REALTIME X-RAY FLUORESCENCE MOVIE OF CALCIUM AND IRON IN GROWING CHEMICAL GARDEN

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The present talk reports a realtime X-ray fluorescence (XRF) movie technique developed in laboratory, which enables the observation of chemical diffusion in growing chemical garden. Chemical garden has a plant-like structure, which grows upward from metallic salt particles in sodium silicate solution. For a long time, the growth process of chemical garden is monitored by photos or videos, which only show the appearance change but are not able to reflect the spatial distribution change of different elements. The present research developed a laboratory XRF movie technique and then utilized it to visualize the diffusion of calcium and iron in chemical garden. The X-ray source is a monochromatic copper Kα beam from a 1.5 kW X-ray tube of copper target. The beam size is 1 mm (V) × 5 mm (V). The grazing-incidence angle between the X-ray beam and the vertical sample surface is 6 °, hence the illumination area on the sample is 10 mm (H) × 5 mm (V). A 50-μm-dia pinhole is placed in front of the sample to project fluorescent X-rays to the detector. The detector is a conventional CCD camera. It serves as a 2D X-ray energy detector when it is used in single photon counting mode. Therefore, it is able to resolve the respective XRF images of different elements. The sample is the chemical garden structure in a thin reaction vessel. The dimension of the reaction vessel is approximately 27 mm (H) × 27 mm (V) × 0.5 mm (thickness). Its side surface which receives the grazing-incident X-ray beam as well as faces to the detector is made of a 50-μm-thick polyester thin film to allow X-rays to penetrate. A pellet of mixed calcium chloride and ferrous sulfate is placed at the bottom of the reaction vessel. To start the experiment, sodium silicate solution is injected into the vessel. In the 15 hours growth process, the detector continuously records the XRF signals from the sample. In the final, the XRF movies of calcium and iron show that they have different diffusion behaviors in the growing chemical garden. After the growth, they concentrate to different positions and therefore they are partially separated, though they are finely mixed before the growth. The typical time resolution of the X-ray movies can reach 3 minutes. The present research helps understand the formation mechanism of chemical garden. In addition, it reveals the great advantages of using the X-ray movie technique for topics on chemical diffusion, as well as the operando analysis for samples in reaction.