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Nanonization: A New Word for a New Process

➤ A recent development in size reduction technology can improve the micronization process by a factor of 10.

by Stephen Miranda, Sales Director, NETZSCH Premier Technologies, LLC; and Thomas Schneider, Marketing Manager, NETZSCH-CONDUX Mahltechnik

Micronization is a word long associated with the miniaturization of particles by jet milling to a range of 2-200 microns (μm). For most dry grinding processes, 2 μm is the practical limit of the technology. However, a recent development in the technology of size reduction through dry grinding can improve this process by a factor of 10.

The new process uses superheated steam as the grinding gas in a fluidized bed jet mill. A steam jet mill can produce dry powders with median particle sizes in the range of 130 nanometers (nm), with 100% of the distribution finer than 400 nm. This patent-pending technology is so novel that it could inspire the coining of a new process name: nanonization.

Searching for Solutions

The drive to improve product performance and develop new products is huge, but it is often the littlest things that make the most difference. The quest for innovation has driven many ceramic industry researchers to look to nanoparticles (materials ground to 0.2 μm and finer in size) to enhance product performance or unlock new applications for ceramic materials.

Production of nanoparticles was achieved years ago, with myriad benefits. Nanoparticles are used to improve reactivity, provide increased surface area, and reduce material consumption, particularly with rare and high-value ingredients.

Media milling technology—the use of grinding beads (media) to break down larger particles into smaller ones—currently plays a significant role in many areas of ceramic processing. Media milling processes typically involve dispersing the materials into a suspension; using media mills for grinding or dispersing; and then drying the suspension to recover the nanoparticles.

Each successive step in the process is energy intensive and can result in product contamination and wear on the equipment. Since dry processing is limited in its capacity to produce particles in the nanometer size range, wet processing is used. This keeps many materials from being produced in efficient commercial-scale manufacturing and limits innovation.

The demand for finer dry powder products in the submicron or nanometer scale has led to the development of a new milling technology that allows real comminution in this range. “Nanonization” of solids in a dry process provides the added benefit of being energy efficient and economical. Unlike conventional dry jet mills, the new system uses superheated steam as the grinding gas.

Using superheated steam as the grinding gas in simple spiral or loop jet mills without an integrated air classifier has been a common practice for decades. Until now, it was not possible to produce materials with a well-defined upper particle size limitation. The new system includes an integrated air classifier for the separation of the exact par-

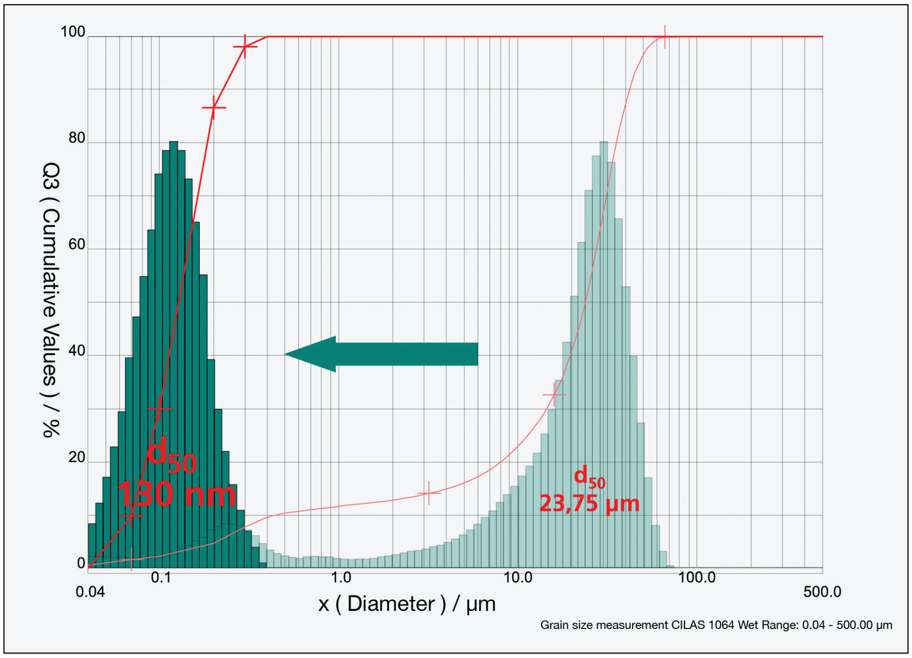

Figure 1. The new system has produced d_{50} of 130-140 nm.

Table 1. A variety of products has been tested in both R&D labs and commercial installations.

Product	Fineness	
	d_{50}	d_{99}
Alumina	0.13	0.35
Barium	0.13	0.34
Iron oxide	0.07	0.37
Glass frits	0.57	1.89
Amorphous graphite	0.16	0.58
Limestone	0.54	1.87
Silicon carbide	0.24	1.04
Wollastonite	0.3	2.6
Zirconium oxide	0.44	2.59

ticle sizes. These two factors make steam fluidized bed jet milling commercially viable.

Benefits

Steam can be provided to a jet mill at very high pressures compared to air. Higher grinding pressures yield higher jet speeds. At a pressure of 40 bar absolute, the jet speed is nearly 1,200 m/sec. (Higher pressures are both possible and beneficial.) Compared to air's near maximum of 600 m/sec, the kinetic energy at the nozzles is approximately four times higher. This increased grinding energy results in much finer size reduction in the milling process, sending finer particles to the classifier.

As a medium for dynamic classification of particles, steam allows a finer cut size than air. The properties of steam, which has a lower dynamic viscosity and lower density than air, as well as a higher speed of sound than air, makes possible a higher flow velocity within a classifier wheel. Thus, the acceleration forces acting on the material being separated are higher, and the possible cut sizes are finer. Since finer particles are already available due to higher jet energy, this is a decisive step in the production of nanometer-sized particles by means of dry grinding.

Higher jet energy can also result in significant increases in capacity and improved energy efficiency. Jet speeds of up to 1,200 m/s, along with the increase of the kinetic impact energy, can lead to significant increases in mill throughput. For example, throughput for an aluminum oxide powder with a fineness of 2.3 µm (d_{99}) is more than tripled. Similar results with other materials have been realized. For materials of the same particle size and with equivalent gas flow rates, throughput rates are increased by two to three times, depending on the friability of the materials.

A variety of products has been tested in both R&D labs and commercial installations (see Table 1). For example, the production of finely ground amorphous graphite with a d_{99} of less than 4 µm by conventional dry milling has, in the past, been problematic. The new system has produced particle sizes significantly below $d_{99} \sim 1.0$ µm.

Another interesting example is in the range of oxide ceramics. Aluminum oxide with conventional dry milling can result in a fineness of about 2.3 µm (d_{99}). The new system has produced d_{50} of 130-140 nm with a d_{99} of 0.34 to 0.35 µm—results that could previously only be achieved through wet grinding (see Figure 1).

These results have led researchers and engineers to rethink applications and materials. With new materials come new markets and expanding opportunities.

The Secret of Success

Steam jet milling provides the ability to grind solids to higher fineness because of higher kinetic energy. However, the precise control of the milling process itself is only possible when a grinding mill has an internal classifier. The classifier controls the particle size by preventing the material from leaving the grinding system until the particle size reaches the correct fineness.

The performance of a conventional classifier with air is limited to a d_{97} of approximately 2 µm. This, of course, varies with the material properties (e.g., specific gravity). Steam also provides the solution to controlling the classification of superfine particles generated with this process.

Two forces act on particles in a classifier field: mass force and drag force. Mass force tends to move particles to the coarse fraction, while drag force tends to move particles to the fines fraction. As previously mentioned, steam has a much lower dynamic viscosity and density than air, as well as a higher speed of sound than air. Steam gas properties of influence both mass force and drag force and allow for the production of finer particle sizes.

The higher speed of sound in steam results in a significantly higher peripheral velocity of the gas flow within the classifier. This increases the acceleration of the particles in the classifier field. For example, if the peripheral velocity increases from 200 to 300 m/s, the equivalent effect of the mass force on the particle is a relationship of the square of the difference. Therefore, in this example, the mass force on the particles is increased by a factor of 2.25, moving the cut size to a finer range.

A classifier wheel.

The reduced dynamic viscosity also reduces the drag force on the particle. Drag force tends to move the particle to the fine fraction. Reducing the drag force lessens the tendency to drag coarser particles to the fine fraction, also moving the cut size to the finer range. Combined, these two factors reduce the median particle size of the product by a factor of 0.3 times finer or more. This means if an air jet mill is capable of producing particles at 1.0 µm, now it is possible to make that same product 0.3 µm or finer.

In the Future

The needs of ceramic companies and suppliers selling into the ceramic industry that are developing dry materials in the nanometer size range can be met with steam jet milling technology. Additional developments in grinding are always possible and ongoing, but steam jet milling is currently the pinnacle in dry grinding technology. ⊕

For more information, contact NETZSCH Premier Technologies, LLC at 125 Pickering Way, Exton, PA 19431; call (484) 879-2020; fax (610) 280-1299; or email info.npt@netzsch.com.

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NETZSCH Premier Technologies, LLC.
125 Pickering Way
Exton, PA 19341
USA
Tel.: +1 4848792020
Fax: +1 6102801299
info.npt@netzsch.com

NETZSCH-Feinmahltechnik GmbH
SedanstraBe 70
95100 Selb
Germany
Tel.: +49 9287 797-0
Fax: +49 9287 797 149
info.nft@netzsch.com

NETZSCH-CONDUX Mahltechnik GmbH
Rodenbacher Chaussee 1
63457 Hanau
Germany
Tel: +496181 506-01
Fax: +49 6181 571 270
info.ncx@netzsch.com

www.netzsch.com