# Development of Three-Dimensional Scanning Photoelectron Microscope at SPring-8 BL07LSU

K. Horiba[1, 2, 3], Y. Nakamura[1], N. Nagamura[1], S. Toyoda[1], H. Kumigashira[1, 2, 4], M. Oshima[1, 2, 3], K. Amemiya[3, 5], Y. Senba[6], and H. Ohashi[6]

[1]*Department of Applied Chemistry, The University of Tokyo*

[2]*Synchrotron Radiation Research Organization, The University of Tokyo*

[3]*Core Research for Evolutional Science and Technology (CREST), Japan Science and Technology Agency*

[4]*Precursory Research for Embryonic Science and Technology (PRESTO), Japan Science and Technology Agency*

[5]*Photon Factory, Institute of Materials Structure Science, High Energy Accelerator Research Organization (KEK)*

[6]*SPring-8, Japan Synchrotron Radiation Research Institute (JASRI)*

With the evolution of nanotechnology, nanoscale analyses of device structures and self-assembled structures are greatly required. In particular, it is important to investigate the nanoscale distribution of electronic structures and chemical states along depth as well as lateral directions in order to understand the mechanisms and characteristics of complicated nanostructure species such as stacking structures in semiconductor devices and the surface/interface reactions of catalytic materials. Here, we report a newly developed scanning photoelectron microscope (SPEM) system with a depth profiling analysis capability for three-dimensional (3D) spatially resolved electron spectroscopy for chemical analysis (ESCA) of solids. We call this system "3D nano-ESCA." The system has been installed at the University-of-Tokyo Materials Science Outstation beamline, BL07LSU, at SPring-8.

The concept of 3D nano-ESCA is illustrated in Fig. 1. A SR X-ray beam is focused to nanometer size on the samples using a Fresnel zone plate (FZP). We use a 200 μm-diameter FZP with an outermost zone width of 35 nm. We can obtain lateral (x and y) distribution of photoelectron spectra by scanning the samples along the lateral directions and acquiring the photoelectron spectrum at each point. This is the typical scheme of SPEM. In addition to SPEM measurements, we simultaneously detect the angular distribution of emitted photoelectrons using an angle-resolved photoelectron spectrometer. In order to achieve high energy-resolution and a wide acceptance angle, a modified spherical photoelectron spectrometer with a two-dimensional (energy and angular distributions) detector and an extremely wide-angle lens with the acceptance angle of 60° is adopted. As an example, Fig. 2 shows the simultaneously-detected angular

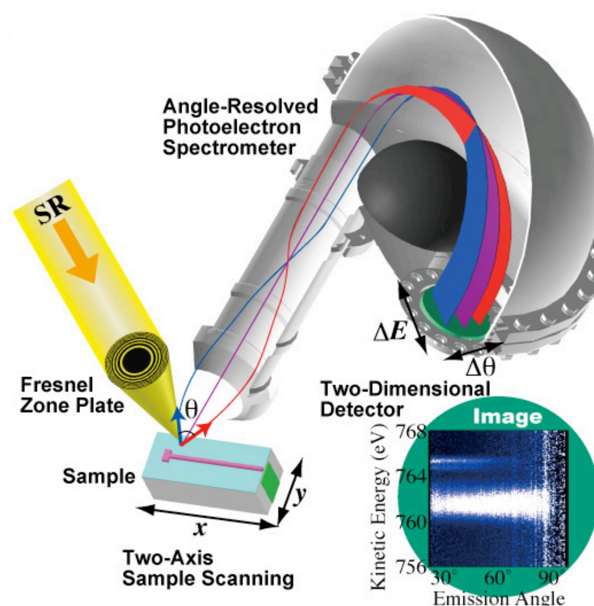


Figure 1. Conceptual scheme of 3D nano-ESCA system

distribution of Si 2*p* photoelectron spectra of 2.5-nm-thick SiON thin films on Si substrates. Sharp two-peak structures at a kinetic energy of around 765 eV and broad peak structures at around 762 eV are derived from Si substrates and SiON films, respectively. By increasing the emission angle, the intensity of the peak from the SiON films increases and the peak from Si substrates disappears, since the grazing-emission condition is more sensitive to the surface. Thus, the angular distribution corresponds to the probing-depth dependence of the photoelectron spectra and can be converted into the depth profiling z information using maximum-entropy methods. Thus, we can obtain three-dimensional x, y, and z distribution of the electronic structure and chemical bonding states of the samples on a nanometer scale.

For demonstrating the capability of our system, we carried out SPEM measurements on nano-patterned poly-Si gate electrodes fabricated on $HfO_2$ films. The line profile of Hf 4*f* core-level photoelectron intensity at the edge of the gate pattern in Fig. 3 is fitted using a step function convoluted with a Gaussian function, and the full width at half maximum of the Gaussian function under the best-fit condition is determined to be 70 nm. Therefore, we have confirmed that the spatial resolution better than 70 nm is achieved.

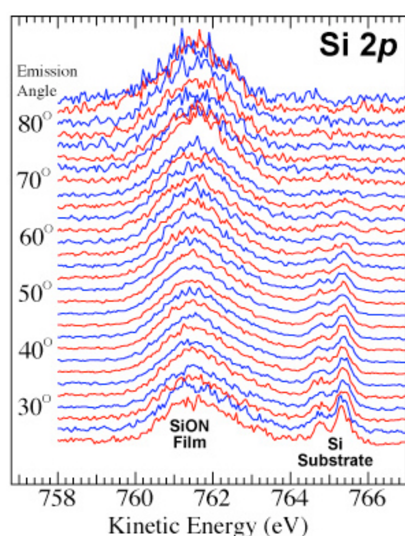


Figure 2. Simultaneously-detected angular distribution of Si 2*p* photoelectron spectra of 2.5-nm-thick SiON thin films on Si substrates obtained at the excitation photon energy of 870 eV

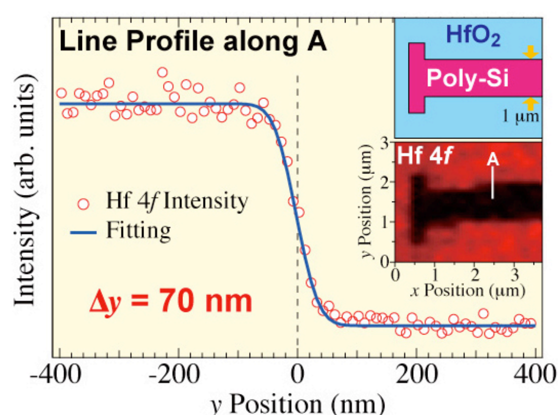


Figure 3. Estimation of the spatial resolution from the line profile at the edge of the gate pattern along line A in the inset of the figure. The inset shows a schematic view of nano-patterned poly-Si electrodes on $HfO_2$ films and Hf 4*f* photoelectron -intensity mapping of the nano-patterned poly-Si/$HfO_2$ structure, obtained at the excitation photon energy of 870 eV.

## References

[1] K. Horiba, Y. Nakamura, N. Nagamura, S. Toyoda, H. Kumigashira, M. Oshima, K. Amemiya, Y. Senba, and H. Ohashi, submitted to Rev. Sci. Instrum.

[2] M. Oshima, S. Toyoda, H. Kamada, T. Tanimura, Y. Nakamura, K. Horiba, and H. Kumigashira, ECS Trans. 33, 231 (2010).

[3] S. Toyoda, Y. Nakamura, K. Horiba, H. Kumigashira, M. Oshima and K. Amemiya, e-J. Surf. Sci. and Nanotech. 9, 224 (2011).