

Micronization
Particle Analysis for Semicon Manufacturers
by Ron Abramshe

Fine particle grading, or micronization, is an important manufacturing process in the semiconductor industry. For example, it is used to produce: diamonds for slicing silicon wafers; submicron diamonds for lapping and polishing GMR read-write heads in computer hard drives; and micron and submicron diamonds for polishing laser and LED caps. Though, while micronization is used to assist in the manufacture of very modern technologies, it must be noted that the process is based on the exact sizing techniques established in the hydraulic principles researched by George Gabriel Stokes, Osbourne Reynolds, John Thomas Fanning, and Ludwig Prandtl.

Through the work of these early pioneers, fine particle measurement has achieved a certain level of standardization. In analyzing the standard statistical techniques for micron and submicron measurement, it is possible to gain a better understanding of the science behind exact sizing techniques. As such, the following will examine the processes of elutriation and centrifugation, both of which serve as a means to an end that is micronization.

Elutriation

Elutriation is the process of grading micron and submicron sizes (usually less than 60 microns) into equal distributions that are normally distributed. Normally distributed means that there is a mean, mode, and median to the number of particles in the population of material being graded. The population of particles follows the Gaussian form of $f(x) = \frac{1}{\sigma \sqrt{2\pi}} \exp[-(X-\mu)^2/2\sigma^2]$. And since the population follows a normal distribution, the control (or standardization) of micron material lends itself very well to statistical process control, as discussed later.

Decantation

Decantation is a simple methodology based on Stokes's principles that: Particles falling under the influence of gravity will be acted upon by drag; and Constant velocity will be reached when these two are in equilibrium [μ ft./sec. = $\frac{gmp(p-p)}{ppA C}$ (where: g = acceleration due to gravity; m_p = mass of the particle; p_p = density of the particle; A_p = apparent density; and C = drag coefficient)].

So by varying the material (density) or the mass of the particle, it is possible to calculate the time that a certain fraction of the distribution of particles will fall through a medium and extract that distribution by decantation. Micronizers are still employing this method of decantation, although mostly in the Far East. Typically this methodology is automated through the use of mixers and computerized drawtubes. These machines are known as auto graders.

For proper elutriation, it is important to provide the following:

- Complete Dispersion of Material in Fluid
- Constant Fluid Velocities
- Short Treatment Times for Given Weight of Material to Be Separated
- Sharp Separation as Measured by Minimum Overlap Between Grades
- Production of Standardized Grades

· Minimum Attrition of Material Being Graded

Dispersion is of the utmost importance in micron and especially submicron separation. If not properly dispersed, fine particles will adhere to other particles and form agglomerates; or they will adhere to larger particles, thus providing a false distribution or loss of yield. In this regard, dispersants are invaluable.

The question, however, is how much dispersant to use. If too much chemical dispersant is added to the system, it can have a reverse effect, creating a flocculated colloid state of fine particles. The use of zeta potential measurements, which determine electrical activity at the liquid/solid interface between the stern layer and shear layer, can be helpful in establishing minimum-use standards for chemicals in the elutriation process.

Some of the popular dispersing agents are saponin, sodium hydroxide, agar-agar, ammonia, tannic acid, etc. The choice of dispersant is dependant on zeta potential and the amount of ionic surface activity that will remain on the particle surface after final processing.

Constant fluid velocities are necessary to avoid fluctuations in the desired cut of material and to minimize the overlap between sizes. Even a very small increase in velocity will cause an errant coarse particle to become entrained in the velocity jet and take on unwanted characteristics in the overall distribution. The use of constant pressure systems with multiple vessels minimizes the effect of varying fluid velocities. Typically, capillary flowmeters are used to control these systems. The use of nozzles or spreaders at the base of the cutting vessels also helps to mitigate parabolic velocity fronts that can occur in the center of the cutting vessel. This is particularly true in single-point elutriation systems.

Separation is a function of elutriation time, end-point constancy of fluid flow, completeness of dispersion, and control of the velocity front in the separator.

Elutriation rates should be constant with sufficient time to yield sharp distributions. Frequent samples should be taken to ensure that minor adjustments are made to keep the system in balance. Because of the stochastic nature of elutriation, the control and subsequent quality of the product lends itself to statistical process controls.

Attrition is generally nonexistent in water-based systems, even for very friable materials, such as proteins and some clays. In air elutriation systems - because of the mechanical action of high-speed rotors - particle impingement on rotor blades could cause attrition to very friable materials.

The scope of this article does not permit detail on the methods of devising "X" and "R" bar charts, other than to say most production control books on this subject will provide this information. Statistical process control techniques are sufficient to determine the quality of the separation using a limited number of measurements. The null hypothesis is generally used and followed for control of the particle size distribution. The methodology reads as follows:

- If one point is out of UCL / LCL, it is noted and nothing is done.

- If one data point per shift is out for four consecutive shifts (all charts), a process change is made.
- If one data point per shift is out for four consecutive shifts (one chart), a population change calculation is made.
 - o Type 1 Error: Deciding from sampled data that an undesired change has occurred when it has not.
 - o Type 2 Error: Deciding from sampled data that no change has occurred when one has.
- After process changes are made, the measuring system, as outlined, is resumed.

Types of Elutriators

Schonene Apparatus: This system is constructed using varied funneled sections followed by long straight sections. A number of funneled sections are followed by a straight section, forming chambers that can be sealed off by stopcocks. The current velocities are measured by piezometric analysis from the overflow section of each chamber. After the appropriate velocity calculations are made for a given feed, a graded size can be collected while the system is in operation.

Mt. Morgan Mines Multi-Tube: Other than single-point elutriation, this is probably the most widely used type of elutriation vessel system. It permits optimal charging, particle spacing, and as close to continuous flow processing as possible. The Mt. Morgan Mine and variations of this type of system have been used successfully for generations. The heart of the system is based on the proper use dispersants and the proper dilution vessel, which can produce very sharp distributions (as evidenced by small standard distributions and small spans) and process large amounts of material. In large-scale production this type of system is preferable because of the near-continuous flow shop conditions.

Andrews Kinetic Elutriator: This system is based on essentially the same funnel and chamber design as the Schonene apparatus, but it has an injector nozzle located in its lowest chamber. The purpose of this nozzle is to provide energy to the system to aid in de-agglomeration of particles in the hindered settling section of the elutriator.

Today, micronization is performed primarily through single-point and multi-tube systems; most likely because of their simplicity in design, operation, and reproducibility. There are, of course, proprietary design modifications available that take into account good engineering and experience. However, it is doubtful that any two micronizers will agree that one system is better than the other.

Centrifugation

As noted earlier in the base equation of Stokes, the falling rate of a particle is influenced by its mass and density relative to gravitational acceleration and the density of the medium the particle is in. Extremely fine separations in the order of 0.05 microns are obtainable by traditional elutriation systems, but they would require a significant amount of time - as much as several years, possibly - to accomplish. Further, anything below 0.05 microns cannot be elutriated. Thus, centrifugation is often used to perform submicron separation.

Centrifugation is accomplished by imparting high gravitational forces that run perpendicular to

the general direction of sedimentation through rotational speeds. Specifically, centrifugation is the application of mechanical force to overcome random Brownian movement. The base calculation is given by the form $F_c = 0.0000142n^2Db$. The magnitude of the force (F_c) exerted is given in terms of multiples of the standard force of gravity (where: n = rotational speed; and D = bowl or container diameter). By substituting relatively high rotation speed for moderately sized bowls or containers, it is possible to generate substantial G force.

While this article will not get into the specifics of centrifuge design, it is important to note that the high force involved in centrifugal systems requires thorough attention to materials of design and operation. Critical speeds, as well as materials of construction, should be carefully engineered to match the requirements of the materials to be processed.

For centrifuge selection, several methods described by Moyers (Chem. Eng., vol. 73, 182, 1966) can be invaluable. They are as follows:

Particle Analyzers: There are a number of particle size analyzers that are available on the market. Some of the more prominent analyzers include: electro-zone; disk centrifuge and laser; laser and visible-light. While all of these analyzers work well on spherical products, most materials are not truly spherical. As such, it is important to understand the optics of the analyzer, its capability to measure nonspherical particles, and its best attributes.

One methodology that Moyers suggests is to establish a standard of the material to be measured. This is accomplished by image analysis of micron and submicron materials. For micron sizes of greater than five microns, standard light microscopy with image analysis capability can be used to establish the true attributes of the material to be analyzed. For submicron sizes, scanning electron microscopy with image analysis capability should be used.

Aspect ratios, circularity index, and roundness are but a few of the parameters to study. The samples (standards) must then be analyzed by the most appropriate particle size analyzers. Once a database is established using standard statistical techniques (described earlier), gauge studies can be used to check reproducibility and repeatability.

Gauge Analysis: In verifying the error in measurement systems, both repeatability and reproducibility must be examined. The 10 percent rule does not discriminate the tolerance level. The measurement instrument has no feasibility to measure the process variation if the percent tolerance by total R&R does not exceed 10 percent. If it does not exceed 10 percent, the measurement is excellent. Between 10 percent and 30 percent, the process is in control with acceptable results. Over 30 percent means errors may lie in the system.

About the Author

Ron Abramshe is the plant and product manager at Warren/Amplex Superabrasives, a Saint-Gobain company based in Olyphant, Pa. He earned his bachelor's degree in Industrial Engineering at the University of Dayton and his master's of Science Engineering at Polytechnic University of New York. Ron also presents information seminars and is currently scheduled to speak at the MicroTEC 2004 Conference and Expo, Oct. 13-15, at the Gaylord Palms Hotel in Orlando, Fla. Mr. Abramshe can be reached at ron.a.abramshe@saint-gobain.com or 570 383-

8973; www.warrendiamond.com

EDITOR'S NOTE: For more information on micron and submicron particle grading, see Perry's Chemical Engineering Handbook, the Handbook of Mineral Dressing by Arthur F. Taggart, and Fluid Mixing Technology by James Y. Oldshue.