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Extraction of difficult to grind components when grinding rare earth powders

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In this age of electric mobility and miniaturisation rare earth alloys and the permanent magnets made from them are constantly gaining in importance. A decisive factor for the quality and properties of permanent magnets is a narrow particle size distribution with the lowest possible fraction of finest (<2 μm) and coarsest particles (>8 μm).

With jet mills and ultra-fine classifiers made by NETZSCH, sensitive Nd-Fe-B-compounds and Sm-Co-compounds or other rare earth alloys can be ground reliably to fine powders under inert gas operation giving a narrow particle size distribution and defined upper particle size limit with reproducible results.

For grinding these alloys NETZSCH has developed the optimised high-density bed jet mill *M-JET*. This machine also offers the possibility of the automatic extraction of components which are difficult to grind during operation without contaminating the plant peripheral parts with impurities. This feature makes rapid and problem-free product change possible at all times.

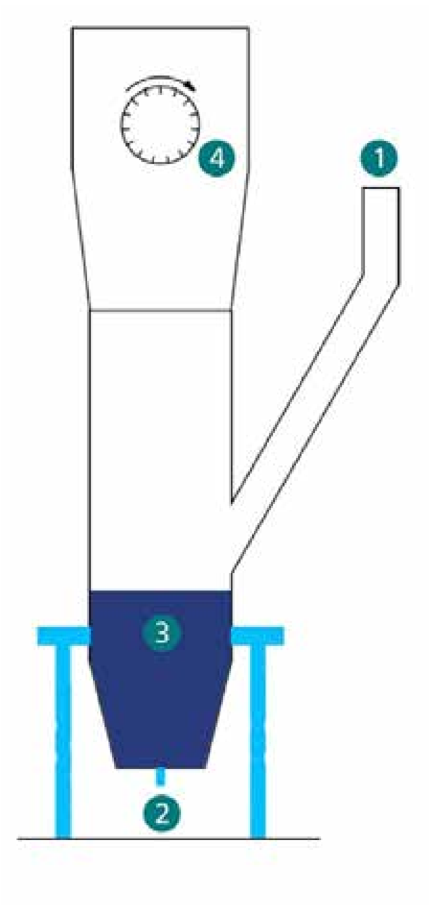

Figure 1: Diagram of a Fluidized Bed Jet Mill with Floor Nozzle
Product feed (1), floor nozzle (2), grinding chamber (3), classifier wheel with drive (4)

Difficult-to-grind components when grinding rare earth alloys

One of these disadvantages is the formation of residues which are difficult to grind during grinding of rare earth alloys used for the manufacture of magnets. These residues consist mainly of neodymium or other rare earth fractions and iron. The residues are ductile and are difficult to grind in jet mills. Instead they tend to accumulate in the grinding chamber. Consequently, the throughput capacities decrease during the grinding process.

When grinding rare earth alloys to be used for the manufacture of magnets (or other applications) residue deposits of difficult-to-grind components are frequently formed. In the case of Nd-Fe-B-alloys these consist mainly of neodymium, other rare earth fractions or iron. The residues are ductile and are difficult to grind in jet mills. Instead they tend to accumulate in the grinding chamber. Consequently, throughput capacities decrease during the grinding process and the productivity of the complete plant diminishes.

In addition to this, if these components contaminate the final product, this has a negative effect on its magnetic characteristics. Furthermore, the accumulation of difficult-to-grind components can also modify the particle size distribution which also influences product quality. Another problem is the shift in the composition of the alloy of the rare earth powder due to selective grinding in the fluidised bed. Therefore, depending on the quality of the rare earth alloys it is necessary to remove the components which are difficult to grind from the grinding chamber at regular intervals.

Extraction of Difficult to Grind Components from conventional Fluidised Bed Jet Mills

The extraction process in conventional jet mills is frequently a very time-consuming and complicated one which is usually carried out dependent on the speed of the mill – typically when 50 % of the standard throughput capacity has been reached.

It is well-known that in the case of counter-rotating jet mills difficult-to-grind components are extracted by reducing the classifier speed. This process is often assisted by using floor nozzles (Figure 1), which, however, are very easily prone to blockage due to their structural design installed on the floor of the machine.

The complete extraction process in a conventional jet mill can take several hours, which in turn, due to the reduction of the classifier speed – can cause contamination with coarse, difficult-to-grind components in the complete plant. Therefore, the extraction process must always be followed by an adequate rinsing time. This means that production must be interrupted for a longer period.

At the end of the extraction process, the fluidised bed must once again be filled with powder. During filling, shifts in the particle size distribution and throughput capacity and selective grinding can occur (Figure 3), which can lead to a shift in the alloy components of the ground powder.

Due to the large volume of the grinding chamber (fluidised bed) of fluidised bed jet mills the process of mixing and homogenisation of the fluidised bed can take up to one hour. With these mills, constant throughput capacities and particle size distributions are not obtained until after this time.

When using fluidised bed jet mills, the best product quality can always only be obtained if the mill is operated without interruption with a fluidised bed of a constant mass. However, this is only possible for a limited time period.

The Solution from NETZSCH: Extraction of Difficult to Grind Components from Spiral Jet Mills of type *M-JET*

To combat the disadvantages of conventional fluidised bed jet mills mentioned above, the engineers at NETZSCH developed a spiral jet mill with an integrated dynamic air classifier. The *M-JET* combines the advantages of a fluidised bed jet mill with those of a spiral jet mill and is therefore the ideal mill for grinding rare earth powders. A highest possible reproducible fineness can be obtained independent of the load in the gas jets. The problem of extraction of difficult-to-grind components has been elegantly solved by experts and is carried out directly from the grinding chamber during operation of the *M-JET*.

Figure 2: Laboratory plant M-JET 10 with extraction pipe in the dust filter

This process can easily be automated. By decreasing the duty cycle, reductions in the throughput capacity can be detected. When a specified value is reached, a flap installed in the piping to the filter opens, and the product is pressed out of the mill by the overpressure in the grinding chamber. During this process, the classifier wheel operates via the bypass. The contents of the mill are then discharged directly into the downstream dust filter via a separate piping, which is only used for emptying the mill.

As the good product is taken directly from the cyclone, there is no contamination of the plant. Figure 2 shows the layout of the extraction piping in a laboratory plant of type *M-JET* 10.

A further application problem is the rapid and complete emptying of the grinding chamber when changing the product. This system can also be used effectively for this purpose although the process of emptying the grinding chamber can naturally also be initiated manually.

Thanks to the small volume of the grinding chamber in the spiral jet mill *M-JET*, phenomena, which usually occur when using a fluidised bed jet mill, such as shifting of the product size distribution of the ground powder and sinking of throughput capacity are extremely rare (Figure 3).

In comparison to classic fluidised bed jet mills, the grinding chamber volume of the *M-JET* -series is lower by a factor of 25 to 40. Thus, an *M-JET* 50 with a gas throughput of between 1200 and 1500 m³ / h requires an active filling of approximately 6 - 10 kg of powder during grinding. The filling of similar sized jet mills is between 150 and 250 kg for Nd-Fe-B-alloy powders.

The Extraction Process in the *M-JET* in detail

In Figure 4 the extraction process of the active powder filling from the grinding chamber is depicted in a graph: the grinding chamber of the laboratory mill *M-JET* 10 was emptied at set time intervals. The decrease of the classifier power during and after the emptying process can be seen clearly. After each emptying the fineness of the cyclone product and the mass and fineness of the extracted powder in the filter was measured. The fineness (d50) of the cyclone product was around d50 = 2.95 μm and remained relatively constant over the complete operating time. It was also shown that there were almost no fluctuations in the d50 fineness of the contents of the mill, collected in the dust filter. On average the extracted mass was around 330 g. The extracted product does not leave the inert closed loop process. Therefore, there is no danger of powder burning during the extraction process and no additional dust filters are necessary, as the dust filter installed in each plant is used for this purpose.

After the contents of the grinding chamber have been extracted, the mill

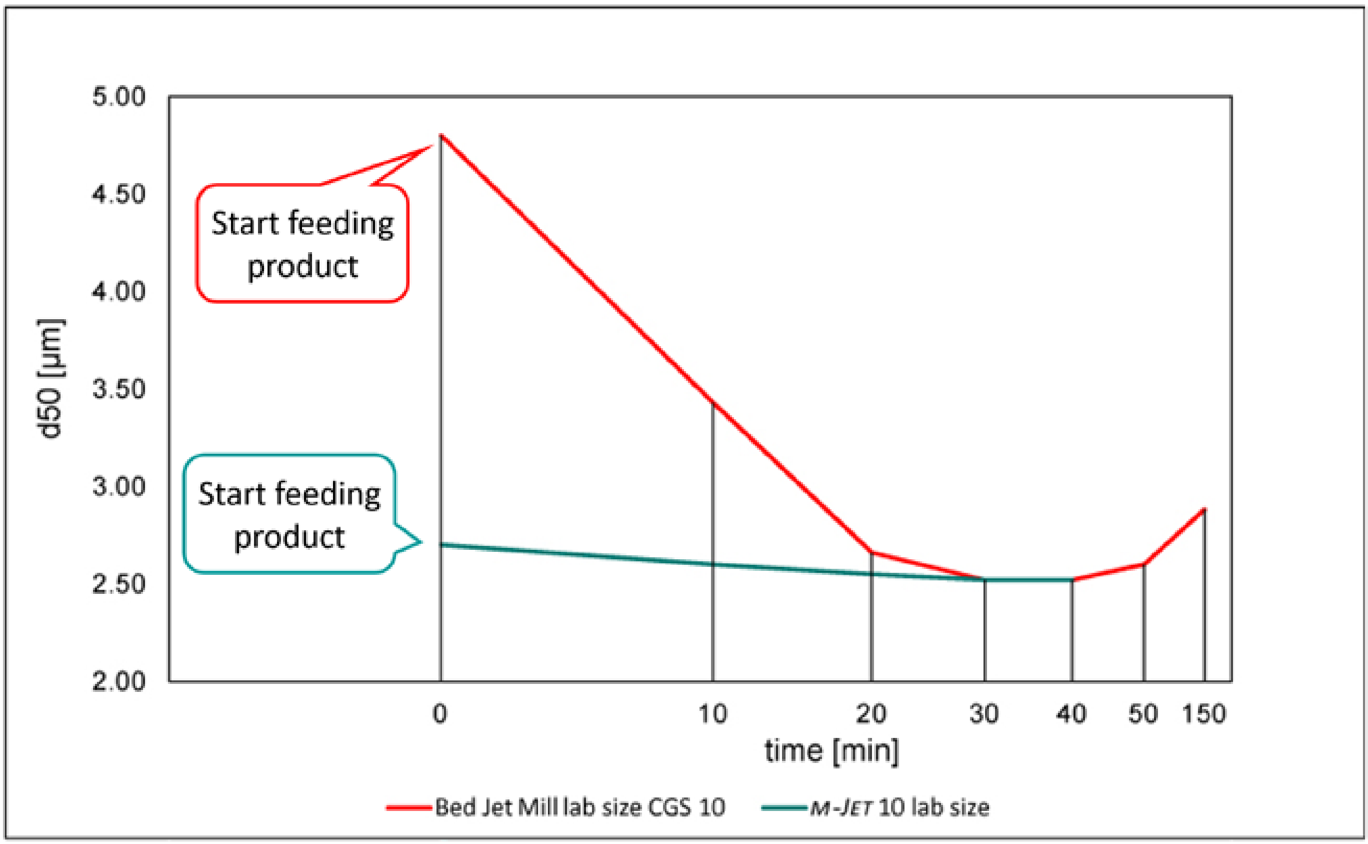

Figure 3: Comparison between M-JET and Fluidized Bed Jet Mill during start-/stop-procedures

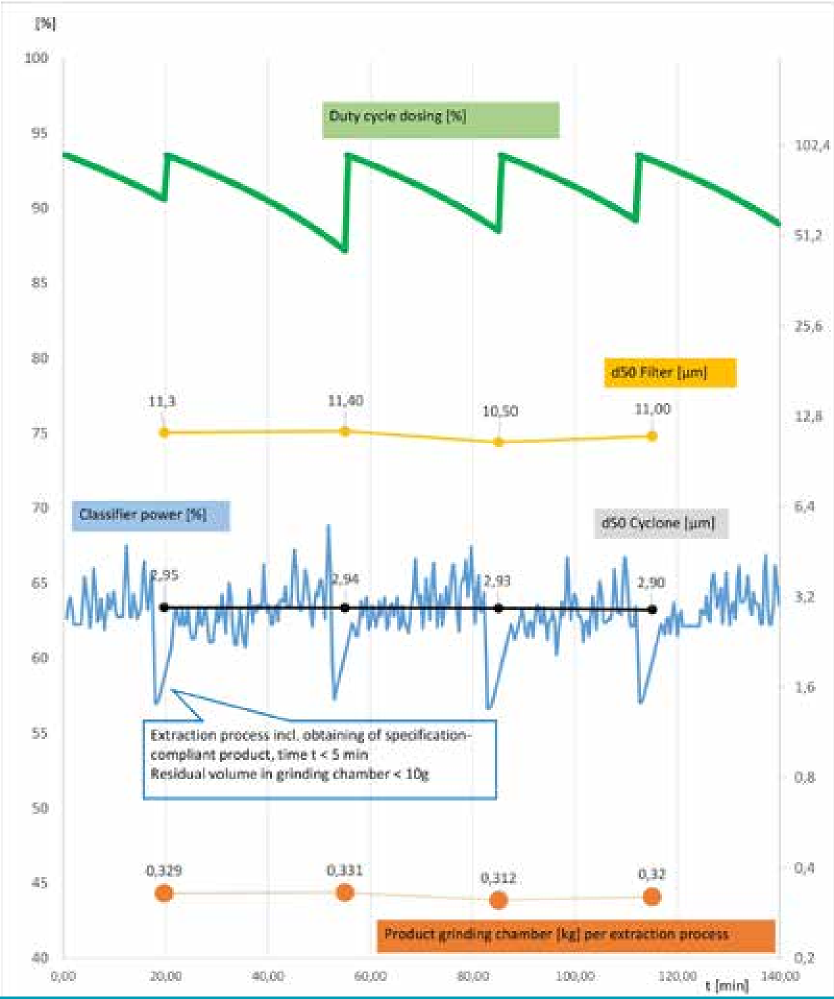

Figure. 4: Extraction of difficult-to-grind components from the Spiral Jet Mill M-JET 10

delivers specification-compliant product again within 10 minutes. This represents a significant saving of time in comparison to conventional fluidised bed jet mills. The time required for the extraction of the product out of the grinding chamber as well as the reaching of a constant product quality can be transferred 1:1 to *M-JET* production scale mills.

In Hanau near Frankfurt in Germany, NETZSCH Trockenmahltechnik has a well-appointed laboratory for tests with rare earth powders. In this lab, it is possible to carry out grinding tests on the fluidised bed jet mill of type CGS and on the spiral jet mill with integrated classifier of type *M-JET* with integrated extraction of product components which are difficult to grind. Further optimisation of the particle size distribution is carried out by classifiying on the High-efficiency Fine Classifier m-CLASS in inert atmospheres. Many different analyses such as Malvern and REM can also be carried out in Hanau as well as ONH, OCN, ICP analyses in cooperation with a renowned institute. If required and requested by our customers, we can also carry out complete sinter programs including the identification of the magnetic properties of the sintered magnets in a Permagraph.