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Detecting Localized Variation of Chemistry via Atomic-Resolution Secondary Electron Imaging

Jane Howe[1], Yoichiro Hashimoto[2], Xue Wang[3], Madeline Vara[3], David Hoyle[1], Tom Schamp[4], Younan Xia[3], and David Joy[5,6]

[1.] Hitachi High-Technologies Canada Inc, Toronto, Canada.
[2.] Science System Design Division, Hitachi High-Technologies Co., Hitachinaka, Ibaraki, Japan.
[3.] The School of Chemistry and Biochemistry, Georgia Institute of Technology, Atlanta, USA.
[4.] Hitachi High-Technologies America Inc, Gaithersburg, USA.
[5.] Department of Materials Science and Engineering, University of Tennessee, Knoxville, USA.
[6.] Center for Nanophase Materials Sciences, Oak Ridge National Laboratory, Oak Ridge, USA.

Secondary electron microscopy (SEM) has been widely used for characterization of a broad range of materials. Conventional SEM operates at an accelerating voltage ranging from a few hundred to 30 kV, commonly using secondary electrons (SE) for imaging. Secondary electrons, defined with an energy of less than 50 eV, are assumed to result from inelastic scattering between electrons. The energy loss is small and the SE could be generated a few nanometers away from the interaction. And both the generation and emission of SE are considered delocalized events [2-3]. In 1980, Pennycook and Howie reported that a high fraction of the energy loss in thin foils occurred close to atomic sites and resulted in the production of secondary electrons [4]. They further predicted that "ejected secondary electrons would show a localized or crystallographic effect". SE imaging at high voltages up to 300 kV can also be obtained in a scanning transmission electron microscope (STEM) equipped with an SE detector. Thanks to recent advances in aberration corrected STEM, Zhu and colleagues demonstrated atomic-resolution SE imaging of crystals and single atoms at 200 kV [1]. This result supports Pennycook and Howie's earlier predication.

Based on the current understanding that generation and emission of secondary electrons is both a localized and delocalized event, David Joy proposed the hypothesis that layer-by-layer SE imaging would reveal localized variation of chemistry and other defects in a crystal [5]. In order to verify Joy's hypothesis, we carry out SE imaging of a series of nanocrystals with well-defined chemical variation and defects. The atomic-resolution SE imaging has been undertaken using a Cs-corrected Hitachi HD-2700A STEM equipped with a secondary electron detector. In Fig. 1, we present a set of SE and annular dark field (ADF) images taken from Pd@Pt core-shell cubes at 200 kV. The outer ~3 atomic layers of these 18-nm cubes are Pt, as revealed by the contrast as well as the EDS mapping. Comparing the SE image of Fig. 1b and the ADF image of Fig. 1c, the SE image revealed less contrast difference of Pt versus Pd than that of the ADF. In bright-field TEM study, image contrast changes as a function of focal length and Cs. In this study, we strive to carry out a through-Cs series of SE imaging. Particularly, we are going to explore whether we can obtain atomic-resolution SE images under negative Cs.

As Zhu *et al.* suggested that secondary electrons produced by energy-loss events in the several keV range, such as innershell ionizations, as being the enabling step for localization [1], it is reasonable to speculate that localized SE event would be diminished if the primary beam energy is too low to produce energy-loss events in the several keV range. What is the cut-off energy of a certain element? Let us see if we can find it out.

References:

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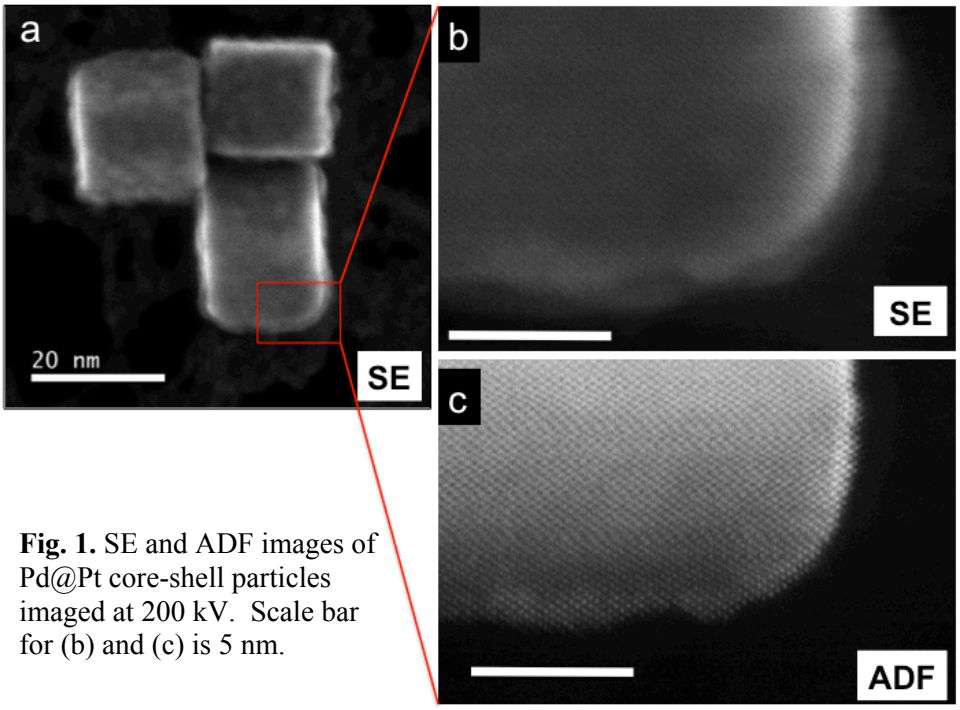

Fig. 1. SE and ADF images of Pd@Pt core-shell particles imaged at 200 kV. Scale bar for (b) and (c) is 5 nm.