TEM Observations of Coherent Stacking Relations in Smectite, I/S and Illite of Shales: Evidence for MacEwan Crystallites and Dominance of $2M_1$ Polytypism

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Abstract: TEM characterization of stacking relations in I/S of expanded shale samples from the Gulf Coast and Michigan Basin was carried out to address the issues of the degree of coherency and the nature of layer stacking sequences in smectite, I/S and illite. The two-dimensional lattice fringe images obtained from this study show that cross fringes are commonly observed to be continuous over at least 3 – 4 layers for smectite, 6 – 7 layers for ordered I/S and 9 – 10 layers for illite-rich I/S. This demonstrates that such sequences are coherent, or at least semi-coherent (in smectite) units (MacEwan crystallites). The observations demonstrate that so-called fundamental particles are fragments of MacEwan crystallites formed primarily as a result of disaggregation along weakly-bonded smectite interlayers. However, both 0k1 and h01 reflections may coexist in selected area electron diffraction (SAED) patterns. The frequency of occurrence of the coexistence in SAED patterns decreases in the order smectite, I/S and illite for Gulf Coast samples. This is consistent with the presence of turbostratically-related interfaces in packets of all of these materials. Therefore, any given layer sequence in smectite, ordered I/S and illite may have both turbostratic and coherent interfaces. The proportion of coherently-related layers increases with increasing proportion of illite-like layers. The concept of fundamental or elementary particles is not related to layer sequences in non-disaggregated, original rocks. Indeed, it implies relations that are not valid.

The lattice fringe images, SAED and optical diffraction patterns demonstrate that where layers are coherently-related, $2M_1$ is the dominant polytypic sequence in all samples. However, this periodic $2M_1$ stacking is so frequently interrupted by stacking faults in smectite that it gives rise to apparent $1M_d$ polytypism. The degree to which the periodic $2M_1$ sequences are interrupted by stacking faults decreases with increasing proportion of illite-like layers. The SAED patterns of I/S and illite unmodified since its formation are diffuse parallel to c^* and have poorly-defined, non-periodic reflections for indices $k \neq 3N$ as a measure of local ordering superimposed on poorly-ordered coherent sequences with a turbostratic component. X-ray diffraction (XRD) patterns, as integrated over domains with a range of heterogeneous stacking relations, do not represent simple mixtures of discrete 1M and $2M_1$ polytypes.

The observations of this study imply that dissolution-crystallization is a dominant mechanism for the smectite-to-illite transition. The semi-coherent stacking of smectite-like layers in smectite-rich samples implies that either a dissolution-crystallization process took place subsequent to deposition of detrital smectite or that Gulf Coast smectite is an in-situ alteration product of volcanic ash.

Key Words: $2M_1$ Polytypism • Coherent • Fundamental Particles • Illite • I/S • MacEwan Crystallites • Optical Diffraction Patterns • SAED • Smectite • Stacking Relations Cross Fringes • Transmission Electron Microscopy (TEM)

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