Observation of the Hydrated Form of Tubular Halloysite by an Electron Microscope Equipped with an Environmental Cell

Norihiko Kohyama, Kurio Fukushima and Akira Fukami

National Institute of industrial Health, Ministry of Labor, Nagao, Tama-ku, Kawasaki 213, Japan
Department of Physics, College of Humanities and Sciences, Nihon University, Sakurajosui, Setagaya-ku, Tokyo 156, Japan

Abstract: The hydrated form of tubular halloysite [halloysite (10 Å)] was observed by a conventional electron microscope equipped with an environmental cell (E.C.), by which the "natural" form was revealed without dehydration of the interlayer water. This study mainly reports the selected area electron diffraction (SAED) analysis of the halloysite (10 Å) and its morphological changes by dehydration. The SAED pattern showed halloysite (10 Å) has two-layer periodicity in a monoclinic structure with the unit cell parameters of a = 5.14 Å, b = 8.90 Å, c = 20.7 Å, β = 99.7°, in space group *Cc*, and almost the same structure as the dehydrated form of halloysite [halloysite (7 Å)]. This means that the dehydration of the interlayer water did not greatly change or affect the structure of halloysite (10 Å). Accompanying the dehydration of the interlayer water, there appeared along the halloysite tube axis clear stripes that were about 50–100 Å in width. The diameters of the tubular particles also increased about 10%. From the results of various experiments, such as a focussing series, observation of the surface structure by the replica method, observation of end-views of the tubular particles, and others, these two phenomena were explained as follows: Halloysite crystals have "domains" along the *c*-axis direction, the thicknesses of the "domains" vary ca. 50–100 Å. They are tightly connected with each other when the halloysite is hydrated, but are separated from each other by the dehydration of the interlayer water, whereupon the stripes come into existence along the tube axis. Taking these considerations into account, a model of dehydration is proposed. Moreover, a new method of calculating the β-angle is proposed in the Appendix.

Key Words: Dehydration • Diffraction • Halloysite • Kaolinite • Tubular

Clays and Clay Minerals; February 1978 v. 26; no. 1; p. 25-40; DOI: 10.1346/CCMN.1978.0260103

Clay Minerals Society (www.clays.org)