Environmental TEM, A CTF Approach for Atmospheric Lattice Imaging

Makoto Suzuki* and Toshie Yaguchi*

* Hitachi High-Technologies Corp., 882 Ichige, Hitachinaka, Ibaraki 312-8504, Japan

High-resolution transmission electron microscopy (HR-TEM) imaging under atmospheric pressure is one of the challenging issues in biological and material sciences. Environmental TEM (ETEM) enables one to observe specimens in various gas conditions with wide ranges of pressure and temperature. Now the gas-capsulated cell with a pair of electron-transparent thin films (E-cell) is extensively used for this purpose [1], stimulated by the progress of micro electrical and mechanical system (MEMS) technology.

We propose a novel method of evaluating the high-resolution imaging in ETEM, based on contrast transfer functions (CTFs) and unscattered fraction of electrons (UFE). In this model, the effect of the electron-gas interaction in E-cell and the electron-solid interaction in electron-transparent films are treated as scattering objects for incident and transmitted electrons, leading to the image deterioration. They are separated into two parts, pre-specimen scattering object above the specimen, and post-specimen scattering object below it. The former object induces energy and angular spreads, thus the CTF of the microscope is modified. The latter object breaks the phase information of the transmitted electrons, and only the UFE carries the phase contrast amplitude.

In order to make a comparison with experiments, we employed a newly developed E-cell [2], installed in 300 keV TEM H-9500. It is equipped with thin (<30 nm) Si₃N₄ amorphous membranes and gas flow tubes. The distance between two membranes is 1.0mm. The specimen is placed on the heating element in the middle of the cell. The air (N₂:O₂=4:1) is used as a gas in the cell, and the pressure is measured by a MEMS pressure gage. Figure 1 shows typical ETEM images of a Si particle. The lattice fringe of 0.314 nm is clearly observed in the condition of 1x10⁴ Pa (0.1 atm) and 600°C.

Figures 2(a) and 2(b) show the calculated CTFs of the microscope used, which are modified after passing through the pre-specimen scattering object. In Fig. 2(a) the effect of the 30-nm-thick Si₃N₄ membrane is shown. The CTFs modified by the angular and energy spreads are shown by the (red) solid and (blue) dashed curves, respectively. The (black) dotted curve represents the original CTF without E-cell. The CTFs modified by the 0.5-mm air space of 1x10⁴ Pa are presented in Fig. 2(b) in the same manner. In both cases the modification of the CTF is minor, and a reasonable amount of the amplitude still remains, supporting the experimentally observed lattice image shown in Fig. 1(b). Figures 2(c) and 2(d) show the calculated UFEs for Si₃N₄ membrane and air, respectively. The threshold above which the lattice imaging becomes possible is not obvious. In the present case (i.e., 30-nm-thick Si₃N₄ and 0.5-mm air space of 1x10⁴ Pa), however, the total UFE yields 33 %, giving one of the reasonable criteria of UFE for high-resolution imaging in ETEM.

References

[1] e.g., T. Kamino et al., J. Electron Microsc. 54 (2005) 497-503.

[2] T. Yaguchi et al., Microsc. Microanal. 16 (Suppl 2) (2010) 302.

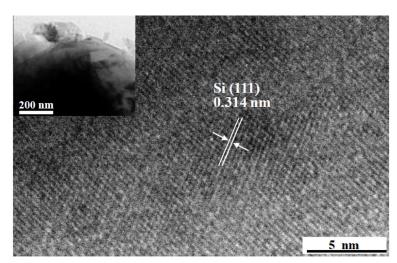


FIG. 1. HR-TEM imaging of a crystalline silicon particle observed at 600°C in the air pressure of 1x10⁴ Pa with thin (<30nm) Si₃N₄ windows 1.0 mm apart. Inset: Low magnification image.

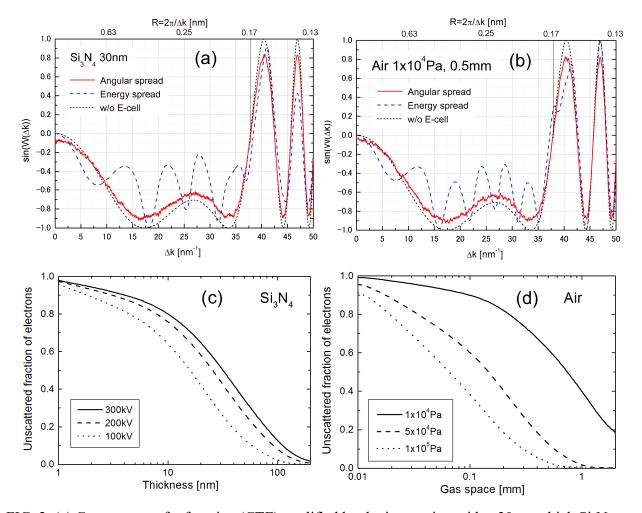


FIG. 2. (a) Contrast transfer function (CTF) modified by the interaction with a 30-nm-thick Si_3N_4 window, (b) CTF modified by the interaction with 0.5-mm air space of 1x 10^4 Pa, (c) Unscattered fraction of electrons (UFE) for Si_3N_4 membrane, (d) UFE for air.