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METHODOLOGY

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INTRODUCTION

This report covers methods that have been developed, are presently being used, or are being investigated by the Colorado School of Mines Research Institute relative to the examination of talc samples. The main emphasis of this study was to develop methods to accurately determine the mineralogy, particle size, and particle shape distribution of finished product talc samples.

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MICROSCOPE TECHNIQUES

GENERAL

A Leitz polarizing microscope is the microscopic tool used in the examination of talc samples. The lens system of the polarizing microscope is in many respects similar to the lens system of the usual compound microscope but has several modifications such as: (1) polarizing devices, both above and below the stage, (2) a rotating stage, and (3) accessories such as a mica plate, a gypsum plate, and a quartz wedge (etc.). This type of microscope is extensively used to examine mineral fragments, small crystals (etc.). It is extremely useful in the determination of optical properties of individual crystals, mineral associations, and size and shape of mineral grains.

The microscopic procedures herein outlined were all performed at 500X in an index oil having a refractive index of 1.600. This oil was chosen because the refractive index of the a and b axis of the platy talc examined approached 1.590. When the index of the oil matches that of the mineral being observed the mineral is not visible in ordinary light. The spread of 0.01 between the 1.600 oil used and the 1.590 of the talc made the talc minerals barely visible in ordinary light, thus facilitating their identification. The other nontalc minerals were clearly visible due to observable negative or positive relief.

Other optical properties such as optical sign, interference figures, extinction angles, birefringence (etc.) were also used for mineral identification. 10X oculars were used in all studies. One ocular contained a cross

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hair and one contained a graduated scale that was calibrated from a stage micrometer.

The lower resolution limits of our optical microscope is approximately 0.5 microns, therefore all data procured refers to particles above that size.

SAMPLE PROCUREMENT AND PREPARATION FOR POINT COUNTING

Many attempts were made to split a representative sample from a standard finished product (-325 mesh, Tyler) using a variety of standard sample splitting devices. None were satisfactory due to severe dusting and material caking problems in the devices. It was found that a small sample procured from near the center portion of the submitted sample proved the best method for obtaining a representative sample for microscopic analysis. Assays have shown this type of sampling to be representative.

The preparation of the sample for microscopic examination can be outlined as follows:

1. Place a clean, dry standard glass microscope slide on a flat surface. It is extremely important that the slide be washed with acetone or alcohol and wiped clean with a tissue that will not leave adherent fibers on the slide.

2. Place one drop of the 1.600 refractive index oil near the center of the slide.

3. Place the talc sample in the oil and gently stir the slurry until the particles are evenly distributed by visual observation.

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4. Place a clean, dry 2 cm x 2 cm glass cover glass on the slurry of oil and talc.

5. Place the sample on the rotating microscope stage containing a standard mechanical stage mechanism to control the amount of horizontal and vertical movement during the microscopic examination.

6. Calibrate the mechanical stage so that the entire 2 cm x 2 cm cover glass area will be traversed during the 500 grain point count.

7. Adjust the microscope lights in such a manner that will produce transmitted light. It was found a blue filter between the light source and the sample produces better results in regard to mineral identification (etc.).

8. Observe the sample at 100X. It will become apparent upon close observation that many plates will be standing on edge giving the effect of needles.

9. With a dissecting needle gently touch the cover glass. This will usually cause the plates to rotate and lay flat. Much caution must be used in this procedure that no extreme downward exertion be applied. Too much downward exertion will cause a squeezing effect and many particles will be moved to the outside of the cover glass.

10. Now observe the sample at 500X and move the slide so as to start the examination at one corner of the cover glass. Using the intervals determined in No. 6 (above) move the slide and make determinations of the particle under the cross hair at each movement interval.

11. It may be necessary to use the dissecting needle occassionally throughout the examination to touch the cover glass to help orient the grains. Again much caution must be used so as not to produce a "squeezing effect."

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MINERALOGY

Each particle examined is identified by using standard optical techniques and placed in one of the following categories. (It is advisable to make tables similar to the one shown in Figure 1 to record all data. Enough of these sheets are necessary to accommodate 500 grains.)

1. Plates (talc and/or chlorite) -- Particles that could positively be identified as plates.

2. Talc Shards -- These are thin pieces that could definitely be defined as being pieces broken off of plates. This entails a very detailed study of each piece to determine if ragged edges (etc.) are present. These particles have length/width ratios of 5:1 or less.

3. Talc Needles -- These are particles that have (a) parallel extinction, (b) refractive indices approaching 1.590, and (c) length/width ratios greater than 5:1.

4. Nontalc Needles -- These are particles that had: (a) nonparallel extinction, (b) refractive indices above or below the 1.600 oil used, and (c) length/width ratios greater than 5:1.

5. Quartz -- This mineral was identified by standard optical techniques.

6. Carbonate -- This mineral was identified by standard optical techniques.

7. Diamond-Shaped Particles -- These are minute (1-2μ) particles that are definitely diamond shaped and have refractive indices above 1.600. These were tentatively identified as carbonate or pyroxenes.

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Figure 1. Typical Type of Table Used in Point Count Analyses

Grain No.	Remarks	Platy Talc and/or Chlorite	Talc Needles	Nontalc Needles	Talc Shards	Carbonate	Quartz	Dark Opaque	Diamond Shaped Particles	Granular Masses	Other

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8. Other Particles -- These are birefringent particles usually in the 1.0-0.5 micron range that could not be positively identified optically.

9. Dark Opaque -- These are particles that do not transmit light.

10. Granular Masses -- These are masses of nearly submicroscopic particles that appear to be tightly bound to each other and not merely poorly dispersed masses of fine particles.

PARTICLE SIZE AND SHAPE

On the table shown in Figure 1 the following was recorded under each mineralogical category noted: (1) maximum length of each particle observed, (2) maximum width of each particle observed. These measurements were recorded as direct ocular micrometer measurements. They were later converted to millimeters or microns for size distribution analyses.

PARTICLE SIZE AND SHAPE DISTRIBUTION

The size distribution of particles is dependent upon the ultimate information desired (shortest dimension, area, number of grains, etc.). The things that have to be decided before making the distribution may be outlined as follows:

1. What type of size data is needed:
   (a) Size based upon smallest dimension.
   (b) Size based upon longest dimension.
   (c) Size based upon square mm or square micron area.
2. What upper and lower size limits and size increments are needed in the study.

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3. Is the study to be confined to individual mineral groups or a combination of mineral groups.

4. Is the data to be presented as number of particles or the percentage of particles in each category (etc.).

In our studies a Pacific Data Systems 1020 tape computer was used for the conversion of ocular units to millimeters or microns and the sorting of particles into each size range required.

The distribution of particles based on particle shape involves only the ratio of length to width based upon the ocular units; the conversion to millimeters or microns is not necessary. Again limits must be set and again we used a Pacific Data System 1020 computer to sort the ratios obtained into the desired categories.

If the volume or weight percent of various types of grains is desired from optical microscopy, care must be exercised to insure that the most meaningful type of data are obtained. If grains are simply counted without regard for their size, then the data are not applicable to volume or weight percent analysis unless all of the particles have the same volume and specific gravity. This may be illustrated by the following example. A sample consists of 2 grains in which grain A has a volume of 100 cubic microns and grain B has a volume of 1 cubic micron. On the basis of grain count alone, each grain comprises 50% of the sample. However, on a weight basis there is 100 times as much of grain A as there is of grain B (assuming equal specific gravities). Ideally, the volume of each grain should be measured and its weight computed from its specific gravity. However, this is not practical by

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optical microscopy since only 2 grain dimensions are observed. The best approach, therefore, is to measure the observed cross-sectional grain area for quantification of each grain type. This was the approach used in our determinations of various talc products.

X-RAY ANALYSIS TECHNIQUES

GENERAL

Brindley, 1961, states "The basic principle underlying the identification of materials by x-ray or by electron diffraction is that each crystalline substance has its own characteristic atomic structure which diffracts x-rays or electrons in a characteristic pattern. The recognition of the pattern establishes uniquely the diffracting substance."

The two methods of x-ray analysis most generally used are x-ray powder diffraction techniques utilizing a scanning goniometer and circular powder camera techniques. Basically both methods produce similar data although one may be superior to the other in certain instances. In both techniques a generator of x-rays is needed. In our studies Cu radiation filtered with a Ni filter and a goniometer scanning speed of 2°/minute were utilized in the powder diffraction. Cu radiation was also used in the circular powder camera work.

POWDER DIFFRACTION TECHNIQUES UTILIZING A GONIOMETER

In this process about 0.5 g of sample is pressed into a standard sample mounting plate. This sample is then scanned, using a goniometer, at 2°/minute and the diffracted x-rays that indicate "d" spacings (lattice spacings)

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are traced on a graph that is moving in phase with the speed of the goniometer. The advantage of this type of analysis is that a relatively large sample can be examined. This increases the chance of a representative sample being analyzed. The disadvantage is that because the goniometer is moving at 2°/minute, minor to trace amounts of material may not be detected.

CIRCULAR POWDER CAMERA TECHNIQUES

In this process a very small amount (±0.1 g) of sample is either mounted on the end of a glass filament or mixed with a material invisible to the x-ray beam and rolled into a needle containing the mineral fragments, or packed into a glass capillary tube. This sample is then placed in the center of the powder camera and rotated in the collimated x-ray beam. The diffraction lines in this technique are recorded on a piece of strip film placed on the peripheral inside edge of the camera. The advantage of this method is that the exposure time can be varied, thusly producing a situation where very minor amounts of material can be detected. The disadvantage is that because of the small sample size much care must be exercised to assure a representative sample is being analyzed. It is advised that both methods be used to examine samples in question.

ELECTRON MICROSCOPE TECHNIQUES

GENERAL

The electron microscope is a tool used to show directly the morphology of very small particles. This technique is usually used on finely dispersed material resting on a collodion substrate.

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POSSIBLE USES

This tool has been used extensively to observe the morphology of submicroscopic particles in various samples. Although some work has been done on the morphology of submicroscopic particles in talc samples, this tool will probably prove invaluable in regard to particle shape in the submicroscopic fractions of talc. Again, as in circular powder camera analyses, there may be serious problems in the procurement of a representative sample if the tool is used for size distribution (etc.). This may be more of a problem in electron microscopy than in powder camera analyses since a smaller amount of sample is used in electron microscopy.

ELECTRON DIFFRACTION TECHNIQUES

GENERAL

This technique is similar to x-ray diffraction but utilizes electrons for diffraction rather than x-rays. The combination of electron diffraction and the electron microscope provides a means to identify both the shapes and crystal type of observed particles. It is possible to isolate single grains and do single crystal electron diffraction analyses to identify individual grains.

POSSIBLE USES

This tool, coupled with the electron microscope, could prove invaluable in the identification of submicroscopic particles.

ELECTRON PROBE MICROANALYZER TECHNIQUES

The electron probe microanalyzer concept is an outgrowth of the following: (1) electron microscopy, which introduced techniques f...

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and controlling electron beams; (2) x-ray spectroscopy which provides a relatively simple instrumental method for chemical analysis; and (3) light optical microscopy, which provides for viewing a specimen and visually selecting areas for analysis.

POSSIBLE USES

Since this technique is capable of making nondestructive chemical analysis on particles down to approximately 1 micron in size, it can be very useful in chemically analyzing small particles. The technique is also capable of making 1 micron wide line scans across particles to determine compositional variations. A visual display, showing the distribution of various elements over selected areas of a specimen, can be provided. Photographs of the elemental distribution can be made for permanent records.

NONMICROSCOPIC TECHNIQUES TO DETERMINE PARTICLE SIZE AND SHAPE DISTRIBUTION

GENERAL

The possibility of size distribution analyses of talc using nonmicroscopic methods has been a matter of much debate and examination. Light optical microscopic methods of particle size and shape distribution is theoretically accurate but it is time consuming and is limited to particle size above 0.5 micron.

POSSIBLE USES

Many types of particle size and shape methods have been examined and most contain numerous pitfalls. In regard to standard sedimentation (Stokes law), particle shape, differential particle sizes, and temperature

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play such a critical role that the validity of results is debatable. Studies at CSMRI have indicated that even the most gentle dispersion and centrifuge techniques tend to produce fines in talc. Some methods have been investigated that do not involve agitation (etc.). These methods basically involve mounting a specimen on various media and scanning them with various light or radiation sources. These methods appear most usable at this point in time but one major drawback can be recognized. As was mentioned in the section on light microscopic examination of talc samples many of the talc plates tend to stand on edge giving the shape effect of needles. These particles are properly oriented in the light optical microscopic examination before measurement. It is debatable if this degree of proper orientation can be achieved in the sizing devices mentioned above.

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