MYTOS & TWISTER

INSTRUMENTS FOR DRY IN-LINE PARTICLE SIZE ANALYSIS BY LASERDIFFRACTION INCLUDING REPRESENTATIVE SAMPLING AND DRY DISPERSING

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1.0 Abstract

The classical approach for dry particle size analysis including representative sampling, dry dispersion and laserdiffraction is combined in MYTOS & TWISTER as a complete system for process applications. Modular components are available for optimum adaptation to the process. In-line solutions cover pipe diameters from 40 to 1000mm. Automated on- and at-line options complete the MYTOS & TWISTER family to satisfy the demands of the process and application.

2.0 Introduction

Particle size is very often the quality criteria for dry powders and a decisive parameter for production processes. Therefore real time particle analysis is able to optimize processes and can be applied to close the control loop. In industrial applications demanding challenges can be found.

Most industrial processes use pipes as connections between different process stages. Without sampling it is quite often not possible to measure directly in the process since the flowthrough in production pipes extends the capabilities of laserdiffraction instruments for particle size analysis. The sampling has to be representative in order to obtain meaningful results and not random numbers. That is why continuous sampling should be preferred [1-2].

The sample preparation must be adaptable to the demands of the product to be analyzed. The dispersing of the sample must be complete but breaking or milling of particles has to be avoided in order to achieve the correct information about the product and the process.

Not only because of the value of certain products it is very often preferred to keep the sample inside the process pipe but also wear, cleaning aspects and even the influence of the process conditions themselves are under full control when the complete instrument is installed inside of the pipe. Of course there are applications were the sampled product is wanted outside of the process for different analysis. In this case the in-line sampler can be connected to an on-line laserdiffraction instrument outside of the pipe.

The applied laserdiffraction sensor has to present results that are easily checkable by reference materials to ensure the functionality of the instrument and the comparability to similar instruments in the laboratory.

The following chapters will present MYTOS & TWISTER, a rugged instrument family that meets the above mentioned demands and covers a variety of applications were in-line particle size analysis is crucial for the product and process quality.

3.0 Preconditions

As explained, in-line particle size analysis consist of three main stages: Sampling, dispersing and laserdiffraction analysis. Each of these has to fulfill certain precondition to form an in-line instrument at the end [3].

3.1 Representative Sampling

The sampler has to scan the complete production pipe, all positions within the pipe should contribute equally weighted and sampling should be isokinetic. It becomes clear that this cannot be obtained by a static, single point sampler but a more advanced approach is required.

3.2 Laserdiffraction and Dispersion

For laserdiffraction the optical concentration or obscuration has to be in certain limits. On one hand it must be high enough to achieve a good signal to noise ratio, on the other hand it should not exceed a certain limit which is defined by the detector dynamics and the algorithm treating multiple scattering. Therefore possibilities to adapt the concentration are required. Laserdiffraction of dry powders also requires a powerful dry disperser since agglomerates are usually not the point of interest for the user. Finally it should be possible to interrupt the sample flow for periodical background measurements.

4.0 In-line Sampler TWISTER

It was pointed out that a dynamic sampling finger being far more representative is to be preferred. Figure 1 shows the top view of the sampler inlet having the inlet diameter *d* in a process pipe with a diameter *D*. From equation 1, were *Phi* is the area related flux, it becomes clear that the amount of sample $\dot{m}$ is only dependent on the sampler's inlet diameter *d*.

$$\dot{m} = \frac{\frac{d^2}{4}\pi}{\frac{D^2}{4}\pi}\dot{M} = \frac{d^2}{D^2}\Phi\frac{D^2}{4}\pi = \frac{\pi}{4}\Phi d^2 \qquad (1)$$

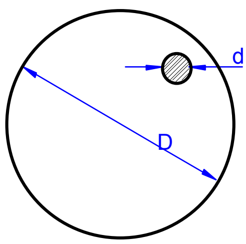

Fig.1 top view of sampler inlet

Since the inlet tip of sampler is adaptable to the demands of the process and the requirements of the laserdiffraction instrument [4] the adaptation and also the possibilities of scaling up are simple. A pre-sampler is obsolete.

Moving the sampling finger in the process pipe of course should be performed without the need of moving gaskets which would be destroyed by particles in a very short period of time. A new approach for the movement of a sampling finger in a process pipe is presented in figure 2. Since all positions in the pipe should contribute equally weighted a modified spiral was chosen since no singularities, leading to unrepresentative results, occur with this form of movement. Since the sampling finger is mounted in the middle of the pipe no moving gaskets are required at all to perform the spiral movement. A shielded parking position mounted at the inner wall of the process pipe closes the sample inlet and cuts off the sample stream, which allows a reference measurement of the in-line laserdiffraction instrument described in chapter 5.

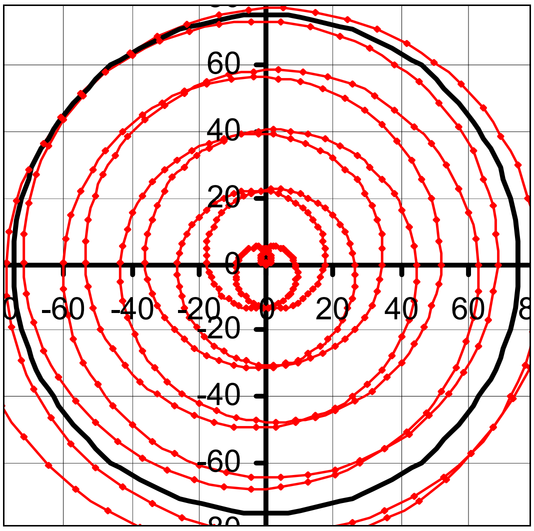

Fig. 2 Sampling profile

By variation of the lever geometry it is possible to adapt the sampler to different pipe diameters and still ensure a representative sampling using the identical concept. Figure 3 presents a cut away view of the in-line sampler in its parking position. It becomes obvious that the sampled material remains inside the process pipe when it is transported to the laserdiffraction stage during the sampling procedure. Therefore the pressure inside of the pipe does not influence the sampling itself. Wear is reduced since no elbows are necessary.

Because no gaskets but metal bellows are used, is suitable for hazardous or explosive environments as well. A scraper integrated in the parking position cleans the tip of the sampler and blockage is avoided.

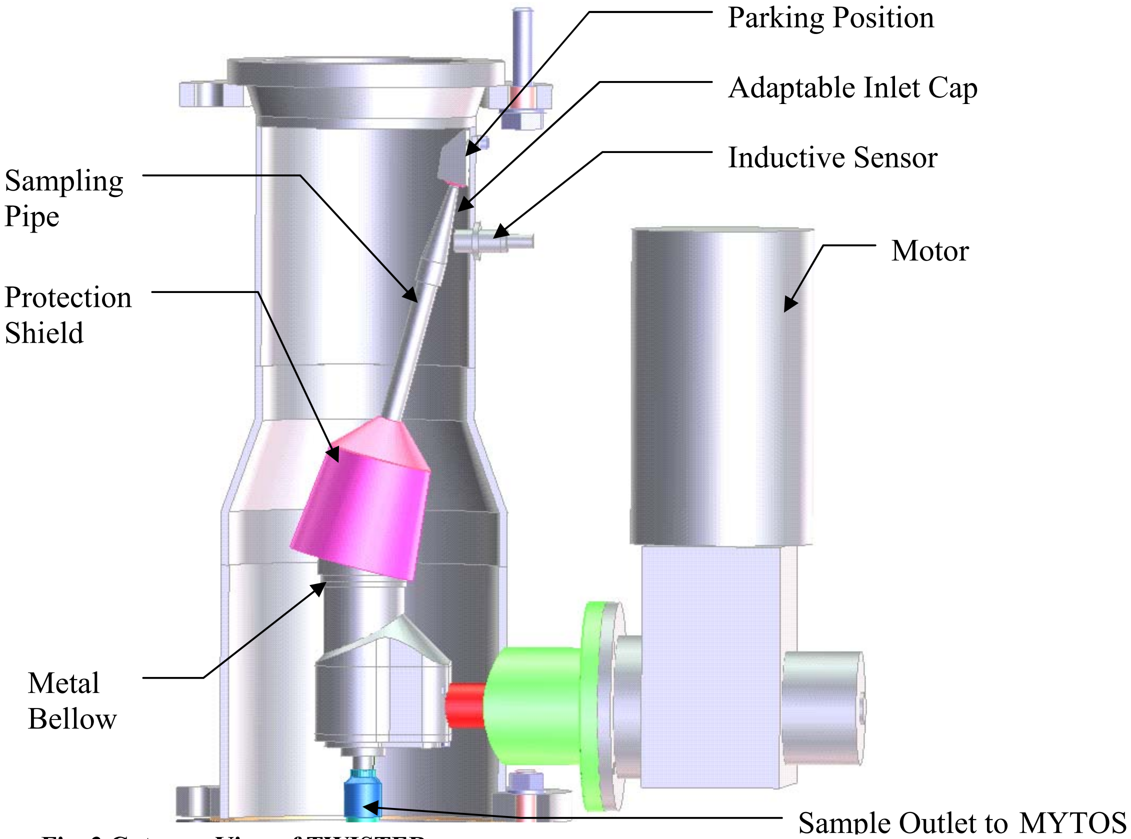

Fig. 3 Cutaway View of TWISTER

To keep the speed of the particle at the entrance of the sampler constant, an adjustable flow pump is required. This flow pump is part of the dispersing- and laserdiffraction system described in chapter 5.0.

5.0 In-line Disperser and Laserdiffraction System MYTOS

For the dry dispersing of the particles [5], the well established Sympatec RODOS dispersing injector is integrated into MYTOS. Since wear is proportional to the numbers of measurements a lifetime of more than two year is guarantied even for abrasive products. Integrated backblowers keep the dispersing unit clean.

For the laserdiffraction part of the unit the standard components of the Sympatec HELOS laserdiffraction particle sizer are mounted into the instrument. Since all parts used are identical to the HELOS & RODOS laboratory equipment the results of MYTOS & TWISTER are guarantied to be identical too.

Figure 4 presents MYTOS in a cutaway view.

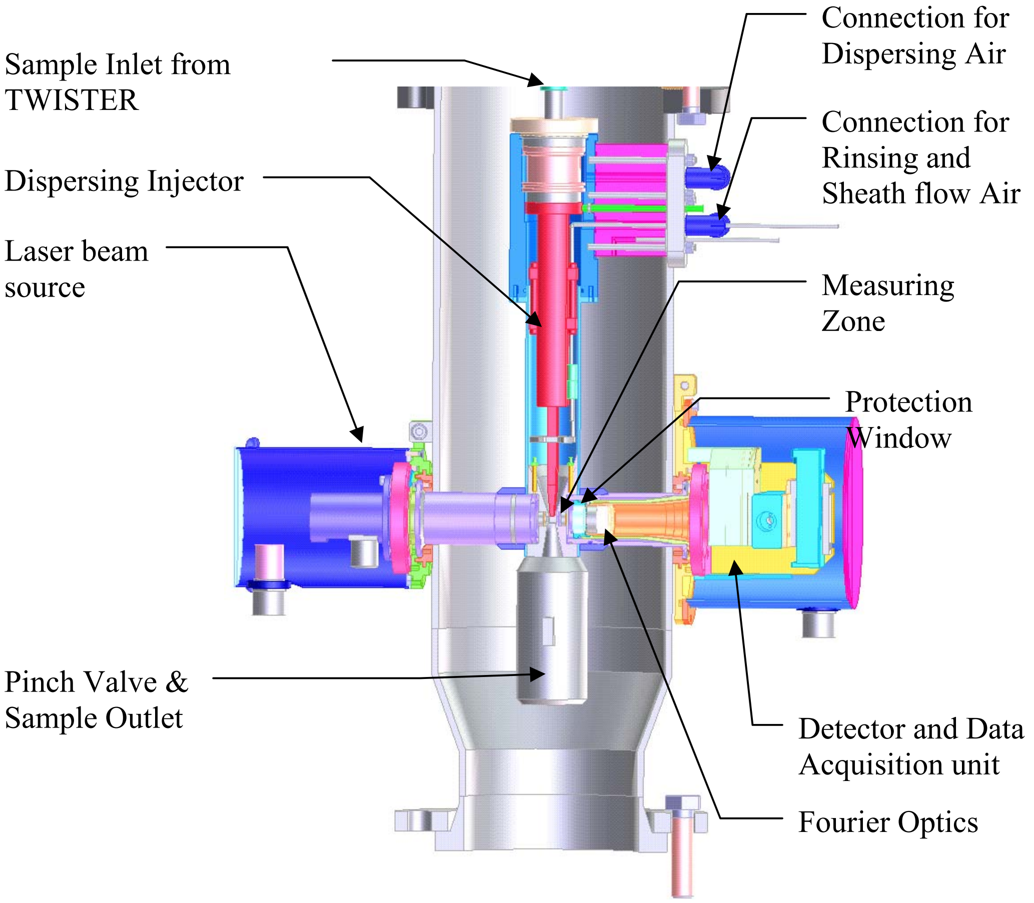

Fig. 4 Cutaway view of MYTOS

The measuring zone of the laserdiffraction unit is equipped with a second injector system that uses compressed air for rinsing and to build up a sheath flow acting like a dynamic focus of the aerosol in front of the protection windows. Experiences prove that cleaning of the protection windows is required less than every three month if necessary at all. This cleaning procedure can be done within seconds without the need of shutting down the process.

6.0 MYTOS & TWISTER Experience

Figure 5 presents a complete MYTOS & TWISTER in-line / in-line instrument installed in a process pipe and ready to measure. Today the unit is engineered to cover process pipe diameters from 40 to 1000 mm in all orientations and a measuring range from 0.25 to 3500 microns.

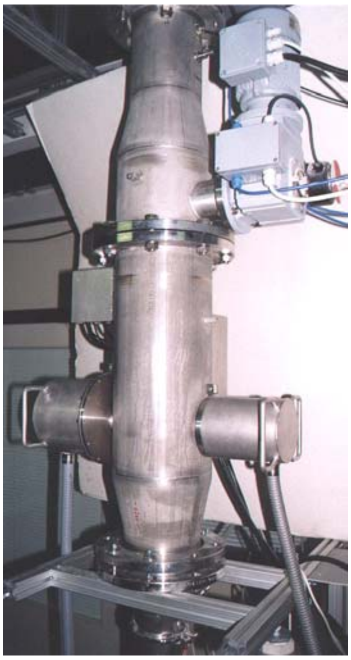

Fig. 5 MYTOS & TWISTER

Figure 6 presents results of reference materials analyzed by the presented in-line unit compared to measurements of the same product using a HELOS & RODOS off-line laserdiffraction instrument.

Since exactly identical optical components and the same dispersion units are integrated in both systems, the results must be identical as well. This is guarantied by the system specification.

Results of measurements in production are presented in Figure 7. In this graphic the trend of the characteristic values (x_{10}, x_{50} and x_{90}) of the measured particle size distributions are displayed. The process presented is a jet-milling process of a polymer powder. A change of process parameters can be detected immediately and in real time. This leads to a new dimension of process-control of particulate matters within seconds instead of hours and an improvement of the product quality.

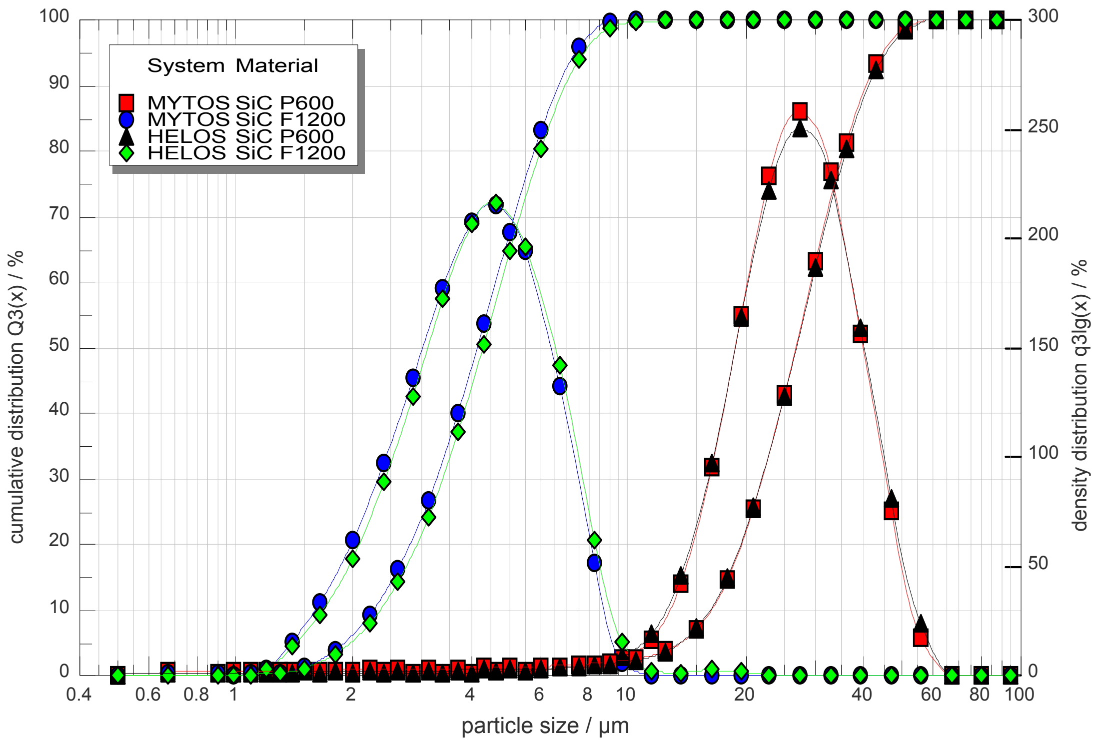

Fig. 6 Comparison in-line (MYTOS & TWISTER) and off-line (HELOS / RODOS) Particle Size Analysis

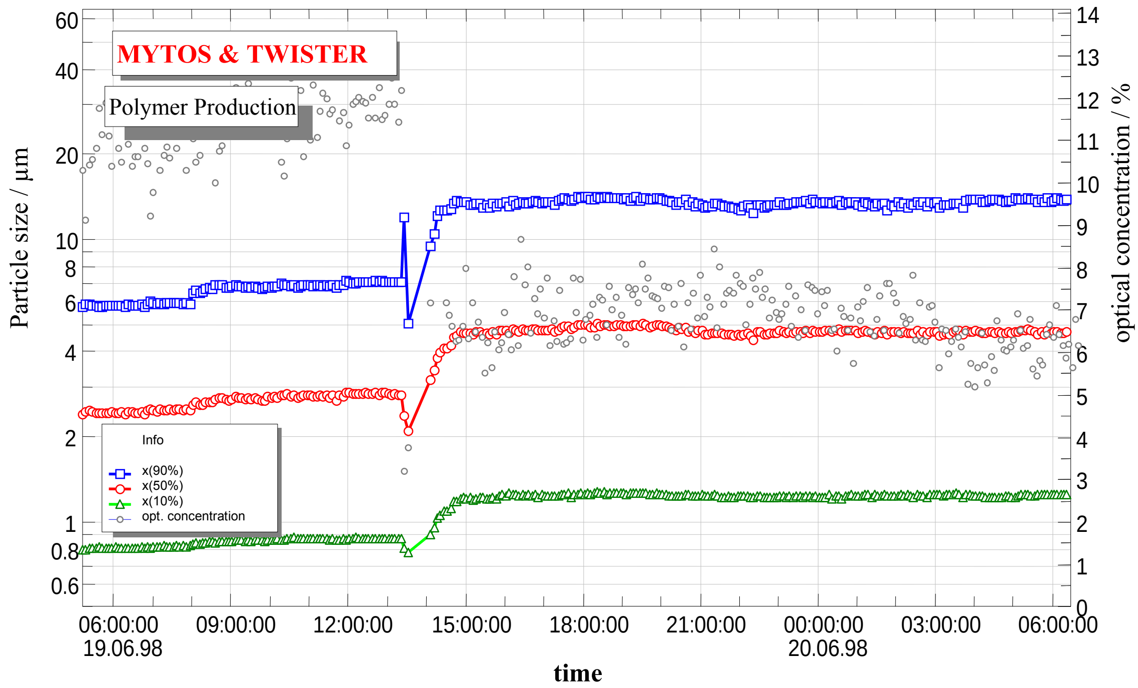

Fig. 7 Change in a Milling Process monitored as a trend of characteristic values

7.0 MYTOS & TWISTER Family

Recently the MYTOS & TWISTER concept has been extended to a complete family of instruments that cover a wide range of applications and different adaptations to the process. The following examples should present an overview about the different members of the family. In case the height for a complete in-line / in-line system is not available as encountered sometimes in existing production sites or if the analyzed product should be available outside for other analyzing purposes as well, the laserdiffraction instrument can be operated outside of the pipe as an on-line system as well. In this case the sampler will be equipped with an outlet tube. Figure 8 presents TWISTER & MYTOS as an in-line / on-line combination.

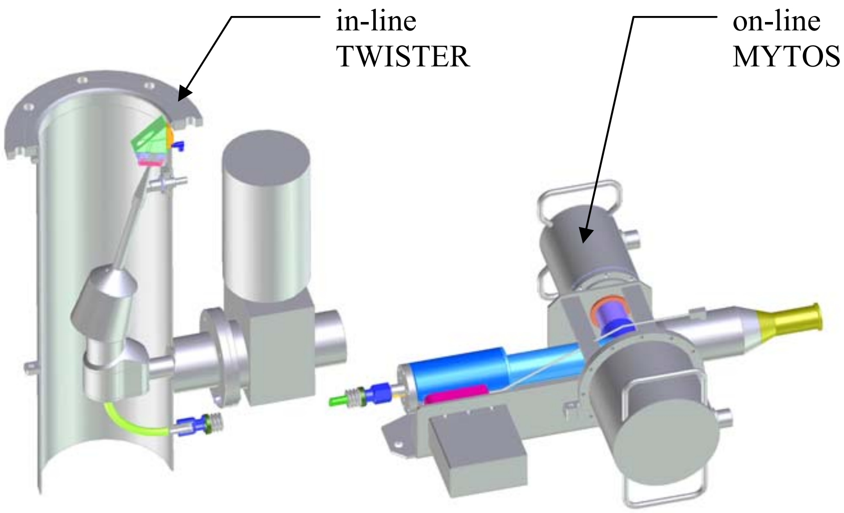

Fig. 8 In-line TWISTER and on-line MYTOS

Even if representative sampling is of crucial importance, very often different kinds of samplers (e.g. screw-samplers) are installed already. Using the presented laser diffraction with the dosing device VIBRI as presented in figure 9, the laserdiffraction technology can be adapted directly to existing samplers or operate as a wall mounted stand alone on-line solution. To complete the family MYTOS & VIBRI can be housed in (figure 10) as module for automated particle size analysis in a fully automated environment.

Fig. 9 MYTOS & VIBRI

Fig. 10 MYTOS & VIBRI Module

8.0 Conclusion

The MYTOS & TWISTER concept for real time particle size analysis combines representative, isokinetic sampling, powerful dispersion and well known particle size analysis of dry powder by laserdiffraction in the center of the process pipe. The sampler's parking position allows cutting off the product flow and performing background measurements as well as the reduction of wear. Since MYTOS & TWISTER in-line systems use the same components as Sympatec HELOS off-line systems, the results are guarantied to be identical.

Different MYTOS versions allow the access to a variety of processes and different conditions i.e. hazardous or explosion proof areas and also GMP [6] applications.

9.0 References

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