with these, covering development of an objective test for gloss or sheen of fine-ground mica, in order to more completely gauge success of grinding research in terms of product quality.

In terms of product use the following principal questions appear to present themselves:

1) Would there be increased market for a product having low bulk density hitherto unattained, even if it had poor slip and sheen?

2) Is the objectively-measured characteristic of slip, previously described, a reliable index of higher level of a desired commercial quality or qualities?

Connected with the above are questions about optimum processing conditions to attain highest quality. For example, can further research in polishing and in gloss measurement yield a product which is superior by all significant criteria, whether subjective or objective?

COMMERCIAL FEASIBILITY OF PROCESS DESCRIBED

The processes employed in the Laboratory were carried out using odd small pieces of equipment put together in an impromptu manner in order to effect preliminary demonstration of certain basic grinding concepts. To gather from these some indication of commercial feasibility of process, certain mathematical data related to production in the laboratory can be used. Still beyond possibility of calculation is the required energy level to grind a given quantity of mica. That must await activation of small prototype production units. However, some figures related to ground mica production rate per cubic foot of mill of a given type can be theorized.

The small attrition mill used in tests cited employed two rods, each 4 7/8 inches long, in contact with the inner surface of a rotating cylindrical mill. Thus, there was a theoretical linear contact totalling 9 3/4 inches under which grinding took place. It should be emphasized here that the mill and rods, not being precision-made, could not have had constant linear contact. Had constant contact actually occurred, grinding rate would have been higher. However, for present purposes the actual grinding rate of this mill is used as a conservative basis for calculations. (Data cited is from Table 2).

The rate of revolution of this mill (55 rpm) and its inner diameter (7 3/4 inches) yield the calculation that the traverse of the rod media within the shell equals 111 feet per minute, or 6675 feet per hour.

Test No. 3569-41N, which produced a mica of 6.6 pounds per cubic foot, was operated for a total of five runs each of 18 hours duration, or for a total of 90 hours (see Table 7). During that time, the following was produced:

Oversize	- 73 grams
Product	- 173 grams
Fines + Loss	- 99 grams
Total Processed	- 345 grams

This test was an attempt to simulate continuous production to some degree. After each stage involving removal of essentially minus 325 mesh material, fresh head feed was added to replace weight lost, and a new run started. It is felt that the fines were actually a result of overgrind and poor classification. Had a closed circuit been used involving short residence time in mill, plus combined screening and hydroclassification, the fines would instead have been product to a major extent. Therefore, for purposes of calculation here, the major portion of the fines is to be calculated as product. By adding the weight of product cited above (173 grams) to the greater part of the fines (87 grams), a total of 260 grams is estimated. This quantity of product, then, could theoretically be produced by this mill in 90 hours if closed circuit with hydroclassification had been possible to employ. This rate equals 2.9 grams of product per hour.

Putting together preceding data, it can be surmised that 9 3/4 inches of linear grinding contact moving 6675 feet (per hour) across a surface can yield 2.9 grams of product. Thus, ten inches of linear grinding surface moving across 10,000 feet should yield 4.5 grams of product. This can be used a a convenient unit of calculation.

Various mechanical designs can be evolved to facilitate maximum contact of stationary media with a moving surface which is flat beneath the media. A horizontal cylindrical mill with intake and discharge ends, and with media pressing around the entire shell circumference, is one possibility. A second one - and this is chosen here as the basis for further planning - involves the concept of a horizontal flat rotating disc upon which would rest the maximum number of radially-arranged stationary rods (permitted by space) - in the manner of spokes. These rods would be fastened in place to a stationary upper disc which would be subjected to the established needed downward pressure for optimum grinding. Mill feed would enter either by way of holes in the center of the upper disc, or else through holes radiating out from the center of the rotating shaft, which would be hollow. The mica feed would then flow out upon the under element of the mill(rotating disc) to be attrition-ground beneath the stationary rods fastened to the upper disc. Centrifugal force would move the feed outward from center, to be discharged at the periphery for classification and recycling.

Using the mechanical concept cited, certain dimensions and specifications are now assumed as a basis for performance calculations. A disc 30 inches in diameter might have, resting upon it radially, a group of 100 small rods each ten inches long (i.e. not reaching all the way to the center). Thus, the underlying rotating disc would have resting upon it 1000 inches of linear grinding contact. Assuming, for the under-disc, a rotational rate of 200 rpm (quite slow for a conventional disc mill), an average linear traverse rate can be calculated, using peripheral speed against inner and outer extremeties of the rods: inner, 524 feet per minute; outer, 1572 feet per minute; average, 1048 feet per minute, or 62,880 feet per hour.

For this mechanical grinding unit, rate of production of fine-ground mica can be calculated by combining its structural and mechanical features with unit performance figures of the small laboratory mill:

$$\underset{\substack{\downarrow \\ \text{(ft/hr)}}}{\frac{62{,}880}{10{,}000}} \times \underset{\substack{\downarrow \\ \text{(linear in.} \\ \text{grinding} \\ \text{contact)}}}{\frac{1000}{10}} \times \underset{\substack{\downarrow \\ \text{(grams)}}}{4.5} = 2{,}830 \text{ grams per hr of product}$$

Converting, a production figure of 6.3 pounds per hour is obtained, or 151 pounds per 24-hour production period.

It is recognized that a single-disc unit of this sort does not exhibit maximum space efficiency. Therefore, some sort of consolidation is indicated. It is seen as mechanically feasible to stack a group of such circular units above one another on a single common shaft. Assuming a shaft six feet in height, and the spacing of one unit in an eight-inch vertical space, there would be at least eight such units in six feet, providing a production figure of slightly over 1200 pounds per day.

As previously stated, a rotational speed of 200 rpm is rather slow for a disc mill. If a speed of 400 rpm is theorized, then the production figure can be calculated as possibly doubled - to over 2400 pounds per day of wet-ground mica. The establishment of more precise figures than these must await development of a prototype. A continuous mica-grinding unit occupying a space 2.5 by 6 feet and turning out a low bulk density product at a rate between 1200 and 2400 pounds per day would appear worth considering, especially in comparison to the muller, which is a batch device.

Addition of Slip and Sheen

The question still remains as to the nature of an additional piece of equipment to impart slip and sheen. It cannot be assumed at this time that low bulk density, slip, and sheen are obtainable in one stage, although there are still possibilities to try toward that end. A specialized device to impart slip and sheen, following grind to low bulk density, seems called for at present.

The most successful test (No. 45N) aimed at improvement of slip and sheen involved contact of already-fine-ground mica with a resilient surface which appeared not to grind very much but rather caused gentle attrition among the mica flakes, resulting mainly in removal of rough, curled edges. To conduct extensive tests in this area, it is first necessary to have relatively generous batches of attrition-ground mica on which to work - implying, again, the need for a small production prototype attrition mill. With sufficient sample batches available, further tests can be run in quick succession, trying polishing variables in the small Abbé mill, plus various other more unusual techniques. One such would be placing fine-ground mica pulp in a steel container with rubber balls and agitating it by means of a paint shaker in a paint store. If this were to yield good results, then a production unit to accomplish the same thing is now avilable: a multi-chamber vibratory tube mill, able to operate wet or dry - which could be charged with soft or resilient media. It is presently felt that, if the practicability of attrition-grinding mica in the manner described is established in terms of commercial feasibility, then the problem of imparting acceptable slip and sheen will be relatively easy to solve.

Space requirements of a polishing circuit cannot be estimated at this time.

CONCLUSIONS

Using a wet attrition-grinding technique with properly-controlled variables, it is possible to fine-grind and to delaminate muscovite to lower bulk density than by current commercial techniques, even if that muscovite is of a mineralogy involving relative softness coupled with a strong interlaminar potassium bond.

This wet attrition-grinding technique appears to be commercially practical in terms of production rate as calculated from actual production figures of a small laboratory batch mill: this with the assumption of certain structural and performance features for a continuous-production unit.

The failure of the described attrition-grind process to impart the characteristics of slip and sheen can be compensated by a second processing circuit involving smoothing and polishing by soft media. There is some doubt this can be done in the same circuit as the attrition grind. This polishing step should be relatively easy to optimize.

Objective measurement of the characteristic of slip appears possible, using instrumentation and technique developed at the Minerals Research Laboratory. This can be used to evaluate the characteristic of slip in assorted mica products turned out, compared to commercial products. An equivalent test for mica gloss, or sheen, is yet to be developed.

In addition to being a preparatory step to creating good wet-ground mica of high slip and apparently good sheen, the attrition-grind stage alone of this process can yield a product of very low bulk density which should be put out for end-use research. Further, the applicability of this process should be evaluated for such minerals as synthetic phlogopite, talc, pyrophyllite, anthophyllite asbestos, and other laminar or fibrous minerals.

RECOMMENDATIONS

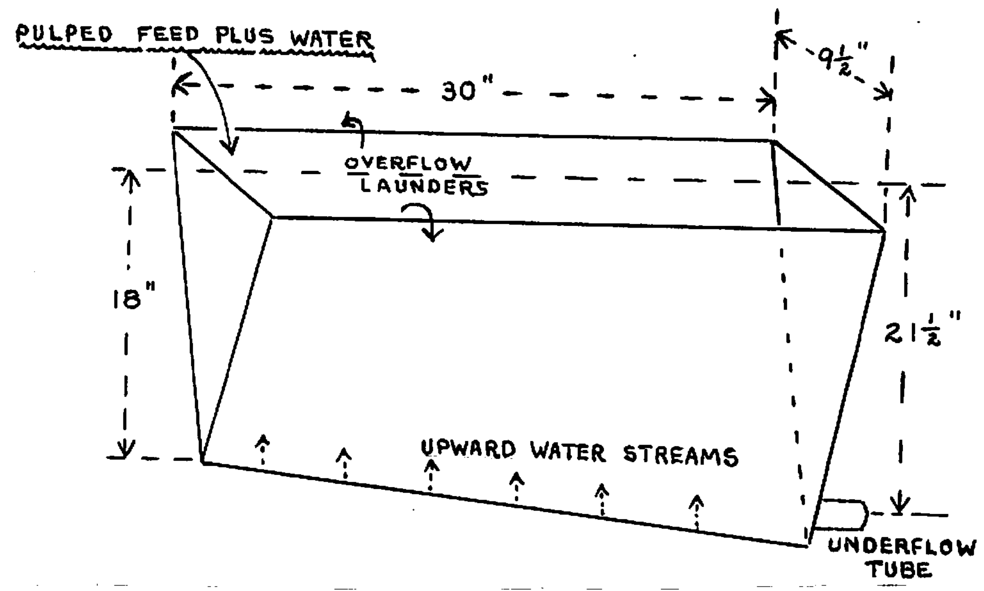
It is recommended that research be continued by construction of a prototype horizontal disc attrition mill as previously described. This mill would embody a single disc having an outside diameter between 10 and 11 inches, with 4 to 8 media giving a total linear contact of from 12 to 24 inches. Its theoretical production rate would be between 12.8 grams and 51.2 grams per hour, depending on total media units (4 to 8, 3 inches each) and operating rpm (200 to 400). It is further recommended that screening and hydroclassifying be set up to operate in closed circuit with this unit so as to minimize fines and to secure product rapidly. This total unit should then be capable of giving production and power consumption figures having close relationship to production potential of this system on a commercial scale.

Primary hydroclassification of product on a batch basis during tests of this report was carried out in a cylindrical vessel close to 12 inches in diameter by 12 inches deep. Primary settling was for 10 minutes, indicating that a particle of milled mica settling slower than 12 inches in 10 minutes, or 1.2 inches per minute, came under the category of either fines or product. Thus, if a pulp flow rate of $\frac{12^2 . \pi}{4}$ x 1.2 cubic inches per minute overflows from an aperture equal in area to a 12-inch circle, this should carry off a ground mica product close in size to that secured by the batch technique just described, and sinks should likewise be comparable. The figures cited resolve to 143 cubic inches of pulp per minute overflowing across 113.1 square inches of area, or 1.26 cubic inches of pulp flow per minute per square inch of surface area.

For performing this hydroclassification with the prototype mill described, a V-box hydroclassifier owned by the Minerals Research Laboratory appears applicable. Figure 3 is a schematic diagram of this V-box.

FIGURE 3

Schematic Diagram of MRL V-Box Hydroclassifier



This V-box will provide a surface overflow area in excess of that needed to be equivalent to the batch settling process just described. The overflow area can be decreased by use of a movable V-shaped core. Overflow containing milled product can be rapidly filtered in batches with the Laboratory's Galigher pressure filter. Underflow, removed from the underflow tube, can be regulated either by a pinch clamp on rubber tubing, or by a slowly-rotating screw.

The hydroclassification procedures just mentioned will require change if commercial production is undertaken. The most critical operation will be dewatering of a very thin overflow pulp containing product, probably between 0.25 percent and 2.0 percent solids. Tests in the Laboratory, using No. 4 Whatman filter paper and a vacuum aspirator, gave certain figures regarding water throughput of a 0.25 percent solids pulp of fine-ground mica (Sample No. 3569). Assuming that a commercial vacuum filter belt would equal this performance on a unit area basis, it is calculated that such a device, 9 feet wide and moving 12 to 13 inches per minute, could dewater 100 pounds per hour (2400 pounds per day) of mica to a filter cake. Among additional equipment to supplement or replace a vacuum filter are high-velocity hydrocyclones, centrifuges, spray dryers, and horizontal filter presses. At this time it is desired only to indicate that existing technology can adequately handle this type dewatering; details can be worked out later.

When such an attrition-grind system is constructed and shaken down, variables such as feed residence time, rate of feed, pulp density, rotational speed, media pressure, linear versus broad-area media contact, slippery vs. "dragging" reagent additives, and others should be investigated. Factors possibly imparting high slip and sheen in this circuit should be investigated. Along with the preceding, batch tests should be run on product from the attrition mill to establish optimum conditions in a second circuit for imparting high slip and sheen. In addition, an objective test for reliably determining relative gloss or sheen of fine-ground mica should be worked out.

At some point when sequence of procedure for creating desirable product is well understood, samples should be submitted outside the Minerals Research Laboratory to knowledgeable commercial mica users, for their evaluation.

Patentability of certain features of this attrition-grinding process, and of any subsequently-developed polishing process, should be investigated and acted upon, although additional exploration and research seem indicated prior to this. Securing of data from the proposed prototype, plus evaluation of product samples from it, appear to be wise as preceding steps.

In the event of construction of, and research with, a prototype setup by the MRL, a future report should detail features of construction and performance tests.

SUMMARY

Continued experiments with a small wet-attrition mill, employing non-cataracting rod media to fine-grind flotation or spiral mica, indicated that this technique, applied with proper variables control, yielded a mica product of very low bulk density. It could easily delaminate a mica known to be hard to delaminate due to softness combined with strong interlaminar bond. Products from such a mica, in the minus 325 mesh range, had bulk densities below six pounds per cubic foot, and optimum variables had not yet been confirmed.

The attrition-grind procedure yielded a product of low bulk density, but with poor characteristics of "slip" and "sheen". A second polishing procedure (wet-milling with a rubber-covered rod) enhanced these characteristics but raised bulk density of the product.

To objectively measure slip of fine mica, a slide block test was designed. It was found that mica products having observably high gloss and a "soapy" feel, when subjected to this test, had a higher slip angle than dry-ground micas of lusterless appearance and "chalky" feel. A sample of hard-to-delaminate mica, attrition-ground, had some characteristics which did not fully correlate with those of other micas: low bulk density, moderately high slip angle, but observably poor gloss or sheen. After polishing, this product increased in bulk density, but acquired visible sheen and also the highest slip angle of any fine-ground mica tested.

Photographs from a scanning electron microscope were useful in determining a number of conditions in different fine-ground micas.

Majac-ground mica particles were jagged, irregular, blocky, and laden with superfine debris. Water-ground (mulled) mica particles were smooth, thin, uni-planar, and relatively debris-free. Mica attrition-ground in the Laboratory was relatively unilaminar and thin-flaked, but with frayed, curled edges. The same mica after polishing closely resembled water-ground mica, although traces still remained of frayed or split edges.

From the characteristics observed in the SEM photographs, it was theorized that water-ground mica which felt "soapy" to the touch ("high slip") nevertheless gave a high slip angle reading because it had more flat particles exerting frictional bearing on the sliding block. By this token, dry-ground mica could not bear as many flat surfaces against the block, which consequently slid at lower inclination. The attrition-ground laboratory mica had a somewhat poorer (lower) slip angle than commercial wet-ground mica due to the curled edges of particles, although still superior to Majac-ground. With the curled edges removed by polishing, it had a higher slip angle than any other mica, raising basic questions as to its true quality in comparison with commercial wet-ground products.

The evolution of a second objective test - for gloss or sheen - is seen as necessary for proper future evaluation of Laboratory products in comparison with others.

Based on encouraging results to date, especially in delaminating and polishing a mica hitherto considered impossible to process, the theoretical performance of a disc-type production unit was calculated, based on batch-mill performance in the Laboratory. These calculations lead to a recommendation that a small prototype production unit be constructed to obtain meaningful data regarding power needs, plus ultimate product quality and grinding rate. Theoretically, a mill occupying 37.5 cubic feet could grind between 1200 and 2400 pounds per day of mica to grade.

The possible application of this grinding technique to other laminar or fibrous minerals is seen as well.