# Mechanical Production of Nanodispersions

NETZSCH

## Introduction

Manufacturers of machines and plants for grinding, dispersing and mixing of solids in liquid and pasty products understand the importance of grinding media size. For comminution 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, "mild dispersion" processes are typically required. In some cases, grinding media as small as 30 µm is used, and a highly developed grinding media separation system is needed.

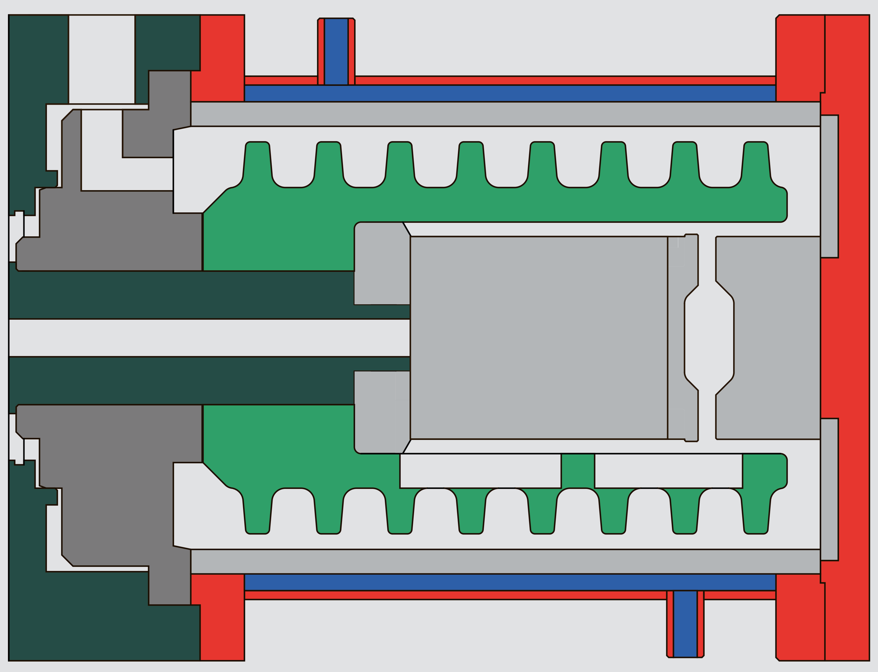

Figure 1 NETZSCH *ZETA® RS* Mill Technology with ODC (Open Dynamic Classifier) System

## *ZETA® RS*, the mill for the use of very small grinding media

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 bead entering and seizing the mechanical seal and doing great damage to the seal face. The new design is a fundamental change of the construction of the seal, successfully preventing even the smallest grinding beads 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 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. 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.

Figure 2 Agitator bead mill in positions for filling, operation and discharge

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

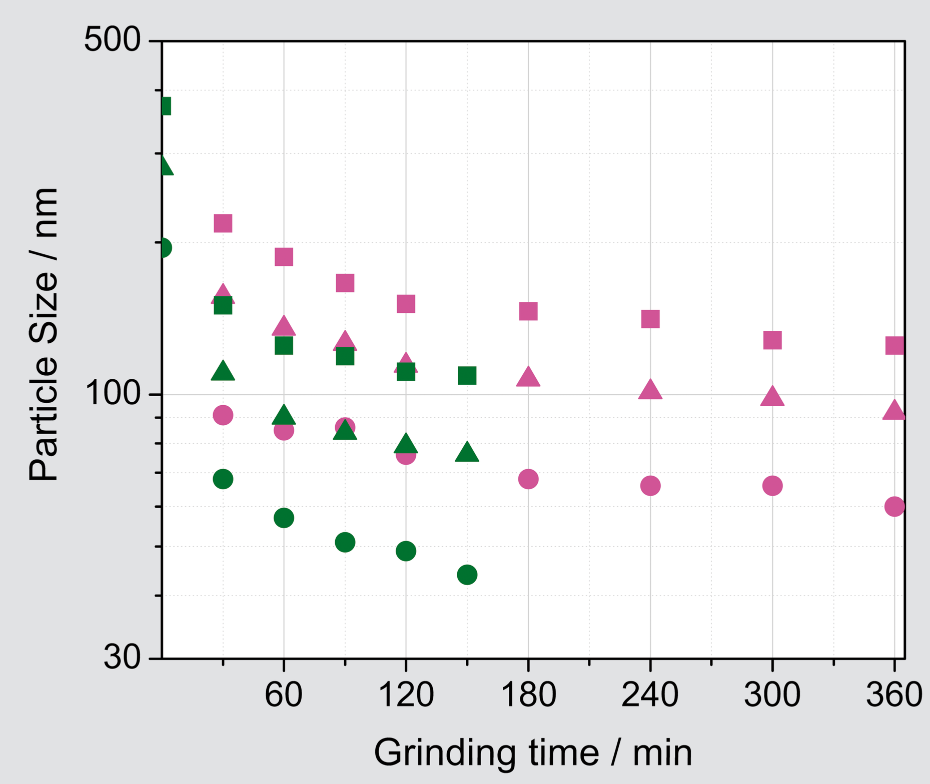


Figure 3 Real comminution results of titanium dioxide with grinding media of different size related to the grinding time

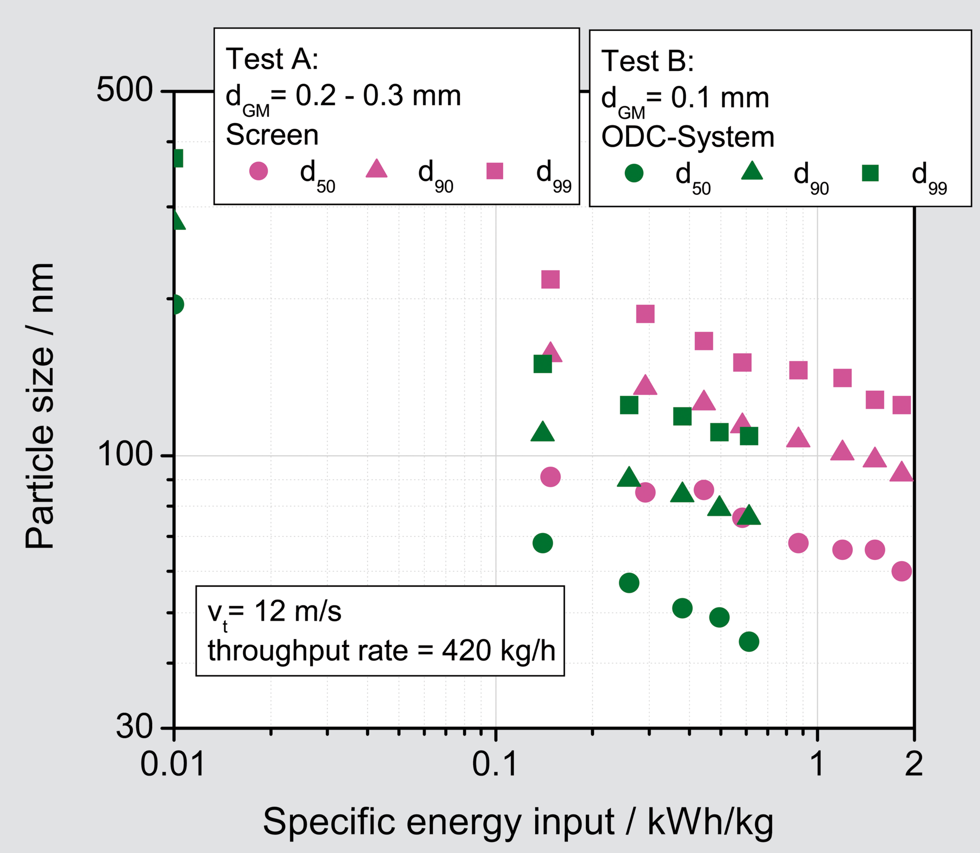


Figure 4 Real comminution results of titanium dioxide with grinding media of different size related to the specific energy input

## Open Dynamic Classifier (ODC) System

Nanotechnology applications often call for metal-free grinding or dispersion. For these applications, a special grinding media separator system called ODC (Open Dynamic Classifier) was developed. This screenless system is suitable for low-viscosity suspensions and offers the following advantages.

- Variable grinding media sizes can be used without having to change the screen
- Screen plugging is impossible
- Significantly low pressure increase in the mill results in higher throughput rates
- The decrease of the grinding media size by wear during long operation is reduced
- Impurities from the product, as well as a small amount of very coarse feed material are no problem as they are flushed out of the machine together with the product suspension
- The ODC-System can be completely disassembled and is easy to clean

## 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 wt%, which was stabilized by an appropriate additive. Two grinding tests were carried out in a *ZETA® RS* 4 agitator bead mill (4 liter 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 and Figure 4. 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.

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.

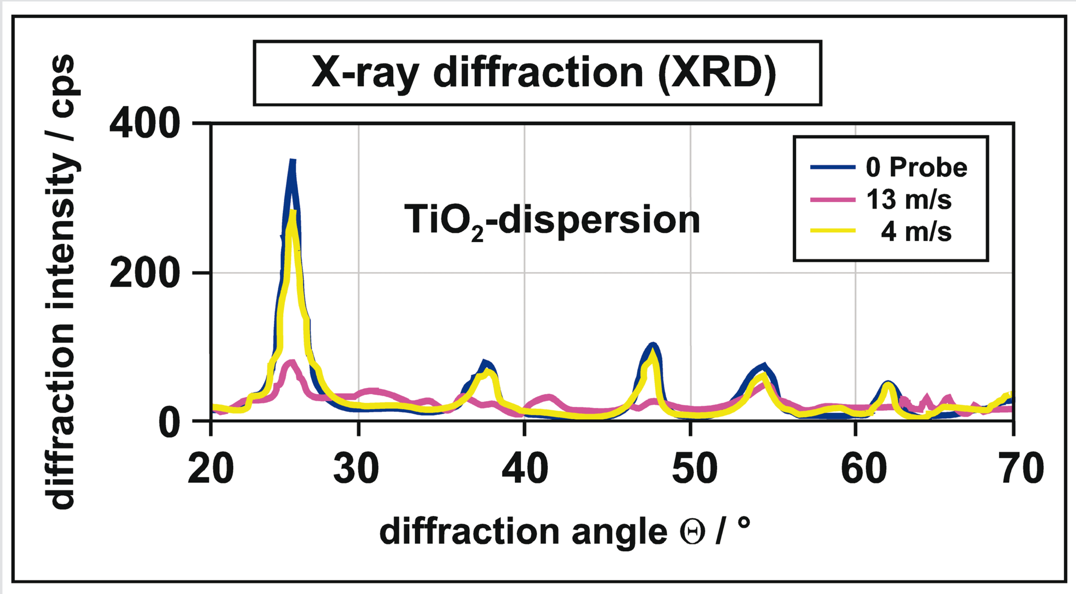


Figure 5 A comparison of mild dispersion vs. real comminution on the structure of $TiO_2$

## Conclusions

As the process of comminution and dispersion to finer particle sizes requires the use of ever smaller grinding media, the ability of the mill to separate the grinding media and the operator to easily handle small grinding media gains importance.

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