School on Nanostructured Materials
Characterization by Synchrotron Light based Techniques

18th - 23rd November 2007. La Plata, Buenos Aires. Argentina

In-situ Time-resolved X-ray Diffraction and the Characterization of Nanomaterials

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Abstract

Investigations at Brookhaven National Laboratory have established the feasibility of conducting sub-minute, time-resolved *in situ* x-ray diffraction (XRD) experiments under a wide variety of temperature and pressure conditions (80 K < T < 1200 K; P< 50 atm) [1]. This important advance results from combining the high intensity of synchrotron radiation with new parallel data-collection devices [1]. Using time-resolved XRD, one can get information about [1-3]:

- Phase identification and composition of catalysts under reaction conditions
- Kinetics of crystallization of nanoparticles and bulk solids
- Crystallite size as a function of time/temperature
- Identify crystalline or amorphous intermediates during phase transitions occurring in nanoparticles or bulk solids
- Real-time crystal structure refinement

Examples of problems studied to date with time-resolved XRD and related to catalysis include [1,4-15]:

- Hydrothermal synthesis of zeolites
- Hydrothermal conversion of zeolites
- Binding of substrates and nanoparticles in zeolites
- Reduction/oxidation cycles in oxide catalysts
- Phase transformations in oxide catalysts for the partial oxidation of hydrocarbons and the water-gas shift reaction.
- Sulfidation of oxide precursors for HDS catalysts

- Regeneration of S-poisoned oxide catalysts
- Synthesis of metal phosphide catalysts

In this presentation, we will review the instrumentation for *in-situ* time-resolved X-ray diffraction and a series of recent works [9-15] that illustrate its uses for studying the behavior of different oxide nanostructures under reaction conditions.

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