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ALUMINA LAPPING ABRASIVE STUDY

PDO 6984846, Topical Report

R. D. Wagner, Project Leader

Published October 1977

Prepared for the United States Energy Research and Development Administration Under Contract Number EY-76-C-04-0613 USERDA

Bendix Kansas City Division

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Department 812

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ALUMINA LAPPING ABRASIVE STUDY

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Prepared by R. D. Wagner, D/812, under PDO 6984846

To achieve greater quality control over lapped finishes, the 3- and 0.3-μm alumina abrasives used in rough and finish lapping were studied. The 3-μm abrasive was found to actually average 5 to 10 μm. The particle size distribution of the 0.3-μm abrasive could not be determined with sufficient accuracy. Recommendations for procurement, storage, dispensing, and mixing were made to provide increased abrasive uniformity and freedom from contamination.

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SUMMARY

Alumina abrasives used in lapping processes must retain lot-to-lot uniformity for process control and must be free from contamination. The particle size distributions of two commonly used abrasives were investigated using available techniques, and recommendations were made on size control and handling methods. Nominal 3-μm abrasive was found to be 5 to 10 μm in size; available methods were not satisfactory for evaluating 0.3 μm abrasives. Procurement of large quantities of abrasive from a single manufacturing lot was recommended as a particle size control technique. Methods for storage, dispensing, and mixing were recommended.

DISCUSSION

SCOPE AND PURPOSE

The objective of this project was to determine the particle size distribution, occurrence of agglomerated particles, and presence of contamination in abrasive powders used in lapping operations. This information was to provide the necessary background for establishing quality control procedures at a future date. The two particular abrasives considered were #30 grit (3.0 μm) alumina supplied by Kansas City Abrasives Co., and Linde 0.3A (0.3 μm) supplied by Linde Corporation, San Diego, California. Of secondary interest was the recommendation of procedures and equipment for dispensing, handling, and mixing abrasive powders.

PRIOR WORK

No technical information for the 3.0-μm abrasive was available. Information available on the 0.3-μm abrasive was not engineering data.

ACTIVITY

Abrasive Manufacture

The Kansas City Abrasives Co. facility was visited to obtain background information on the manufacture of 3.0-μm abrasive. The process starts with the purchase of raw alumina in the form of a by-product of grinding wheel manufacturing. The raw alumina comes from several sources and varies in color, shape, and size with each of the vendors and with each shipment. The raw alumina is crushed in a ball mill, separated by size using a water elutriation process, and dried in a furnace. The entire manufacturing scheme is geared to produce several tons of abrasive per day. Size control is dependent entirely on a constant water flow rate in the elutriation process.

The manufacturing process for the 0.3-μm abrasive is proprietary and was not disclosed.

Usage

Abrasives are mixed with appropriate fluids to form a lapping slurry. Precision lapping processes require uniform abrasive particle size distributions in order to maintain process control for each group of parts processed. Otherwise, material removal rates must be empirically established for each change in abrasive particle size to determine the appropriate lapping process.

Particle Size Determination Techniques

Several particle size determination techniques were considered. Actual implementation, however, was limited to three methods considered to be least subject to operator influence: electrolytic resistivity (Coulter Counter), settling rate (Micromerograph), and image recognition (Quantimet). The electrolytic resistivity technique consisted of dispersing the abrasive powder in a conductive fluid (typically a bio-medical saline solution) and then passing the dispersion through a very small orifice. A particle in the fluid changed the resistivity of the volume of fluid in the orifice proportionally to the size (volume) of the particle. The Coulter Counter operation was automated in such a manner that each time a particle caused the resistivity to rise above the background level, a signal pulse was stored in an accumulator set at a threshold corresponding to a predetermined particle size.

Fifteen accumulators were available and were set at thresholds appropriate to the particle size range being investigated. The accuracy of the Coulter Counter technique depended upon the particles being well dispersed and nonconductive, and the assumption that the particles pass single file through the orifice. Samples were prepared by dispersing 1 ml of abrasive in 100 ml of saline solution using ultrasonic agitation and then mixing 1 ml of the first solution with an additional 100 ml of saline solution. While the Coulter Counter manufacturer claimed that the electrical resistivity technique was valid for particles larger than 0.5 μm, references indicated that disagreement between the Coulter Counter results and measurements using other techniques are not unusual for particle distributions smaller than 15 μm.

The cumulative settling rate method for obtaining a particle size distribution consisted of allowing a volume of dispersed abrasive powder to settle in a still column of air. As the abrasive settled to the bottom of the column, the cumulative weight was recorded by a sensitive balance system as a function of time. The application of a modified version of Stoke's law for a falling particle in a gas could then be used to develop a particle size distribution. In practice, the commercial equipment used for this evaluation (Micromerograph) provided charts which allowed direct conversion from cumulative weight to particle size distribution based on the density of the particles. The use of the Micromerograph for particles smaller than 1.0 μm with the density of alumina was not recommended by the manufacturer. The relatively long settling time necessary plus static attraction of the particles to the column walls and each other were cited as the reasons.

Particle size measurement by photographing the abrasive at a known magnification and then scaling the photographs was determined to be an ineffective technique due to several problems, most of which were associated with the quality of photographs obtained. Depth of field problems associated with the relatively high magnification microscope objective necessary (45X, 0.65 numerical aperture [n.a.]) prevented all the particles in the field of view from being sharply focused at the same time.

The depth of field, S (also resolving power), is

$$S = \frac{1.22\lambda}{2(n.a.)} ,$$

where λ is the average wavelength of the illuminating light. Giving λ a value of 0.56 μm (for white light), then

$$S = \frac{1.22 \times 0.56\ \mu m}{2 \times 0.65} = 0.53\ \mu m.$$

For the smaller of the two abrasives, it wasn't possible to distinguish a single large particle from two small particles separated by a distance less than a particle diameter. Also the pack-type Polaroid film used with the available metallurgical microscope probably wasn't flat enough for this application use since small deviations of the film from the image plane of the camera produced an out-of-focus condition at the total equipment magnification used (560X).

Subsequently, a Quantimet image analyzer was used for direct particle size measurement. Electronic magnification of the field of view of the Quantimet permitted the use of lower optical magnification. This produced a better depth of field than was obtained with the Reichert microscope. A sample preparation technique was also developed which placed most of the particles on the same plane. The Quantimet's principle of operation was to determine the number of points on a cathode-ray tube image defining the area and circumference of a particle and then use this information to compute the average diameter. The operation of the Quantimet was semiautomatic but required manual reduction of the data to obtain a particle size distribution. The resolving power of the Quantimet, while improved over that of the metallurgical microscope, wasn't satisfactory for use with the nominal 0.3-μm abrasive.

Data Presentation Methods

Several methods of data presentation were available for particle size distributions. The method favored by the manufacturers of the Coulter Counter and Micromerograph were graphs of cumulative percent of the particles by weight versus the logarithm of particle size (Figure 1). Figure 2 is the plot of the data taken from the previous figure but plotted as a histogram of percent of the particles by weight versus arithmetic particle size. A histogram

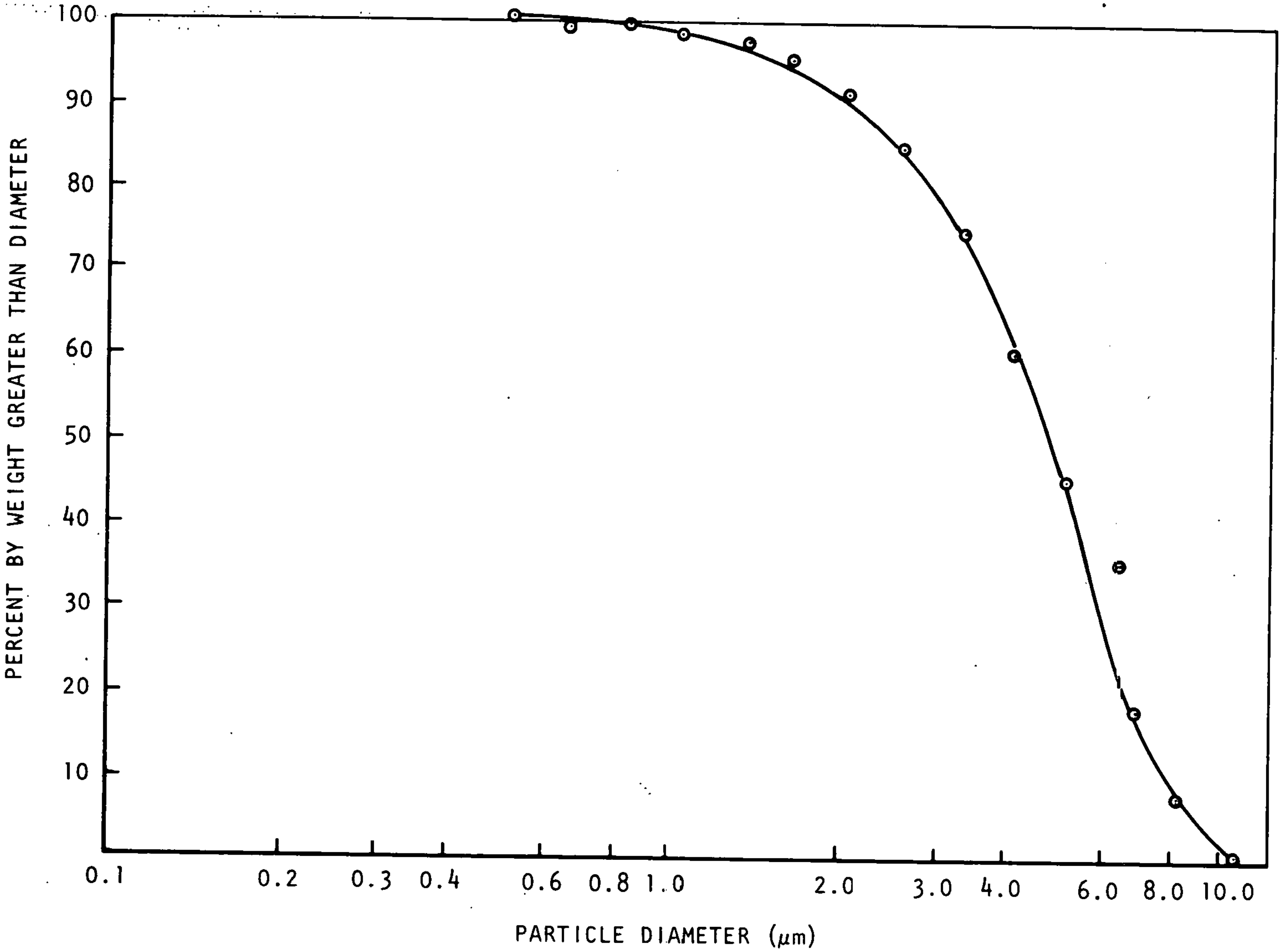

Figure 1. Coulter Counter Data, Percent by Weight Versus Log of Particle Size for 3-μm Alumina

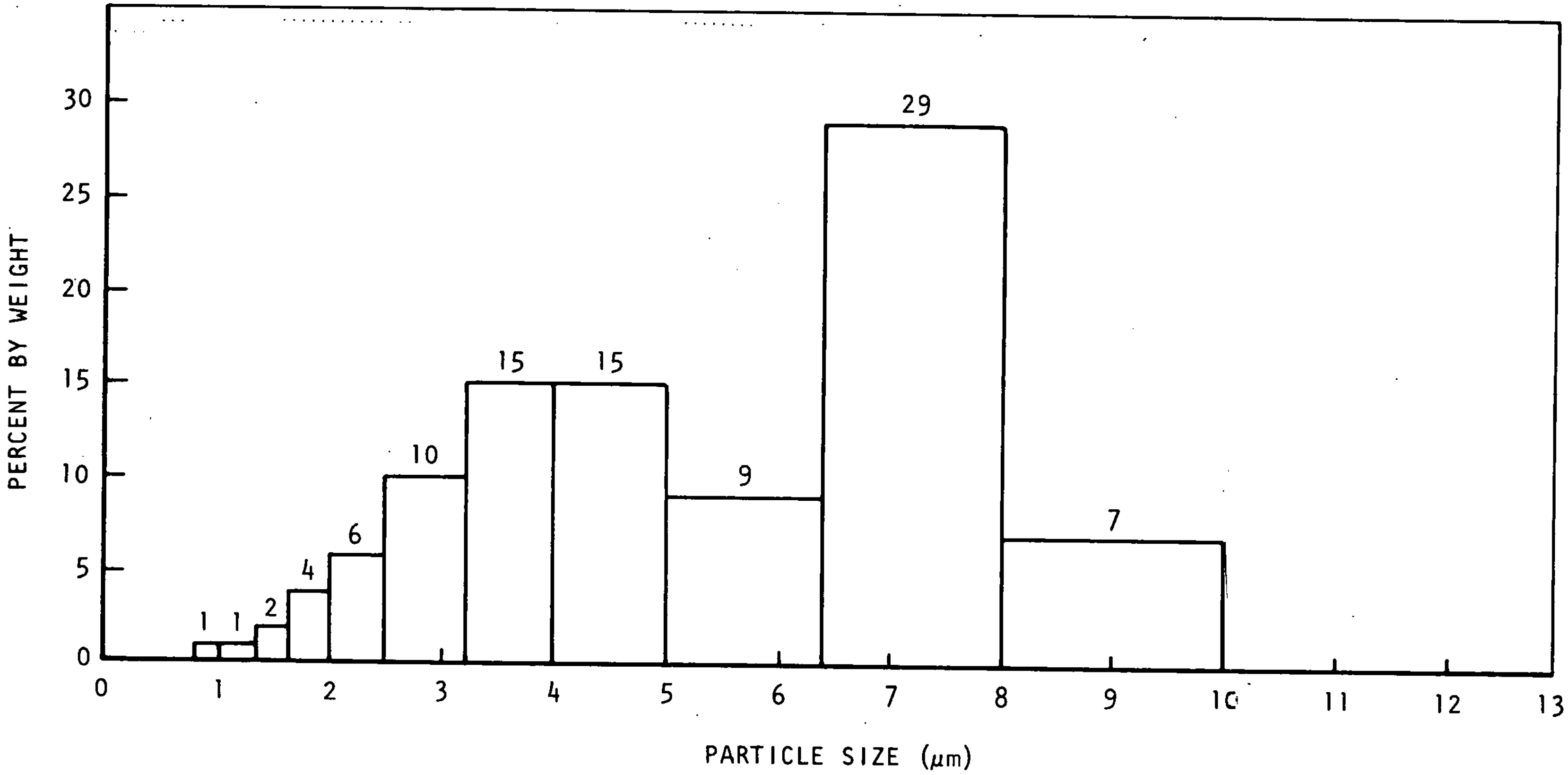

Figure 2. Histogram of Figure 1 Coulter Counter Data, Percent by Weight Versus Particle Size for 3-μm Alumina

with a percent as the ordinate can be somewhat misleading when comparing data if the cell sizes along the abscissa are not equal (as they weren't for this instance). The points along the abscissa could have been equally spaced, however, by establishing the appropriate thresholds on the equipment which performed the particle measurements.

Another presentation method was a histogram of percent of population versus the particle size, which was obtained by plotting data generated by the Quantimet (Figure 3). Since the weight distribution is proportional to the cube of the population distribution, the two distributions yield results quite different in appearance. It was initially undecided whether it was desirable to present the distribution as a percent by weight or percent by population versus particle size, so several sets of data were converted from one form to the other for comparative purposes. Conversion from a weight distribution to population distribution was accomplished by the following.

$$(\text{Percent of Population})_x = \frac{N_x}{\sum_1^N N_x} , \qquad (1)$$

where N_x is the number of particles in the cell (size range) between X_N and X_{N+1}.

Multiplying numerator and denominator by

$\frac{\rho}{W_t}$, where

W_t = total sample weight, and

ρ = particle density, then

$$(\text{Percent of Population})_x = \frac{\frac{\rho N_x}{W_t}}{\sum_1^N \frac{\rho N_x}{W_t}} , \qquad (2)$$

and

$$(\text{Percent of Weight})_x = \frac{W_x}{W_t} = \frac{N_x V_x \rho}{W_t} , \qquad (3)$$

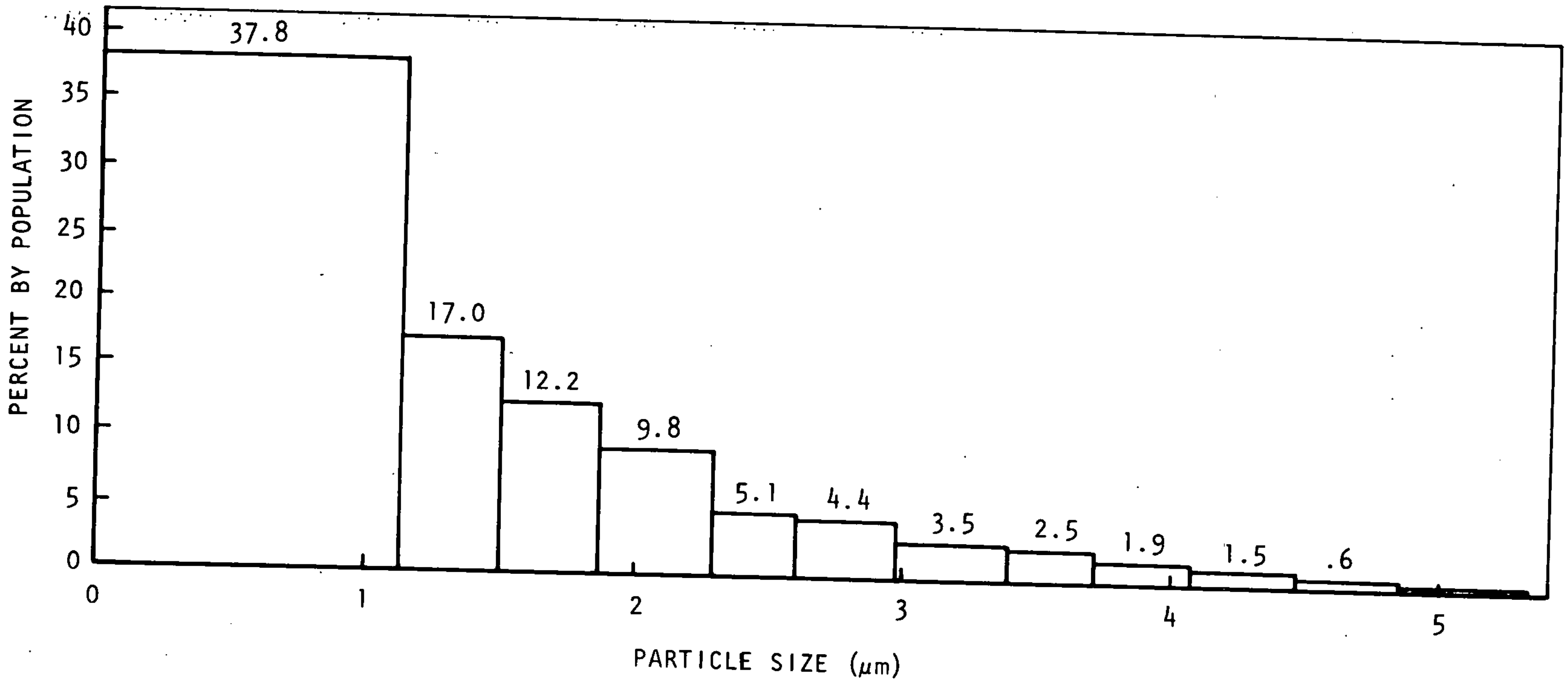

Figure 3. Quantimet Data, Percent of Population Versus Particle Size for 3-μm Alumina

where

W_x = weight of all particles in the cell, and

V_x = volume of a particle in the cell.

Rearranging terms and substituting in Equation 2 as follows

$$\frac{\rho N_x}{W_t} = \frac{(\text{Percent of Weight})_x}{V_x} , \qquad (4)$$

then

$$(\text{Percent of Population})_x = \frac{\dfrac{(\text{Percent of Weight})_x}{V_x}}{\displaystyle\sum_1^N \frac{(\text{Percent of Weight})_x}{V_x}} . \qquad (5)$$

$$V_x = \frac{\pi}{6} D_x^3 , \qquad (6)$$

where $D_x = (D_N + D_{N+1})/2$.

Thus,

$$(\text{Percent of Population})_x = \frac{\dfrac{(\text{Percent of Weight})_x}{D_x^3}}{\displaystyle\sum_1^N \frac{(\text{Percent of Weight})_x}{D_x^3}} . \qquad (7)$$

Conversion from a population distribution was more straightforward:

$$(\text{Percent of Weight})_x = \frac{W_x}{W_t} = \frac{\rho(V_x N_x)}{\rho \displaystyle\sum_1^N (V_x N_x)} = \frac{N_x D_x^3}{\displaystyle\sum_1^N (N_x D_x^3)} . \qquad (8)$$

While the conversion from a population to weight distribution was accurate, the reverse was not true. Due to the unknown lower limit for particle diameter, conversion from weight to population distribution based on the mean diameter of the cell size caused a bias usually in favor of the small diameter particles. (All particles less than the minimum threshold were accumulated as if a single size.) For this reason, comparison of the three particle size measurement techniques was limited to weight distribution.

For application to lapping abrasives, the weight distributions are probably more meaningful than population distributions. With the weight distribution, the presence of a few relatively large particles is apparent while de-emphasis of the very fine particles (less than 10 percent of the nominal size) is probably of little concern since, during lapping, the "fines" probably have little effect on the cutting characteristics of the abrasive.

Distribution in 3-μm Alumina

Two samples of 3-μm alumina were taken from each of two available 11-kg (25 pound) containers of the abrasive as received from the manufacturer. One container was approximately 75 percent used when the sample was removed; the other container was unopened prior to obtaining the sample. Both containers were from the same vendor lot. Each sample taken was approximately 50 cm^3 and was dried for 16 hours with heat (60°C) and vacuum. No significant differences in particle size distribution were found between samples from the two containers. Since one set of samples was from the top of a container and the other set from near the bottom, it was concluded that separation of the particle sizes during transit was unlikely. The comparative data presented herein is from a single sample taken from the partially empty container.

Particle size distributions for the 3-μm alumina as obtained from the Coulter Counter, Micromerograph, and Quantimet are presented as histograms of percent by weight in Figures 2, 4, and 5 respectively. Generalizing, the primary peaks of the distributions occurred between 5 and 8 μm. With each distribution, approximately 50 percent of the particles (by weight) were between 5 and 10 μm, which would classify the abrasive as 8-μm rather than 3-μm. Due to the numerically large quantity of particles less than 1 μm and other reasons subsequently discussed, it is probable that Figures 2 through 5 represent true particle size rather than agglomerated particles.

Distributions of this type are classified as being modified log-normal and appear to be encountered in particle size distributions which result from crystallization and/or crushing. A feature common to histograms obtained from the three independent

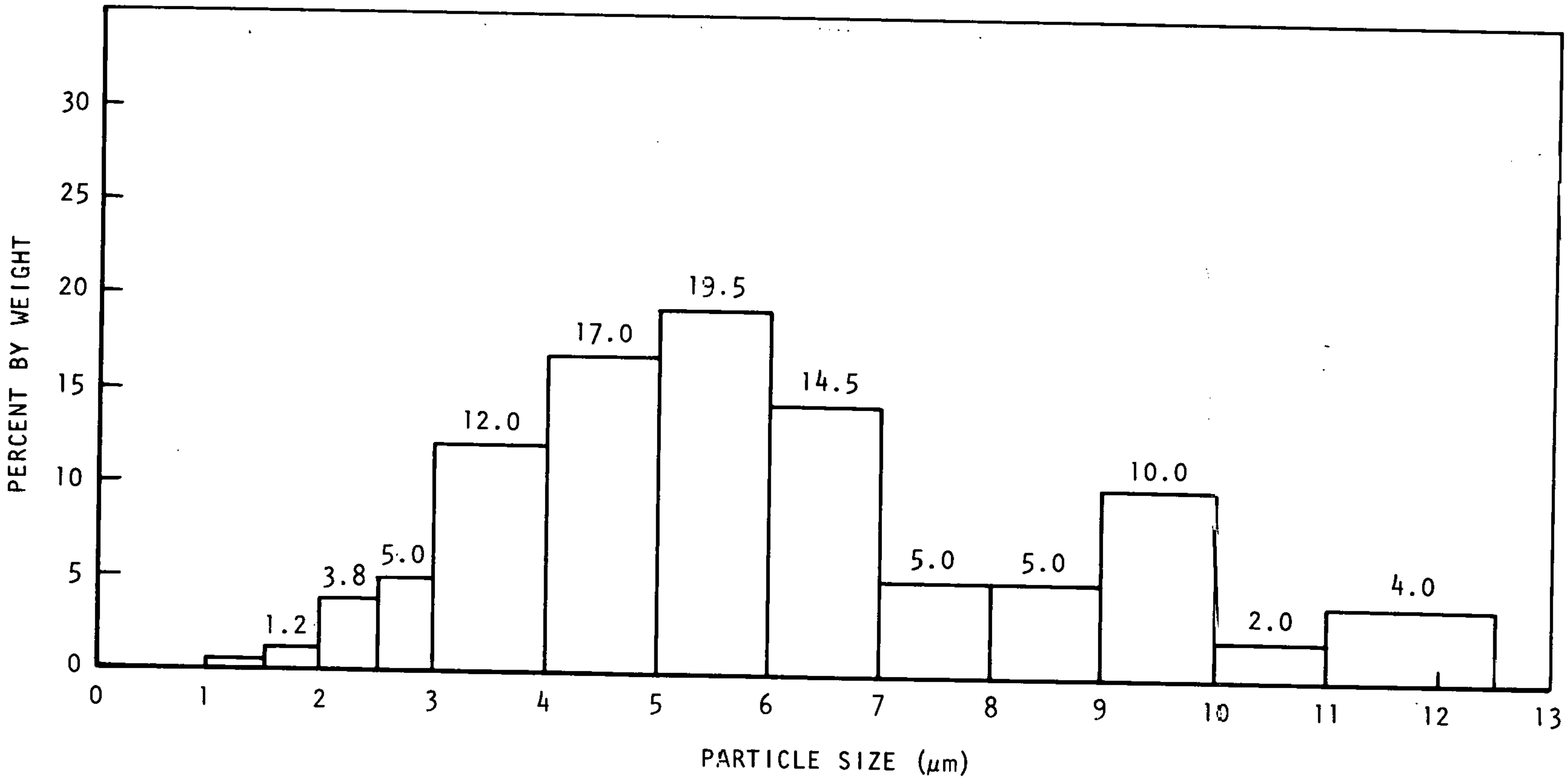

Figure 4. Micromerograph Data, Percent by Weight Versus Particle Size for 3-μm Alumina

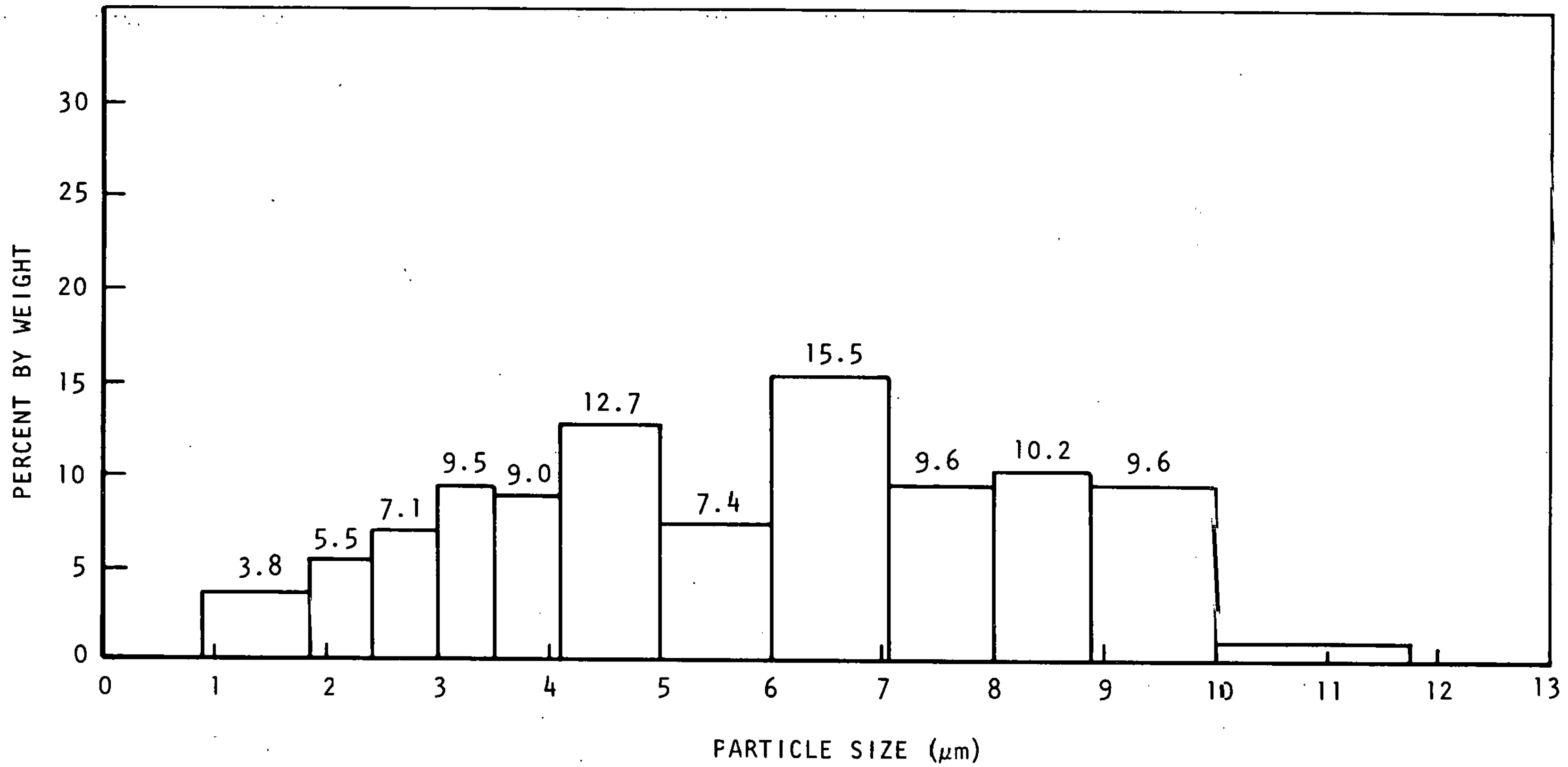

Figure 5. Quantimet Data, Percent by Weight Versus Particle Size for 3-μm Alumina

techniques is a secondary peak occurring about two microns smaller than the primary. This may indicate that two separate abrasive sizes have been mixed to obtain the commercial formulation supplied by the vendor.

Distribution in 0.3-μm Alumina

None of the particle measurement systems were considered adequate to measure particles in the range expected to be encountered with 0.3-μm alumina. Samples were evaluated on the Micromerograph and Coulter Counter, but the results were not considered meaningful. In addition to equipment limitations, the particles were probably agglomerated. Two techniques which have the capability for the measurement of particles in the range below 1 μm are centrifugal sedimentation and electron microscopy. Centrifugal sedimentation equipment was not available, and sample preparation techniques necessary for electron microscopy were not developed. Electron microscopy appears to be the most desirable technique for particle measurement under 1 μm if an accurate method for distributing the alumina in a layer one particle deep can be developed.

Agglomerates in 3-μm Alumina

Optical microscopy was used to determine whether agglomerated particles existed in the 3-μm (#30) alumina. Samples were prepared by making a dispersion of the alumina powder in one of several fluids and then smearing a drop of the dispersion onto a glass microscope slide. The dispersion fluids were distilled water with detergent, John Crane oil (Crane Packing Co., 6400 West Oakton, Morton Grove, Ill., 60053), Sunnen oil (Sunnen Products Corp., 7910 Manchester, St. Louis, Mo.), and microscope immersion oil. Mixing was accomplished by vigorous shaking and ultrasonic agitation. For some specimens, the oils were diluted with compatible solvents such as electronics grade acetone (CH_3COCH_3) or trichloroethane (CH_3CCL_3). Slides of dry abrasive powder were also prepared; however, no acceptable technique was found for dispersing the dry powder in a layer one particle deep. The literature indicated that this problem was not uncommon.

The resulting survey of the slides (oil dispersion) gave many indications of two or more particles agglomerated but it couldn't be determined whether the particles were bonded together or simply lying adjacent to each other. The magnification used for cursory examination of all slides was 562X, while individual areas of interest were examined at higher magnifications. This process sometimes revealed two discrete particles which had initially appeared to be agglomerate.

When using water with detergent as the dispersive fluid, the alumina was more easily dispersed than with either the John Crane or Sunnen oil. In only a few instances were two or more particles found together usually at the edge of what had been a droplet prior to evaporation of the water. It was observed that as the concentration of alumina in the dispersion was reduced, the ratio of suspected agglomerates to discrete particles appeared to decrease. It was concluded the particles were not agglomerated while in a water dispersion but were probably brought together as a result of water surface tension as it evaporated. Since the particle size distributions also indicated the existence of many particles smaller than 1 μm but few particles in excess of 10 μm, it is unlikely that significant agglomeration exists.

Agglomerates in 0.3-μm Alumina

Specimens were again prepared by making a dispersion of the alumina powder in John Crane oil and in distilled water with detergent added as a wetting agent. In both cases, apparent agglomeration was revealed at the 562X magnification; however, the limit of resolving power of the microscope precluded making a judgment. The apparent relative amount of agglomeration seemed to be little affected by the concentration of alumina in the dispersive fluid or the duration of mixing. This was later substantiated by Coulter Counter particle size analysis which showed 25 percent of the particles to be in excess of 1.5 μm in diameter. However, in each case the presence of multiple particles clustered together could possibly be attributed to improper dispersion of the particles during sample preparation. Since, again, the bond strength of the multiple particles couldn't be measured, the true state of agglomeration couldn't be determined.

Recommendations for Particle Size Control

The capability to determine the particle size distribution in 3-μm alumina has been demonstrated. By improving those capabilities through standardization of testing techniques, sample preparation, and data collection, it should be possible to make a reliable comparison between an unknown alumina sample and a specification and/or previously used material.

The major problems in particle size control are the inability of the manufacturer to closely control the size separation process and the manufacturer's limited enthusiasm for improving the quality of a product for a customer that purchases relatively small quantities. A recommended approach (for both abrasive sizes) is to buy a large quantity of the abrasive (perhaps a 5-year supply) from a single lot, depending upon lot uniformity to maintain particle size control. When necessary to purchase another supply of alumina, minor process adjustments may be necessary to accommodate a different particle size distribution, but, if appropriately large quantities are procured, these adjustments would be infrequent.

Recommendations for Abrasive Handling

Three areas of abrasive handling related to the lapping process which required procedures and equipment were abrasive storage, dispensing, and mixing. The approach taken was to determine the optimum quantity of abrasive slurry to be mixed in a single container so that equipment and practices could be structured around the volume of the mixing container. Since it is planned to use comparatively large quantities of abrasive slurry in the future, the possibility of mixing the quantity of abrasive supplied in a vendor 8-liter shipping container was first considered. The pre-mixed slurry could then be drawn from the bulk container when required for individual lapping processes. Using the typical 4:1 volumetric ratio of lapping fluid to abrasive would require a 40-liter (10 gallon) mixing container.

While the bulk mixing approach would require less frequent measuring of the components and would provide a convenient ready-mixed supply of abrasive slurry, the problems associated with this option were considered prohibitive. To prevent the abrasive from settling, the slurry would require constant agitation which would be a problem during weekend power outages necessary for plant maintenance. Remixing large quantities of slurry which had been allowed to settle could be a difficult operation. Since it would be necessary to maintain at least three mixing stations to accommodate the currently used abrasive/carrier combinations, a relatively large floor space would be required. In addition, OSHA requirements for the containment of oils in case of spillage could increase the floor space above that necessary for the mixing equipment alone. Finally, each mixing container would hold about 75 kg, the inertia of which could make starting and stopping difficult without a large motor and braking system.

In view of the problems likely to be encountered in mixing large quantities of slurry, it was proposed to structure the abrasive handling system around the quantity of slurry necessary to fill a lapping machine. This volume is about 1.4 liters, which suggests the use of commercially available half-gallon containers. After reviewing some of the available types of containers, it was recommended that a half-gallon rectangular polyethylene milk bottle with a 38-mm screw lid be used. This container is available locally at $0.10 each, which compares favorably with the presently used round polyethylene bottle costing $1.70 each. Disposing of a container after a single use would be a cost savings over cleaning a container and would prevent the possibility of cross contamination.

It is also recommended that mixing be accomplished by making a tumbler to rotate the bottle about its longitudinal axis. This mixing technique has previously proven satisfactory when using rectangular containers, and equipment could be designed for mixing several containers simultaneously.

One problem associated with the use of small containers for mixing abrasive slurry has been considerable spillage of the abrasive while pouring the correct volume of abrasive into the bottle through a funnel. The use of a funnel specifically designed for powders has reduced this problem. The powder funnel differs from a regular funnel by having sides inclined at a steeper angle and a larger diameter spout with vertical sides.

The recommended procedure for storage of abrasive would be to fill enough of the half-gallon containers (about 28) to completely use an 8-liter shipping container of abrasive. The correct carrier fluid could then be added to the bottle just prior to mixing. Adequate storage space exists to use this concept for abrasive handling. Since dispensing the abrasive into the bottles would be a batch rather than single time operation, it is possible to have a single operator assigned to maintain the abrasive as a part of his overall duties. This would result in less spillage and cleaner operations. Finally, contamination storage problems of bulk abrasives should be greatly reduced, since it will be necessary to store only unopened shipping containers or capped mixing containers.

ACCOMPLISHMENTS

Alumina abrasive presently identified as nominally 3-μm by the vendor should be appropriately identified as 5- to 10-μm; satisfactory techniques for determining the particle size of 0.3-μm abrasive are not presently available.

The 3-μm alumina contains few agglomerated particles of any consequence. The evidence for agglomeration of 0.3-μm alumina was inconclusive.

The following steps are recommended to improve lapping process control.

1. Micromerograph and Coulter Counter particle size determination methods should be used to determine the distribution for each new lot of 3-μm abrasive used.

2. Particle size control for each of the two nominal sizes should be improved by procuring sufficiently large quantities from a single lot.

3. Mixing of abrasive should be performed in the quantities required to fill an individual lapping machine. Half-gallon disposable containers should be used to prevent contamination and as a cost savings.

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