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Incommensurate modulation and the crystal structure of ganophyllite

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ABSTRACT

Incommensurate modulation in ganophyllite crystals was investigated with selected-area electron diffraction (SAED), high-resolution transmission electron microscopy (HRTEM), analytical electron microscopy (AEM), and single-crystal X-ray diffraction (XRD) techniques. The XRD data were used to perform a new subcell (a = 5.550 Å, b = 13.539 Å, c = 25.134 Å, β = 93.928°) refinement of the structure with a higher precision than previous refinements (R = 0.041). Although supercell reflections were too weak for collection with the diffractometer, the supercell was modeled by comparing experimental SAED patterns and HRTEM images to their simulated counterparts. These simulations indicate that incommensurate modulation arises from offsets in the location of inverted tetrahedra between adjacent [100] strips. The true supercell of incommensurate crystals involves a tripling of the subcell **a** axis and at least a twelvefold increase in the **b** axis; previously identified supercell reflections are actually aggregates of extremely closely spaced reflections in incommensurate crystals. Unlike commensurate crystals, the subcell **c** axis of incommensurate ganophyllite is not doubled. AEM data suggest that the occurrence of commensurate or incommensurate forms is not compositionally dependent.