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TEM-assisted dynamic scanning force microscope imaging of (001) antigorite: Surfaces and steps on a modulated silicate

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Abstract

Ultra-high vacuum dynamic scanning force microscopy (dynamic SFM) has been performed on in situ cleaved and as-grown (001) surfaces of low-*T*, *m* = 18 and *m* = 20, antigorite from the Kovdor Mine, Russia. The internal microstructure of the same crystal before cleavage has been checked by conventional TEM on FIB-cut cross-sections. The structural wave is imaged by dynamic SFM with a ~0.25 nm topographic amplitude (outcropping tetrahedral sheet) on cleaved and as-grown surfaces, and with a ~0.5 nm topographic amplitude (outcropping tetrahedral + octahedral sheets) mostly found on cleaved surfaces. Atomic resolution imaging was successfully applied on the cleavage surface through imaging individual atomic features on the outer hexagonal net of the emerging (Mg, O, OH) octahedra of the half-wave. The antigorite cleavage crack undulates through a single octahedral sheet, thereby avoiding rupture of strong Si-O bonds. The two tetrahedral reversals, which form the edges of the modulation repeat, are found to be strongly non-equivalent in structure: across <010>, one reversal is sharp as expected from the standard models of the antigorite structure, whereas the other reversal is unexpectedly "extended." The latter suggests some scheme of anti-polar positioning of silicate tetrahedra along <010> at the 6-membered ring reversal. High-resolution transmission electron microscopy (HRTEM) structure imaging of antigorite viewed down to <010> confirms spread out electron densities at this reversal. Numerous step height measurements on (001) surfaces show incremental results as integral multiples of 0.25 nm, the spacing between O,OH surfaces along **c***. Many of them differ in height from integral multiples of the unit cell repeat along **c*** and could be explained from carving the bulk wave structure. For all surfaces and steps, local stoichiometry and global electro-neutrality of the surface are satisfied.

Keywords: Antigorite, scanning force microscopy, surfaces, steps