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Observing beam effects and their thresholds in the environmental transmission electron microscope

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Environmental transmission electron microscopy or ETEM is becoming an increasingly applied method to investigate materials in an environment approaching operating conditions [1]. In a dedicated ETEM, a sample can be exposed to various gases at pressures of up to 10^3 Pa. Using dedicated windowed sample holders, pressures 2 orders of magnitude higher can be achieved while imaging. The much higher gas density under such conditions enable a whole host of phenomena when the gas molecules react with the beam and the sample. When performing experiments in liquids, the density is even higher and the observed phenomena can be even more extreme.

The effect of the beam, or looking from the other side: the effect of the gas molecules on the electron beam, is firstly to scatter electrons in the entire high-pressure zone, i.e. the objective lens gap in the case of a dedicated ETEM. Scattering is a function of both gas pressure and the gas specie [2]. The result of this scattering process is a net loss of electrons both above and below the sample, see Figure 1.

In order to extract reliable data from experiments conducted with a gas surrounding the sample, a series of control experiments must be performed. These can be done in various ways. Generally, the sample is imaged prior to exposing the samples to a given reactive environment to establish the initial state of the sample. The sample is then exposed to the same environment used in the in situ experiment for the same duration of time without exposing the sample to the electron beam. After reestablishing high-vacuum conditions around the sample, the sample is imaged again and the results compared to the initial state. Any observed changes are then compared to changes observed in an in situ experiment where the sample is exposed to reactive conditions in combination with the electron beam.

With the proper analysis and control experiments, ETEM can be a powerful technique for extracting details of samples under reactive environments. Such information can guide the development of functional materials of various classes.

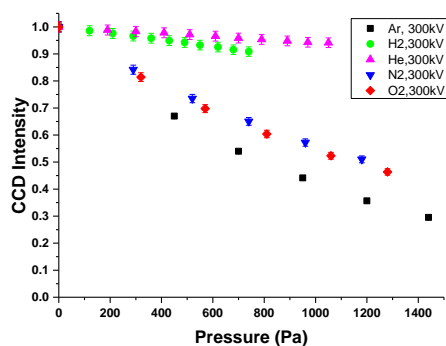


Figure 1: Normalized intensity measured on a CCD camera in various gases at various pressures.

References

- [1] T.W. Hansen and J.B. Wagner, eds. *Controlled atmosphere transmission electron microscopy*. 2016, Springer. 329.
- [2] T.W. Hansen and J.B. Wagner, *Microscopy and Microanalysis* **18** (2012), p. 684.