Electron Energy-Loss Spectroscopy in an Environmental TEM

Thomas W. Hansen*, Jakob B. Wagner, Marco Beleggia, Chris B. Boothroyd, Rafal E. Dunin-Borkowski
DTU Cen, Fysikvej, building 307, DK-2800 Kgs. Lyngby, Denmark
*twh@cen.dtu.dk

Abstract

The increasing use of environmental transmission electron microscopy (TEM) in materials science provides exciting new possibilities for investigating chemical reactions and understanding both the interaction of fast electrons with gas molecules and the effect on high-resolution imaging in the presence of gases. A gaseous atmosphere in the objective lens of the microscope alters both the incoming electron wave prior to interaction with the sample and the outgoing wave below the sample. Whereas conventional TEM samples are usually thin (below 10 nm), the gas in the environmental cell fills the entire gap between the pole pieces and is thus not spatially localized, as is the case for a conventional TEM sample. By using an environmental FEI Titan TEM equipped with a monochromator, we investigate the interaction between fast electrons and gas molecules using electron energy-loss spectroscopy.

Introduction

State-of-the-art aberration corrected TEMs provide electron micrographs with high spatial resolution. The apparent interpretability of such images encourages microscopists to analyze data more quantitatively. Such an analysis requires a detailed knowledge of the entire path of the electron along the microscope column. The effect of gas on the electron wave in the objective lens is not well understood and needs further attention. EELS can contribute to the understanding of the electron-gas interaction, by monitoring the relation between energy-loss and scattering angle.

Method

By using a differentially pumped FEI Titan 80-300 Titan ETEM, electron energy-loss spectra were acquired from several gases at various pressures, ranging from ca. 10^{-5} mbar to 15 mbar. The gases were let into the environmental cell using digitally controlled mass flow controllers, providing accurate and stable control of the pressure in the cell. Spectra were acquired using 80-300 kV primary electrons to elucidate the variation in interaction cross section. Furthermore, beam intensities were recorded on the pre-GIF CCD camera in order to investigate the loss and/or scattering of electrons when passing through the high-pressure region of the column (ca. 5mm) and its effect on the signal to noise ratio in high-resolution images.

We will present results from various elemental as well as di-molecular gases and their effect on imaging and spectroscopy in the environmental transmission electron microscope.

Figure 1. Fraction of inelastic intensity in the electron energy-loss spectrum of as a function of argon pressure.

Figure 2. Low-loss electron energy-loss spectrum of 3 mbar molecular oxygen measured with a conventional FEG electron source and a monochromated FEG electron source.