TEM/AEM study of the structural and chemical evolution of mixed-layer illite/smectite through illite in Basque-Cantabrian pelites

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The evolution of smectite through illite has been studied using a continuous sequence of argillaceous sediments from 8.000 m of core from the Basque-Cantabrian Basin. XRD data demonstrate that the sequence is uniquely complete from R = 1 illite/smectite throgh illite with < 5 % smectite; i.e.nearly pure illite (Arostegui et al., 1991). Because of collapse of smectite layers in the transmission electron microscope (TEM), both smectite and illite give similar 10-A 001 lattice fringes, but Guthrie and Veblen (1989) have determined the conditions under wich contrast differences in lattice fringe images can be obtained that permit differentation of illite and smectite layers.

We present compositions determined by analytical electron microscopy (AEM) and corresponding HRTEM images, obtained under overfocus conditions in order to differentiate between illite and smectite layers, for the sequence from R = 1 I/S through nearly pure illite. Photographs in which illite and smectite layers can be differentiated (e.g., Fig. 1) demonstrate that, although illite and smectite layers are locally ordered into regions with, for exemple, R = 1 order, the sequence of layers is quite variable on a local basis. Even in very small areas there is continuous change from true R = 1 I/S (50 % illite) through R>>3 illite-rich material. No true R = 2 or R = 3 I/S has been identified.

Two AEM analyses were obtained for each analyzed area; 30 second analyses were obtained for K contents in order to minimize volatization, and 200 second analyses for other cations. The analyses demostrate that the interlayer composition of I/S is extremely heterogeneous within a given sample, but with a mean value in agreement with the proportion of I/S predicted from XRD data (total interlayer cations for 10 oxygens: illite sample - 0.80; R = 1 sample - 0.42). The variation of proportions of other cations within I/S of a given sample is as great as that between the shallowest sample (R = 1 I/S) and the deepest (pure illite). However, the average TOT - layer composition of R = 1 dominant material is approximately equal to that of illite-rich material; that is, there is no general relation between proportion of illite layers and Si/Al ratio. The range of composition of ordered I/S is independent of proportion of illite layers.

The data collectively imply the presence of two metastable phases: (1) ordered R = 1 I/S (rectorite-like) (2) R>>3 I/S (nearly pure illite) with more than 90% illite layers. These phases occur intergrown and interlayered in the same sample, but the proportion of R = 1 I/S decreases with increasing depth, as a result of a temperature-controled sequential reactions governed by Ostwald step rule. The relative proportions of illite and smectite as determined by XRD are qualitatively in agreement with those determined by TEM, and are determined by the ratio of R = 1 I/S and nearly pure illite in the bulk sample.

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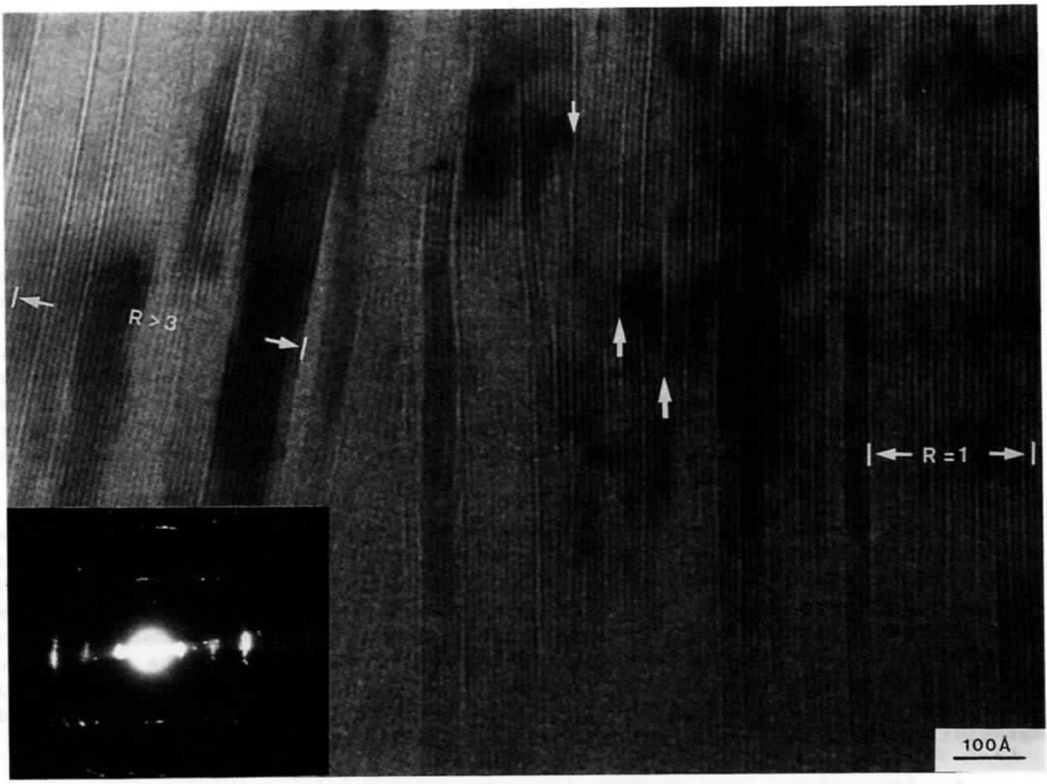

Fig. 1- HRTEM image showing coexisting R1 and R > 3 mixed layer I/S. Large arrows: separation of smectite layers due to beam-specimen interaction. Small arrow: Edge dislocation. Inset: 001 diffraction pattern of mixed-layered I/S, with weak chlorite and calcite reflections.

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