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Atomic-Level Chemical Analysis by EELS and XEDS in Aberration-Corrected Scanning Transmission Electron Microscopy

M. Watanabe[1], E. Okunishi[2] and T. Aoki[1,3]
[1] Dept of Materials Science and Engineering, Lehigh University, Bethlehem. PA 18015
[2] JEOL Ltd, Akishima, Tokyo, 196-8558, Japan.
[3] JEOL USA Ltd. Peabody, MA 01960

Chemical analysis at atomic-level spatial resolution with single-atom detection sensitivity is one of the ultimate goals in materials characterization. Such atomic-level materials characterization would be feasible by electron energy-loss spectrometry (EELS) and X-ray energy dispersive spectrometry (XEDS) in the latest aberration-corrected scanning transmission electron microscopes (STEMs) because more probe current can be added into the incident probe by aberration-correction. Especially for EELS analysis, sufficient amounts of core-loss signals can be generated within a short acquisition time by higher current probes, and hence atomic-resolution EELS mapping has already been applied [e.g., 1-3]. For XEDS analysis, spatial resolution reaches ~ 0.4 nm [4], which implies atomic-level analysis is feasible, in aberration-corrected STEM. In this paper, the latest attempts to obtain atomic-level elemental distributions by EELS and XEDS approaches in aberration-corrected STEM will be presented.

For atomic-level analysis, the STEM probe must be positioned above individual atomic column sites during acquisition, which requires relatively long-term instrumental and environmental stabilities. The newly developed JEOL ARM-200F aberration-corrected STEM instrument is designed to perform the atomic-level chemical analysis with improved instrumental stabilities. An HAADF-STEM image from an interface in a $LaMnO_3/SrTiO_3$ multilayer thin-film is shown in Fig. 1(a). The bright and slightly fainter spots appearing in this HAADF-STEM image correspond to heavy atom columns of La or Sr and to Ti-O or Mn-O columns in the perovskite structure, respectively. An EELS spectrum-imaging (SI) data was acquired from the same field of view with 186 x 26 pixels and 1350 channels for a dwell time of 0.1 s using a Gatan Enfina spectrometer. After applying multivariate statistical analysis (MSA) [5] to enhance weak signals in the dataset, elemental maps were extracted by power-low background subtraction. From the extracted elemental maps, two RGB color-overlay images were constructed as shown in Fig. 1(b, Red: Ti $L_{2,3}$, Green: Sr $M_{2,3}$ and Blue: O K) and 1(c, Red: Mn $L_{2,3}$, Green: La $M_{4,5}$ and Blue: O K), which represent $SrTiO_3$ and $LaMnO_3$ layers, respectively. Elemental distributions at individual atomic columns can be clearly distinguished. Especially the Ti distribution is terminated at the $LaMnO_3/SrTiO_3$ interfaces relatively sharply, whereas the Mn distribution seems diffused toward $SrTiO_3$ layers.

Atomic-level chemical analysis is even more challenging in the XEDS approach since detection of X-ray signals is more limited than that in EELS (~100 times difference). An XEDS SI dataset was recorded from a [100]-projected GaAs specimen and then MSA was applied to improve weak signals in the dataset. Figure 2 shows a HAADF-STEM image (a) and X-ray maps of Ga Kα and As Kα lines (b), and EELS maps of Ga $L_{2,3}$ and As $L_{2,3}$ edges (c), which were also recorded with the XEDS SI dataset simultaneously. Although the signal levels are still very limited in comparison to EELS analysis, the atomic-level XEDS analysis is now possible in combination of aberration-corrected STEMs with advanced statistical analysis such as MSA.

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Fig. 1

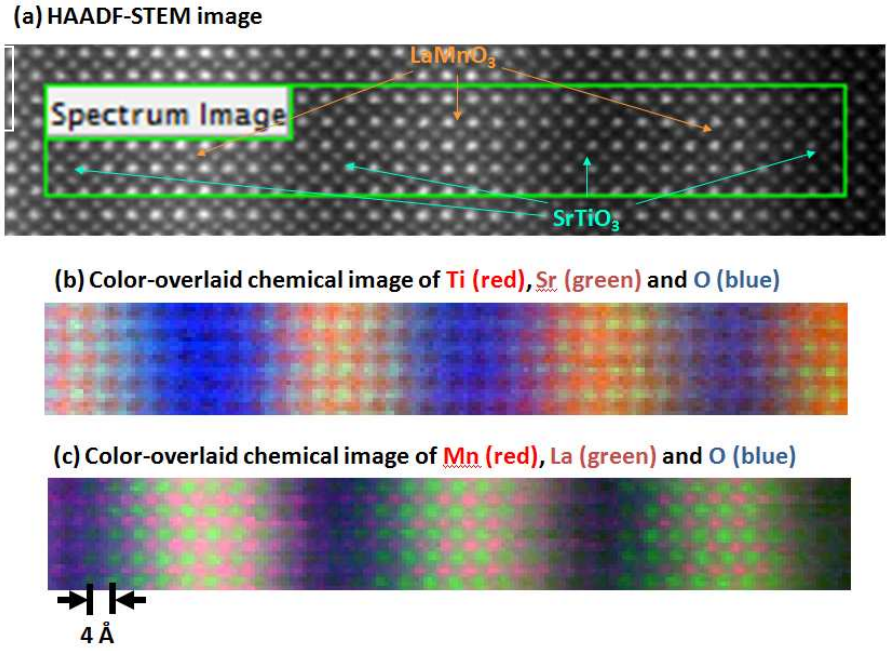

Fig. 2

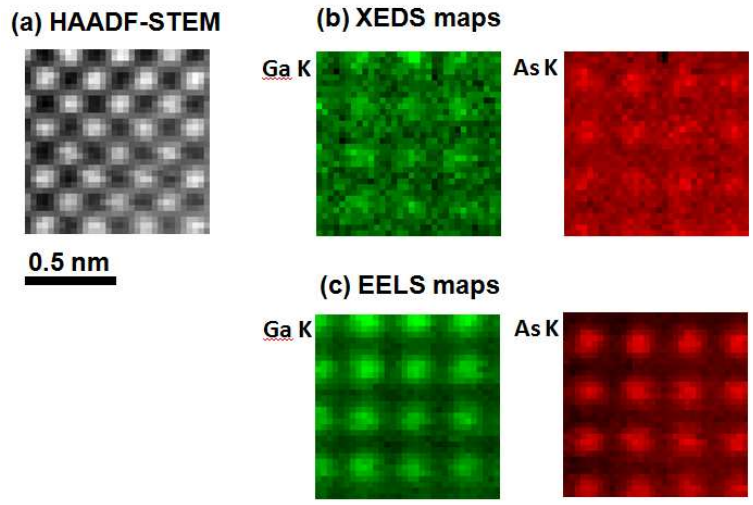

Figure 1: (a) An HAADF-STEM image from a $LaMnO_3/SrTiO_3$ interface, (b) RGB color-overlay image of $SrTiO_3$ and (c) RGB color-overlay image of $LaMnO_3$, obtained by the EELS approach.
Figure 2 (a) An HAADF-STEM image from a GaAs specimen, (b) XEDS elemental maps of Ga Kα and As Kα lines, and (c) EELS maps of Ga $L_{2,3}$ and As $L_{2,3}$ edges.

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