ONLINE MONITORING OF CRYSTALLIZATION PROCESSES USING DIGITAL HOLOGRAPHY

T. Khanam, A. Rajendran, V. Kariwala, A. K. Asundi [a]

School of Chemical & Biomedical Engineering, Nanyang Technological University, Singapore (arvind@ntu.edu.sg)
[a] School of Mechanical & Aerospace Engineering, Nanyang Technological University, Singapore

Keywords: Crystal size distribution, Digital holography, Image analysis algorithm, Imaging technique, Online monitoring

Particle imaging has been proven to be an effective technique for online particle size and shape measurement.[1,2] These methods extract the information regarding particle size and shape from the captured images through image analysis. The successful application of image analysis algorithms for conventional imaging systems, however, is often hindered by the problems arising due to limited depth of focus imposed by required magnification as well as undetermined perspective position of the particles in the solution [3]. This issue becomes even more pronounced for non-spherical particles, especially needle-like particles, as their orientations cannot be measured. Thus, conventional imaging systems provide 2D information about the 3D particles, which can result in inaccurate inference of particle's 3D size and shape [3,4].
Digital holography (DH) is a three dimensional imaging technique that overcomes some of the limitations associated with the conventional imaging techniques. DH uses coherent light to record the interference pattern between the light that is transmitted through the object and that is diffracted by the objects. This interference pattern can be numerically focused (reconstructed) at different planes of the scene. Thus, 3D information can be obtained using a single hologram by reconstructing it at different depths [5]. Thus, DH is free from the out-of-focusing and the problem of undetermined perspective position of the object in a 3D scene.

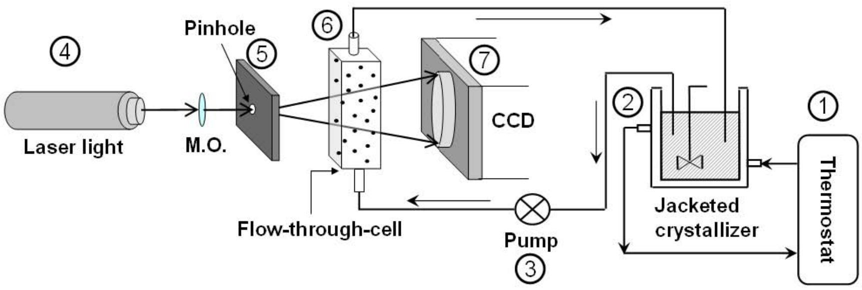

Figure 3. Schematic set-up for on-line imaging of crystallization using digital holography: 1. Thermostat; 2. Jacketed reactor placed on a magnetic stirrer; 3. Liquid circulating pump; 4. Laser; 5. Pinhole; 6. flow-cell and 7. Imaging device, CCD.

In our earlier work, we have studied the measurement of spheres [6] and fibers[7, 8] in solution using digital holography. The unique advantage of holography in measuring real lengths of randomly oriented microfibers in a solution without *a priori* information about their orientation has been presented and validated. With the use of a microscope objective, we have also demonstrated the application of this technique to measure near-micron sized particles.

In this work, we demonstrate the use of digital holography for online monitoring of crystallization process. The measurement system developed for this study, shown in Figure 1, consists of a jacketed crystallizer with a sampling loop. A portion of the solution is pumped through a flow cell, where the holograms are recorded. This information is processed using a customized image analysis software that extracts metrics, *viz.* size, and shape, from the recorded holograms. The cooling crystallization of oxalic acid from water is considered as a model system. Different cooling rates (rapid and slow) have been applied and the growth of the crystals has been monitored. From the holograms, the growth rate of crystals and the evolution of crystal shapes are measured quantitatively. The measurements from the holographic system are compared with those obtained from microscopy, and the two are found to be in excellent agreement; see Figure 2.

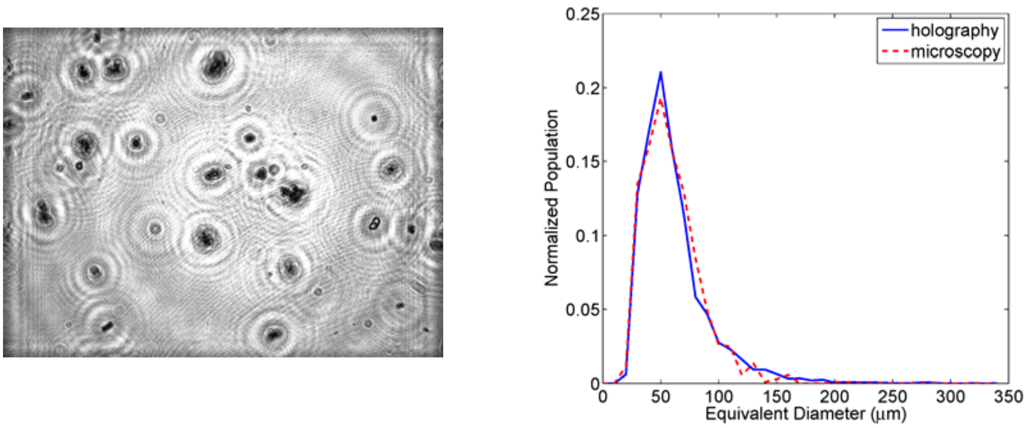

Figure 2. Measurement of transparent crystals of oxalic acid. (Left) A reconstructed image of oxalic acid crystals in solution; (Right) Crystal size distribution of population of transparent crystals in saturated solution compared to those obtained from microscopy.

References:

[1] Eggers, J., Kempkes, M. and Mazzotti, M. Measurement of size and shape distributions of particles through image analysis. *Chem. Eng. Sci.*, **2008,** *63*, 5513-5521.

[2] Larsen, P. A. and Rawlings, J. B. The potential of current high-resolution imaging-based particle size distribution measurements for crystallization monitoring," *AIChE J.*, **2009,** *55*, 896-905.

[3] Wang, X. Z., Roberts, K. J. and Ma, C., Crystal growth measurement using 2D and 3D imaging and the perspectives for shape control, *Chem. Eng. Sci.*, **2008**, *63*, 1173-1184.
[4] Kempkes, M., T. Vetter, and M. Mazzotti, Measurement of 3D particle size distributions by stereoscopic imaging. *Chem. Eng. Sci.*, **2010,** *65(4),* 1362-1373.
[5] Schnars, U. and Juptner, W., *Digital Holography: Digital Hologram Recording, Numerical Reconstruction, and Related Techniques*, Springer, Berlin, 2005
[6] Darakis, E., Khanam, T., Rajendran, A., Kariwala, V., Naughton, T. J. and Asundi, A. K., Microparticle characterization using digital holography, *Chem. Eng. Sci.*, **2010**, *65*, pp. 1037-1044
[7] Kempkes, M., Darakis, E., Khanam, T., Rajendran, A., Kariwala, V., Mazzotti, M., Naughton, T. J. and Asundi, A. K., Three dimensional digital holographic profiling of micro-fibers, *Opt. Express*, **2009**, *17*, 2938-2943.
[8] Khanam, T., Mohammad, N.R., Rajendran, A., Kariwala, V., Asundi, A.K. Accurate size measurement of needle-shaped particles using digital holography, Chem. Eng. Sci, Under Review.