

s5.m17.02 **Direct Determination of Defect Structures in Kaolin Minerals by High-Resolution Transmission Electron Microscopy (HRTEM).** Toshihiro Kogure, *Graduate School of Science, the University of Tokyo, Japan. E-mail: kogure@eps.s.u-tokyo.ac.jp*

Keywords: Kaolin; HRTEM; Stacking Faults

Kaolinite is among the most abundant clay minerals and an important resource for industries. In addition, the mineral has been attracting many clay mineralogists because it shows diverse structural variations related to stacking defects. In X-ray powder diffraction, these defects or stacking disorder are mainly evaluated by the diffraction profiles with $k \neq 3n$. However, these stacking defects have not been completely understood yet and discussion has been continued since, e.g. Brindley and Robinson (1946) [1]. In the present study, near-atomic resolution TEM imaging has been successfully applied to determine the stacking defect structures in kaolin minerals, especially in kaolinite [2]. The specimen studied is at mid-stage of the depth-related kaolinite-to-dickite transformation in a sandstone reservoir. Although electron radiation damage is a serious obstacle, a number of high-quality images were recorded on films, in which the tetrahedral and octahedral positions in a kaolinite unit layer were clearly resolved. Electron diffraction and high-resolution imaging of dickite showed that few stacking defects exist in this polytype. On the other hand, kaolinite crystals contain high density of stacking defects. These defects or stacking disorder are formed by mixture of the two kinds of lateral interlayer shifts, t_1 (approximately $-a/3$) and t_2 ($-a/3 + b/3$), between adjacent layers. Disorder by the coexistence of B layer and C layer, or dickite-like stacking sequence was never observed. These results provide not only an unambiguous settlement for the long controversy of the defect structures in kaolinite, but also a new clue to understand kaolinite-to-dickite transformation mechanism.

- [1] Brindley, G.W. and Robinson, K. (1946) *Transaction of Faraday Society*, **42B**, 198-205.
 [2] Kogure, T. and Inoue, A (2004) *American Mineralogist*, in review.

s5.m17.03 **Pushing the Limits of Lab X-ray (Micro) Diffraction.** Caroline Kirk and Gordon Cressey, *Department of Mineralogy, The Natural History Museum, Cromwell Road, London SW7 5BD, United Kingdom*

Keywords: Mineralogy; Microdiffraction; High-Temperature

In the Department of Mineralogy, we have coupled a high brightness X-ray source (Bede MicroSOURCE) to a rapid X-ray detector (INEL CPS120). This combination has allowed us to trade off some of the brightness and use a smaller beam for microdiffraction-type applications or use the full intensity of the beam to investigate very rapid phase transitions at non-ambient temperatures.

Lab microdiffraction is not a new concept; Debye-Scherrer and Gandolfi methods have been routinely carried out for decades. However, the main drawback with these techniques is that data collection times are long (many hours-days). Data collection times with the Bede MicroSOURCE/Inel Position Sensitive Detector are short; only a few minutes are required.

This presentation summarises a few of the problems tackled with this set-up:

Within a broken sample of Carbonado Diamond, a patch, approximately 300 μ m in diameter, of delicate crystals (10 μ m or less in diameter) with different morphologies, was discovered. Electron Microprobe analysis found the crystals contained varying amounts of **Ag, Ti, Cu** and **N**. In order to identify the phases present, *in situ* XRD was carried out; five phases were identified in this specimen three of which were found to be new minerals. This demonstrates how particles, or grains can be analysed *in situ* as a specific part of a polished block/rock sample.

Dilute microdiffraction was carried out to analyse particulates collected on a polycarbonate filter, used to monitor the amount of Pb in contaminated air. Elemental analysis was carried out using SEM-EDS, but XRD was required to identify the phases present. The SEM analysis determined the approximate amount of particulates on a 1cm² piece of filter to be 147 μ g. By rotating the sample non-centrosymmetrically, a large area of the filter was irradiated by the X-ray beam.

The Bede MicroSOURCE/Inel Position Sensitive Detector system can also be coupled to an Anton Paar Heating Stage. This allows very rapid phase transitions of materials to be investigated at non-ambient temperatures over subtle changes in temperature by taking “snapshots” of the diffraction data every few seconds. The dehydration stages of Melanterite, FeSO₄·7H₂O, have been investigated using this set-up. At temperatures close to ambient (25-40°C), very rapid structural changes to other hydrated iron sulphates, e.g. Rozenite, FeSO₄·4H₂O, have been detected.