

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

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## Visitors

Prof WEGNER, Gehnard MPI for Polymer Science, Germany, 28 May 2007  
Prof CHEN, Jen-Sue Department of Materials Science and Engineering, National Cheng Kung University, Taiwan, 8–9 June 2007  
Dr LO, Shen-Chuan ITRI, Taiwan, 13–19 October 2007

## 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 2007)

### Publications

Yoshida K, Minamikawa H, Kamiya S, Shimizu T, Isoda S: Formation of Self-Assembled Glycolipid Nanotubes with Bilayer Sheets, *J. Nanosci. Nanotechnol.*, **7**, 960-964 (2007).

Koshino M, Kurata H, Isoda S: Stability of Peripheral Halogenation among Phthalocyanine Complexes, *Microsc. Microanal.*, **13**, 96-107 (2007).

Minari T, Seto M, Nemoto T. *et al.*: Molecular-packing-enhanced Charge Transport in Organic Field-effect Transistors of Semiconducting Porphyrin Crystals, *APL*, **91**, 123501 (2007).

### Presentations

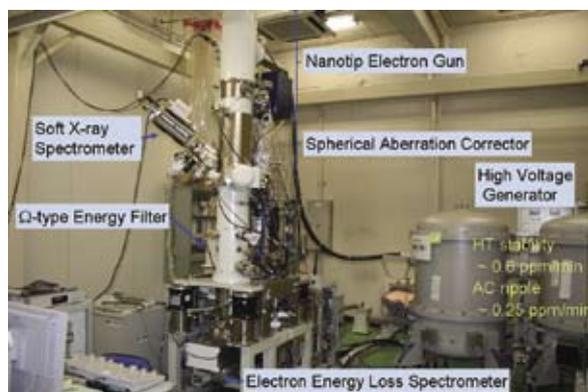
Porphyrim Single-crystal Field-effect Transistors, Seto M, Nemoto T, Isoda S, et al., China NANO, 4–6 June 2007, Beijing, China.

Direct Observation of Iodine-doped C<sub>60</sub> Crystal by Scanning Transmission Electron Microscopy, Haruta M, Kurata H, Yoshida K, Isoda S, IWFAC'2007, 2–6 July 2007, St. Petersburg, Russia.

Morphology-Controlled Titania Nanocrystals and Application for Dye-sensitized Solar Cells, Isoda S, Jiu J (Osaka Univ.), Adachi M (Doshisha Univ.), Yoshida K, Kurata H, KJF-2007, 27–29 September 2007, Seoul, Korea.  
Nanostructuring of BTQBT Derivative Co-adsorbed

## Development of Cold-FEG with a Nanotip for 200kV TEM/STEM

Electron energyloss spectroscopy (EELS) combining with an STEM makes it possible to extract local electronic structure as well as elemental information from a small area in solids. Because of the small inelastic scattering cross-section, however, it is still difficult to measure spectrum image data with an electron probe of atomic scale. Recent development of spherical aberration correctors is one of promised approaches to overcome this problem, because it can provide an increased probe current in addition to a sub-angstrom probe size. As another way to get a brighter electron source, we have developed a cold-FEG for a 200kV TEM/STEM (Figure 1) with a sharpened  $\langle 111 \rangle$  orientation tungsten tip. The method producing a sharpened tip is to heat it under applying a strong electric field (thermal field, TF, treatment). It promotes the diffusion of surface atoms and the faceting of low index planes. The diffusing atoms gather around the edge region to gain the polarization energy due to the applied electric field, so that the protrusion is formed as a pyramid structure along the  $\langle 111 \rangle$  direction with a few nm in height. The probe current at the specimen position is about 50–100 pA, which is almost one order higher than that of the Schottky type

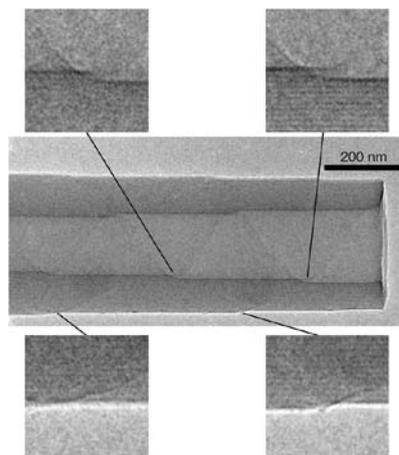


**Figure 1.** A 200kV TEM/STEM with nanotip electron gun.

of FEG. If the bright electron gun is combined with a spherical aberration corrected STEM, one can expect the increased probe current at a sub-angstrom probe size, which will be promising to perform STEM-EELS analysis with a high efficiency.

## Formation of Self-Assembled Glycolipid Nanotubes with Bilayer Sheets

Rolled-up morphology of bilayer sheets in a self-assembled glycolipid nanotube (LNT) in water was carefully examined by using a cryogenic transmission electron microscope (cryo-TEM) with a rapid-freezing specimen-preparation technique. The LNTs were obtained under a series of self-assembly conditions: boiling of an aqueous dispersion of glycolipid *N*-(11-*cis*-octadecenoyl)- $\beta$ -D-glucopyranosylamine, subsequent gradual cooling, and incubation at room temperature for several days. Cryo-TEM images revealed that the LNT walls consist of a multilayer structure with interlayer distance of about 4.7 nm. (Figure 2) These layers correspond to constituent lipid bilayers. From the result of precise cryo-TEM observations and analyses, we confirmed the rolled-up morphology of the lipid bilayer sheets in a complete self-assembled glycolipid nanotube.



**Figure 2.** A cryo-TEM image of the LNT rolled up near its end.

with Solvents, Chiba Y, Nemoto T, Yamashita Y (Tokyo Inst. Tech.), Isoda S, ICSPM15, 6–8 December 2007, Atagawa, Japan.

### Grants

Kurata H, Development of an EELS/XES Electron Microscope for Electronic Structure Analysis, Leading Project, The Ministry of Education, Science, Culture and Sports, Japan, 1 April 2004–31 March 2007.

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.

### Award

Isojima S, Best Poster Awards, The 63rd Annual Meeting of the Japanese Society of Microscopy, “LAADF of Iron Oxide Nanowire”, The Japanese Society of Microscopy, 21 May 2007.