LASER-DIFFRACTION RESULTS FROM DYNAMIC IMAGE ANALYSIS DATA

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ABSTRACT

How to handle the influence of different particle shapes on laser diffraction results is an issue in powder technology since long. In all laser diffraction systems an angular variation of the intensity pattern of the diffracted light is recorded. It is generally difficult to interpret the relationship between the diffraction pattern obtained and the equivalent size distribution of spherical in the case of non-spherical particles. Thus it is often assumed that the performance of a laser diffraction system can only be verified with spherical reference material.

Sympatec relies on more than 20 years of documented experience with non-spherical particles as an internal reference material for the HELOS laser diffraction system family. If this material is measured with methods other than laser diffraction, a significant bias may result. It is often assumed that there are no true traceable size values for non-spherical particles and therefore such material cannot be used to quantify the accuracy of a laser diffraction system. This assumption is generally true, if this material should be applied for instruments using different types of dispersing systems. We will show a method allowing for the transfer of results between our families of laser diffraction and image analysis instruments.

For shape and size characterisation Sympatec has introduced the QICPIC dynamic image analysis system. The dispersers for dry powders and suspensions are modular and interchangeable between QICPIC and HELOS. Today's instruments can handle particle numbers of more than 100 million particles per measurement and reach statistical relevance of the results comparable to laser diffraction.

The direct comparison of image analysis and laser diffraction measurements under identical particle dispersing conditions is now possible. For arbitrarily shaped particles an equivalent HELOS laser diffraction pattern is calculated from the images of the QICPIC measurement. The absolute scale of both systems may now be verified by direct signal comparison, without even relying on evaluation modes or inversion procedures. Furthermore, this pattern can be numerically detected using a virtual detector geometry and feed as input to the existing HELOS evaluation, resulting in a direct representation of laser diffraction size distributions measured by dynamic image analysis. This approach enables a direct comparison of both techniques - independent of the particle shape.

1 INTRODUCTION

In all laser diffraction (LD) systems for particle size measurement a radial variation of the intensity pattern of the diffracted light is recorded. The evaluation procedure is generally based on Fraunhofer diffraction for spheres. Diffraction is a valid approximation if the particles are opaque and their shapes are sufficiently represented by their two-dimensional projection areas. For non-spherical particles the LD method reports a size distribution in which the predicted diffraction pattern for the volumetric sum of spherical particles matches the measured diffraction pattern. It is generally a challenge to interpret the relationship between the diffraction patterns obtained and the equivalent size distribution of spherical in the case of non-spherical particles.

It has been shown using simulations that the microstructure of the shape has little influence on the particle size distribution in contrast to the macrostructure, i.e. the axis ratio of ellipsoids (Mühlenweg 1998). If these simulations are applied to images of real particle systems it will be possible to compare the results with the results of a laser diffraction instrument directly. Until now the comparison of digital image analysis (IA) with LD has been limited because of differences in dispersion, average particle orientation and statistical confidence of the IA results.

At PARTEC 2004 a new concept of IA was presented (Witt 2004), for the first time combining high-speed image analysis with powerful dry dispersion in a table-top instrument (fig. 1). The short exposure time of 1 ns makes it possible to apply the dispersing devices originally developed for the standard laser diffraction line of instruments (fig. 2). The high frame rate of up to 450 fps at full resolution of 1024x1024 pixels and the fast handling of large particle numbers per measurement (> 10^8) fundamentally overcomes the weakness of typical image analysis systems – low particle numbers resulting in large statistical errors (Witt 2005). Now for the first time image analysis data is available with the same statistical significance as of LD results. The results of both instruments can be obtained under identical dispersion conditions. A direct comparison of both methods is possible.

With the help of this method we have compared the focal length of the laser diffraction systems to the magnification of the image analyzer using non-spherical reference material (Köhler 2008).

Fig. 1: OASIS, the wet (SUCELL) and dry (RODOS) dispersing system set-up in the QICPIC image analysis sensor.

Fig. 2: OASIS, the wet (SUCELL) and dry (RODOS) dispersing system set-up in the HELOS/BR laser diffraction sensor.

2 LINK BETWEEN THE TWO METHODS

2.1 Fraunhofer Diffraction Theory

Fraunhofer diffraction is a well known model describing forward light scattering by opaque particles at a large distance compared to the size of the particle and it provides a direct relation between the projection images and the corresponding diffraction patterns. Analytical solutions exist for the Fraunhofer diffraction integral at circular or rectangular shaped objects. For general shapes we write the diffraction integral as a Fourier integral in terms of (x, y)-coordinates in the object plane and $(\tilde{x}, \tilde{y})$-coordinates in the focal plane of a lens with a focal length of f.

$$U\left(\frac{\tilde{x}}{\lambda f}, \frac{\tilde{y}}{\lambda f}\right) = C \cdot \iint G(x, y) e^{-2\pi i\left(\frac{\tilde{x}}{\lambda f}x + \frac{\tilde{y}}{\lambda f}y\right)} dxdy \qquad (1)$$

The term $\tilde{x}/f$ is an approximation of the direction cosine of the wave vector, λ is the wavelength of the light and C is constant. The object function $G(x, y)$ represents the spatial transmission function of the particle projections. If an opaque particle is present at (x, y) then $G(x, y) = 0$, and $G(x, y) = 1$ in the case of no particle. The result U of the transform represents the scalar complex field at the detector. The measurable diffraction pattern of the intensity is the square of the field's amplitude.

A binary digital image represents a sampled and discretized form of the object function which is scaled to the object plane with the optical magnification. We can now simulate diffraction for a large amount of real particles in different orientations recorded by an imaging system with the help of the Discrete Fourier Transform. An equivalent LD signal can then be calculated by applying a digital representation of the detector geometry.

2.3 Instrument set-up and experimental conditions

The same dispersing systems and conditions were used for both IA and LD. In IA the depth of focus must be considered carefully. We used a cuvette for IA with an optical path length of 1 mm, and the same cuvette for the LD measurements. In LD, contamination of liquid is subtracted as a background signal with the help of a reference measurement. In IA a particle filter on shape and size may be applied to recognize and eliminate air bubbles but such a filter was not used in the experiment.

It was not possible to use the same optical concentration. LD requires a strong and reliable signal and therefore relatively high particle concentrations. In contrast, IA requires that overlapping particles must be strictly avoided. In IA the optical concentration is defined as a geometrical obscuration. In LD systems it is defined as the extinction of the laser beam in focus. According to the Fraunhofer diffraction theory this value is twice the geometrical obscuration of an IA system.

3 RESULTS

3.1 Comparison with spherical particles

Opaque spherical glassy carbon particles having a narrow size distribution have been selected for the first comparison between QICPIC and HELOS sensors. The result of the laser diffraction analysis is obtained with the advanced FREE iterative method. The IA evaluation is based on the equivalent projection area of a circle (EQPC).

The particle size distributions are presented in fig. 3. For spherical particles the results of both evaluation methods are nearly identical.

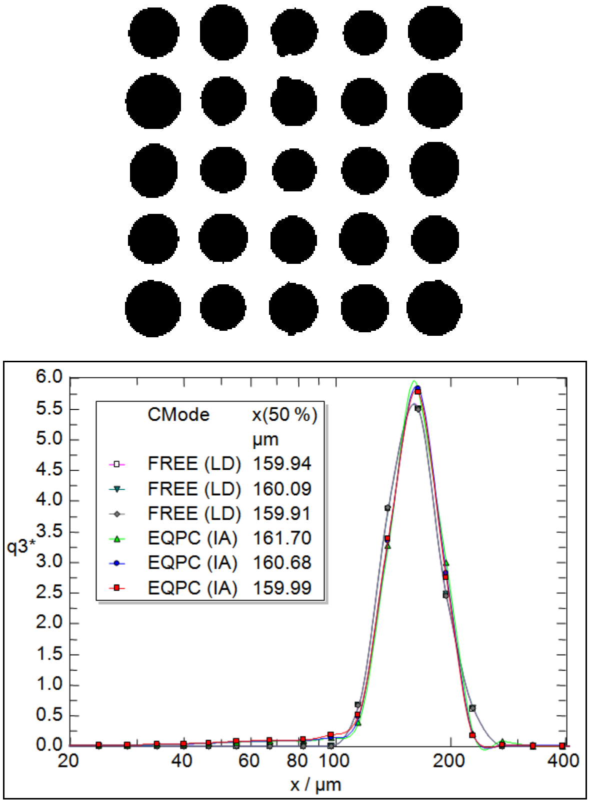

Fig. 3: Size distribution of opaque spherical glassy carbon particles. Sample images are shown above. The HELOS (LD) result is obtained with the FREE evaluation mode. The QICPIC (IA) result is obtained with the EQPC evaluation mode.

3.2 Comparison with irregular particles

Irregular silicon carbide particles are used by Sympatec as an internal reference material (RM) to certify and recertify IA and LD sensors according to Sympatec's own specifications. The main advantage compared to spherical material is more realistic wet and dry particle feeding characteristics, resulting in a better overall system test. With the introduction of the QICPIC small but noticeable differences in the results of LD compared with IA have been observed. A very small amount of these differences may be attributed to differences in feeding and particle orientation, but even if exactly the same dispersing conditions are used the results are not identical (fig. 4). This is an experimental demonstration that for non-spherical particles the EQPC and the equivalent spherical diameter measured by LD are not the same.

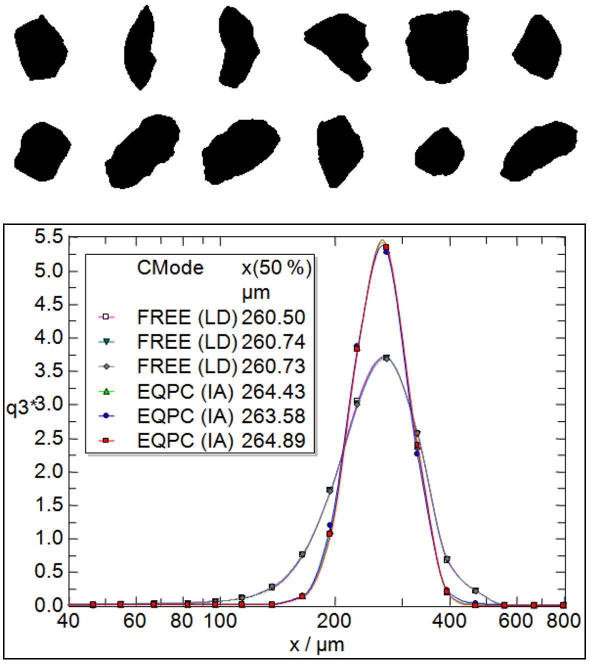

Fig. 4: Size distribution of irregular SiC-P80 particles. The HELOS (LD) results are obtained with the FREE evaluation mode. The QICPIC (IA) results are obtained with the EQPC evaluation mode.

To overcome this basic difference more than 10 000 images of the same QICPIC measurement are converted by a Fast Fourier Transform after removal of edge touching particles. The results are accumulated to an equivalent laser diffraction signal. These values are passed through the LD inversion algorithm. This procedure leads to a very good agreement of both results (fig. 5).

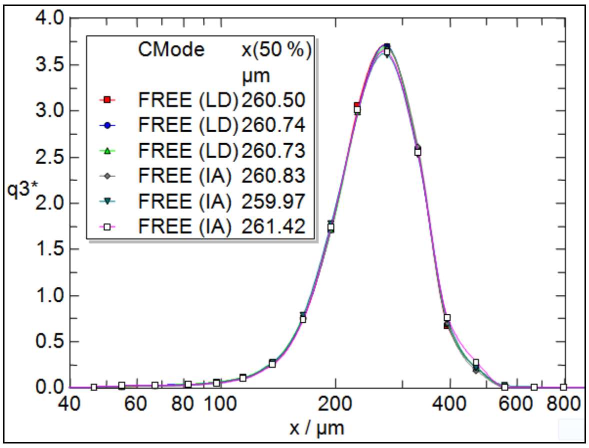

Fig. 5: The QICPIC (IA) results are obtained after calculating an equivalent diffraction pattern by Fast Fourier Transform and using the FREE laser diffraction evaluation mode. The HELOS (LD) results are the same as in Fig. 4

4 CONCLUSIONS

A non spherical material has been characterized as an equivalent laser diffraction result through use of transformed image analysis data. This approach has finally solved the fundamental problem of how to handle the influence of the infinite number of appearances of particle shapes on LD results. The results show that the observed main differences between LD and IA arise primarily from the effect of the particle shape on the evaluation procedure

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