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Report:

Time resolved in-situ synchrotron X-ray powder diffraction is a very powerful tool for studying synthesis and chemical reactions involving microcrystalline materials. We have designed and constructed a facility for time resolved in-situ powder diffraction studies in cooperation with the GILDA beam line, and the present experiments were the first experiments performed using this facility. The facility consist of a capillary based micro reaction cell, a hot air heater, and a Translating Imaging Plate (TIP) camera for collecting time resolved powder diffraction data.

The aim of the present experiments were to follow hydrothermal syntheses of zeolites and microporous aluminophosphates in-situ, in order to gain information regarding crystallization kinetics and mechanisms. The experiments are part of a project studying nucleation and crystallization of aluminosilicate zeolites in zero gravity.

Three systems were investigated: synthesis of zeolite LTA using activated clay materials as starting materials, formation of zeolite P from a clear aluminosilicate solution, and formation of microporous aluminophosphates and transition metal substituted aluminophosphates..

The syntheses were performed in 0.7 mm quartz glass capillaries heated using a hot air stream. In order to obtain hydrothermal conditions at temperatures up to 200°C, a nitrogen pressure was applied inside the capillaries.

The temperature dependence of the crystallization kinetics for the aluminosilicate zeolites was investigated by performing isothermal experiments. Integrated intensities of diffraction lines were used to determine crystallization curves. Kinetic analysis will be performed.

For the hydrothermal synthesis of microporous aluminophosphates, the dependence of crystallization behaviour, end product, intermediate and precursor phases were investigated. Temperature ramps from RT to 200°C were used. The template used was di-n-propylamine, and except for the type of transition metal, the chemical composition of the starting gel was not varied. Crystallization of pure aluminophosphates as well as Mg, Co, Zn, and Mn substituted materials were studied. A pronounced structure directing effect of the transition metal cation was observed. The occurrence of precursor and intermediate phases was also strongly dependent on the type of transition metal cation present.

The figure shows an example of a 3-dimensional representation of the time resolved powder diffraction patterns during hydrothermal synthesis of the Mn-substituted microporous aluminophosphate MAPO-11. The disappearance of a precursor phase, and the crystallization of the end product is clearly visible.