Micro-diffraction
GeniX Cu High Flux

Application Note n° AN-G9

Xenocs
Reflecting Future Technology

A b s t r a c t

Monitoring fast kinetic reaction in case of clays dehydration is an issue of major importance. Traditional X-ray sources performances are not sufficient to reveal very short time scale events.

At NHM a microfocus sealed source was replaced by the high brightness GeniX Cu High Flux beam delivery system which made it possible to well-resolve XRD patterns within one second-snapshots even with a 100 µm illumination on the sample.

NHM is now able to resolve all three water-interlayer states and sequential transitions within an experiment of only 200 seconds. Latest results acquired with the GeniX indicate that the real lattice response in clay on water loss is probably much quicker and more uniform than previously reported with the previous setup.

NATURAL HISTORY MUSEUM

Fig. 1 : The GeniX Cu High Flux X-ray source installed at the Mineralogy Dept. of the Natural History Museum in London.

Monitoring of dehydration kinetics in clays

Data courtesy of Dr Gordon Cressey & Dr Jens Najorka, Dept. of Mineralogy, Natural History Museum, London, UK.

I n t r o d u c t i o n

The dehydration of clay was studied on Na-montmorillonite. Under humid conditions, this clay can adsorb up to 3 interlayers of water into its structure. Removal of interlayer water is accompanied with a step-wise collapse of the clay lattice which is detectable using X-ray diffraction techniques (Fig. 2). However, the transition from one hydration state to the other occurs within seconds and is very difficult to follow (with conventional X-ray sources).

In this study, we were able to resolve such ultrafast transitions by applying extremely short counting times, which were achieved by using a GeniX Cu High Flux X-ray source in combination with a static position sensitive detector.

E x p e r i m e n t

A slurry sample of Na-montmorillonite (Kunipa-F) was prepared and a thin film was placed on a zero background holder. A GeniX High Flux (Cu Kalpha) system equipped with FOX2D CU 10_30P mirror was used and operated at 50kV and 1mA (Fig. 1). Exceptionally strong intensities of the clay diffraction peaks were observed, which made it possible to reduce counting times down to 1 second per pattern. A sequence of 200 one-second ‘snapshots’ was recorded. Measurements of this study were restricted to a rather small area of the sample by using a 100µm pinhole. This was advantageous because it minimized effects from locally different onsets of clay dehydration because of slightly uneven water distribution in the sample.

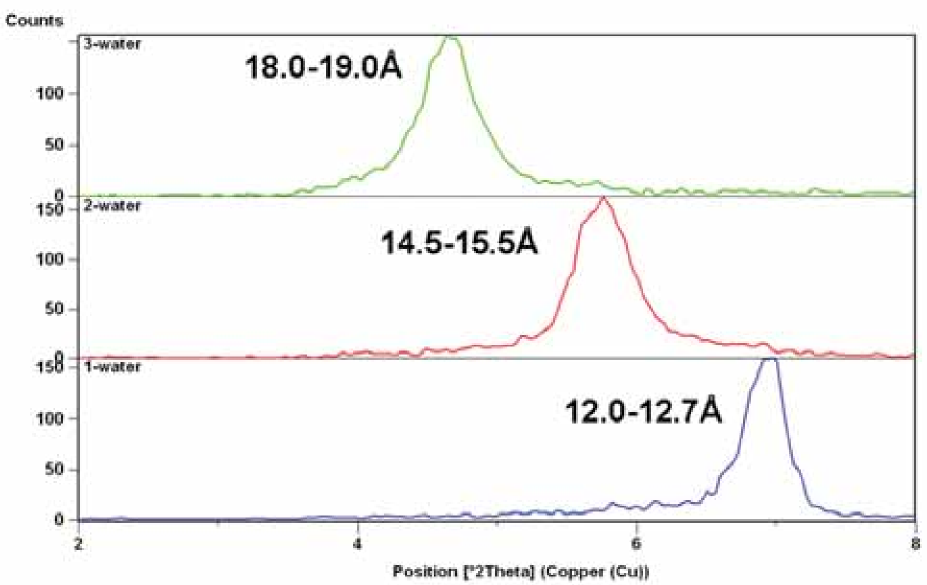

Fig. 2 : The basal (001) reflection of Na-montmorillonite clay at 3 different states of dehydration: Top…3-water-interlayer, Middle…2-water-interlayer, and Bottom… 1-water-interlayer. Removal of interlayer water results in collapse of the clay lattice which decreases the d(001) spacing.

Figure 3 shows the evolution of the basal (001) reflection with progressing dehydration of Na-montmorillonite. The dehydration starts with formation of the 3-water interlayer state (Peak at 18.8Å). After 40 seconds, this 3-water layer peak becomes less intense and the 2-water layer peak forms (14.7-16.0Å). After 90 seconds, the 1-water layer peak (12.7Å) develops at the expense of the 2-water layer peak. The final 1-water interlayer state represents the stable configuration for the Na-montmorillonite clay at given conditions (standard RT and RH).

Figure 4 shows a 2D-projection of Figure 3. The sequential character of the dehydration reaction becomes clearly visible. One state dominates at a time. The transition from one state to another takes place within seconds. Increase of peak intensity within one hydration state is due to increasing alignment/degree of ordering within the clay interlayer. Release of interstitial water could explain additional peak shifts (e.g. 14.7-16.0Å).

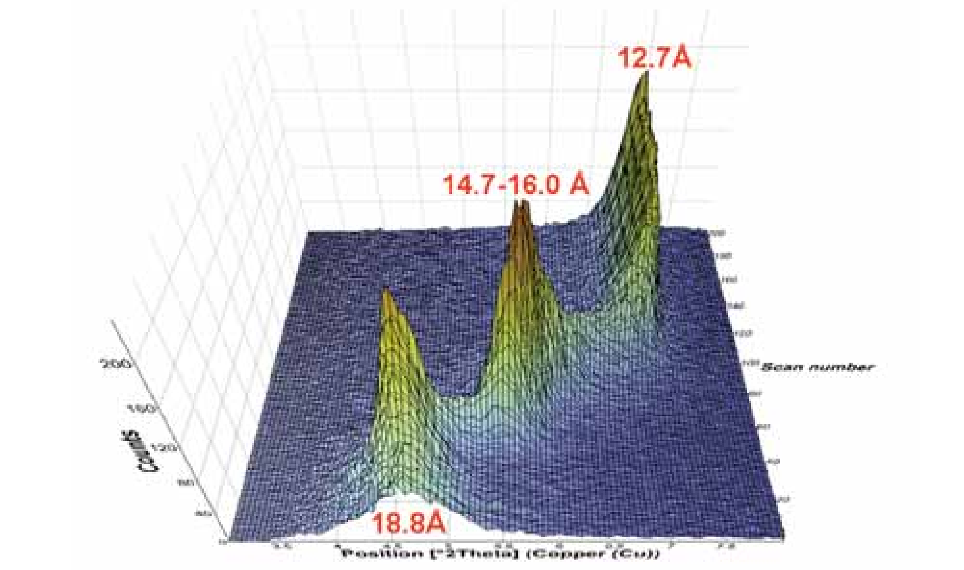

Fig. 3 : Development of the basal (001) reflection with time during dehydration of Na-montmorillonite. All three states of clay dehydration (3-, 2-, 1- water interlayer) become resolvable using one-second X-ray snapshots.

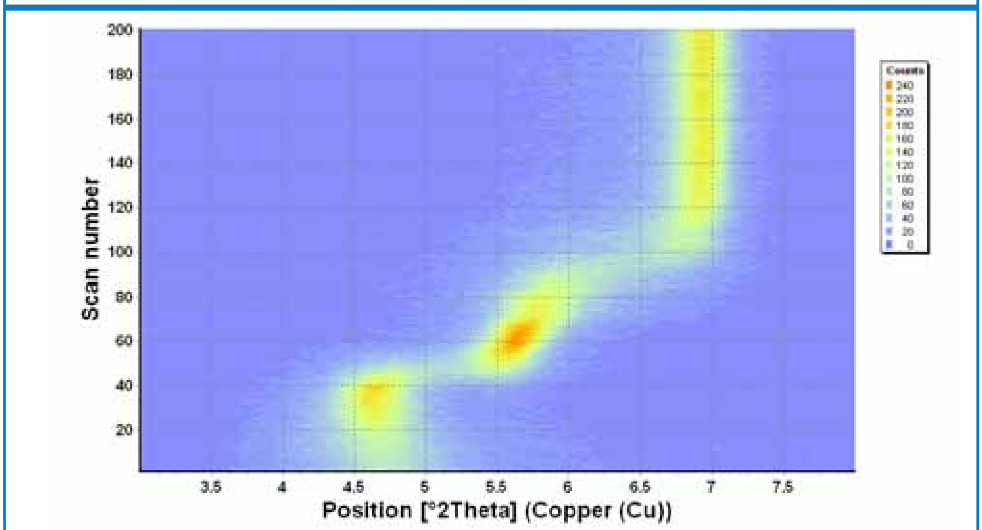

Fig. 4 : 2D-projection of Fig. 3. The dehydration follows a sequential-type of reaction path. Transitions with two dehydration stages are restricted to very short periods of time.

C o n c l u s i o n

Owing to the high brightness of the GeniX X-ray source, well-resolved XRD patterns could already be obtained within one second-snapshots (peak intensity up to 150 counts). The system is capable to monitor extremely fast kinetic reactions. In case of clay dehydration, the rapid-type of data collection made it possible to resolve all three water-interlayer states and sequential transitions within an experiment of only 200 seconds. It indicates that the real lattice response in clay on water loss is probably much quicker and more uniform than previously reported.

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