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The development of synchrotron-assisted scanning probe microscopy at NSRRC

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Abstract. Synchrotron-based X-ray microspectroscopy is a technique that brings together microscopy and X-ray spectroscopy. It can be considered as an experimental approach capable of extracting X-ray spectrum from a finite area, or an alternative way of constructing images with spectroscopic contrast. The goal of this project is to integrate the functions of scanning tunnelling electron microscope (STM) with near edge X-ray absorption fine structure (NEXAFS) spectroscopy. Here, we describe our experimental setup, followed by recent results that demonstrate the feasibility of acquiring NEXAFS spectrum with a SiO₂ coated STM tip.

1. Introduction

Scanning tunneling microscopy (STM) is a technique capable of resolving the surface structure at atomic scale. However, while sensitive on current detection, the STM does not generally discriminate the kinetic energies of electrons. As a result, there is little chemical information can be extracted from the STM images. On the other hand, the synchrotron-based photoelectron spectroscopy is known to have a high chemical sensitivity through the incorporation of either an electron energy dispersive device or an energy tunable photon source. As it is not a trivial task to focus soft X-ray into nanometer scale, the photoelectron spectroscopy is hardly the technique of choice to examine a single structure prepared in nanometer dimension. Nevertheless, considering both techniques detect electrons, it seems reasonable to explore if it is possible to integrate STM with synchrotron radiation (SR) so that both element specific and spatially-resolved information can be accessed via a single hybrid instrument. The concept of integrating a scanning probe microscope with other spectroscopy techniques has been under pursuit since 1990s [¹-⁸]. At NSRRC, the effort in constructing a synchrotron-assisted scanning probe microscope was initiated jointly in 2009 by National Central University and NSRRC. In this paper we report the status and recent progress of the SR-STM station at Taiwan Light Source (TLS).

2. Experimental setup

The SR-STM system at TLS is powered by a 3.9 m long undulator, U50, which delivers 3.6x10¹⁷ photons mm⁻² mrad⁻² at 360 mA storage ring current [⁹]. Using a spherical grating monochromator, the beamline BL09 hosting U50 is operated within an energy window of 80 to 1200 eV. To enhance the
photon brightness, a set of Kirkpatrick-Baez (KB) mirror was installed at BL05A2 branch to focus incoming photon down to a 20 \( \mu m \times 70 \mu m \) spot at the sample position. The STM system used in this study consists of a UNISOKU USM 1000 system, a sample preparation chamber, and a fast load lock system. Optical alignment was performed when the system is connected to the beamline to ensure a proper X-ray illumination on STM tip/sample. In current setup, we have a 5° of X-ray incident angle measured from the sample surface. Figure 1 shows the optical layout of SR-STM at BL05A2.

![Figure 1. Schematic drawing of SR-STM setup at TLS beamline BL05A2.](image)

3. Insulated tip preparation

One challenging issue in developing the synchrotron-assisted STM (ST-STM) is to collect only those electrons from regions of interest. However, the grazing incidence of micrometer-sized photon beam means that the area capable of emitting photo-excited electrons is in the order of millimeter. To avoid the collection of electrons emitting from regions far away from the tip, the metallic tip needs to be modified\(^{[10,11]}\). Our approach is to cover the tip with a thin glass layer except its apex. Following is a brief description of how the glass-covered tip was fabricated; a tungsten (W) tip of 0.5 mm in diameter was inserted into a glass tube that has inner and outer diameters of 0.75 and 1.0 mm, respectively. The glass surrounding tip apex was made thinner by pulling the glass tube at a temperature close to its melting point. A 50 ML hydrofluoric acid was use to etch away the final glass layer covering tip apex. Glass covered W-tip with less than 5 \( \mu m \) opening at apex was successfully fabricated. Figure 2 shows the schematic drawing of our in-house setting for tip modification. In (I), (a) is an aluminum made frame which supports of all the installation, (b) is a macro ceramic which holds up the electrodes (c), power connector (d) and filament(e), (f) is an O.D. 1mm I.D. 0.75mm capillary glass tube, (g) is an O.D. 2mm capillary glass tube and it connects with (f), (h) is a screw used to fix (f) in order to stops the its sliding , (i) is a plastic tube connected to pumping system, (j) is a ~3g metal plate for pulling the capillary glass(g). In (II), it illustrates the detail arrangement between tip and filament. (III) and (IV) are SEM images of a glass-coated W tip before and after the HF treatment.

![Figure 2. Schematic drawing of the apparatus that was used to fabricate tip insulation.](image)
4. Results and discussions

Fig. 3(a) is a STM image of Cu(100)/Co (200 nm x 200 nm) recorded with a standard W-tip at constant current mode (0.2 nA). The Co islands were clearly observed before X-ray irradiation. However, upon the irradiation of energetic photon (778 eV), the image gets distorted immediately. Such a phenomenon is understandable based on the facts that X-ray illumination would cause both W-tip and surface to emit photoelectron. The tip would first experience a sudden increase in current upon the arrival of photon, followed with a response of fast retraction from the proximity of surface. What was unexpected in fig. 3(a) is the appearance of strip patterns within the period of time that X-ray irradiates. As the spatial modulation seen in image corresponds to a 60 Hz modulation in time domain, we believed the periodic patterns seen in fig. 3(a) are actually originated from the responses of STM electronics trying to accommodate photon-induced current. However, unlike the tunneling current is a quantity sensitive to tip-surface separation, the photon-induced current is not. As a result, the STM feedback system would lead to a period pattern as depict in fig. 3(a). Besides the image acquisition, a STM tip can in principle act as an electron detector. Our next attempt is thus to extract localized NEXAFS spectra by approaching a miniature detector (STM tip) to the area of interest. This is what depicted in fig. 3(b) in which NEXAFS spectra were recorded with a glass coated tip positioned a little beyond the tunneling distance but remains close to the Cu(100)/Co surface. During the spectra acquisition, the feedback loop in STM controller was turned off. Fig. 3(b) shows the cobalt L-edge absorption resonances recorded at four different bias (bias is applied on sample). As expected, a large negative bias would repel more electrons away from the surface to cause larger tip current. We are now working on improving the vibration isolation of our SR-STM system, and designing a chopping system to discriminate tunneling and photo-excited electrons.

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