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PREPARATION AND CHARACTERIZATION OF
ULTRAFINE PARTICLES FROM MATERIALS
OF HIGH HARDNESS

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Nuclear Division

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ABSTRACT

A method has been developed for producing ultrafine ($< 10 \mu$) particles from high-hardness materials by different methods and classifying these particles by ultrasonic acceleration of the particles in a fluid medium through a ten-micron micromesh sieve. The powder is collected in a semicontinuous super-centrifuge. Further classification is made by a sedimentation classifier to produce powders that are less than three microns in diameter.

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SUMMARY

Methods have been developed that can prepare and classify ultrafine particles ($< 10 \mu$). Emphasis was placed on innovations in fluid-energy-mill techniques and improved classification of ultrafine particles by the use of an ultrasonic generator to accelerate the particles through a ten-micron micromesh screen. Collection was accomplished by means of a semicontinuous supercentrifuge. An explosive reduction of particle size was achieved and a study was made of a sedimentation particle-size classifier ($0.5 - 10 \mu$) utilizing shutter valves.

INTRODUCTION

Studies involving particle technology are of importance to many industries which are directly or indirectly concerned with the use of powders, including ceramic and metallurgical industries. The properties of particles are important in determining the sinterability of powders, since the reactivity of solids depends on the surface area, and since smaller particles exhibit higher vapor pressures than larger ones.⁽¹⁾ The rate of a diffusion-controlled reaction is proportional to the surface area (Fick's law), an important consideration in sintering reactions and cement hydration. The absorption of vapors by solids because of chemical or van der Waals forces depends on the particle size, which is important in the surface hydration of ceramics (viz, MgO and CaO) as well as the surface oxidation of metals. Particle technology is vital in determining the strength of compacts (particle packing, sinterability or particle reactivity, porosity or pore-size distribution), fluid or flow properties of solids, wettability of different materials (metal-to-glass bonding), and flocculation and dispersion of fine particle suspensions (slip casting).⁽¹⁾

The purpose of this report is to describe the methods used at the Oak Ridge Y-12 Plant^(a) to prepare and classify ultrafine ($< 10\mu$) particles of such hard materials as ceramic aluminum oxide and crystalline boron without contamination.

(a) Operated for the US Atomic Energy Commission by the Union Carbide Corporation's Nuclear Division.

PREPARING AND CHARACTERIZING ULTRAFINE PARTICLES

PARTICLE-SIZE REDUCTION BY MECHANICAL FORCES (COMMINUTION)

Large Particles to Intermediate Particles

Compacts or large particulates of aluminum oxide and crystalline boron were ground by conventional techniques utilizing iron components in order that the iron contamination could be removed by magnetic or leaching methods.

Intermediate Particles to Fine Particles

A rather unique method was used to reduce the size of particles greater than 200 mesh ($74\ \mu$). This method involved the reduction of particle size by shock waves produced by an explosive detonation.⁽²⁾ The procedure consisted of loading the desired particulate material into a metallic container, sealing the container, and wrapping the loaded specimen in an envelope of plastic explosive. The charge density was two grams of explosive per square inch of surface area (a higher charge density caused rupture of the Type 304L stainless steel container). The components of the assembly are shown in Figure 1. In operation, the center rod was removed and the end piece, which is the small disc at the center of the rod in Figure 1, was welded to the remainder of the cylinder. Next, the rod was inserted, but set slightly ajar, leaving a small space for the particles to be admitted. As much of the granular material as possible was fed through the space, and the steel rod was forced into position flush against the end of the cylinder. Finally, the rod was welded to the cylinder at both ends. After the explosive charge was affixed, the capsule was suspended by a wire in a water tank from an overhead crane, the hole in the upper end of the steel rod serving as a handle for this purpose. The explosive was detonated by an electric impulse. Sample retrieval was achieved by simply cutting open the assembly. This method was successful in reducing the particle size of material that was originally 100 percent greater than 200 mesh to the state where 50 percent by weight was less than 500 mesh ($26\ \mu$) and 90 percent less than 200 mesh. This decrease in particle size may be a contributing factor to the reported lower sintering temperatures and high densities⁽²⁾ obtained with explosively compacted materials, which involves a process similar to the one described here. It should be noted that when this process was attempted on material already fine ($90\% < 38\ \mu$, $50\% < 15\ \mu$), the material remained essentially the same after an explosive shock ($90\% < 38\ \mu$, $50\% < 13\ \mu$). It is concluded that the explosive-shock method is of considerable value in reducing large particles, but is not capable of reducing fine powder to a still smaller size. This conclusion is in line with expectations, since the small particles would have a tendency to cushion the impact of the shock.

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Figure 1. STEEL JACKETS FOR REDUCING POWDER SIZE BY SHOCK WAVES.

Fine Particles to Ultrafine Particles

Particles less than 200 mesh (74μ) were reduced effectively in the fluid-energy or jet-mill^(b) arrangement seen in Figure 2. In this system, two opposing high-velocity jets of argon gas cause intense local velocity gradients, and intense interactions are set up within the circulating material.⁽³⁾ The particle reduction is thus caused by the particles impinging upon each other, and no contamination is introduced by the grinding. The gaseous fluid supplying the grinding energy is withdrawn at an inward point, tending to cause the particle-laden gas to travel spirally. By this action, the smaller particles are carried out with the gas and the coarser particles are thrown to the periphery where they are subjected to further reduction. Thus, the grinding chamber also serves as an internal classifier. Most commercial models cause the exit gas from the mill to enter a cyclone separator⁽⁴⁾ (a conical chamber) tangentially and leave through a central opening; thus, the heavier particles, by virtue of their inertia, tend to move toward the outside separator wall from which they are led to a receiver, usually

(b) Manufactured by the Trost Equipment Corporation, Newtown, Pennsylvania.

Figure 2. FLUID ENERGY MILL GRINDING ARRANGEMENT.

a bag or product bin. The cyclone is essentially a settling chamber in which gravitational acceleration is replaced by centrifugal acceleration. However, most of the ultrafine particles produced by the fluid energy mill are carried out through the cyclone exhaust.

The bag-type collection system proved to be very inefficient, since many particles escaped through the weave of the bag and since removal of the product was very time consuming and awkward. The system was especially inadequate if recycling of the particles was necessary to obtain a particular particle size distribution. Particles were normally introduced to the fluid energy mill through a venturi tube by the operator or by a complicated vibratory feeder. Because of these inadequacies in commercial fluid energy mills, the arrangement shown in

Figure 2 was designed. Particles were recycled in a closed-loop fashion until they left through the cyclone exit, where they were trapped by being bubbled through a liquid (usually ethyl alcohol). Some particles were so fine that they were transported by the vapor and gas in the bubbler to the filter outlet ($0.3\ \mu$ filter) where they were trapped. In practice, particles trapped in the fluid were found normally to be less than ten microns, whereas particles from the filter were somewhat smaller. Particles were recycled for as long as 32 hours, depending on the fineness desired. It should be noted that the rate of grinding decreases as the fineness of the grinding proceeds.⁽⁵⁾ This fluid-energy-mill arrangement would be ideal for toxic or radioactive powders, since it is completely enclosed, has automatic feeding, and all particles are trapped. Also, this particular arrangement can readily be scaled up to accommodate production-scale batches. Figure 3 shows the fluid energy mill and accompanying equipment before the automatic feeding arrangement was used. The 0.3-micron filter is not shown.

A typical mass-weighted size distribution of particles obtained from the bubbler fluid after long recycling (32 hours) is given by the graph of Figure 4; Figure 5

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Figure 3. FLUID ENERGY MILL ARRANGEMENT BEFORE THE AUTOMATIC FEEDING ATTACHMENT WAS ADDED.

shows the particle size distribution of the -500 mesh ($26\ \mu$) starting material. A great reduction occurred (approximately four fold, based on the ratio of the mean particle sizes). All distribution analyses of ground or classified material were obtained by the Coulter counter.^(c)

CLASSIFICATION OF ULTRAFINE PARTICLES

Particles Larger Than Ten Microns

Standard sieves were used to classify particles to -500 mesh ($26\ \mu$), and standard vibrating sieves or automatic shakers were used to aid the particles in flowing through them. A commercial sonic-sifting device was used that utilized acoustic-frequency air waves (an oscillating air column with a frequency of 50 or 60 cps) to diffuse particles through sieves; however, when the apertures were less than 20 microns, significant cohesion or agglomeration of the powder occurred, causing a great reduction in the fraction capable of passing through the sieve. The sonic-sifting method was recently studied by Suhm⁽⁶⁾ and was utilized mostly for particles greater than 37 microns.

Particles Less Than Ten Microns

Classification by Felvation - A process called "felvation"⁽⁷⁾ (a process combining the advantages of fluidization, elutriation, and sieving) can be used to classify particles that are less than 10 microns, since the powder is fractionated by fluidization and elutriation in a fluid flow upward toward a sieve surface. However, careful control of the elutriation conditions is necessary to produce a selective presentation of particles to the sieve apertures. This step must be performed in such a way that the smallest particles carried by the fluid-flow streams are presented first to the apertures in the sieve, then pass through the holes. As the sieving process continues, the elutriation velocity is increased so that the larger particles are presented selectively to the sieve aperture. Ultimately, a condition is reached where the particles are of sufficient size to be retained by the sieve. At this point, the particles capable of passing through the sieve have done so and the sieving process is complete.

The classification system used for ten-micron particles is shown in Figure 6. The following features should be mentioned:

-
- (c) An electronic counting technique where a very dilute, well-dispersed suspension is caused to flow through a small aperture, with essentially one particle at a time passing a screening device. Particles passing through the screening device are detected by the change they produce in the electrical conductivity of the medium. The threshold value of the change in conductivity which will be counted can be varied, thus providing a count of the number of particles above the chosen setting.

Figure 4. PARTICLE SIZE DISTRIBUTION OF MATERIAL OBTAINED FROM THE FLUID ENERGY MILL BUBBLER. (By Coulter Counter)

Figure 5. PARTICLE SIZE DISTRIBUTION OF - 500 MESH STARTING MATERIAL. (By Coulter Counter)

1. The liquid containing the fine particles from the fluid-energy-mill bubbler previously described is added to the left compartment of the ultrasonic vibrator tank.
2. Ethyl alcohol is used as the fluid medium.
3. An air-driven stirrer is used to keep the particles in a uniform slurry.
4. The ultrasonic vibrator tank diffuses the particles through the screen.

The micromesh sieve^(d) for the ten-micron screen was formed by the electro-deposition of nickel. A supporting grid was made in the same manner and

Figure 6. TEN-MICRON CLASSIFICATION SYSTEM.

(d) Manufactured by the Buckbee Mears Company, St. Paul, Minnesota.

bonded to the micromesh sieve. The resulting screen (8" x 8") has very uniform square apertures certified to be ± 2 microns from nominal. Both a ten and five-micron sieve were obtained. Particles less than ten microns pass through the ten-micron screen and are pumped from the right side of the tank to a semi-continuous supercentrifuge, then through a clarifier bowl where the fine particles are removed. As Figure 6 indicates, the clear alcohol is returned to the ultrasonic tank on the coarse side of the filter, adding to the motion of the fluid through the screen.

The screen was sealed to the ultrasonic tank with a silicone sealant. Pumping was obtained by utilizing rollers to compress the plastic tubing, creating a vacuum which pumped the alcohol solution to the centrifuge. It was necessary to machine an aluminum facing for the pump to prevent the alcohol from reacting with the plastic facing supplied with the pump. The air-turbine centrifuge provided a direct-driven rotation of 50,000 rpm, or an acceleration equivalent to 62,000 g's. The ultrafine particles ($< 10 \mu$) thus were driven against the walls of the clarifier bowl by the centrifugal force, and the clarified liquid was exhausted from the spout. The liquid from the spout became cloudy when the bowl became filled. At this point the bowl was removed, a new one inserted, and the process continued until all the ultrafine particles were removed. Heating of the ethyl alcohol in the ultrasonic tank sometimes occurred during the classification operation, but this could be controlled by adding small amounts of dry ice to the alcohol slurry in the tank. A typical particle-size distribution of the product which passed through the ten-micron micromesh screen and collected in the clarifier tube is indicated by the graph of Figure 7.

Classification by Wet Sieving - A wet-sieving method, utilizing electroformed sieves for determining the particle size distribution of materials, was recently reported.⁽⁸⁾ This method, designed for very small samples, involved alternate surges of pressure and suction to create a pulsing action of the sieving liquid to move the particles through the sieve; however, "blinding" of the screens was experienced. It was stated that the sieves were ultrasonically cleaned, but damage to the screens was noted from the cleaning operation. In this study, no evidence of deterioration of the screen was found after many operations with the ultrasonic acceleration used (frequency of 25 kHz). From Figure 7, it is seen that 88 weight percent of the particles passing through the sieve were less than twelve microns, the upper limit of the variation from the nominal aperture size of ten microns for the micromesh sieve. The twelve weight percent of material above this limit (12μ) probably penetrated the screen because the particles had an elongated shape.

A sample from the fluid-energy mill was used to test the efficiency of the apparatus. The starting material had a particle-size distribution of: 90 percent less than 16 microns, 50 percent less than 6 microns, and 10 percent less than 3 microns. Samples of the material on the coarse side of the filter were taken at intervals

Figure 7. PARTICLE SIZE DISTRIBUTION OF MATERIAL THAT PASSED THROUGH A TEN-MICRON MICROMESH SCREEN. (By Coulter Counter)

after the vibrator was started. After 45 minutes, the distribution was found to be: 90 percent less than 20 microns, 50 percent less than 15 microns, and 10 percent less than 11 microns. These results indicate that the particles less than 10 microns in diameter were able to pass through the filter very effectively.

This system offers the advantage of an ultrasonic acceleration of the particles, with wet screening providing an optimum removal of product while preventing particle agglomeration and "blinding" of the screen.

Classification by Sedimentation - A new sedimentation particle-size classifier was constructed for the classification of particles less than ten microns. The design, shown in Figure 8, utilized two shuttle valves. After the classifier was filled with an appropriate settling medium, the upper valve was closed and a slurry of the settling medium and the particles to be classified was poured into the apparatus above the valve. This valve was then opened, allowing the particles to settle in the static medium. Stokes' law was used to calculate the time required for a particle of the desired size to reach the lower valve. After the particles settled for this period of time, the lower valve was closed, preventing further settling. The slurry in the region between the upper and lower valves was then either poured or syphoned off and allowed to pass through a fritted-glass filter.

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Figure 8. SEDIMENTATION PARTICLE SIZE CLASSIFIER.

The product obtained in this manner should contain only particles less than the particle size calculated by Stokes' law. If both an upper and lower limit of a particle size were required, the product was rerun and the time was calculated on the basis of the lower particle size. In this case, the slurry between the two valves contained particles smaller than the required lower particle size and was discarded, whereas the slurry below the lower valve contained particles in the desired range. By adding a third valve at an appropriate distance from the second valve (depending on the lower particle size required), a particle-size range can be established with one run.

Boron (density, 2.34 gms/cc) was initially used to investigate the operation of the classifier. Because the theoretical requirements of Stokes' law are almost impossible to completely fulfill, it was expected that the required time to obtain the correct particle-size fraction would be somewhat different from that calculated from the law. Thus, in order to find the best multiple of the calculated time to obtain the optimum yield and the correct particle size, several settling times were employed. The multiples of the time calculated by Stokes' law were: 0.75, 1.00, 1.25, and 6.28. To obtain good separation, it was necessary to use a starting powder with a median particle size equal to the upper particle size required in the desired range to be separated. Since the boron powder had a median particle size of 5.5 microns, the times were calculated on the basis of 5.5-micron particles. The Coulter counter method was used for determining particle sizes of the products obtained. The results (Table 1) show that for the calculated time by Stokes' law, 85 percent of the particles in the product were less than 5.5 microns; whereas, for the 1.25 multiple, 91 percent were less than 5.5 microns. Although the 6.28 multiple had 94 percent less than 5.5 microns, the amount of product was significantly reduced. Thus, the 1.25 multiple of the calculated time was concluded to be optimum. A repetition of the sample using the 1.00 multiple (theoretical time) showed identical results with the previous sample. The products obtained for the 1.00, 1.25, and 6.28 multiples of the calculated time all had a median particle size of about 3.0 microns. A separation at the 3.0-micron maximum particle size level (using a 1.25 multiple of the time calculated by Stokes' law) yielded a product with about 88 percent of the particles less than 3.0 microns. A repetition gave equivalent results. Thus, particles can be fractionated down to 3.0 microns and, based on the reported size for Brownian movement, possibly can be fractionated down to 0.5 micron. The efficiency of separation varied from 56 to 63 percent (as shown in Table 1) using a two-gram sample; the higher efficiencies were obtained with the separation at 5.5 microns.

This apparatus was readily scaled up to accommodate 20 to 50 grams for starting samples. A scaled-up version is shown in Figure 9, where three air-driven solenoid shutter valves were used in order to classify a range of particles below

Table 1
SEPARATION OF PARTICLES LESS THAN 5.5 MICRONS USING THE
SEDIMENTATION PARTICLE SIZE CLASSIFIER⁽¹⁾

Sample Number	Time (fraction of Stokes' law)	Time (hrs)	Percent Less Than 5.5 μ	90 Percent Less Than (μ)	50 Percent Less Than (μ)	10 Percent Less Than (μ)	Efficiency (%)
1	0.75 X	2.253	78	12.0	3.1	1.9	56.1
2	1.00 X	3.0	84	8.0	3.1	2.1	59.6
3	1.00 X	3.0	85	9.6	2.7	1.9	62.5
4	12.5 X	3.75	91	5.2	2.9	2.0	59.1
5	6.28 X	18.74	94	4.4	3.1	2.2	57.7

(1) Alcohol medium with boron particles (density, 2.34 gm/cc); median particle size of starting powder was 5.5 microns.

ten microns in one run. The middle and lower valves were connected to automatic timers, thus closing the valves at the appropriate time for a run which has been calculated by Stokes' law. For every different system of particles it is necessary to find the best multiple of the calculated time to obtain the optimum yield and the correct particle size. Also, different liquids of different densities and viscosities should be used to obtain the best efficiency for a particular system.

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Figure 9. SCALED-UP VERSION OF THE SEDIMENTATION PARTICLE SIZE CLASSIFIER.

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