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Unraveling the stacking structure in tubular halloysite using a new TEM with computer-assisted minimal-dose system

Toshihiro Kogure,[1,*] Kiyofumi Mori,[2] Yoshihide Kimura,[2] and Yoshizo Takai[2]

[1]Department of Earth and Planetary Science, Graduate School of Science, The University of Tokyo, 7-3-1 Hongo, Bunkyo-ku, Tokyo 113-0033, Japan
[2]Department of Material and Life Science, Graduate School of Engineering, Osaka University, 2-1 Yamada-Oka, Suita, Osaka 565-0871, Japan

Abstract

The stacking structure of tubular halloysite, which is easily damaged by electron-beam radiation, has been investigated by high-resolution transmission electron microscopy (HRTEM) using a new TEM with a computer-assisted minimal-dose system. The new TEM is equipped with a high-speed beam blanker by which the specimen is beam-irradiated only during image acquisition by the recording media. Using a few images taken by a CCD camera with a short exposure time (0.04 s), TEM operations such as area search and brightness adjustment are completed by the computer-assisted system, which can decrease the electron dose delivered before recording final HRTEM images by a factor of 100. The specimen used was halloysite (7 Å) with a tubular morphology from Eureka, Nevada, U.S.A. HRTEM images were taken with the incident electron beam perpendicular to the tube axis, which is parallel to the Y_i-axes of the dioctahedral 1:1 layer of halloysite. Lattice fringes in halloysite were almost extinguished with an electron dose of ~2000 electrons (e)/Å^2, and it was almost impossible to record crossed lattice fringes without radiation damage using conventional TEMs. By contrast, HRTEM images of high quality and without damage could be recorded with ~400 e/Å^2 in the new TEM system. The stacking structures recorded in such images were generally disordered, but ordered packets with one-layer periodicity were often observed. Among them, a stacking sequence that has never previously been observed in kaolinite was identified.

Keywords: Halloysite, HRTEM, minimal-dose system, radiation damage, stacking structure