

Advanced Research Center for Beam Science - Electron Microscopy and Crystal Chemistry -

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Chiang Mai University, Thailand, 21–22 May 2009

National Taiwan University, Taiwan, 1 July–30 September 2009

NanoMEGAS, Belgium, 10 September 2009

Shanghai Jiao Tong University, China, 15 September 2009

Université du Sud Toulon Var, France, 29 October 2009

Scope of Research

Crystallographic and electronic structures of materials and their transformations are studied through direct imaging of atoms or molecules by high-resolution spectromicroscopy which realizes energy-filtered imaging and electron energy-loss spectroscopy as well as high resolution imaging. It aims to explore new methods for imaging and also obtaining chemical information in thin films, nano-clusters, interfaces, and even in solutions. By combining this with scanning probe microscopy, the following subjects are urging: direct structure analysis, electron crystallographic analysis, epitaxial growth of molecules, structure formation in solutions, and fabrication of low-dimensional functional assemblies.

Research Activities (Year 2009)

Publications

Haruta M, Kurata H, Komatsu H, Shimakawa Y, Isoda S: Site-resolved Oxygen K-edge ELNES of Layered Double Perovskite $\text{La}_2\text{CuSnO}_6$, *Phys.Rev.*, **B80**, 165123 (2009).

Haruta M, Kurata H, Komatsu H, Shimakawa Y, Isoda S: Effects of Electron Channeling in HAADF-STEM Intensity in $\text{La}_2\text{CuSnO}_6$, *Ultramicroscopy*, **109**, 361-367 (2009).

Yoshida K, Isoda S, Kamata T (NAIST): Epitaxial Orientation of Dimethylglyoximatoplatinum(II) on Various Substrates, *Crystal Growth & Design*, **9(6)**, 2582-2587 (2009).

Presentations

Electron Crystallography of Organic Materials by TEM, Ogawa T, Isoda S, The 26th MST Annual Conference, Chiangmai, Thailand, 28 January 2009.

Local State Analysis by Electron Energy-loss Spectroscopy in a Scanning Transmission Electron Microscope, Kurata H, NSYSU-KU Bilateral Symposium on Materials Chemistry, Kaohsiung, Taiwan, 22 September 2009.

Effects of Electron Channeling on HAADF-STEM Intensity in $\text{La}_2\text{CuSnO}_6$

Atomic resolution imaging using the high-angle annular dark-field scanning transmission electron microscopy (HAADF-STEM) can be applied to analyze atomic structures of materials directly. This technique provides incoherent Z-contrast usually with the atomic number of the constituent elements. In the present work, however, unique contrasts that make intuitively interpreting the HAADF-STEM image to be difficult were observed in double perovskite oxide $\text{La}_2\text{CuSnO}_6$. Multislice simulation confirmed that this occurred as an effect of the channeling process of electrons in combination with the effect of Debye–Waller factors. This was confirmed because in the $\text{La}_2\text{CuSnO}_6$ crystal, two independent Sn atoms and four independent La atoms in the unit cell had different Debye–Waller factors, and the La columns consisted of pairs of columns with a small separation, whereas the Sn atoms were arranged straight.

Furthermore, the image contrast was examined systematically by multislice simulation on virtual structures in which two atomic La columns in the unit cell were separated by certain distances in a projected plane. As a result, the HAADF intensity did not decrease constantly

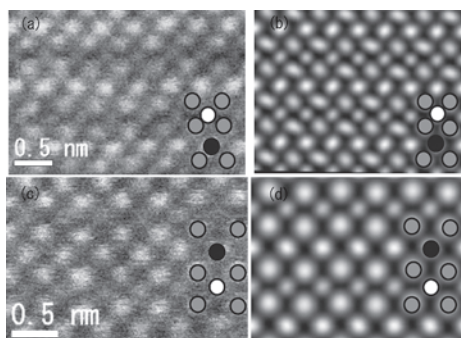


Figure 1. Experimental HAADF-STEM images of $\text{La}_2\text{CuSnO}_6$ along (a) the b-axis projection and (c) the c-axis projection. Simulated images along (b) the b-axis projection and (d) the c-axis projection.

with the increase in column separation, with the exception of a very thin sample, which could be interpreted by the specific change in the electron-channeling process.

Photochemical Synthesis of Silver Particles in Tween 20/Water/Ionic Liquid Microemulsions

Metal particles of silver (Ag) were synthesized by the photoreduction of silver perchlorate (AgClO_4) in water-in-ionic liquid (ILs: [BMIm] $[\text{BF}_4]$, [OMIm] $[\text{BF}_4]$) microemulsions consisting of Tween 20, water and ionic liquids. The time evolution of Ag particle formation by photoreduction using UV-irradiation was investigated by UV–Vis, cryo-TEM, extended X-ray absorption fine structure (EXAFS) and small angle X-ray scattering (SAXS) measurements. The average diameter of the metallic Ag particles prepared in the water-in-[BMIm] $[\text{BF}_4]$ and water-in-[OMIm] $[\text{BF}_4]$ microemulsions was estimated from TEM to be 8.9 and 4.9 nm, respectively, which was consistent with that obtained from the SAXS analysis. Using Guinier plots in a low q-range ($<0.16 \text{ nm}^{-1}$), we demonstrate that the average diameter of the water droplets that consisted of aggregates of ionic precursors of AgClO_4 before reduction and Ag particles after reduction, in the microemulsions, was estimated to be about 20–40 nm. The diameter of the water droplets increased as a function of photoreduction time because of the formation of Ag particles and their aggregates.

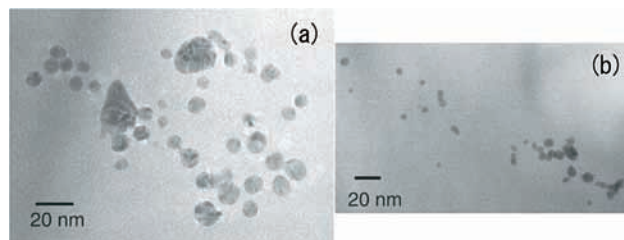


Figure 2. Cryo-TEM images of Ag particles prepared by the photoreduction in the presence of Tween 20 in (a) the water-in-[BMIm] $[\text{BF}_4]$ and (b) the water-in-[OMIm] $[\text{BF}_4]$ microemulsions.

Grants

Kurata H, Local State Analysis of Defects and Interface Regions by Spherical Aberration Corrected STEM and EELS, Grant-in-Aid for Scientific Research (B)19310071, 1 April 2007–31 March 2010.

Isoda S, Nanotechnology Support Project, The Ministry of Education, Science, Culture and Sports, Japan, 1 April 2007–31 March 2011.

Isoda S, Development of Observation Method of Polymer Composite Materials without Staining by Scanning Trans-

mission Electron Microscope, Grant-in-Aid for Scientific Research (C) 20550188, 1 April 2008–31 March 2011.

Award

Haruta M, Kurata H, Komatsu H, Shimakawa Y, Isoda S, Best Poster Award, Site Resolved Oxygen K-edge ELNES of Layered Double Perovskites $\text{La}_2\text{CuSnO}_6$, EDGE2009: International EELS-Workshop Committee, 21 May 2009.