Direct Imaging of Very Fast Particles Opens the Application of the Powerful (Dry) Dispersion for Size and Shape Characterization

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ABSTRACT

Image analysis is widely in use for the characterization of shape and size of particles. Using the commercially available components the method is limited to the analysis of static or slow moving particles. Proper dispersion techniques, which usually accelerate the particles to high speeds (up to 100 m/s), are not applicable and often replaced by time consuming image processing algorithms. The limited amount of images results in low particle counts and large statistical errors.

Proper dispersion units are well established for particle size analysis by means of laser diffraction. So a new imaging sensor has been developed, which allows for the direct use of these dispersers. It combines an innovative light source with an exposure time of less than 1 ns and an adaptable optical system for perfect illumination and imaging of the fast particles on a high speed camera in the range of 1 to 10,000 µm. An integrated image pre-processor and a Gigabit digital transmission line to the computer allow for the acquisition of up to 500 fps.

For the first time, particles can be imaged and analyzed directly at the output of the well established and proven dry dispersing injection system. A powerful selection and display facility of particles is implemented.

1 INTRODUCTION

For the characterization of real particles the particle shape becomes more and more important in addition to particle size. Image analysis (IA) can give valuable service.

Static image analysis (SIA) is characterized by non-moving particles, e. g. on a microscope slide. The advantages are: The depth of the sharpness ε is well defined resulting in high resolution for small particles, and the method is well established and standardized in ISO 13322-1 [1]. But this method can only handle small amounts of data, the particles are oriented by the base, overlapping particles have to be separated by time consuming software algorithms and the tiny sample size creates a massive sampling problem resulting in very low statistical relevance of the data.

Dynamic image analysis (DIA) images a flow of moving particles. This enlarges the sample size. The particles show arbitrary orientation and the number of overlapping particles is reduced. So several companies offer systems which either operate in reflection or transmission, with wet dispersion or free fall, with matrix or line-scan cameras. The current limitations are: The free fall systems are limited to well flowing bulk materials only, dry dispersion is not established. Systems with wet dispersion only allow for smallest samples sizes and slow particles. Common to all are small particle numbers which result in poor statistics. This type of instruments is going to be standardized in ISO 13322-2 [2].

2 CONCEPT

The optimum IA system would use DIA combined with effective dispersion and good statistics.

2.1 New Approach

The task is the combination of effective dispersion with DIA. As dispersion adds energy to the particles, i.e. creates fast particles, they have to be imaged clearly. An example is the dry disperser RODOS [3], which combines particle-to-particle, particle-to-wall and centrifugal forces caused by velocity gradients. It is proven for dry dispersion down to 0.1 µm and generates a particle aerosol beam with up to 100 m/s. Taking an image with a typical flash lamp with an exposure time τ of 100 µs would result in a motion blur of up to 10 mm. The best available flash lamps with $\tau \approx 1$ µs still would result in a motion blur of up to 100 µm. So an exposure time $\tau < 10$ ns is required for an acceptable motion blur < 1 µm.

In order to obtain a representative sample and a good statistical reliability, a large number of particles have to be acquired in short times. As we are dealing with number statistics, 1% precision requires 10,000 particles within one size class. With 10 particles per image, 1000 images have to be taken resulting in 40 s of measuring time at 25 fps. For 30 equally populated classes this would result in an unacceptable measuring time of 1200 s or 20 minutes.

Increasing the frame rate to 500 fps and the number of particles to 30 per frame (due to better dispersion) would reduce the measuring time to acceptable 30s, only. But this would result in extreme data volumes, e. g. 500 Mbytes/s for a camera with 1 Mpixel resolution at 256 grey levels.

2.2 Optics

In order to process massive data volumes, clear particle images are essential. So the optical path has to be designed with care.

While front illumination creates very realistic images, the reflections depend on the particles' material and shape resulting in a difficult and error-prone image analysis. The frequently used illumination box still creates reflection at the particle borders and a small aperture stop is needed to reduce reflections. This requires a high light intensity.

The best contrast is produced by rear illumination. But even a perfect parallel beam will not produce a clear shadow of a small particle as illustrated in Fig. 1.

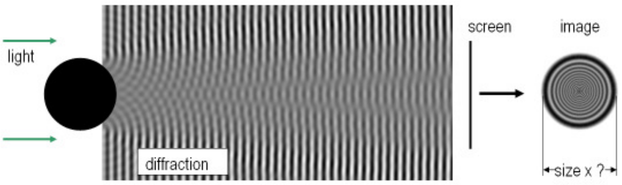


Figure 1: Calculated image of a particle created by a perfect parallel beam without imaging optics

The intensity pattern depends on the distance between the sensor and the particle. So either the sensor must be very close to the particle or an imaging optics has to be used. This cannot be a standard imaging lens. If the distance of the object plane is varied with respect to the lens the images are out of focus and the magnification varies with the object position.

The optical set-up of Fig.2 overcomes this behavior.

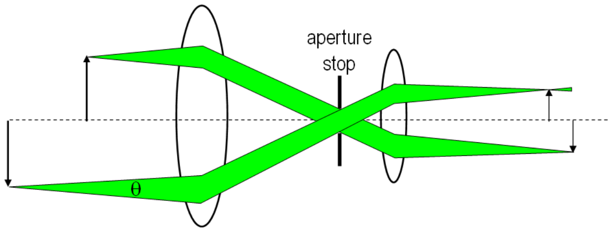


Figure 2: Special imaging lens with aperture stop

Only ray fans parallel to the optical axis are used for the image and the aperture stop controls the aperture angle θ. The size of the image is no more depending on the object position and the image is less sensitive to defocus. Even transparent particles are imaged with high contrast, as the refracted light is not imaged. This strongly reduces the calculation effort.

3 REALISATION

3.1 Principle Set-up

The set-up used is displayed in Fig. 3. The light of a pulsed light source is expanded by an adaptable beam expansion unit (ABU) which creates a parallel beam. This illuminates the dispersed particle flow which is created by the disperser. The particles are finally imaged by an imaging objective to a high speed camera.

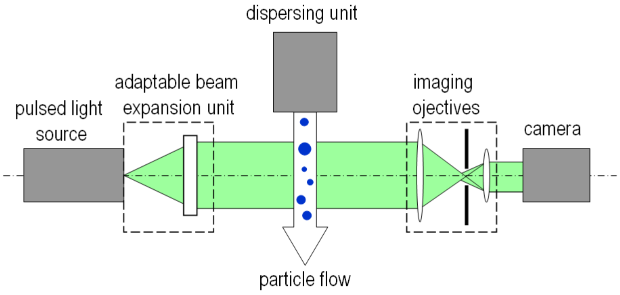


Figure 3: Set-up of the new DIA system

With this set-up smallest aperture angles can be used, resulting in high contrasts. The aperture stop blocks only stray light and light diffracted to large angles. It has no influence on the brightness of the background, so only small light energies are required. The depth of the field is limited by diffraction only, resulting in wide working distances.

The innovative light source creates stable visible light pulses with $\tau \approx 1$ ns at an output power of about 0.15 nJ/pulse. The repetition rate is adjustable from 1 to 500 Hz meeting the specifications of the high speed CMOS camera which operates at up to 500 fps synchronous with the light source. One image is composed out of 1024 x 1024 pixels of 10 µm x 10 µm area with 256 gray levels (8 bit).

Imaging objectives for different magnifications (2:1, 1:1, 1:3, 1:10 etc.) are mounted on a carousel for simple selection of a measuring range by software. The ABU adapts the illumination beam diameter width in a way that the light intensity on the camera is nearly independent on the magnification used.

3.2 Data Processing

The biggest challenge is the extreme data throughput of up to 500 Mbytes/s generated by the camera. As an off-the-shelf PC should be used, the bottle neck is the PCI-bus. While the internal memory bus has transfer rates in the GByte/s regime the PCI-bus is typically established as 32 Bit bus and operates at 33 MHz, only. So the maximum theoretical throughput is about 128 MByte/s. This value is even further reduced by the operating system to about 60 MByte/s.

So a real-time data pre-processing has to be implemented that shrinks the data volume by more than a factor of 10. Latest signal processing and programmable logic designs have been used for the image capture, background and threshold treatment, and subsequent binarisation, which shrinks the data volume already by a factor of 8. A subsequent binary compression reduces the amount of data without loss of information to a total compression factor of about 10 to 100. The factor is proportional to the number and size of the particles in the image.

The resulting data stream of up to 50 Mbytes/s is transferred via a single twisted pair cable to a specially designed bus-master PCI interface inside the PC at data rates of 1.25 GBit/s.

3.3 Physical Appearance

The complete unit is housed in a table-top stainless steel housing, as displayed in Fig. 4 and 5. The light source and ABE are located on the left, the carousel with the optical modules and the camera are located in the right.

Figure 4: Set-up of the DIA system QICPIC™ with dry disperser RODOS/L™ and dry feeder VIBRI/L™

Figure 5: Set-up of the DIA system QICPIC™ with gravity disperser GRADIS/L™ and dry feeder VIBRI/L™

The open measuring zone in the center allows for the adaptation of different dispersing units for dry and wet dispersion or the investigation of inhalation devices or sprays. Well established units e.g. those known from the laser diffraction particle sizer HELOS™ can be used with minor adaptations.

4 OPERATION & EVALUATION

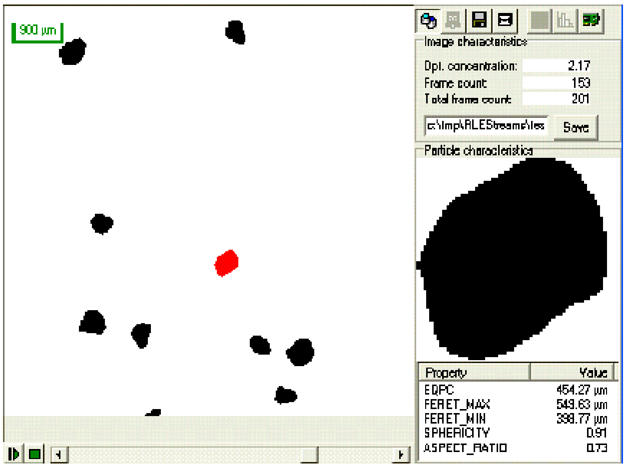
4.1 Software Structure

The high data throughput of 5 to 50 Mbytes/s requires a powerful data handling. A fast commercial database module (INTERBASE™) is used and established as a powerful stand-alone database server. A built in application server manages the storage and retrieval of the data as well as the filing of all measuring parameters.

All application programs are organized as clients and can work concurrently with the server. The software is fully compliant with CFR21 Rule 11 as requested for pharmaceutical applications.

4.2 Signal Test

A signal test window allows for the inspection of the images captured during and after the acquisition. As the complete video stream is stored, each individual particle is accessible. Fig. 6 displays one image in combination with the size and shape parameters of a specific particle.



Opl. concentration: 2.17
Frame count 153
Total frame count 201
c:\tmp\RLEStreams\tes

Property	Value
EQPC	454.27 µm
FERET_MAX	549.63 µm
FERET_MIN	338.77 µm
SPHERICITY	0.91
ASPECT_RATIO	0.73

Figure 6: Signal test window displays video stream of particles.

4.3 Evaluation

All particles in the video stream are identified and their size and shape parameters are calculated. The circle of equal projection area (EQPC), Feret diameters (max., min., averaged, 90° to max. or min.), length and width of the minimal enclosing rectangle, chord length (vertical, horizontal, max., min, 90° to max. or min., averaged) are calculated as selected. They are sorted and stored in 10,000 internal size classes allowing for subsequent rearrangement of application or user specific size classes without any loss of accuracy (even if the video data were deleted). The aspect ratio and/or sphericity are used for the characterization of shape.

5 RESULTS

5.1 Particle Size

All particle size distributions (PSDs), e.g. the EQPC can be displayed in graphical or table format as number, length, area or volume distribution.

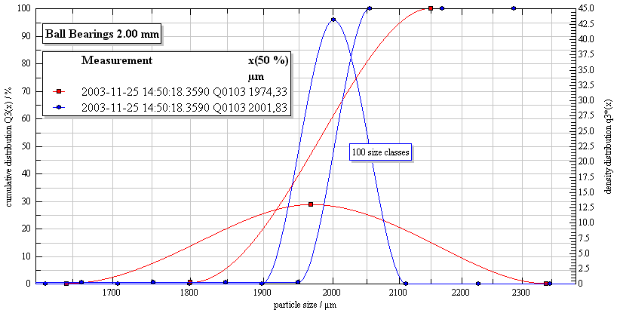


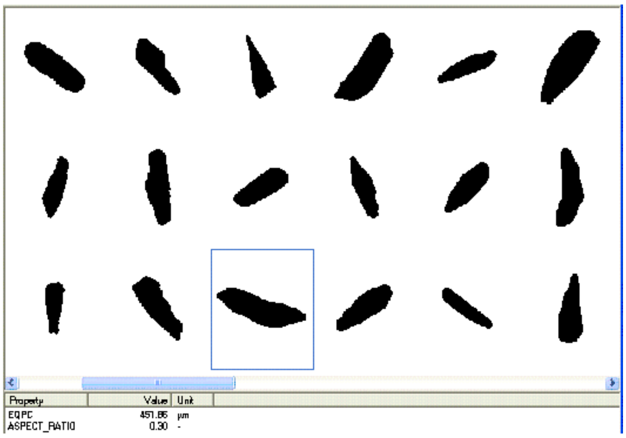
Figure 7: Size distributions Q3(x) and q3*(x) for ball bearing beads with 2000 µm nominal width 31 size classes (squares) and 100 size classes (circles)

5.2 Particle Shape

The shape parameters aspect ratio or sphericity are displayed as distribution function or as a function of particle size.

5.3 Particle Gallery

From the video stream an image gallery of individual particles can be generated, which fulfill a combination of rules defined by the user. Fig. 8 shows the selection of particles with 100 µm < EQPC < 1000 µm and aspect ratio < 0.4.



Property	Value	Unit
EQPC	451.86	µm
ASPECT_RATIO	0.30	-

Figure 8: “particle gallery” of SiC particles, the parameters of the marked particle are displayed in the bottom lines

This feature can be used for finding the “needle in a haystack”. Back traceability enables the link of an individual particle of the particle gallery to the image in the video stream with this particle highlighted.

5.4 User defined Evaluations

This feature allows for the evaluation of size and shape information for those particles only, that satisfy a combination of definable rules (i. e. like with the particle gallery). For example it is possible to calculate size distribution of the components of mixtures, as long as they differ in size or shape parameters. Further more, ball bearing beads can be used for in-situ calibration by adding them to the sample and focusing the evaluation to perfect spheres only.

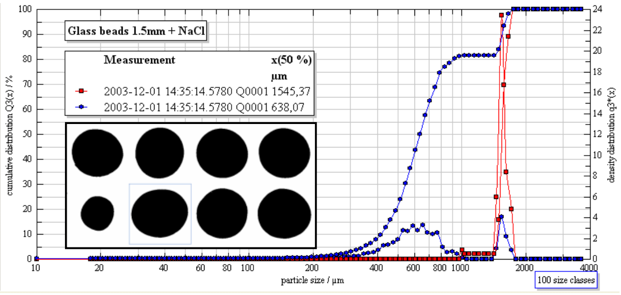


Figure 9: Mixture of glass beads and NaCl. Selection of articles with EQPC > 1000 µm and sphericity > 0,9

Fig. 9 extracts for example all spherical glass beads from the cubic Sodium Chloride and calculates the size distribution for the beads only (squares).

6 CONCLUSION

A new analyzer for particle size and shape was developed, which combines for the first time DIA in the range of 1 µm to 10 mm with well established dry and wet dispersing units. This was possible due to a new developed pulsed light source which generates exposure times of about 1 ns, so clear images of fastest particles are taken. In addition, the high acquisition rate of up to 500 images per second guarantees high particle counts resulting in a high statistical relevance of the results.

The effective data compression without loss of information enables retrospective modifications of the evaluation and visualization. Various different size and shape factors can be used. Features like “particle gallery” and “evaluation of user specific fractions” are powerful tools for the investigation of specialties. The use of a very fast data base in combination with a strict client-server structure of the software accomplishes high data volumes and manages concurrent operability with Laser Diffraction, Ultrasonic Extinction and Photon Crosscorrelation sensors on the same data base.

REFERENCES

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[3] K. Leschonski, S. Röthele, U. Menzel, Entwicklung und Einsatz einer trockenen Dosier-Dispergiereinheit …; Part. Charact., 1, 161-166 (1984)