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Time-Resolved In-Situ XANES of Nanoparticle Growth
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Introduction: In nanoparticle syntheses, the products are often critically dependent on reaction conditions, including the time for which the reaction is allowed to proceed. For this reason, it is important to obtain the kinetics and mechanism of the reaction. One powerful tool for doing this is x-ray absorption spectroscopy, since the local environment of many elements can be determined as the reaction proceeds.

Methods and Materials: Nanoparticle of cobalt, iron, and iron oxide are being synthesized by two methods. In the polyol synthesis, metal precursors such as iron carbonyl are suspended in polyol and the solution is taken to reflux, resulting in the nucleation and growth of nanoparticles. In the reverse micelle process, water, a hydrocarbon, and a surfactant are combined so as to produce water-filled micelles on the nanoscale. When water-soluble precursors are added to the solution, nanoparticle growth occurs until the micelle is filled.

In both cases, the solution was pumped through a custom-designed flow cell as the reaction was proceeding, with the x-ray absorption near edge structure (XANES) being collected in transmission at roughly two-minute intervals. In some cases extended x-ray absorption fine structure (EXAFS) was instead collected at ten-minute intervals.

Results: For the polyol process, time-resolved XANES showing the formation of iron oxide particles was successfully obtained (see Fig. 1). In the case of the reverse micellar processes, the reaction evolves gas, which adds noise to the transmission spectra. Efforts are currently underway to overcome this problem.

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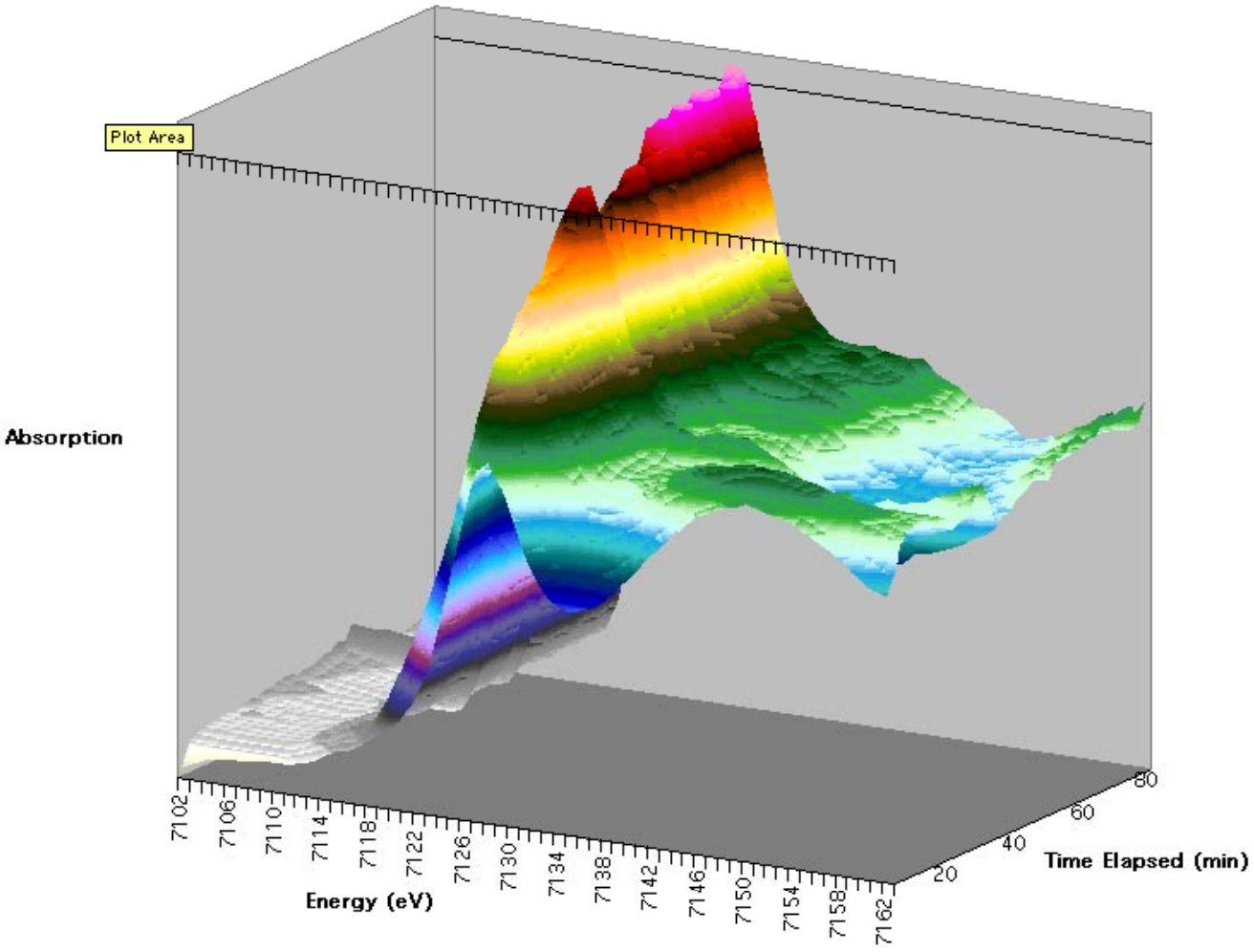

Figure 1. Time-resolved XANES of iron oxide formation using a polyol process. The starting material is iron carbonyl.