

A backscatter imaging and light scattering system for monitoring of crystallization processes

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A miniature optical probe (16 mm in diameter and 300 mm length) is developed for diagnostics of crystallization processes. A probe volume is produced near the optical window at the end of the cylindrical probe. The probe volume consists of a CW laser beam spot of about 50 micron diameter, which is viewed by a photodetector. Crystals crossing an ellipsoidal region of 50 μm diameter and 100 μm length scatter CW light into the photodetector. Increase in the photodetector output (caused by the light scattered off a crystal) triggers a camera and a pulsed laser. The pulsed laser spot size and the camera viewing area are significantly larger (~500 μm in diameter) than the CW spot size, so the crystals are fully illuminated and imaged. Multimode optical fibers are used to bring the CW light and the pulsed laser light to the end of the cylindrical probe. A multimode fiber and an imaging fiber bundle are used to collect the scattered CW light and the crystal image respectively. A thermistor is also located at the end of the probe for monitoring the crystallizer temperature. All the measured data is transferred to a computer via IEEE 1394. A software displays images in real-time and is capable of post-processing and displaying data in various forms, such as correlation between crystal size and temperature. Measurements of a crystallization process have shown that the sudden growth of crystallization nuclei is accompanied by a rise in the solution temperature due to the heat of crystallization, as shown in Figure 1. Further, correlation between the measured crystal size and the aspect ratio is used to predict the three-dimensional structure (habit) of the crystals. In the results reported here, a positive correlation is found (see Figure 2), i.e. aspect ratio increases with the effective particle size. This correlation is indicative of rod-like crystals. A negative correlation is expected for plate-like crystals. In-situ determination of crystal habit is valuable in pharmaceutical applications; e.g. plate-like crystals are suitable for making drug tablets as they provide larger area for adhesion.

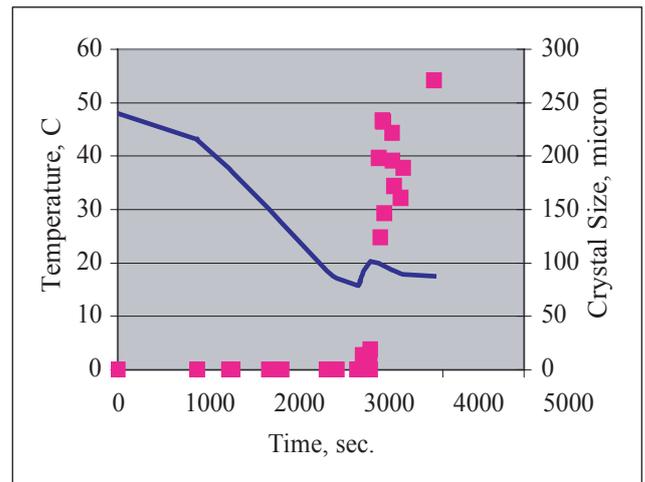


Fig. 1 Crystallizer temperature and the crystal size as a function of time

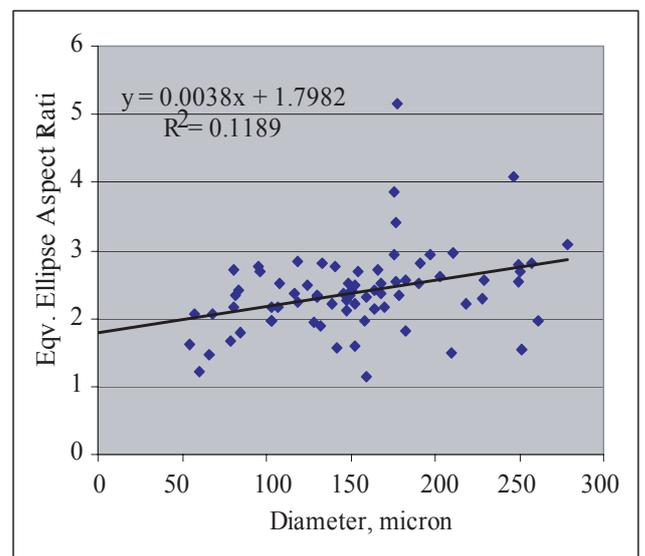


Fig. 2 Aspect ratio versus crystal size (Equivalent Diameter) validating rod-like crystal structure