

On the gas-dependent image resolution in an aberration-corrected ETEM

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Currently, energy and environmental technologies receive an increasing attention. In order to rational design new, improved functional nanomaterials for said applications, a detailed understanding of their structure-performance relationships is inevitably needed and atomic-scale insight into the geometrical, compositional and electronic structures of the nanomaterials is therefore of paramount importance. With the recent advancements, atomic resolution transmission electron microscopy (S/TEM) has become a powerful and indispensable tool for characterizing nanomaterials with a spatial resolution in the sub-Angstrom range and a sensitivity for detecting even single atoms [1-3]. The imaging techniques may be beneficially combined with capabilities for in-situ observations of the nanomaterials in their working state. One important advancement for in situ electron microscopy is the environmental transmission electron microscope (ETEM) facilitating time-resolved, atomic-scale observations of catalytic and environmental nanomaterials during exposure to reactive gas environments [4].

Here, we examine the conditions to obtain such in situ observations in the high-resolution transmission electron microscopy (HRTEM) mode with an image resolution of 0.10 nm [4]. The HRTEM image resolution threshold is mapped out under different gas conditions, including gas types and pressures and under different electron optical settings, including electron beam energy, dose and dose-rate. Specifically, nitrogen (N_2) or hydrogen (H_2) was admitted to the differentially pumped specimen area of the ETEM to a pressure, P_e , ranging from 0.2 mbar to 20 mbar (N_2). HRTEM images were acquired with an incident electron beam energy of 300 or 80 keV, respectively, with a higher electron dose rate (HD) of ca. $1 \cdot 10^6$ electrons/ nm^2s and a lower dose rate (LD) of ca. $1 \cdot 10^5$ electrons/ nm^2s and with exposure times of the charged-coupled device (CCD) camera of 0.1 s to 10 s for comparison (see Fig. 1).

Our main finding is that the microscope's inherent resolution limit can be maintained under the condition of image sensitivity (signal-to-noise ratio) consistent with the *Rose* criterion. The 0.10 nm resolution seems to be retainable for H_2 over the entire accessible pressure range of 1-10 mbar. Even for N_2 , the 0.10 nm resolution threshold can be reached up to at least 10 mbar under optimized imaging conditions. Surprisingly the results show that the 0.10 nm resolution limit is also highly sensitive to the electron dose-rate in such a way that the LD conditions facilitate 0.10 nm resolution threshold over the broadest pressure interval (see Fig. 2).

A discussion on the electron-gas interactions responsible for gas-induced resolution deterioration is given based on interplay with complementary electron diffraction (ED), electron energy loss spectroscopy (EELS) as well as scanning transmission electron microscopy (STEM) data. The corresponding optimal optical and environmental conditions may be reached for any gas-solid experiments in an ETEM with a careful optimization following the presented approach. [5]

References:

- [1] JR Jinschek, *et al*, Carbon **49** (2011) p. 556.
- [2] C Kisielowski, *et al*, Angewandte Chemie Int. Ed. **49** (2010) p. 2708.
- [3] K Urban, Nature Materials **10** (2011) p. 165.
- [4] JR Jinschek, S Helveg, Micron, doi:10.1016/j.micron.2012.01.006, in press (2012).
- [5] The authors would like to acknowledge the encouragement from both their organizations to pursue this project. The authors gratefully acknowledge Sven Ullmann (Haldor Topsøe A/S).

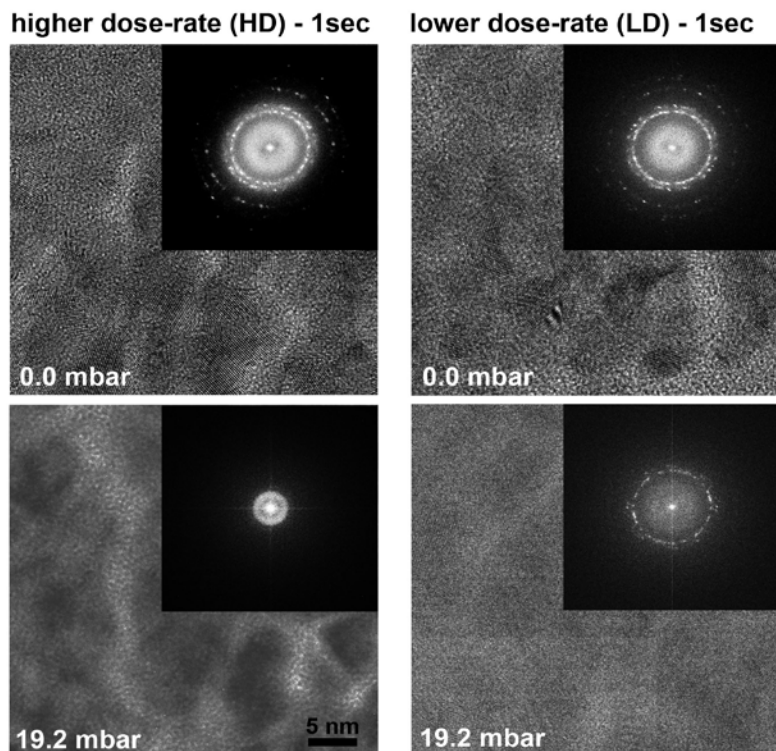


Figure 1. HRTEM micrographs of a Au/C cross grating obtained at 300 keV. Images are acquired in situ during the exposure to 0.0 and 19.2 mbar N₂ under higher dose-rate (HD = 10⁶ e⁻/nm²s) and lower dose-rate (LD = 10⁵ e⁻/nm²s) illumination conditions [4].

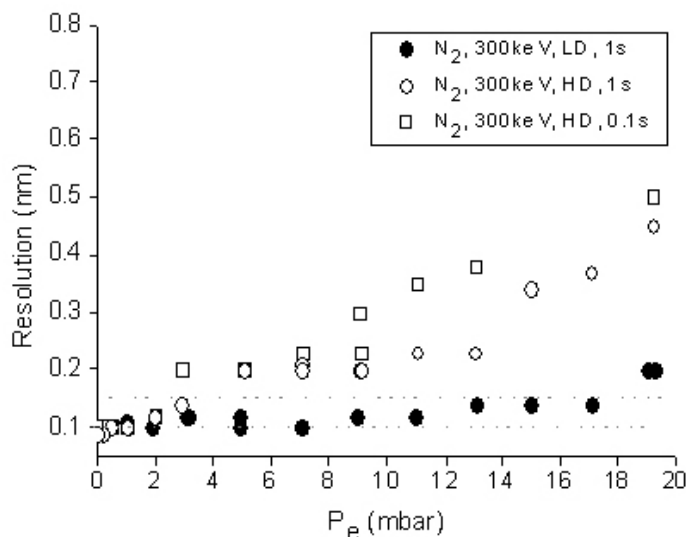


Figure 2. In situ HRTEM imaging performance with a N₂ environment under the HD and the LD settings at 300keV. Image resolution limits are shown versus the N₂ pressure in the ETEM sample area [4].