## **NOTE**

## **A mixed layer kaolinite- smectite from Lower Silesia, Poland**

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MODERN mineralogical literature contains many papers on interstratified minerals, hut very few in which 1:1 (one tetrahedral per one octahedral) layers participate in the interstratification. Brindley and Gillery (1954) reported a mixed-layer kaolin-chlorite, and Sudo and Hayashi (1956) and subsequently Shimoyama *et al.*  (1969) described randomly interstratifled kaolin-montmorillonite from an acid clay in Japan. In the last mentioned cases, 2:1 (two tetrahedral per one octahedral) expanding layers of montmorillonite were distinguished from l : l layers, but no differentiation was made between halloysite, metahalloysite and kaolinite.

In Jeglova, Lower Silesia, western Poland, kaolin clay and well developed quartz crystals fill veins and cavities in metamorphic shield rocks associated with the granite intrusion known as the "Strzelin intrusion". This kaolin is a very plastic clay, fine grained and mostly white in color, but sometimes yellow or brown depending on the iron content. From the chemical composition and differential thermal curve (Morawiecki, 1953, 1962), the white clay was thought to be composed of kaolinie relatively free of chemical and mineral contaminations apart from quartz; a microscopic study found only well crystallized anatase, mica and quartz.

Recently this kaolin clay has been carefully examined by X-ray diffraction and rather unusual properties were discovered which cannot be attributed to a. normal kaolinite.

The X-ray diffraction pattern of a fully disoriented aggregate (Fig. 1) does not resemble that of a pure kaolinite. It differs in the number of reflections, their relative intensities, and the exact angular positions of the basal reflections. Such a pattern might result from a mechanical mixture of metahalloysite and kaolinite rather than pure kaolinite (Brindley *et al.* 1963). Electron micrographs, however, show a platy material (Fig. 2).

The platy shape of the individual grains is the main reason for the excellent orientation obtained by sedimentation on a glass slide surface. These characteristics indicate a kaolinite rather than a hallovsite type mineral.

It appeared during sample preparation, that the volume per gram of the wet clay from Jeglova was much greater than that of any other kaolin. Further swelling properties of the mineral in water and organic liquids were discovered. Swelling provides a good means for studying the nature of the structural layers. Oriented aggregates saturated with Na, Ca, ethylene glycol, glycerol were prepared. Samples also were heated to 380°C and to 600°C to check respectively on the layers after collapse and the remaining structure after kaolinite dehydroxylation. The oriented aggregates gave several orders of basal X-ray reflections. By comparing the results with those of the untreated and unheated sample, it was found that all the diffraction data were changed. The (001) peaks were shifted very distinctly, some towards lower, some towards higher diffraction angles. Deviations from an integral series of a type  $d_{000}/n$ , where *n* is an integer, were even greater than for the untreated sample.

Interpretation of the X-ray data has been performed by a visual method (Meting, 1949). The peak positions have been represented in reciprocal space (Fig. 3), and the angular breadth  $(\Delta\theta)^\circ$  of the reflections measured at half maximum intensity is indicated. From considerations of the non-integral series of X-ray reflections, the directions of peak shifts and the  $(\Delta\theta)$  values for four "orders" of reflection for the differently treated samples, I came to the conclusion that the observed  $X$ -ray data are due to a statistically random mixture of non-swelling kaolinite and swelling smectite (Brindley, 1966) layers. To confirm this hypothesis, the theoretical positions of four (001) orders of basal spacing of kaolinite equal to  $7.15 \text{ Å}$ (Brindley and Robinson, 1946) and (001) orders of



Fig. 1. X-ray diffractogram of a fully disoriented air-dried aggregate,  $d_{(001)} \approx 7.3 \text{ Å}.$ 

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Fig. 3. Schematic representation of observed and theoretical (00l) orders in reciprocal space. Along each horizontal line are shown the positions of integral Bragg reflections from the separate components, e.g.  $12.5 \text{ Å}$  and  $7.15 \text{ Å}$  components. When these occur randomly intermixed, broad non-integral scattering maxima are likely to occur as indicated by the heavy lines. The positions and widths of the observed maxima are indicated.

smectite with monolayer water  $(d_{(001)} = 12.5 \text{ Å})$ , two layers of water  $(d_{(001)} = 15.4 \text{ Å})$  and without interlayer water  $(9.8 \text{ Å})$  and also of ethylene glycol- and glycerolsmectite complexes have been marked in Fig. 3. It is easily seen in Fig. 3 that the experimental data are consistent with the calculated peak positions (thick horizontal lines) due to the various mixed-layerings. The angular breadths of the observed peaks also are very significant, being small if component orders are close together, and larger when they are farther apart.

The diffractogram of the sample heated to  $600^{\circ}$ C is specially significant and may be interpreted with the aid of the concept of Ross (1968). According to this idea we may explain the structure by assuming that the 2:1 type layers do not dehydroxylate during heating while the 1:1 layers do dehydroxylate; in this way we observe diffraction from domains composed of 2:1 layers separated by X-ray amorphous metakaolinite material.

An attempt was made to evaluate the proportions of kaolinite and smectite lavers in the clay size fraction, and was found to be about 80-90 per cent of kaolinite.

Further studies are being undertaken on the "statistical" structure of this material and also on the structure of the individual mixed-layer domains.

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Fig. 2. Electron micrograph (dispersion replica). Magnification 16,500  $\times$ .