A Quantum Leap into the Nano Class

Wolfgang Simon

Planetary ball mills have long been a popular tool for the finest comminution of powders down to the micrometer range. In many industry segments, however, this is no longer sufficient. Demand now exists for the creation of nano particles (1nm = 10^{-9}m).

For example, the pharmaceutical industry is interested in the production of new medications with active ingredients at the nano scale. Active ingredients at the nano scale are taken up by the human body much more rapidly than conventional preparations; in other words, the better bio-availability makes it possible to significantly reduce the dosages in medication. Despite lower quantities of active ingredients, the same therapeutic effects are achieved while reducing undesired side-effects at the same time.

Nano particles are needed in materials research as well. For example, numerous research groups worldwide are active in the development of new nano materials for efficient storage of hydrogen. This is an important step on the way to the automobile of the future.

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SUNKEN GRINDING BOWLS - PREMIUM PERFORMANCE

With the Fritsch *premium line* planetary mill, the decisive step into the nano class has now been taken. The revolutionary new feature of the *premium line* is the sinking of the grinding bowls into the sun disk of the mill.

The grinding of materials down to the nanometer range requires very high application of energy and therefore significantly higher rotational speeds than those allowed by typical planetary mills.

Conventional planetary ball mills are characterised by grinding bowls that are clamped to the sun disk. This limits the maximum possible rotation speed because as of a specific speed limit, the centrifugal forces acting on the bowls will be so great that the clamping of the bowls can no longer hold. Damage to the mill and the bowls would result.

Sinking of the grinding bowls into the sun disk of the mill solves these problems! Now, the centre of gravity of the bowls lies in the plane of the sun disk. The centrifugal forces arising generate significantly lower overturning moment, which in turn allows the mill to run at a significantly higher speed.

As a result, the new Fritsch *premium line* attains a speed of up to 1100rpm, generating up to 150% higher energy application. This significantly reduces the grinding time to reach the nanometer range. For certain materials, only this level of energy application even allows the creation of nano particles with planetary ball mills.

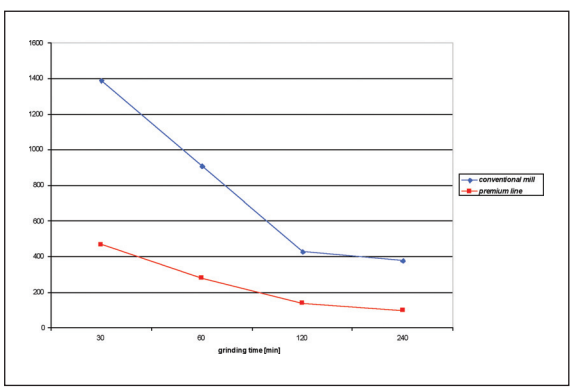


Figure 1. Comparison of the new premium line (see red line) with a conventional planetary mill (see blue line)

Figure 1 shows a grinding test with the new Fritsch *premium line* compared with a conventional planetary mill. Aluminium oxide Al_2O_3 was ground in suspension using 0.5mm zirconium oxide grinding balls.

The d_{50} value of the initial material was 21µm. After only 60 minutes of grinding with the Fritsch *premium line*, the d_{50} value of the particle size distribution was under 300nm. This value is not reached with the conventional planetary mills even after 240 minutes.

At the end of the test series (240 minutes), a d_{50} value for particle size distribution of 99nm was achieved with the new *premium line*. To generate still smaller particles, we recommend using even smaller grinding balls in a following step.

The premium line planetary mill

Table 1: d_{50} value depending on grinding time

Time [min]	*premium line* d_{50} [nm]	conventional mill d_{50} [nm]
30	450	1390
60	280	910
120	140	420
240	99	380

SelfLOCK TECHNOLOGY - PREMIUM SAFETY

The sunken bowls also simplifies the handling and improves the process safety. The grinding bowls are clamped into the mill using the **SelfLOCK technology** with only two hand motions. Incorrect operation is impossible. An RFID-chip integrated into the lid of the bowl also automatically identifies the grinding bowls used. The rotation speed is optimised and impermissible grinding settings are prevented. Naturally, a balance check is integrated which prevents start up of the mill if it is loaded incorrectly.

The completely newly developed special bowls allow for secure sealing. Any overpressure arising during grinding can be released in a controlled fashion through a built-in valve in the lid.

In other words, the best conditions for achieving precise and reproducible grinding results down to the nano range.

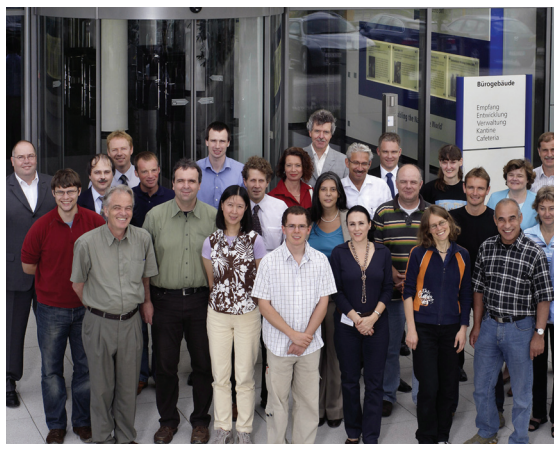
GRINDING PROGRAMS AND COMPUTER INTEGRATION - PREMIUM SIMPLE

For routine operation, the *premium line* is naturally also capable of generating and saving various grinding programs. The variable parameters are speed, grinding time, pause times and reverse mode. It is possible to generate SOPs (Standard Operating Procedures) that can be protected with a password. The *premium line* has USB, Bluetooth and Ethernet interfaces for integration into the IT structure of the laboratory. Naturally, all grinding parameters can be recorded and saved by special software. This makes it possible to validate the grinding process.

Grinding bowls in the sizes 20ml, 45ml and 80ml are available for the new Fritsch pulverisette 7 *premium line*. A series of different bowl materials are available for the widest range of applications, of course. From agate to zirconium oxide, from steel to hardmetal tungsten carbide, the right material exists for every application. In addition, it is possible to select from an entire range of different ball sizes. It should be noted in particular that increasingly small grinding balls are also required in order to achieve ever smaller particle sizes.

The new planetary mill generation will be expanded already next year with the pulverisette 5 and pulverisette 6 *premium line*. The new concept described here will then also be realised for large grinding bowls up to 500ml.

Successful EFTEM User Workshop



21 guests met for the first EFTEM User Workshop in the new Hans Mahl Nanosolution Center of the Nano Technology Systems (NTS) Division of Carl Zeiss SMT. Under the motto "Efficient – Fascinating – Top-class – Easy – Microscopy", element analysis with ESI and EELS on the ZEISS LIBRA® 120 Energy Filter TEM was the focus of the three-day workshop.

In an informal sequence of lectures and practical demonstrations about electron energy loss spectroscopy, the guests were offered the possibility of obtaining information about innovations and developments in the field of EFTEM technology.

"A key aspect was the practical exchange of experience among the guests and with our application specialists," explains workshop organiser Marlene Thaler from NTS Product Management. "This concept has been very well received. We intend to offer our customers and users such workshops more often in the future."

Circle no. 76

Nanotechnology Milestone Achieved for Quadrupling Terabyte Hard Drive

Hitachi Ltd and Hitachi Global Storage Technologies have developed the world's smallest read-head technology for hard disk drives, which is expected to quadruple current storage capacity limits to four terabytes (TB) on a desktop hard drive and one TB on a notebook hard drive.

Researchers at Hitachi have successfully reduced existing recording heads by more than a factor of two to achieve new heads in the 30-50 nanometer (nm) range, which is up to 2,000 times smaller than the width of an average human hair (approx. 70-100 microns).

Called current perpendicular-to-the-plane giant magnet-oresistive (CPP-GMR) heads, Hitachi's new technology is expected to be implemented in shipping products in 2009 and reach its full potential in 2011.

Hitachi continues to invest in deep research for the advancement of hard disk drives as we believe there is no other technology capable of providing the hard drive's high-capacity, low-cost value for the foreseeable future," said Hiroaki Odawara, Research Director, Storage Technology Research Center, Central Research Laboratory, Hitachi, Ltd.

"This is an achievement for consumers as much as it is for Hitachi. It allows Hitachi to fuel the growth of the 'Terabyte Era' of storage, which we started, and gives consumers virtually limitless ability for storing their digital content."

Circle no. 78

The Smallest Details Ever Seen by Electron Microscopy

The highest-resolution images ever seen in (S)TEM electron microscopy have been recorded using a new instrument developed jointly by U.S. Department of Energy national laboratories, FEI Company and CEOS GmbH, in Heidelberg, Germany. This breakthrough in electron microscopy - with 0.5 Ångström and below performance - is a result of The TEAM Project (Transmission Electron Aberration-corrected Microscope) supported by the U.S. Department of Energy's Office of Science Basic Energy Sciences.

One-half Ångström is one one-billionth of five centimeters. To put that in perspective, the DNA helix is approximately 20 Ångströms in diameter, a carbon atom is around two Ångströms. The width of an average strand of human hair ranges from 500,000 to one million Ångströms.

Electron microscopes can be used to observe fine details of the inner structure of materials. The ability to characterise the atomic-scale structure, chemistry, and dynamics of individual nanostructures makes this type of microscope a very powerful tool for scientists in all disciplines.

With the extraordinary 'vision' of the special TEAM microscope it will become possible to study how atoms combine to form materials, how materials grow and how they respond to a variety of external factors. These constitute many of the most practical things that science needs to know about materials and will improve designs for everything from better, lighter, more efficient automobiles, to stronger buildings and new ways of harvesting energy. See page 39 for further details.

Circle no. 77

New ALD System is Launched and Ordered

Oxford Instruments is pleased to announce the launch and first customer orders of its new OpALTM atomic layer deposition (ALD) system. Following on from Oxford Instruments' successful launch of its FlexAL® ALD process tool in 2006, the OpAL system offers a compact open-loading system to complement the load-locked FlexAL.

The OpAL system is based on Oxford Instruments' renowned Plasmalab®80Plus open-loading process tool platform, and can handle from small wafer pieces up to full 200 mm (8") wafers – making the OpAL tools equally suitable for academic and industry R&D.

The base thermal ALD system can be upgraded with the addition of a remote plasma ALD source. The remote plasma option allows for the widest possible choice of precursor chemistry with enhanced film quality; plasma enables low-temperature ALD processes while the remote source maintains low damage.

Liquid or solid precursors can be heated to 200 °C and bubbled with argon, and are housed inside an extracted stainless steel cabinet providing safe management of hazardous precursors, located within the tool to minimise delivery line length.

Five orders have already been received from customers across the USA, Europe and Asia, for both thermal OpALT and combined thermal/plasma OpALRPT systems, and are now being delivered. With its global service and support team, Oxford Instruments gives worldwide hardware and process support to its customers in addition to the process guarantees provided from its own Applications Laboratory.

Circle no. 79

Circle no. 80