

Technology Offer

Automated real time determination of crystal size and 3D shape distribution to quantify and predict the properties and dynamics of the crystallization processes

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To monitor crystallization processes a special camera setup to measure the crystal size and 3D shape distribution was developed.

The setup consists of two high-speed cameras that are positioned orthogonally to one another such that a precise overlap of both fields of view can be achieved. The setup further contains a specially designed flow-through cell that allows to observe the crystal suspension from both camera directions and given the opportunity to temperate the solution within the cell. In addition also a specialized software-package to determine the crystal size and 3D shape distribution automatically from the collected camera image-sets in real time is available.

Background

In the optimization and monitoring of crystallization processes, the continuous measurement of the crystal size distribution is still a challenge. Image-based methods have proven to be advantageous in this context, since they contain information about the crystal size and shape and the individual crystal faces. In particular, online microscopes, which are fed with the suspension to be measured via a bypass, offer the advantage of evaluating a statistically significant number of objects in a short time. To date, commercially available online microscopes use flow cells for this purpose, in which the crystals flowing past are recorded from one direction via a viewing window by means of a light source and a camera. For complex crystal shapes, overlapping objects or agglomerates, the information contained in the image data may no longer be sufficient to determine the particle size or shape.

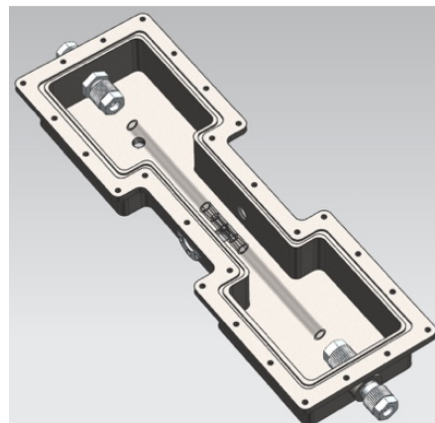
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At the Max Planck Institute for Dynamics of Complex Technical Systems, an image-based measurement system was therefore developed which simultaneously records the suspension to be measured at exactly one point from two different directions. The system consists of a temperature-controlled flow cell, a high-speed camera system, a setup for adjusting and calibrating the camera-cell unit, and a software package for automated evaluation of the recorded image data.

The flow cell contains a glass cuvette, which has a square cross-section with optically favorable properties in the receiving area.

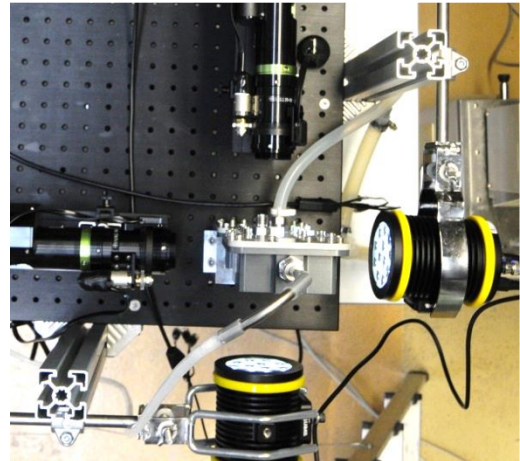
The cuvette is placed in a hollow body parallel to four quartz sight glasses, ensuring an unaffected light beam path.

The closed housing is used to hold a fluid medium for temperature control of the suspension.



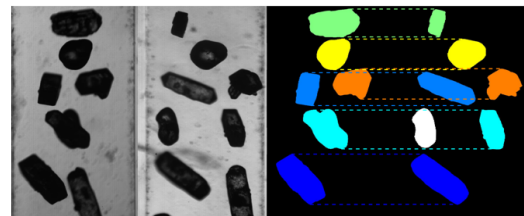


Two high-speed cameras with LED light sources at the height of the viewing windows record the objects carried in the suspension with variable resolution and frequency. Both cameras are controlled so that the same objects are recorded at the same time. This requires precise positioning of the cameras in space and axially to the flow cell sight glass windows and the glass cuvette, for which a special mounting system was designed. A separate calibration block, which can be placed at the position of the cell holder, is used to accurately calibrate the captured image pixels to spatial coordinates via a microscale.



For quantitative evaluation of the image or video sets obtained by the two cameras, it is necessary to identify the detected images of the individual crystals in the image data of the two cameras.

For this purpose, software routines were developed that are able to process these tasks automatically in real time. The developed software also allows to assign different scalar size values to the detected crystals (which are suitable for the determination of a univariate crystal size distribution) and to reconstruct the three-dimensional crystal shape reliably from the obtained image data.



Analysis of overlapping crystals, on the left images from the two high-speed cameras, on the right the correlating schematic representation based on the software calculation.

The crystal population can thus be described by a multivariate crystal shape and size distribution, which can be used to quantify and predict the properties and dynamics of the crystallization process.

Patent Information

"Flow measuring cell"

DE priority application DE102019132218A1 filed 27.11.2019

PCT patent application WO2021105318A1 filed in 27.11.2020

"Method for calibrating a system, system, flow meter assembly and storage medium"

DE priority application DE102019132219B4 filed 27.11.2019, granted 21.10.2021

Additional qualified knowhow and software package

Contact

Dr Lars Cuypers

Senior Patent- & License Manager

Chemist

eMail: cuyper@max-planck-innovation.de