Screenless Agitator Bead Mill for the Use of Smallest Grinding Media

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Abstract

The business unit Grinding and Dispersing of the family-owned enterprise NETZSCH comprises of consolidated companies NETZSCH-Feinmahltechnik GmbH for liquid and pasty products and NETZSCH-CONDUX Mahltechnik GmbH for dry products. The business unit offers a comprehensive product range for all tasks arising in the procedural steps dispersing, de-aerating, wet and dry grinding as well as classifying.

The company NETZSCH-Feinmahltechnik GmbH defines itself as mechanical engineering company aiming at supplying special machines or complete systems to customers from the most different fields of applications. The machine equipment on offer enables the development of products on a laboratory scale just as well as the scale up to production size machines. The machines excel by their long lifetime and hereby guarantee a high reliability.

For comminution processes down to the nanometer size range or for the dispersion of nano-sized particles the use of finer and finer grinding media is necessary. Furthermore for the avoidance of changes in the crystal structure of the product smooth conditions for “Mild Dispersion” processes are very important.

With the new agitator bead mill developed by the NETZSCH-Feinmahltechnik GmbH a very good separation of grinding media with diameters down to 50 µm is possible even for low stirrer tip speeds. From experience we know that in particular nanotechnology applications often call for metal-free grinding or dispersion. For these applications we have developed a special grinding media separator system called ODC (Open Dynamic Classifier).

The differences between real comminution, desagglomeration and disaggregation processes are discussed by the use of real experimental results. Furthermore the advantages of the innovative new screenless ODC-System will be explained.

ZETA® RS, the mill for the use of very small grinding media for real comminution processes with high energy densities and “Mild Dispersion” processes

This mill is characterised by a very easy handling especially for the use of smallest grinding media down to 50 µm, a highly developed grinding media separation system and a continuously enhanced mechanical sealing system.

The media separator as well as the mechanical seal are crucial components in agitator bead mills.

When using grinding media of a size smaller than 200 µm in conventional agitator bead mills there is always the danger of single grinding media entering and seizing in the mechanical seal and doing great damage to the face seal by causing ruptures on the seal rings. Having fundamentally changed the seal's construction even smaller grinding media are successfully prevented from entering and seizing in the mechanical seal.

Furthermore, when using grinding media of a size smaller than 200 µm and with an increasing viscosity of the product suspension there is the danger that the media are transported to the separator screen by the flow forces where they are compressed. Therefore standard agitator bead mills made by NETZSCH-Feinmahltechnik GmbH are equipped with a grinding media separator, which ensures separation of grinding media from the suspension by centrifugal forces. With the advanced mill these centrifugal forces are even increased by a rotating separator screen with agitator shaft (s. Figure 1). This design guarantees safe grinding media separation, in particular, if the viscosity rises during dispersion processes with very fine grinding media and low stirrer tip speeds due to particle-particle interactions in the suspension.

From experience we know that in particular nanotechnology applications often call for metal-free grinding or dispersion. For these applications we have developed a special grinding media separator system called ODC (Open Dynamic Classifier). This system is first and foremost suitable for low-viscosity suspensions and brings about the following advantages listed below:

- Variable grinding media sizes can be used without having to change the slotted pipe (screen).
- Screen plugging is impossible.
- No contamination caused by metal wear debris.
- Significantly low pressure increase in the mill: Therefore higher throughput rates are possible.
- The decrease of the grinding media size by wear during long operation is no problem.

- Grinding media other than spherical shape (flint, coarse fraction of the material to be ground (autogenous grinding)) can be used.
- Impurities from the product (hair, fibres, …) as well as low shares of coarse material are no problem. They are flushed out of the machine together with the product suspension and are separated on the redundant external screen.
- The ODC-System can be completely disassembled and is easy to clean.

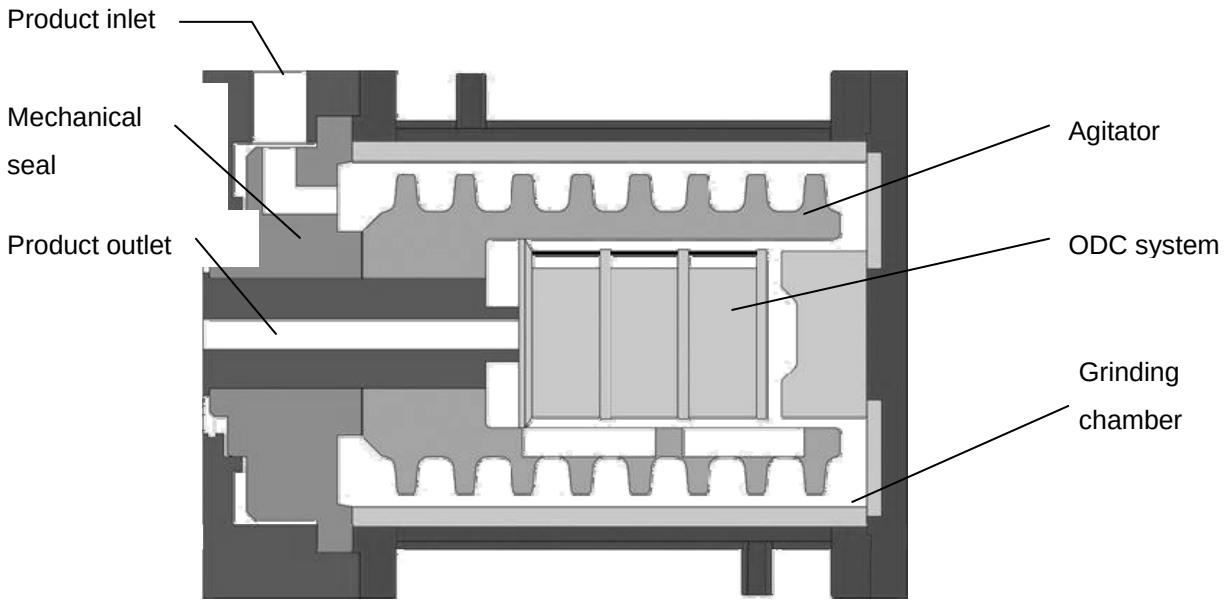

Figure 1: Agitator bead mill, system *ZETA®RS*

For nano applications small product batches with frequently changing products are being processed. This necessitates a frequent change of the grinding media bulk. Especially when working with very small grinding media the emptying and the filling of the grinding chamber is very time consuming. Therefore the agitator bead mill system *ZETA®RS* was designed similar to a laboratory mill, this means, the grinding chamber can be swivelled into different positions for emptying, filling or for operation (s. Figure 2).

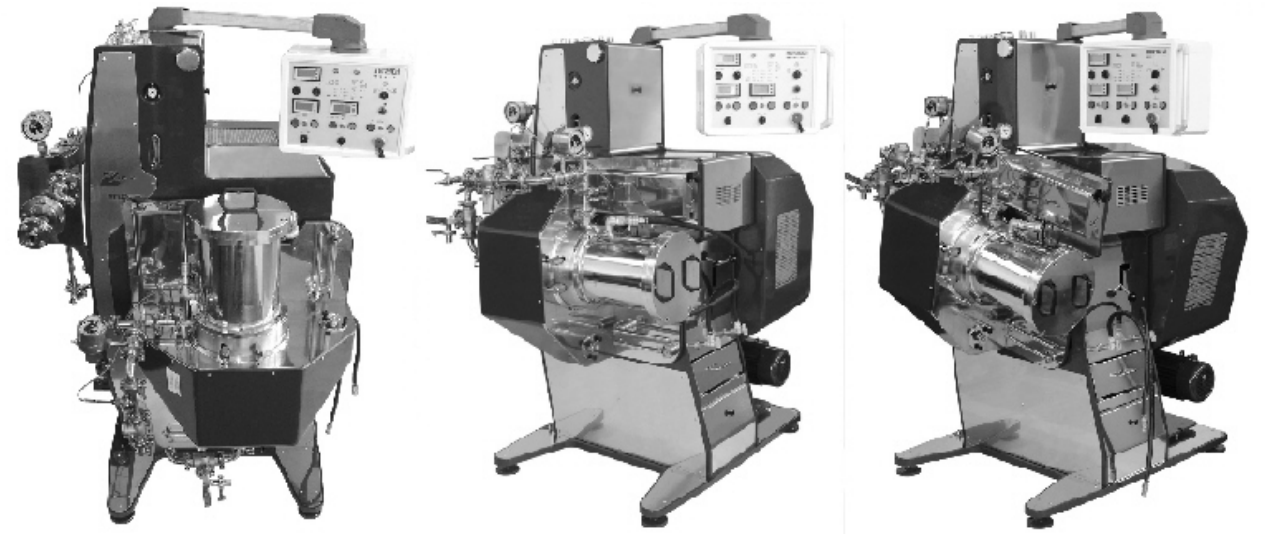

Figure 2: *ZETA® RS 10* in positions for filling, operation and discharge

The *ZETA® RS* agitator bead mill is available in different grinding chamber volumes ranging from 2 to 25 litres and is easy and safe to handle.

Tab. 1: Sizes and technical data of the agitator bead mill type *ZETA® RS*

Type	***ZETA® RS 2***	***ZETA® RS 4***	***ZETA® RS 10***	***ZETA® RS 25***
Power of the motor [kW]	7.5	15	25	45
Agitator speed [min^{-1}]	500 - 2500	300 - 2000	350 - 2000	250 - 1500
Grinding chamber volume [l]	2	4	10	25
Grinding media size [µm]	50 - 300			

Real comminution of titanium dioxide (TiO_2)

For the production of functional coatings titanium dioxide with a median value of the particle size distribution $x_{50,3}$ of about 200 nm and a particle size $x_{99,3}$ of about 375 nm was to be ground as fine as possible. It was a water based suspension with a solids content of 48.5 ma.%, which was stabilized by an appropriate additive. Two grinding tests were carried out in a *ZETA® RS* 4 agitator bead mill (4 litre grinding chamber volume). During test A yttrium-stabilized zirconium oxide grinding media with a diameter of 0.2 to 0.3 mm were used. The grinding media were separated by a rotating slotted pipe in the mill. Test B was made with yttrium-stabilized zirconium oxide grinding media of close fraction with a diameter of 0.1 mm. To avoid a pressure increase

on the suspension inlet of the mill the ODC separating system was used. All further operating parameters were constant. The test results are shown in Figure 3**.** The particle sizes were measured by means of dynamic light scattering with a HORIBA LB 550. For this purpose the defined samples were diluted with deionised water in a ratio of 1:50 and analyzed without any additional ultrasonic dispersion.

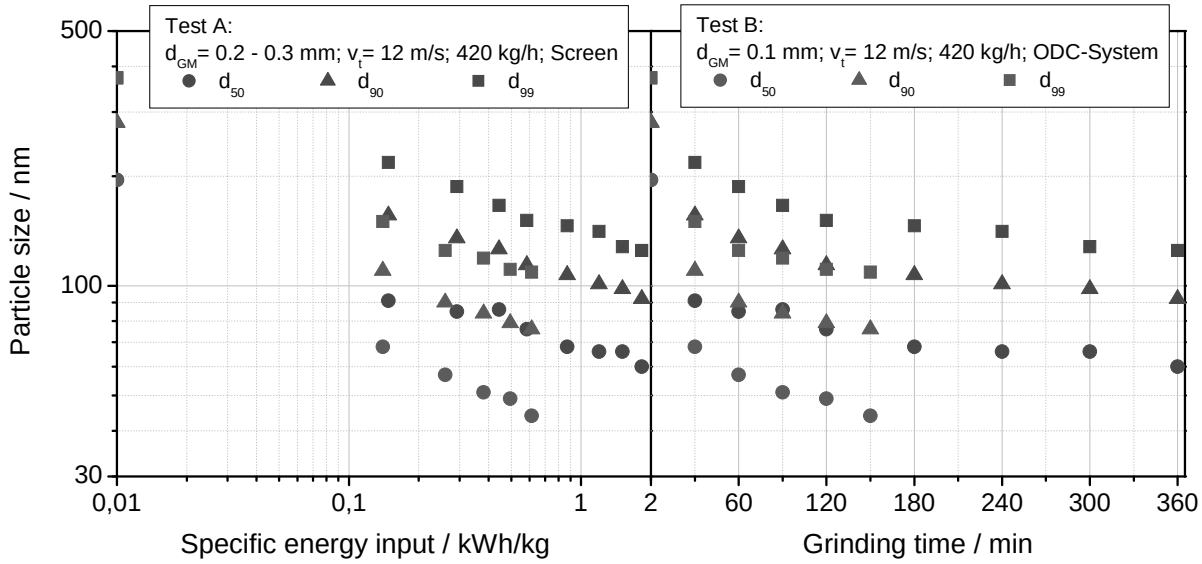

Figure 3: Real comminution of titanium dioxide with grinding media of different size

During the comminution with bigger grinding media (test A) particle sizes $x_{50,3}$ of 60 nm, $x_{90,3}$ of 92 nm and $x_{99,3}$ of 125 nm were obtained after a grinding time of 6 hours and a specific energy input of 1.83 kWh/kg. By using smaller grinding media (test B) a significantly better comminution result could be obtained with one third of the specific energy input (0.61 kWh/hg) and after a grinding time of only 2.5 hours. The obtained results were $x_{50,3}$ 45 nm, $x_{90,3}$ 76 nm and $x_{99,3}$ 110 nm.

Desagglomeration of barium titanate ($BaTiO_3$)

Barium titanate is a material popular with the micro-electronic industry. One possible application is the production of multi-layer ceramic capacitors (MLCC). For this application barium titanate of an always higher fineness is required. The finer the material the more homogeneous layers and thus higher capacities can be realised at a constant height of the element. Moreover, it is the target to further decrease the sintering temperature required for such elements.

Barium titanate was dispersed in a *ZETA*® *RS* 4 with SiC liner and a Si_3N_4 agitator shaft (see

Figure 4). The particle size distribution was measured with a HORIBA LA 950. The analyses were made without adding dispersant and without any additional ultra-sonic dispersion. The zero sample showed a bimodal particle size distribution. Particles of a size of up to 200 µm could be detected.

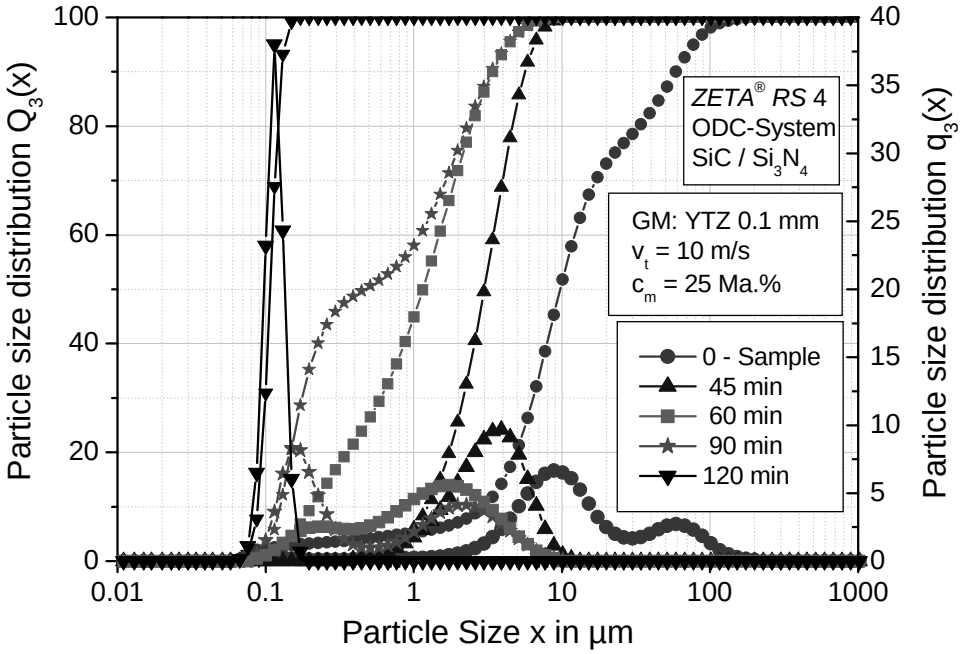

Figure 4: Desagglomeration of barium titanate with the ZETA® RS 4

For being able to disperse this material system in a standard mill it is necessary to pre-disperse it with grinding media of 0.3 mm in diameter, in order to avoid plugging of the separating screen on the product outlet.

Thanks to the ODC system we could do without the pre-dispersion step in this case. We used yttrium-stabilized zirconium oxide grinding media of the company Tosoh with a diameter of 0.1 mm for the comminution of the barium titanate. The solid mass concentration of the suspension was 25 ma.%. The barium titanate was mixed into the suspension via the mill. For that purpose the mill was started with a circumferential speed of the agitator of 15 m/s. The dry barium titante powder was fed into the batch tank for 15 minutes and thus slowly a higher concentration was created. After this mixing process the agitator speed was reduced to 10 m/s. A batch of about 25 kg was dispersed. The throughput rate of the mill was about 420 kg/h.

Subsequently samples of the product were taken and analysed after 30 minutes of circulation grinding each. After a grinding time of 60 minutes an organic dispersing aid was added to the suspension to avoid reagglomeration of the product particles. After 120 minutes of circulation grinding a suspension with the values $x_{50,3}$ of 107 nm and $x_{99,3}$ of 149 nm could be produced.

Conclusions

For real comminution of coarse particles as well as for desagglomeration of powders with primary particles sizes in the nanometer range the use of very small grinding media is very advantageous.

However, these small grinding media call for a special machine design adapted to their size for being able to allow easy and safe handling. The *ZETA*® *RS* agitator bead mill of NETZSCH-Feinmahltechnik GmbH disposes of this special machine design.

By the development of a new screenless open grinding media separation system extremely small grinding media can be used for comminution and desagglomeration processes with at the same time avoiding any metal contact inside of the mill. The discussed examples show that by optimizing the grinding media size both the energy input as well as the grinding time required for achieving a comparable product fineness can be enormously reduced.