INNOVATIONS IN DYNAMIC IMAGE ANALYSIS: DOWN TO 1 µm

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ABSTRACT

Since the introduction of high speed image analysis with powerful dry dispersion at the PARTEC 2004, this technique has been extended into various fields of application. The unique combination of a pulsed light source with light pulses shorter than 1ns, telecentric illumination and imaging, a high speed mega-pixel camera with adjustable acquisition rates up to 500 frames per second, and a powerful database for storage of the image data, measuring conditions, and evaluated results has made the characterisation of particle size and shape possible at unrivalled particle numbers of even sticky materials in a well dispersed aerosol beam – creating results of formerly unimaginable statistical relevance.

This technology has been continuously improved. Today particle numbers of more than 100 million particles per measurement can be acquired, stored and evaluated. The acquisition time for a single measurement has been extended to more than 1.5 hours at a frame rate of about 450 images per second. As all particle images are stored, this allows e.g. for searching the proverbial “pin in a haystack”. As the evaluation software fully supports multi-core processors, the calculation speed is increased to about 110 000 particles per second e.g. for calculating the equivalent projected circle diameters (EQPC) on a 2.4 GHz quad-core Q6600 Intel processor.

Wet dispersion in addition to dry dispersion has recently been introduced. The telecentric illumination and imaging create high contrast images at the particle edges even for highly transparent gel particles. Recently a special telecentric objective has been developed to lower the minimum particle size for wet dispersion down to 1 µm, and in combination with an adapted dry disperser to below 5 µm.

This technology has been expanded into the process environment. With a family of instruments combining representative sampling, dry and wet dispersion and image analysis particles are characterised at impeccable statistical relevance for cycle times down to below one minute per measurement. GMP versions are available as well as versions for hazardous areas.

1 INTRODUCTION

Since the introduction of high speed image analysis with powerful dry dispersion at the PARTEC 2004 by Witt (2004), this technique has been extended into various fields of application. The unique QICPIC combination of light pulses shorter than 1 ns, telecentric illumination and imaging, and a high-speed megapixel camera with adjustable acquisition rates up to 450 frames per second has made the characterisation of particle size and shape possible at unrivalled particle numbers.

A high-performance database for storage of the image data, measuring conditions, and evaluated results allows for creating results of formerly unimaginable statistical relevance.

This technology has been improved continuously. Today particle numbers of more than 100 million particles per measurement can be acquired, stored and evaluated. The longest acquisition time for a single measurement has been extended to more than 1.5 hours at maximum frame rate. Since all particle images are stored, this allows e.g. for searching the proverbial “pin in a haystack”.

As the evaluation software fully supports multi-core processors, the calculation speed is increased to about 110 000 particles per second e.g. for calculating the equivalent area diameter.

2 DISPERSION

Particle dispersion is one of the crucial requirements of particle analysis. The modular design of the dynamic image analysis sensor, QICPIC, is the basis for the application of dry as well as wet dispersing systems. Flexible set-ups allow for an optimum adaption to the requirements of the application or sample respectively.

2.1 Dry Dispersion

In combination with the dry dispersing system RODOS the QICPIC analyses particle size and shape of even sticky materials in a dispersed aerosol beam.

Fig. 1: QICPIC and RODOS

Additionally, for coarse or fragile samples the gravity dispersing system GRADIS is a remarkable supplement.

Fig. 2: QICPIC and GRADIS

2.2 Wet Dispersion

For image analysis applications, wet dispersion in addition to dry dispersion has been introduced in 2006 (Witt 2008). The OASIS combines the principles of dry (RODOS) and wet dispersion (SUCELL) in one automated system (fig. 3), whereas LIXELL offers most flexible setups for all kinds of applications (fig. 4).

Both systems offer an automatic alignment of the flow cell in order to image the moving particles within the depth of field and hence to achieve particle images of maximum sharpness.

Fig. 3: QICPIC and OASIS

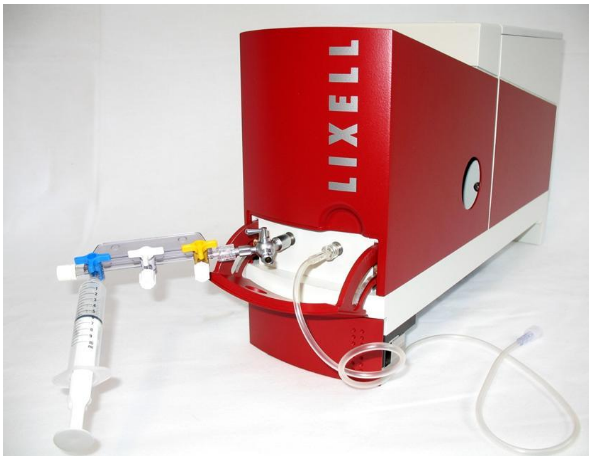

Fig. 4: LIXELL and accessories

3 DEPTH OF FIELD

In optics the depth of field (DOF) is the interval of distances with respect to the lens where all objects appear acceptably sharp in the image. From the theoretical point of view a lens can precisely focus at only one distance. However, the loss of sharpness is gradual on both sides of the focused distance. In other words, the DOF is the interval of distances where blur remains invisible in the image.

Invisible in terms of image analysis means a blur of less than one pixel. The depth of image field Δz_{image} with acceptable image blur depends on the maximum aperture angle Θ' in image space (fig. 5) and the pixel size a. The aperture angle Θ required in object space is given by the angle of the first zero of the Airy diffraction pattern of the smallest particles. The relation between Θ and Θ' (or Δz_{object} and Δz_{image}) depends on the transversal magnification M (Hecht 2001):

$$\Delta z_{object} = \Delta z_{image} / M^2$$

Δz_{image} is given by the setup and hence fixed. The remaining relation shows a reciprocal dependence of the DOF (Δz_{object}) on the square of the magnification.

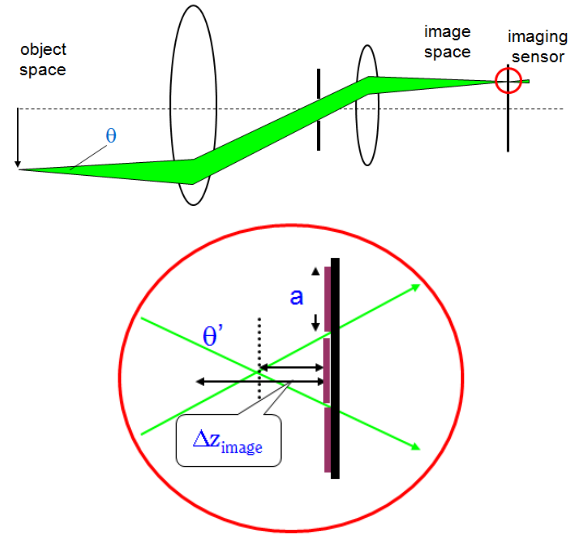

Fig. 5: Relation of magnification, depth of field and maximum blur

From the microscope it is well-known that keeping the particles in the DOF gets more and more important with increasing magnification due to this strong dependence.

4 MEASURING RANGE M3 FOR PARTICLE SIZES DOWN TO 1 µm

As the short theoretical considerations of the preceding section show, increasing the number of pixels as well as the usage of smaller pixels does not overcome these physical laws. Instead the blur would cover more than one pixel then, i.e. high-resolution blurs instead of high-resolution particle edges are the consequences.

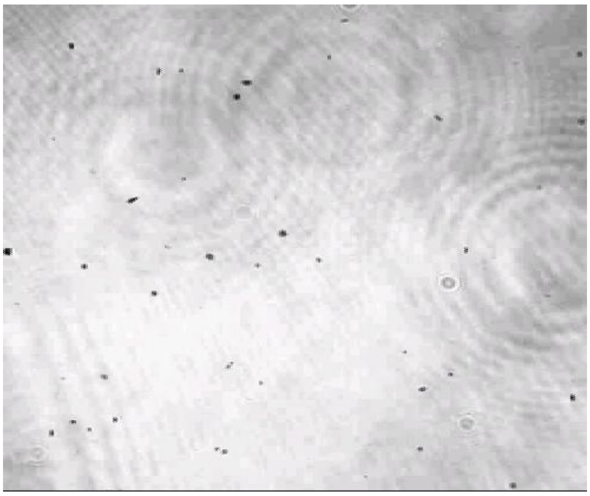

Fig. 5: SiC-F1200 within DOF of M3

The fig. 5 shows a snapshot of F1200 silicon carbide reference particles ($x_{50} \approx 5$ µm) which are well suitable to characterise particle size and shape simultaneously.

Although, in image analysis fine particles naturally consist of only a few pixels, it is possible to obtain sharp particle edges and even meaningful shape information, e.g. the aspect ratio.

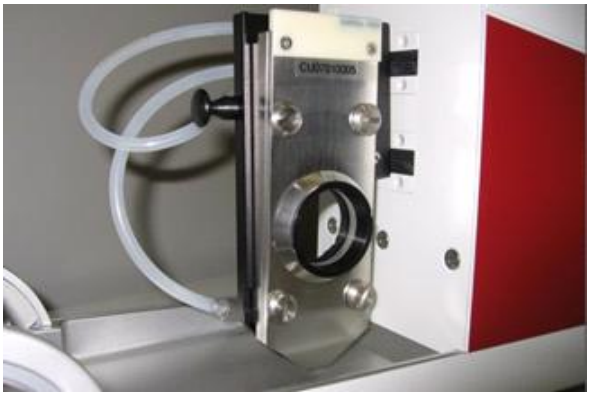

Fig. 6: Flow cell alignment at LIXELL

The combination of proper particle position within the DOF via suitable flow cells (fig. 6), fine positioning and M3 lens overcomes these problems and creates high-contrast images of even fine particles.

Fig. 7 shows the resulting particle size distributions.

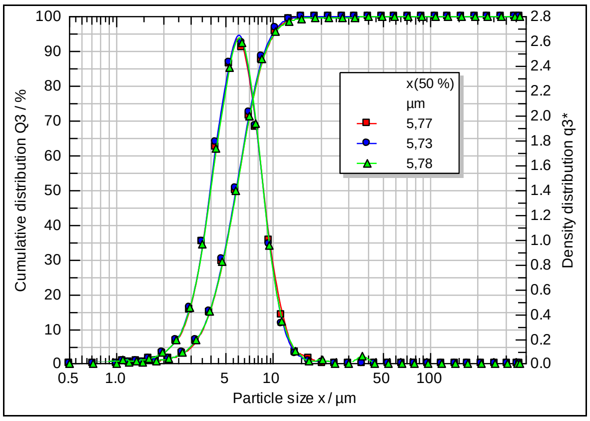

Fig. 7: Particle size distributions Q3 and q3* of SiC-F1200

The aspect ratios vs. particle size are displayed in fig. 8.

The expected irregular shape of a typical abrasive material is confirmed.

Fine particles consist of a comparably low number of pixels. Since the number of pixel arrangements is limited for low pixel numbers the correlating aspect ratios are emphasized peaks in the shape distribution (fig. 9).

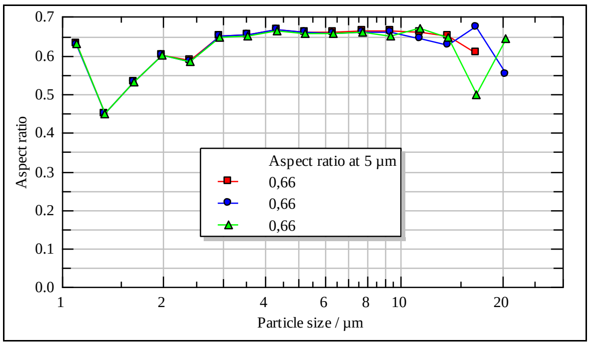

Fig. 8: Aspect ratio vs. particle size

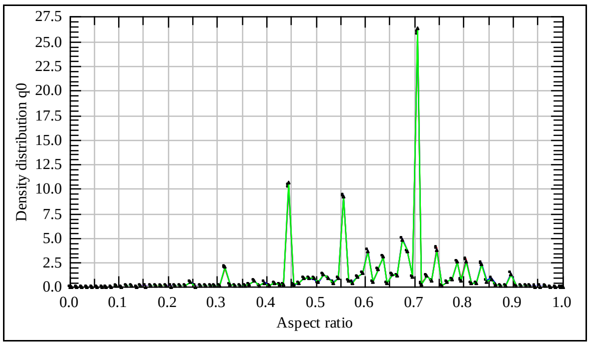

Fig. 9: Aspect ratio distribution q(a)

5 DRY DISPERSION FOR FINE PARTICLES

Whilst particle positioning in wet dispersing systems is realised via special flow cells the situation for dry dispersing systems is different. For gravity dispersing systems, special outlets in combination with an alignment mechanism are applicable. However, this method is of course limited to free-falling non-sticky materials.

Even from sticky material RODOS generates a well-dispersed aerosol beam. However, the free jet normally cannot be guided by a flow cell. Instead, e.g. aerodynamic focussing is an interesting alternative.

4 CONCLUSIONS

The introduction of the M3 measuring range in combination with particle positioning within its depth of field allows for meaningful particle size and shape analysis of high quality in the micron particle regime down to 1 µm.

REFERENCES

HECHT, E., (2001), Optik, 3. vollständig überarbeitete Auflage, München; Wien: Oldenbourg 2001

WITT, W., KÖHLER, U., LIST, J., (2004), Direct Imaging of very fast Particles Opens the Application of Powerful (Dry) Dispersion for Size and Shape Characterisation, PARTEC 2004, Nuremberg, Germany

WITT, W., LIST, J., KÖHLER, U., (2008), Possibilities of Dynamic Image Analysis in the Laboratory and Process Environment, Particulate Systems Analysis, Stratford-upon-Avon, UK