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# The Soft X-ray Scanning Photoemission Microscopy Project at SRRC

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The Synchrotron Radiation Research Center (SRRC) and the Institute of Atomic and Molecular Sciences (IAMS) have initiated a project to construct a scanning photoelectron spectromicroscopy end station at SRRC (SRRC-SPEM). High-brightness soft X-rays will be provided by the U5 undulator beamline. Zone-plate-based soft X-ray optics will be used to focus the beam to form the microprobe. A hemispherical sector analyser with multichannel detection capability will collect the photoelectrons. A total of up to 32 images can be acquired concurrently. The apparatus is also equipped with a sample distribution system for *in situ* sample preparation and characterization in conjunction with other surface spectroscopic techniques.

**Keywords: zone plates; scanning microscopy; X-ray photoelectron spectroscopy.**

## 1. Introduction

Engineering of new materials and the continuous miniaturization of many devices require characterization methods with high spatial resolution to visualize and locate ultrafine material structures. The high brightness of the third-generation synchrotron facilities has enhanced new research efforts in laterally resolved electron spectroscopy, so-called spectromicroscopy and microspectroscopy. The leading groups in this field from the USA, Germany, Italy and Sweden are working on the improvement of the spatial resolution, which is mainly determined by the soft X-ray optics for the scanning type and by the electron optics for the imaging type of microscopes. The technical development in this field, as well as initial scientific results, are illustrated in the recent special issue of the *Journal of Electron Spectroscopy and Related Phenomena* (Ade, 1997).

In 1994, SRRC and IAMS initiated a program to construct a scanning photoelectron spectromicroscope (SRRC-SPEM). The SRRC-SPEM, which is an instrument that combines the techniques of X-ray photoelectron spectroscopy (XPS) and zone-plate (ZP) microfocusing, is designed for performing spatially resolved elemental and chemical analysis (Ko *et al.*, 1997; Klauser *et al.*, 1996, 1997).

A U5 undulator provides a bright photon source (a requirement for scanning microscopy) in the soft X-ray range with a high degree of transverse coherence for zone-plate focusing. The photon energy can be selected with a spherical-grating monochromator (SGM) between 80 and 1200 eV.

A hemispherical sector analyser (HSA) with multichannel detection (MCD) capability will collect the photoelectrons. Elemental and chemical mappings of material surfaces are formed by raster scanning the sample with the focused beam fixed. The system is capable of simultaneously detecting the total electron yield, transmitted flux and sample drain current. The data-acquisition system is designed to acquire a total of up to 32 images concurrently.

This project emphasizes the *in situ* fabrication and analysis of ultrafine structures. This requires flexibility in sample preparation and supplementary diagnostic tools. A special transfer chamber combines the spectromicroscopy chamber with a fast-entry air lock, an analysis/preparation chamber equipped with LEED, XPS and scanning Auger electron microscope (SAM) devices and an AFM/STM chamber. The scientific programs include chemical etching and deposition of semiconductors, interfacial interactions on wide-band-gap materials, metal clusters and thin films in the submicrometre spatial regime.

## 2. U5-SGM beamline

SRRC is a 1.5 GeV third-generation synchrotron radiation facility with sixfold symmetry and triple-bend-achromat lattice structure (Liu, 1995). The spectromicroscopy end station will be installed in the U5 beamline. The U5 undulator is a variable-gap C-type undulator contracted to Danfysik, Denmark, in June 1995, and installed in March 1997 at one of the straight sections. It has a total length of 3.9 m and provides high-brightness radiation, $>2\times$

$10^{17}$ photons $mm^{-2}$ $mrad^{-2}$ (0.1% bandwidth)$^{-1}$ (200 mA)$^{-1}$ in the energy range 80–400 eV for the first harmonics at gaps of 18–50 mm (Table 1).

The U5-SGM beamline includes a vertical-focusing mirror (VFM), moveable entrance and exit slits, a spherical-grating monochromator (SGM) and bendable vertical and horizontal refocusing mirrors (VRFM and HRFM), which are used to steer the focal point (Tseng *et al.*, 1995). Fig. 1 shows the optical layout and the mechanical design. Inside the SGM are four gratings which cover photon energies from 80 to 1200 eV (Table 1). The merit of adopting the bendable VRFM-HRFM design is that the focal point can always be kept fixed at the same location despite changes in photon energies. A maximum photon flux of $10^{14}$ photons $s^{-1}$ (0.1% bandwidth)$^{-1}$ (200 mA)$^{-1}$ and a resolving power of 3500–15000 are predicted (Table 1).

## 3. Zone-plate focusing, beam collimation and beam-position monitoring

After the optics, the monochromatic beam with a spot size of 100 μm × 150 μm to 200 μm × 250 μm FWHM at the focal point of the beamline will pass through a pinhole (in a pinhole array) and the beam-position monitor (BPM) chambers, and finally will be further focused by a zone plate inside the spectromicroscopy end station. The photon BPM is an absolute XUV silicon photodiode, model No. AXUV-PS1, with a 300 μm diameter central opening (we use it for the purpose of beam collimation) from International Radiation Detectors (IRD). The AXUV-PS1 silicon photodiode has four equally spaced detection areas (similar to a position-sensitive detector used in a laser stabilization system). By detecting the signals from each of the detectors, the transverse beam position can be determined. In the future, these signals will feed back to the mirror control console to adjust the mirrors for automatic beam alignment. The pinhole array consists of a series of vertically aligned pinholes, ranging from 20 to 1000 μm in diameter. Zone-plate optics further demagnify the source from this pinhole to form the microprobe. Zone plates are obtained from Gate Micro Technology, Rome, Italy, with expected diffraction-limited resolution of about 80 nm. Zone plates are diffractive optics (Michette, 1986) which require an order-selecting aperture (OSA) to select the desired order for focusing (usually the first-order diffraction is used).

## 4. Spectromicroscopy end station

The entire end station includes the spectromicroscopy system, the sample distribution/transfer system, and the sample analysis system. Fig. 2 shows a diagram of the entire system.

### 4.1. *Spectromicroscopy system*

Major components for spectromicroscopy applications are attached to the spectromicroscopy chamber, which

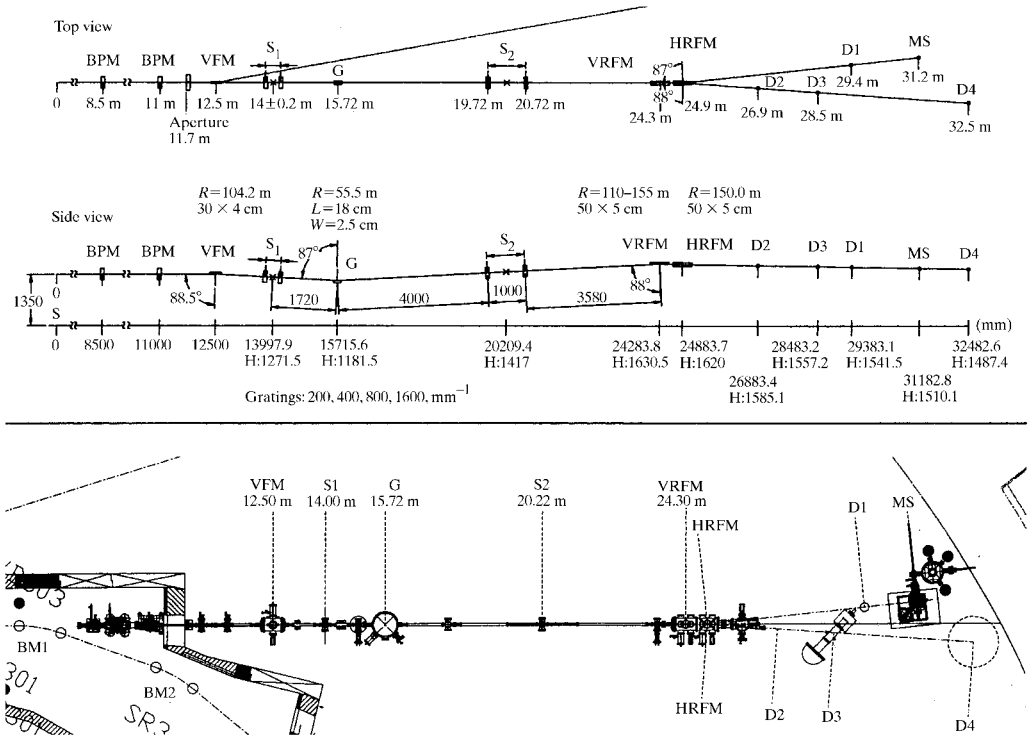


**Figure 1**
The optical layout and the mechanical design of the U5-SGM beamline.

**Table 1**
Beamline specifications.

| Energy range (eV) | Grating type (lines $mm^{-1}$) | Resolving power ($\lambda/\Delta\lambda$) | Flux [photons $s^{-1}$ (200 mA)$^{-1}$ (0.1% bandwidth)$^{-1}$] | Spot size (μm) | Horizontal angular acceptance (mrad) |
|---|---|---|---|---|---|
| 50–200 | 200 | 3500–9500 | $2 \times 10^{14}$ | <95 | 0.06–0.09 |
| 100–400 | 400 | 3500–10500 | $1.6 \times 10^{14}$ | <90 | 0.06–0.09 |
| 200–800 | 800 | 3500–12000 | $2 \times 10^{13}$ | <90 | 0.06–0.09 |
| 400–1200 | 1000 | 3500–14000 | $1 \times 10^{13}$ | <90 | 0.06–0.09 |

includes the following: sample-scanning/manipulation system, sample-transfer system, signal-detection system, electron-energy analyser, sputter gun, electron gun, sample-neutralization gun, and ZP/OSA-positioning device, alignment system. Essential functions of the above components will be described. Some of the components are depicted in Fig. 3.

4.1.1. *Electron energy analyser.* Photoelectrons are generated within the area irradiated by the focused photon beam and then collected by an HSA within an acceptance angle defined by the input lens of the analyser. Since the photoelectrons are energy-dispersed in the exit plane of the HSA, the use of an MCD allows us to monitor photoelectrons of different kinetic energies simultaneously. Each channel of the MCD is spaced equally in energy. The signal from each channel of the MCD can be used for image formation (each MCD channel has its own discriminator and amplifier and runs in pulse-counting mode for digitalization). All 16 MCD channels can be acquired simultaneously for photoelectron image formation, which means simultaneous acquisition of 16 photoelectron images (Ko, 1995; Ko *et al.*, 1995). The scheme is illustrated in Fig. 4. The HSA is from Physical Electronics (PHI) with an Omni V lens. There are two major reasons why we prefer the PHI Omni V electron energy analyser: (i) the geometry of the input lens is suitable for the tight space inside the chamber, (ii) it has a quadruple deflection system in the input lens. This deflection system is used to deflect the electrons (generated from the imaged area on the sample) into the analyser. By rastering the imaged area, an image can be taken with a spatial resolution of about 50 μm. This capability of the input lens greatly reduces the time in aligning the focused beam to the analysis area of the analyser.

4.1.2. *Sample positioning and scanning mechanism.* (*a*) Coarse-scanning scheme. The sample-scanning scheme includes coarse and fine scanning, which is put together by attaching a fine *xy* sample-scanning stage to an *xyz* sample manipulator used as a coarse-scanning device. We use an *xyz* sample manipulator from Vacuum Generators, model Omniax, as the coarse-scanning device because of its high mechanical stability. The advantage of using a commercially available manipulator is the capability of adding auxiliary devices, *e.g.* sample heating and cooling systems.

The VG Omniax manipulator is motorized using a stepping-motor control. Resolution for the half-step movement is 0.5 μm. This manipulator provides ±25 mm travel in the horizontal and 400 mm in the vertical. The 400 mm vertical travel allows samples to be transferred to the lower region of the spectromicroscopy chamber for other sample manipulations or signal detection.

(*b*) Fine-scanning scheme. The fine-sample-scanning stage is based on Physik Instrumente's (PI) model P-731.20 flexure stage modified for UHV with two piezoelectric drives and two capacitive sensors (one for each direction). It has a resolution of 5 nm with close loop operation using capacitive sensors for feedback control and has a travel range of 100 μm × 100 μm. This flexure stage is directly

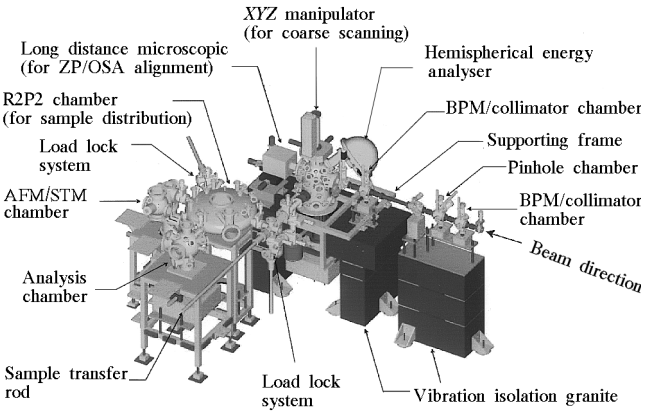


**Figure 2**
The SRRC-SPEM. Major apparatus include the beam collimation/BMP system, the spectromicroscopy system, the sample distribution/transfer system, the AFM/STM system and the analysis system.

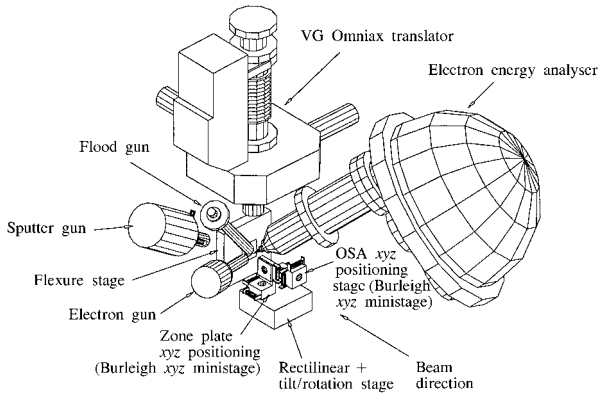


**Figure 3**
Part of the major components inside the spectromicroscopy chamber.

mounted to the supporting tube of the VG Omniax *xyz* sample manipulator.

4.1.3. *ZP/OSA positioning device.* The ZP/OSA positioning device is based on a cross-roller-slide design driven by Burleigh's Inchworm UHV motors. The ZP mount is attached to *xyz* stages (ZP stage) with 25 mm travel in the beam direction and a 10 mm travel range in the transverse directions. The OSA mount is attached to an *xyz* stage (OSA stage) with 10 mm travel in each direction. Both the ZP and the OSA stages are mounted on a linear stage with a 40 mm travel range in the beam direction. This three-stage device is mounted to a 10″ multi-sealed flange as one integrated unit for easy installation and maintenance.

4.1.4. *Other components.* The spectromicroscopy chamber is also equipped with an ion gun for simple Ar sputter-cleaning, a flood gun for sample neutralization and an electron gun for alignment (Fig. 3). More dedicated sample preparation and analysis can be performed by transferring the sample to the specific chambers attached to the sample distribution system.

Detection of sample current, total electron yield and transmitted flux are also implemented. The total electron yield will be detected by a channel electron multiplier, while transmitted flux will be detected by a channel electron multiplier or a silicon photodiode from IRD.

4.1.5. *Alignment of the focusing optics.* Inside the spectromicroscopy chamber and downstream of the zone-plate focusing optics, a Chevron detector (microchannel plates) with phosphor screen will be used to inspect the defocused radiation pattern of the ZP/OSA optics for aligning the ZP and the OSA. This defocused radiation pattern shown on the phosphor screen (it has a doughnut shape if the ZP and the OSA are aligned) will be examined by a long-distance microscope through a view port (Ko, 1995). The Chevron detector, the silicon photodiode and the channel electron multiplier are mounted together on a rotational and linear feedthrough. Users can select one of the detectors by manipulating the rotational and the linear motion of the feedthrough.

### 4.2. *Sample transfer and analysis system*

Applying SPEM as a unique method for solving relevant scientific problems has been previously limited by the lack of *in situ* sample preparation and further characterization. The space inside the microscopy chamber is very much restricted to accommodate optics and alignment tools and leaves only little room for simple sample treatment, such as ion sputtering. We are particularly interested in the investigations of growth and etch processes and cluster nucleation on surfaces. These require, however, an extensive preparation of the sample, such as controlled overlayer deposition and gas exposure. We decided to attach a distribution chamber to the system which allows maximal flexibility in manipulation and analysis of the specimens.

The samples, fixed to a modified sample plate from Omicron, can be removed from the microscopy chamber by a transfer rod and moved to a transfer arm mounted at the centre of a 30″ carousel chamber from VG ('R2P2').

We will connect a fast-entry lock, an analysis chamber and an AFM/STM unit to the system. The ESCA Microscopy group at ELETTRA suggested in an earlier publication (Nataletti *et al.*, 1992) the combination of SPEM with SAM and STM. These three techniques are complementary microscopies aiming at different structural and spectroscopic information and spatial resolution. We have adopted a similar approach. Our SAM has a spatial resolution of 0.1 μm, comparable with that of the SPEM. The combination of these methods is ideal to determine micro- and nanostructures, the effects of cluster size and shape, the location of reactive sites, and the role of catalytic promoters or other additives.

The analysis chamber is a spherical one-level chamber equipped with a VG CLAM4 electron energy analyser and a VG LEG1000 electron gun with scanning unit for SAM. The sample can be annealed by electron-beam heating and cooled through an attachment of an $LN_2$ reservoir. Further chamber ports are for X-ray source, LEED optics and sample preparation, such as atom source and Knudsen cell. The analysis chamber can also be operated independently

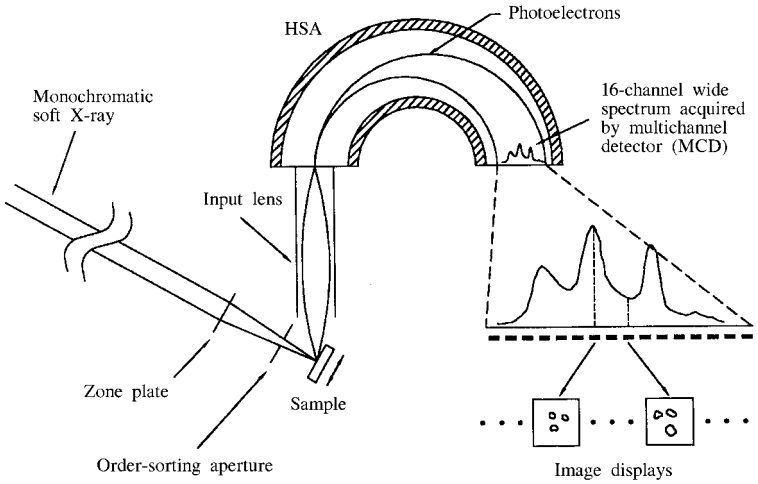


**Figure 4**
Schematic of photoelectron image formation by a hemispherical sector analyser and multichannel detection.

with a separate entry lock and a port for the beamline connection.

The AFM/STM system consists of an adapter chamber attached to the distribution chamber to accept the sample plate from the transfer arm and the AFM/STM unit from Omicron. The adapter chamber has simple facilities for sample treatment, so that the AFM/STM system can also be operated separately.

## 5. Vibration isolation

Vibration isolation is an important issue in the design of such systems. The measured vibrational magnitude of the ground floor is less than 0.1 μm. For this reason, we adopt passive vibration isolation. The whole spectromicroscopy system is mounted on a 2 ton granite bench with vibration isolation pads in between the granite and the legs.

## 6. Electronics and image/data-acquisition system

The image-acquisition system has to incorporate the motion control and the data input. In order to synchronize the data input with the scanning motion, we have adopted an electronic hand-shaking and data digitalization scheme (Fig. 5). For the purpose of digitalization, all signal measurements are converted to frequencies, which can be expressed by the following relationship,

$$f = kS,$$

where $S$ is the magnitude of the measured signal, $f$ is the converted frequency and $k$ is a conversion factor. In other words, $f$ is modulated by $S$. For example, current signals will pass through a current-to-frequency converter. After the signal-to-frequency conversion, the frequency signals will be collected by a multichannel scaler (MCS). In order to acquire digitized scanning images, the data collection of the MCS has to be in synchronization with the scan motion using electronic hand-shaking between the motion control signals and the MCS. Fig. 5 illustrates the concept of synchronization between the signal inputs and the motion controls. Each single MCS channel can form an image. Our acquisition system is designed to collect up to 32 images simultaneously. The 32-channel MCS uses a VME interface to ensure a fast data-transfer rate (up to 30 Mbyte $s^{-1}$) between the MCS module and the host computer. The MCS channel assignments are 16 channels for MCD detection, two for transmitted flux detection (one for signals from the channel electron multiplier and one from the silicon photodiode), one for sample drain current, one for total electron yield, one for a fixed frequency input (clock), and the rest are spare channels, which can be reserved for other signals (*e.g.* the storage-ring current signal, temperature, pressure *etc.*). The purpose of the clock input is to keep record of the motion speed (for a slower speed, more counts are received by the MCS per unit time). The rest of the MCS channels are normalized to this clock to eliminate variation of the scanning speed.

## 7. Conclusions

The SPEM system promises to overcome many of the difficulties faced by electron microprobes. Large categories of heterogeneous materials that are out of reach using the electron microprobe techniques can now be studied by this novel microscopy method. The XPS technique has always been a powerful tool in surface chemistry analysis. Due to the traditional drawback of poor spatial resolution, its applications in microscopy are very limited. However, the advent of the micro-XPS technique provides a new tool for microscopy analysis on material surfaces.

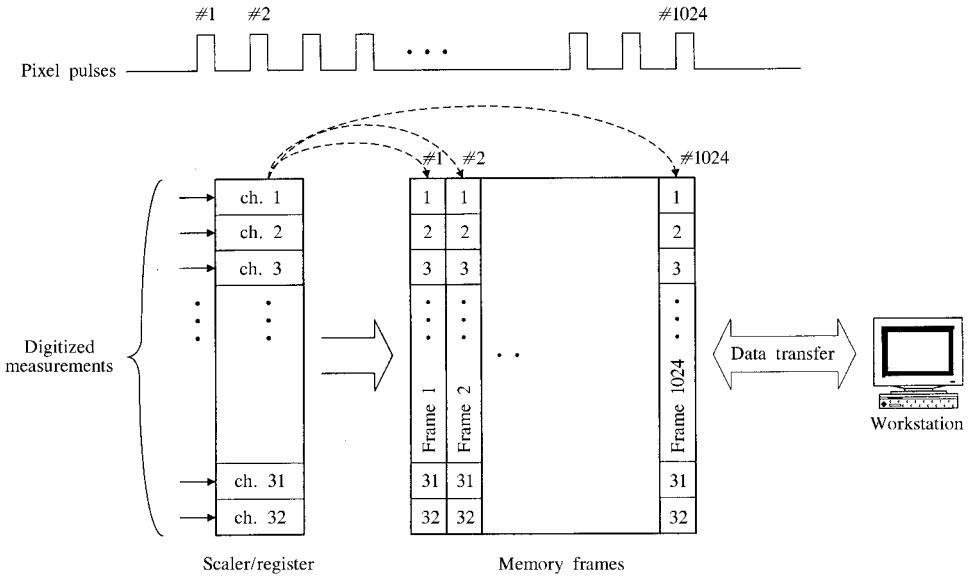


**Figure 5**
Schematic of the hand-shaking mechanism between the motion control signals and the input data signals.

In addition to the basic functions of such an SPEM system, our design guideline has incorporated the following concepts: flexibility, ability for future expansion, and compatibility with outside users' equipment. For example, our current sample-scanning design can be easily converted to zone-plate scanning for future applications. For experiments requiring specimen heating/cooling, the system can be upgraded using commercially available products. With some modifications on the ZP/OSA positioning device, our system is also able to operate in the micro-XANES mode in UHV. The use of Omicron's sample plate as our standard sample plate ensures maximal compatibility with outside users' surface preparation/analysis systems, which can be attached to the sample distribution chamber and become part of our system. The SAM and AFM/STM apparatus provide additional complementary microscopic techniques.

Currently, the U5 undulator and the U5-SGM beamline are installed. The spectromicroscopy system is under construction and expected to be finished before the middle of 1998.

We are very grateful to those who are involved in the U5 undulator project and the U5 beamline design and construction. The support we received from SRRC was enormous and encouraging. The work is supported by SRRC, the Institute of Molecular and Atomic Sciences of Academia Sinica and the National Science Council of the Republic of China under Grant Nos. NSC 85-2613-M-001-001 and NSC 85-2613-M-001-004.

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