Photoemission electron microscopy beamline at the Synchrotron Light Research Institute

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Abstract. Photoemission electron microscopy end station is installed at Beamline 3.2b of Siam Photon Laboratory of the Synchrotron Light Research Institute in Thailand. The system has been tested for sample imaging using synchrotron, a UV lamp and an electron gun as sources with successful results. Multi-disciplinary research at the PEEM end station is reported in this paper. Arc discharge problem which have been found during the experiments is also discussed.

1. Introduction
Photoemission electron microscopy (PEEM) is an imaging technique which provides microscopic images of samples using electrons created from photoelectric effect. PEEM can perform X-ray absorption spectroscopy (XAS) to reveal chemical and electronic information near the surface similar to the total-electron-yield (TEY) mode in conventional XAS measurement, with spatial resolution of typically about one µm or less. XAS measurement by PEEM is surface sensitive characterized by a probe depth of about 10 nm. The spatial resolution and the surface sensitivity especially makes PEEM unique among other techniques at the Siam Photon Laboratory (SPL). PEEM is installed at Beamline 3.2b, the only beamline at SPL which utilizes synchrotron radiation from a planar undulator [1]. The beamline was designed to provide photons with the energy range of 40-1040eV and the flux of more than 1×10⁹ photons/s/100 mA at the energy bandwidth of less than 0.01% suitable for the PEEM technique. Compared to PEEM research at many other synchrotron facilities where elliptically-polarized undulators are used and X-ray magnetic circular dichroism are studied extensively, we focus our effort on XAS measurements of semiconductors, biological samples and, more recently, metallurgical samples using our linearly-polarized undulator.

2. Installation and commissioning results
The PEEM end station is made by Elmitec GmbH in Germany. The system was tested using Hg arc discharge lamp as a source with successful results before being permanently installed at Beamline 3.2b. A gold mesh with 64-µm nominal aperture was installed in front of the sample position to measure the incoming photon intensity during the PEEM measurements. The alignment of PEEM with the photon beam was initially performed using a motorized mechanical support which moved the PEEM chamber in the plane normal to the photon beam direction. Subsequently, the alignments were performed by the adjustments of the Kirkpatrick-Baez (KB) mirrors to compensate the beam shift over...
The beam size at the current sample position provided by the KB mirrors is estimated as 100 µm vertically and 800 µm horizontally, suitable for our typical 75 µm and 50 µm field-of-view imaging. The future mechanical redesign of the mirror holders will allow the KB mirrors to be bent in order to adjust the focal length and thus the spot size. We also plan to install a YAG crystal and a photodiode in addition to the gold mesh for the beam-position monitoring and the total flux measurement, respectively.

One of the major obstacles in the operation of PEEM is the arc discharge under the electric field between the -20 kV biased sample and the objective lens. The degassing of samples during the measurement is suspected as the trigger of this problem. For experiments which require in-situ imaging at high temperature where the sample degassing rate increases significantly, the arc discharge happens more frequently and can be strong enough to damage the sample surface. There are two main solutions to prevent the arc: reducing the bias voltage, and increasing the distance between the sample and the objective lens. Reduction of bias voltage requires new settings of PEEM's optics, and recalibration of the image magnification. We have found that this method results in the severe loss of image intensity, which is not acceptable as the quality of XAS spectra strongly depends on it. In the second method, the sample is moved about one or two mm further away from the objective lens. This requires an adjustment of the objective lens current from the optimal value of 1650 mA, and also a new alignment of the synchrotron beam. It has been found that the latter method works efficiently and does little effect to the image quality at the field of view of 75 µm and 50 µm.

The intensity of electron emission from the sample determines the magnification power of PEEM. At the higher magnification (the smaller field of view of PEEM images), a higher photon flux is needed to generate enough electron emission for imaging. At the energy range of 40-200 eV provided by the 600 lines/mm grating of BL3.2 (where the photon flux is more than 1×10^{12} photons/s/100 mA from the calculation [1]), the field of view could go down to 10 µm with good image intensity. For the higher photon energy range, i.e. 200-600 eV provided by the 1200 lines/mm grating, and 400-1040 eV provided by the 2400 lines/mm grating, the field of view is usually kept at 75 µm and 50 µm. Spatially-resolved X-ray photoelectron spectroscopy (XPS) in PEEM is also performed using an imaging electron energy analyser with the energy resolution of 500 meV. XPS is performed less often than XAS as it requires much longer acquisition time, especially when multiple core levels are measured for quantitative analysis. However, for shallow core levels and valence bands, the time required for the XPS measurements is comparatively shorter and thus can be done more regularly. Angle-resolved photoemission spectroscopy by PEEM is yet to be tested under optimal alignment of the electron optics, and with suitable clean crystalline samples. UV PEEM with Hg arc-discharge lamp is frequently used together with the synchrotron to help identify metal/semiconductor domains by providing strong contrast between them. Low-energy electron microscopy (LEEM) has also been routinely performed. Because the emission from the electron gun can be increased easily by reducing the Wehnelt voltage, the field of view of LEEM images can be as small as two µm without sacrificing image intensity. However, the image resolution in LEEM relies considerably on the good alignment of the electron optics and the flatness of the samples. Selected-area low-energy electron diffraction (LEED) is also performed regularly to determine domain structures in single-crystal and polycrystalline samples.

3. Research focus at PEEM beamline

The first experiments at the SPL's PEEM end station consist mostly of semiconductor samples. The research focuses on strain in Si and SiGe alloy created by the lattice mismatch between Si and Ge. The strain induces a shift in the conduction band which can be detected by measuring L_{2,3} absorption edge of Si near 100 eV [2]. With PEEM, the conduction band can be measured locally so that the distribution of the energy shift can be determined. The project largely involves the use of single-crystal Si/SiGe nanomembranes in which strain can be applied elastically by stretching, allowing for engineering of their electronic properties [3]. The study of GaAs semiconductors is also conducted with the focus on in-situ observation of Ga droplet formation at high temperature [4] in various
constrained conditions. Other research projects include novel oxide semiconductors such as SrTiO$_3$ in which a large electrical conductivity from two-dimensional electron gas is generated by irradiating with high-intensity photons [5], and future electronic materials like graphene [6]. Study of epitaxial growth of graphene sheets on 6H SiC substrates by LEEM have shown that scratch lines left on the surface of the substrates affect the growth rate and the average size of the graphene sheets during annealing.

Future research may involve the closer look at the electronic properties of surface quantum well in semiconductors, which has been studied by the intensity-voltage analysis of LEEM [6] as well as TEY XAS [7]. PEEM and LEEM techniques at SPL will also be used to study organic semiconductors such as pentacene thin film. The project has been started by the installation of in-vacuum deposition system consisting of a resistively heated crucible, sample temperature measurement by a thermocouple, and a deposition-rate monitor inside the load-lock chamber of PEEM (Figure 1). During the film deposition, a substrate is clipped on a 0.25”-diameter W wire and is heated radiatively by a heater coil placed less then 5 mm away. The substrate temperature, which is the most important parameter to determine the final structure of organic films, can be well controlled because the heat loss is limited by the small thermal conduction through the thin W wire clip. The first test of the system exhibited the pentacene growth on SiO$_2$ at the steady rate of 5 ML per minute.

Metallurgical engineering is one of the most promising field of PEEM research at SPL, with collaboration from both academics and the stainless-steel industries in Thailand. PEEM is used to record the formation of micro-crystalline grains, phase transitions and precipitations of materials in stainless steels in real-time [8]. Corrosion behaviors of stainless steels under various coating conditions have also been studied. PEEM gives information about chemical reactions and products on the microscopic domains on the surface where the local corrosion takes place. Different types of coatings for industrial uses are investigated, including TiN, CrN and diamond-like carbon. In the first corrosion study project, CrN film coated on stainless steel samples with various degrees of surface roughness were tested in NaCl solution at pH 2, 7 and 10. PEEM images from the samples help to characterize the corrosion mechanism by the shapes and sizes of the corrosion pits, and the XAS measurements help to identify difference in chemical compositions of the CrN film and the corrosion pits as shown in Figure 2. Initial results suggest the importance of Cr$_2$O$_3$ protective layer in the corrosion resistance mechanism of the CrN film [9].
Figure 2. XAS spectra measured from the corrosion pit and the intact area of CrN film coated on stainless-steel substrates after a corrosion test in NaCl solution at pH 2. The wide-scan spectra were taken with the acquisition time of 1000 ms per energy step, showing the difference between the amount of N, O and Cr in the corrosion pits and the intact area. The inset shows a fine scan with acquisition time of 3000 ms per energy step for Fe L edge.

Measurements of biological samples are difficult in PEEM. They must be freeze-dried, coated with metal films for electrical conductivity, and then left degassing in the loadlock chamber for at least a few days before imaging. Due to the small escape depth of the electrons in the TEY XAS measurement, the thickness of the metal coating is required to be about one nm. This is achieved by a separate mini electron-beam evaporator where the thickness of the metal coating can be controlled precisely. By this method, biological samples such as cells and tissue sections have been imaged by PEEM, and the measurements of N K-edge spectra in some tissue samples have been successful.

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