

# The Photoemission Electron Microscopy Project

Curiosity and inquisitiveness are two of the human great characters that extend mankind's activities towards prosperity. Wondering how the universe was formed, mankind has spent countless efforts gazing into deep space through telescope, hoping to pick up the traces left behind by events occurred billions years ago. On the other hand, pondering what is the smallest unit a material is built upon, people have constructed various kinds of microscopes to look for the ultimate building block. In both quests, image is the favorite method for demonstrating the evidence because a visual presentation makes us feel like we were there watching how it was happened. Such favorite in visual contact is not necessary a human predilection but has a good reason behind it. Taking the X-ray discovered more than 100 years ago as an example, its ability of seeing through object allows medical doctor to make correct life-saving procedures based on the patient's internal condition revealed by X-ray image. The power of X-ray is also appreciated by the scientists. For years, X-ray has been an indispensable analysis tool revealing structural and chemical information of materials of all kinds. Nevertheless, in the field of X-ray imaging, there were not many activities other than medical application due to lack of photon intensity. The situation changed in early 1990s, when the third generation synchrotron radiation facility that is capable of providing intense photons in wide energy range started to appear. The availability of high intense photon beam has since speeded up the pace of evolution of X-ray imaging technique.

In Taiwan, the Taiwan Light Source, a third generation synchrotron radiation light source,

started its operation in 1993, and the effort of establishing X-ray imaging techniques was begun soon after that. In 1999, the Synchrotron Radiation Research Center (SRRC) completed its first X-ray microscope -- Scanning Photoemission Microscopy (SPEM) station. The SPEM, a zone-plate-based microscope, is capable of providing image of the interested area by raster scanning the sample, and X-ray photoemission spectrum (XPS) within sub-micron area. Utilizing the high brightness of photon beams generated from the U5 undulator insertion device, the SPEM station has been very successful in helping perform various research subjects since its operation, as demonstrated in two articles in this issue (see W. F. Pong *et al.* and M. Zharnikov *et al.*). The effort of establishing a second X-ray microscope, X-ray Photoemission Electron Microscopy (PEEM) station, started in year 2000. Using a different approach comparing to SPEM, PEEM offers images of interested area one frame at a time with fixed photon energy, and spectroscopy analysis (X-ray absorption spectroscopy, XAS) with scanning photon energy. At the meantime, in order to develop a better PEEM system with an ultimate performance, SRRC has joined the project of developing an aberration-corrected PEEM system at the Advanced Light Source. The aberration-corrected PEEM system is expected to have an ultimate spatial resolution of better than 5 nm and a transmission efficiency several times of magnitude higher than this newly installed PEEM system discussed here.

The microscope adopted in our PEEM station is a commercial available all-electrostatic-lens OMICRON FOCUS-IS cryo-PEEM IEF system.



As illustrated in Fig. 1, the microscopic feature within the interested area is made observable by an electrostatic lens system. For PEEM, the probe is the photon beam, and the signals collected for imaging are electrons emitted from sample upon photon irradiation. When the sample is exposed to the photon beam, the emitted photoelectrons are first collected by an objective lens, and then aligned and magnified by deflector, stigmator and two projective lenses before reaching the imaging unit. The imaging unit consists of a multi-channel plate for intensifying the electron signal and an aluminum coated YAG crystal screen for transferring electron signal into visible light. The image appeared on the screen is digitally recorded by a thermo-electric cooled slow-scanned CCD camera which has  $1317 \times 1015$  pixels. To enhance spatial resolution as well as provide option for area selection on image, two apertures, contrast aperture and iris aperture, are positioned at the back focal plane and the image plane of objective lens, respectively. Using stick/slip movement, five different sized apertures  $1000 \mu\text{m}$ ,  $500 \mu\text{m}$ ,  $150 \mu\text{m}$ ,  $70 \mu\text{m}$  and  $30 \mu\text{m}$ , can be chosen for desired resolution at the expense of signal intensity.

To maximize the usability of PEEM station, two separated UHV chambers are included in the station: one is the main chamber for housing the PEEM system, and the other is the preparation chamber equipped with a sputtering gun and a load-lock system for preparing and transferring sample. As shown in Fig. 2, the two-chamber system is sitting on a top plate, which is separated from the supporting frame by 9 pieces of  $40 \text{ mm} \times 40 \text{ mm}$  vibration isolation pads, so that the

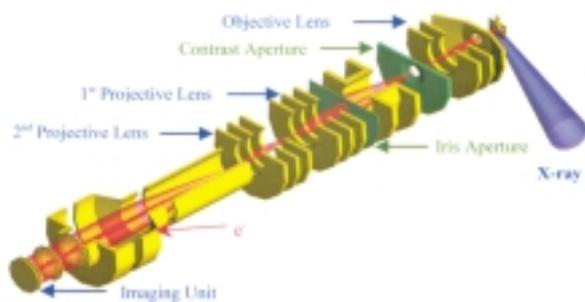


Fig. 1: Cartoon presentation of PEEM optics and electron path.

impact from ground vibration can be greatly reduced. A tightly integrated sample stage located in front of the electron lens system is designed to reduce the image blurring caused by relative motion between sample and lens system. Hence the resolution of this PEEM system is mainly limited by the aberration of electron optics system but not the vibration.

The PEEM station is located at the EPU-SGM beamline, which delivers intensive photon beams with various degrees of polarization generated by an elliptically polarized undulator insertion device. High brightness photon beam is highly desired because, as mentioned earlier, the signal intensity is weak when small aperture is used for higher image resolution. The polarization of the photon beam enables the possibility of magnetic imaging, using the polarization dependence of absorption cross-section in magnetic system. The EPU-SGM beamline is designed to deliver photon beams at

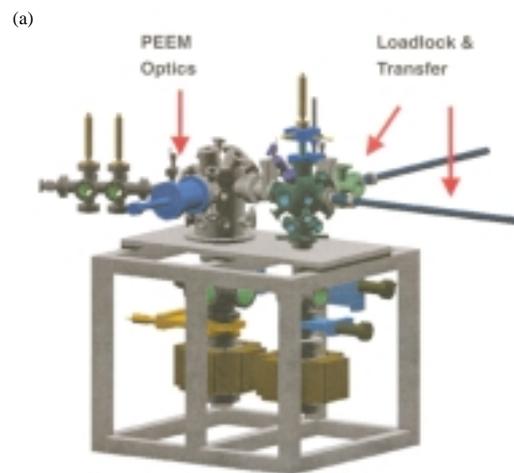
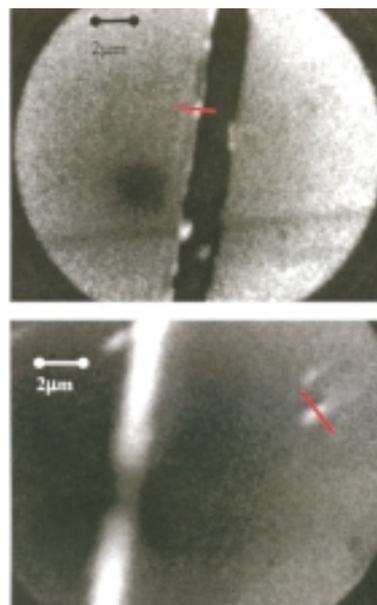


Fig. 2: (a) Schematic drawing of PEEM station. (b) Photo of PEEM station at the EPU-SGM beamline.

energies from 60 eV to 1400 eV with reserved polarization and an average calculated flux of  $10^{13}$  photons/sec/200mA. At the present time, the focused beam size measured at sample is about 1.5 mm  $\times$  0.1 mm (H  $\times$  V), which is much larger than the theoretical value in the horizontal direction. The photon brightness collected at sample will be increased when the horizontal focusing property is improved. With the current photon intensity collected, a PEEM image with reasonable resolution and quality can be recorded in seconds (image acquisition time is inversely proportional to the resolution required).

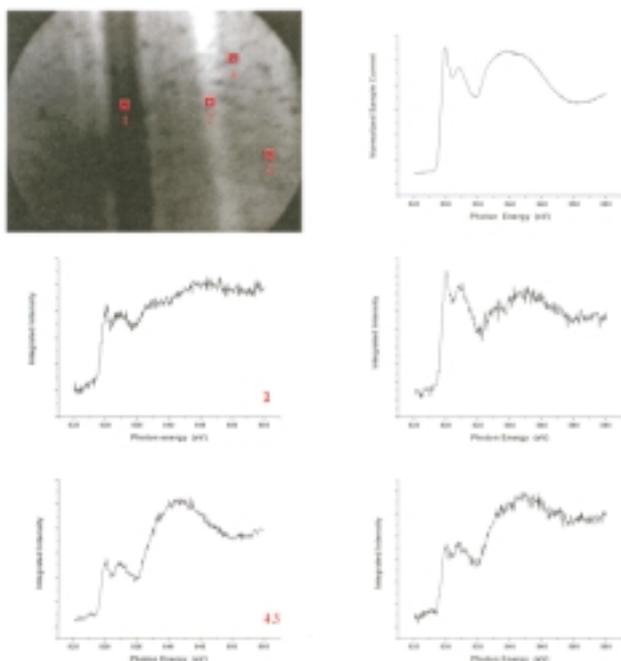
For the performance test of the PEEM system, the spatial resolution of an image and the capability of micro area chemical analysis employing XAS technique were performed. The test sample used for the resolution measurement is a Pd/Si sample, on which the 2  $\mu$  m wide palladium (Pd) strips are deposited on silicon (Si) substrate. The image of Pd stripes are recorded by using both mercury light and synchrotron radiation (SR) light, as shown in Fig. 3(a) and 3(b). A spatial resolution of 0.1  $\mu$  m is obtained with mercury light and 0.18  $\mu$  m with 130 eV photon beam. The reason for better resolution obtained using mercury light is that the photon energy 4.9 eV of the sharp mercury light chosen falls between the work function of Pd (5.22 eV) and Si (4.85 eV) and there is no measurable electron generated from the Pd area, hence better contrast image is obtained. For the case of SR, the intensity of electron signal is supposedly dominated by the absorption of the material exposed to the photon beam. The signal generated from the Si substrate is weaker than that from the Pd area when the sample is exposed to photon beam with energy 130 eV, as shown in Fig. 3 (b). This is possibly because the cross section of generating secondary electrons from Pd is higher than that of Si at photon energy 130 eV, and furthermore the test sample is highly contaminated so that no measurable signal generated from the Si L-edge absorption is obtained.

From a sequence of images taken with continuous change of photon energy, the intensity recorded at each pixel (or group of pixels) is a measurement of absorption spectrum. Considering



*Fig. 3: (a) The image of the Pd/Si sample taken with Hg light. The signal from the Pd stripes is hardly measurable, because the work function of generating secondary electron from Pd is higher than the energy provided by the Hg light. The best spatial resolution obtained in this image is 0.1  $\mu$  m. (b) The image of the Pd/Si sample taken with SR light. The signal from the Pd stripes is higher than that from the Si substrate at  $h\nu = 130$  eV. The best spatial resolution obtained in this image is 0.18  $\mu$  m. The dark area located at lower left part of both images shows the low efficiency of the MCP, which may be damaged.*

each pixel (or group of pixels) of an image contains four parameters, x-coordinate (x), y-coordinate (y), strength of electron emission ( $\sigma$ ), and corresponded photon energy ( $E_p$ ), a snap shot taken at photon energy  $E_p$  is the intensity distribution function  $I(x, y, \sigma; E_p)$ ; and the absorption information at location (x, y) is contained in a spectroscopy function  $S(\sigma, E_p; x, y)$ . We note that the X-ray absorption spectra extracted from a sequence of PEEM images are only meaningful if the selected micro area for counting the absorption intensity is greater than the spatial resolution. As shown in Fig. 4, there are four oxygen K-edge absorption spectra extracted from four different micro areas on a SiN thin film, in which different curve shapes shown in spectra show different chemical states of oxygen existed in different area. The X-ray penetration depth is greater than the electron escaping depth (few nm) so that the absorption spectrum can be used to study the interface properties of multi-



*Fig. 4: Micor-area absorption spectra taken from different areas on a SiN thin film. The TEY spectrum obtained from the sample current is also shown for comparison. Different shapes obtained from different area show different chemical states of oxygen existed in that area.*

layer thin-film system. That is, depending on the contrast selected, PEEM can be used to acquire image and micro-area absorption spectrum from either surface or interfaces to study the local change of chemical or electronic states.

After the assembly and initial test, the PEEM station was connected to the EPU-SGM beamline for commission in early 2002. Up to date, the system has shown spatial resolution of  $0.1 \mu\text{m}$  using mercury light and  $0.18 \mu\text{m}$  using synchrotron radiation light. Better spatial resolution can be obtained if a well-prepared sample is used. The measurement of photo-absorption from a micro area has also been well demonstrated, and is ready for study on the local changes of electronic or magnetic states in the sample.

In the future, significant efforts will be directed to perform scientific and technical orientated studies under collaboration with various research groups. At present, there are two collaboration undergoing: one is the study of artificial microstructure using topographic, elemental, and/or magnetic contrast measurement, collaborating with Prof. Jong-Chin Wu from

National Changhua University of Education; and the other is the study of probing depth of thin film systems as well as finite area XAS analysis on new materials with Prof. Jung-Chun Huang from National Cheng Kung University. At the meantime, data acquisition/analysis software programs and additional experimental options will be continuously upgraded and installed to improve the overall system performance.

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