Type of presentation: Oral

ID-12-O-2691 A Novel Method of Studying Small Particles in Wet Environmental-Cell TEM (WETEM)

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Wet environmental-cell TEM (WETEM) is at the forefront of providing new observational capabilities and technology in a myriad of research science and engineering capabilities. WETEM is capable of imaging wet materials and resolving real-time dynamic environmental processes at micro- and nano-scales. Much attention has been devoted to the development of the TEM sample holder over the last decade. However, the relatively high cost of the commercial environmental TEM holder has prevented many researchers from studying wet materials in TEM. This paper presents a simple, cost-effective method of constructing a wet environmental cell (EC) for WETEM research.

To construct a wet environmental-cell (EC), amorphous carbon films with very fine ridges were fabricated on a special Cu grid that has only a few small holes. To prevent possible breakage of carbon film, the hole that allows the electron beam to penetrate is rather small (~ 0.1 mm). An enclosed EC was built by sealing two pieces of carbon film/grids with carbon side facing each other in a regular TEM holder (Fig. 1). A tiny drop of smectite clay suspension was pipetted onto the bottom side of grid, and then covered with another carbon grid on the top. A very small amount of vacuum grease was applied to seal both grids. Special care was exercised to prevent adhesives from entering into the grids and contaminating the sample. The EC with wet sample was evacuated in a pre-pumping chamber to ensure the integrity of the wet cell before inserting into the TEM (modified JEOL JEM 2010 operated at 200 kV) equipped with a CCD camera for examining wet samples.

Conventional TEM image (Fig. 2a) shows typical smectite aggregates and well-defined particle outlines. Electron diffraction of dry particles reveals a stacking of smectite particles (i.e., aggregate) in a very clear dot pattern. The WETEM micrograph, as shown in Fig. 2b, reveals a blurred image indicating electron scattering by water molecules in the sample and EC. SAD pattern obtained from WETEM also depicts strong diffused electron scattering in the sample/EC. Analysis of SAD patterns obtained from dry (Fig. 2a*) and wet state (Fig. 2b*) illustrates lattice expansion of (h k 0) (Fig. 3). The small lattice expansion in (h k 0) probably resulted from the expansion of (0 0 l) plane due to addition of water molecules in the crystal along c-axis. This unique method provides a simple and affordable method for preparing and investigating wet samples in TEM. These experiments signify the opening of new opportunities in fundamental “in-situ” dynamic processes research.

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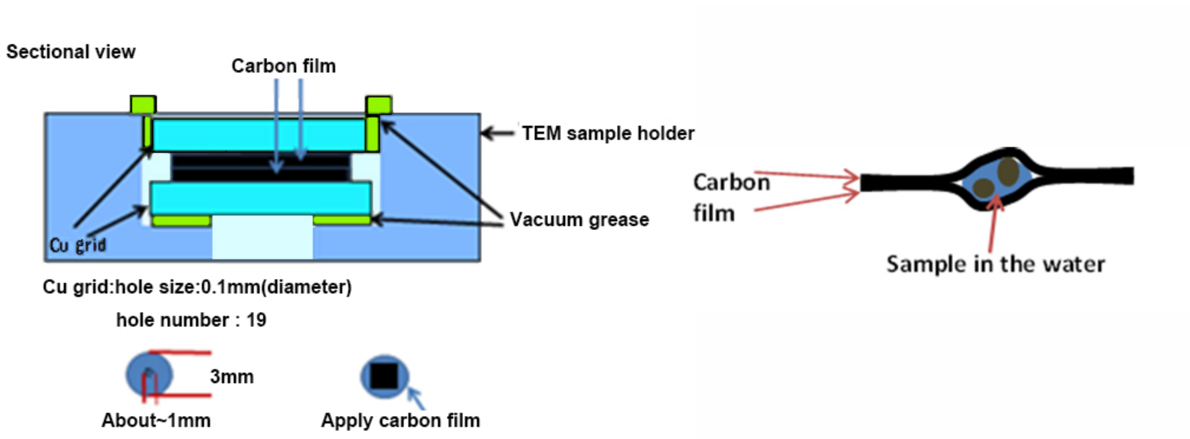

Fig. 1: Schematic diagram shows the basic construction of an EC in a TEM holder (left), and the concept of the nano-wet environmental-cell (right).

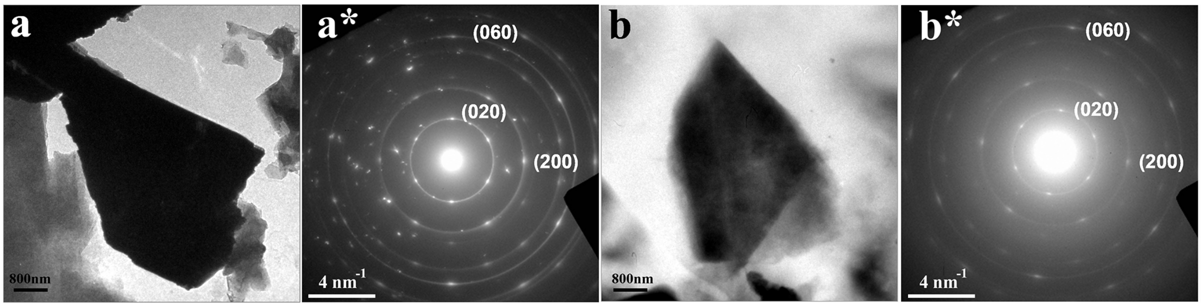

Fig. 2: Conventional TEM micrograph reveals typical morphology of dried particles and aggregates (a), whereas blurred image of smectite particles was observed in WETEM (b). The corresponding SAD patterns (a* and b*) also depict electron scattering due to water molecular in the sample/EC.

direction	0°	±60°		0°	±60°		±30°		90°
(hkl)	(020)	(110)	$(1\bar{1}0)$	(060)	(330)	$(3\bar{3}0)$	(130)	$(1\bar{3}0)$	(200)
Intensity ($\times 10^6$)	1.55	1.48±0.08		1.3	1.1±0.2		1.2±0.3		1.3
half[$\times 10^{-1}$ nm^{-1}]	8.0	9.6±0.4		12.9	14.0±0.2		14.2±0.5		16.3
d [nm]	0.461±0.001	0.460±0.001		0.154±0.001	0.154±0.001		0.265±0.001		0.264±0.001

direction	0°	±60°		0°	±60°		±30°		90°
(hkl)	(020)	(110)	$(1\bar{1}0)$	• (060)	(330)	$(3\bar{3}0)$	(130)	$(1\bar{3}0)$	(200)
Intensity ($\times 10^6$)	1.39	1.67±0.1		1.33	1.08±0.05		1.27±0.3		1.3
half[$\times 10^{-2}$ nm^{-1}]	4.8	8.3±0.7		6.8	9.5±0.5		8.4±0.4		1.1
d [nm]	0.457±0,.001	0.453±0.001		0.152±0.001			0.262±0.001		0.263±0.001

Fig. 3: A comparison of calculated d-spacings of the dried (top) and fully hydrated samples (bottom). Lattice expansion in different (h k l) probably resulted from the intercalation of water molecular in the smectitie crystal.