高輝度 XAFS グループ

Time-resolved XAS Experiment Using a Microfluidic Cell

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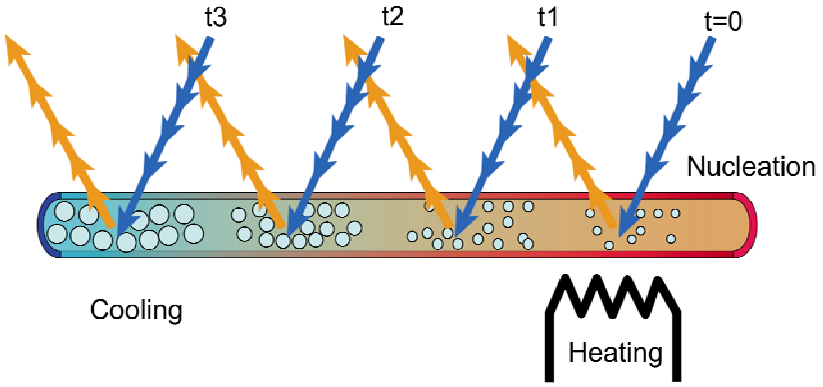
Microfluidic cell used in biomedical research is an ideal research tool for time-resolved chemical reaction in synchrotron radiation research. Originally for *in-situ* observation of a living cell, this technology allowed us to perform time-resolved experiments in a position-sensitive manner. Here we report the *in-situ* XAS studies on chemical reaction used to synthesize nanoparticles. Colloidal semiconductor nanocrystals, sometimes called quantum dots, have attracted significant attention in recent years due to their size-tunable optical properties and a variety of applications ranging from solar cells, lasers, electroluminescence devices to fluorescent labels in biological imaging. One of the most important routes of synthesizing CdSe nanocrystals is chemical growth in colloidal solutionsPrinciple of the measurement system is schematically illustrated below. Along a microfluidic cell (diameter: 250 microns), x-ray beam spot of the same size is shifted and position dependence of fluorescence yield is detected as a function of photon energy.



For precise positioning, high precision XY stage (usually used for monochlomator) is computer controlled over an area of 400 mm x 800 mm with a position error within 0.1 micron. As a fluorescence detector, silicon drift diode detector (SDD, Amptek XR-100SDD) and x-ray filter are used to record the fluorescence line intensity. Experiments were performed at BL13B (MPW) of Photon Factory and at NW2 (Undulator) of AR. As microfluidic reactor records time-dependent spectral change as position-dependence,[1] one can record time-resolved EXAFS spectra without degrading statistics.[2] We find that detailed analysis of kinetics yields diameter *D* and concentration *N* of nanocrystals.[3] As the proposed method can provide fundamental parameters (*D*, *N*) of nanocrystals *in-situ*, initial process of nanocrystal growth became feasible.[3] We measured the Se *K*-edge EXAFS spectra for CdSe nanocrystals along a microfluidic reactor channel which indicated strong time-dependence of the nucleation and growth upon the initial process of the reaction. A rapid increase of the reaction yield within several seconds was observed. It is found that after injection of starting materials, the nucleation occurs abruptly and the CdSe nuclei concentration reaches the maximum and then declines rapidly. Detailed analysis of EXAFS data combined with UV-vis spectra led to reliable estimation of fundamental Nanocrystal parameters which are used to optimize growth condition. Current status of microfluidic *in-situ* EXAFS and future prospects are discussed.

[1] H. Nakamura *et al.*, *Chem. Commun.* 2844 (2002).
[2] M. Uehara *et al.*, Appl. Phys. Lett. 94, 063104 (2009).
[3] Z. Sun *et al.*, J. Phys. Chem. C 113, 18608 (2009).