Increasing the quality of metal powder with qualitative particle size analysis

As explained by Manfred Puckhaber*, particle size is one of the decisive quality parameters of metal powders. One should be aware that there is no powder with only monosized particles – a collection of particles always represents a size distribution which determines flowability and reactivity as well as compressability and hardenability – hence the most important characteristics of the sintered material.

After almost 30 years of rapid development the laser diffraction method for particle size analysis not only gained a leading position in the laboratory but also gained the potential to face new challenges directly in the process. Whilst off-line application is suitable for laboratories with different products, in-line application integrates representative sampling and supplies the particle size information immediately for continuous monitoring and automated process control.

Instrumentation

Particle size analysis with laser diffraction offers simple operation and wide usage possibilities for most materials, from under 0.1 micron to the centimetre range (fig.1). Above all, the qualitative features such as reliable reproducibility, high dissolution, shortest analysis periods and virtually maintenance-free operation offer significant advantages over screen and sedimentation processes. Products can be measured in air or a carrier gas, or as suspension. The quality of particle size analysis depends on different aspects: sampling; sample handling; dispersion; detection; evaluation and representation of results. A modular laser diffraction system has the advantage of being able to apply different dispersing modules with the same diffraction sensor.

Decisive for reliable results is that the particles analysed reflect the complete amount of material. Therefore, not only representative sampling is important but also that the number of particles measured is high enough to overcome statistical limitations. Laser diffraction in comparison, e.g. to image analysis, handles large samples and records the diffraction pattern of all sample particles of which the size distribution is finally deconvoluted.

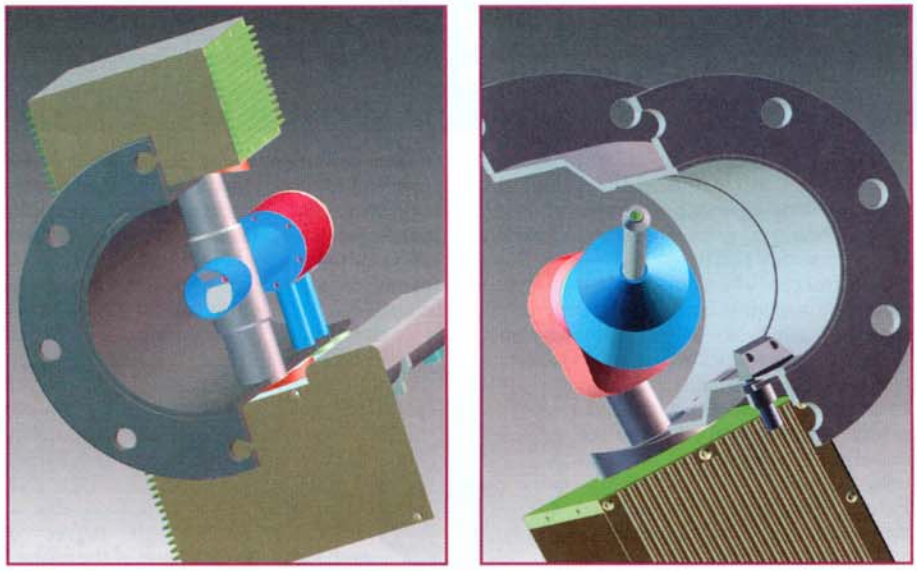
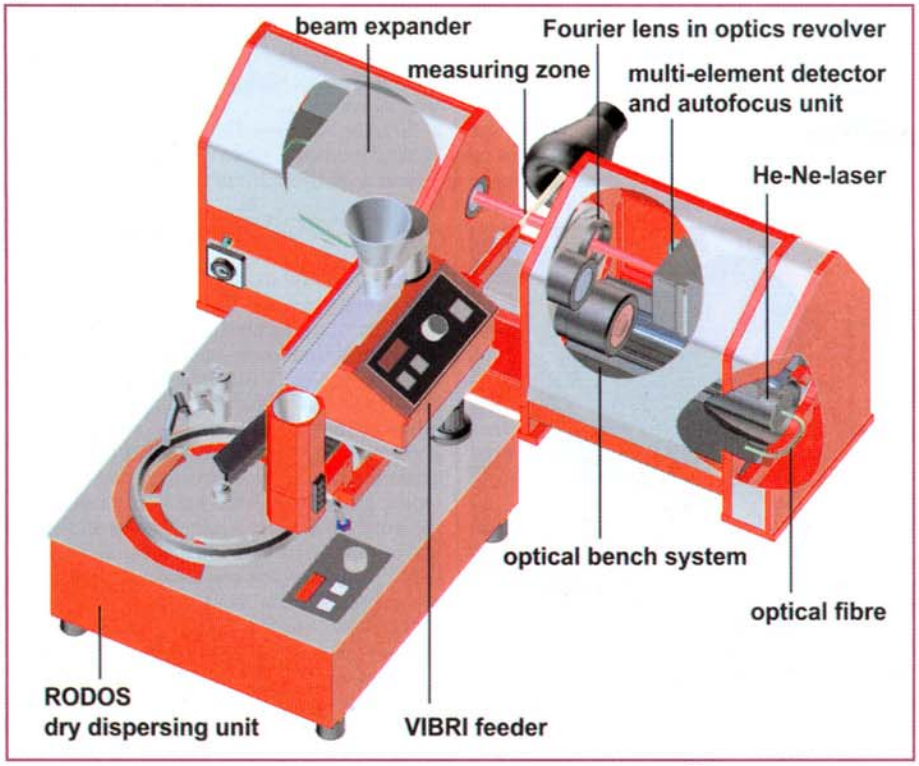


Fig.2: Analysis and sampling of Sympatec MYTOS & TWISTER

Fig.1: Sympatec HELOS and RODOS with VIBRI as example of particle size analysis with laser diffraction and dry dispersion



For both, dry and wet dispersion, controlled dispersion conditions are necessary. Proper dry dispersion for example, uses a controlled combination of shear forces, particle-particle and particle-wall collisions and is able to reliably handle samples down to below 0.1 micron. All dispersing parameters are set up via the operating level, for different products, for example. Standard operating procedures (SOP) which are recalled by pressinga single button can be defined which control the complete cycle of measurement – from reference measuring via sample feed and dispersal to measuring, evaluation and cleansing of the equipment and help to secure identical measuring conditions for comparison tests. State-of-the-art control and evaluation software is built around a versatile database and provides powerful tools for data evaluation, statistics and graphical presentation.

With adequate dispersion, the cycle time for a particle size analysis is typically less than a minute including feeding, dispersion, measurement, evaluation and automatic rinsing.

Quality assurance of instrumentation and analysis results

Production of systems according to the validation rules requires the highest standards of quality assurance. Sympatec began in 1994 with its own QA-system for particle size analysis, meanwhile strengthened by a large number of international applications. The high educational standard of the employees is the basis of its three levels, the documented development of the measuring systems, the ▸

*Manfred Puckhaber is with Sympatec GmbH, System-Partikel-Technik, Burgstatter Straße 6, D-38678 Clausthal-Zellerfeld, Germany. Tel: +49 5323 717 0; Fax: +49 5323 717 229; email: sales@sympatec.com

documentation of all the production steps in the Production Binder and the certification of all components, and the assembled system. With the reference materials a re-certification can be executed after installation and repeated at the customer's site to ensure the full operation whenever requested.

With this extended quality management, Sympatec guarantees extremely high accuracy and system-to-system comparability for its particle size analysers. Hence, the standard deviation of results from different systems of the same type is typically less than 1%. The limits defined by ISO 13320 of max. 3% to 5% have been clearly exceeded and intentionally so since the introduction of the Sympatec QA-system. Significant for the increased awareness of quality aspects is that global competition and increased endeavours for continual production control have transformed the knowledge of particle size distribution of a product from an internal characteristic quantity to an external supply guarantee.

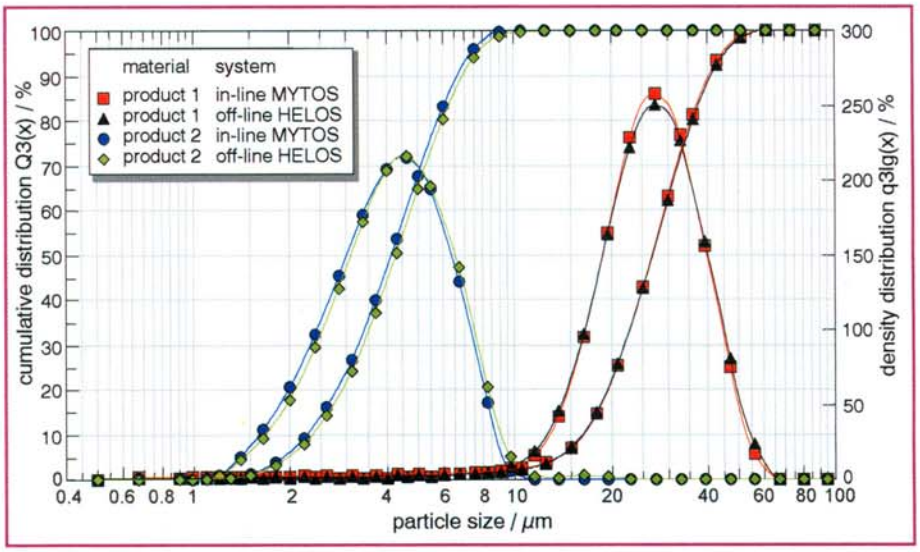


Fig.3: Comparison of results from MYTOS and TWISTER with HELOS and RODOS

In-line process monitoring

For reliable in-line PSA, not only controlled dispersion, sensitive measurement and powerful data evaluation must be performing reliably, but also sampling and sample handling. Therefore, Sympatec has developed a new PSA system with integrated representative sampling (fig.2). 'TWISTER' is the first link in this innovative chain. From its shielded parking position at the side of the pipe the sampling finger starts its spiral track into the centre and out again to the side, drawing a representative sample whilst doing so. The system can cope with a wide range of process conditions, e.g. pressure up to 10bar, temperature up to 100°C.

'MYTOS' comprises adapted components of the established 'HELOS' and 'RODOS' technology, thus also ensuring complete comparability of results to off-line analysis (fig.3). The Sympatec 'WINDOX' software, which records all measurements in a database, can be set up to show just the key features of the distribution as a trend plot against time, thus allowing effective control of important process parameters without losing any detail information. Automated control is provided using a PLC. An intermediate solution can be the combination of MYTOS and 'VIBRI'.