# Characteristics of the MBE1 End-Station at PNC/XOR

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**Abstract.** An end-station for in-situ characterization of thin films at the PNC/XOR undulator beamline, Sector 20 of the Advanced Photon Source, is detailed. The ability to study films in-situ on a beamline enables examination of surfaces and interfaces on freshly-prepared films, without the influence of a capping layer. The MBE1 molecular beam epitaxy system was designed with this in mind. Now in routine operation and available for General Users on a collaborative basis, the primary function of MBE1 is to undertake polarization-dependent XAFS studies on fresh or stored films, but it also has the capability to do X-ray Standing Wave and Reflectivity measurements. The characteristics of the MBE1 system - its ranges of motions and detector options - are described in detail, with example data illustrating its functionality.

**Keywords:** XAFS, surface, thin film, X-ray Standing Wave, Reflectivity
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## INTRODUCTION

There is an enormous benefit in studying surfaces, films and interfaces to be able to conduct such studies in-situ. For such studies at a synchrotron, having a surface-science end-station allows sample preparation and characterization by a variety of techniques [1-6]. One can study fresh, clean surfaces, surface dosing, low coverage films, films without influence of capping layer and the formation of interfaces between films or film and substrate.

Such a system must meet certain requirements – some common to any system, some specific to the techniques being used to characterize the samples. The abilities to prepare and accurately position samples are crucial to all. How they are implemented depends on the desired characterization techniques. At the PNC/XOR beamline, Sector 20 of the Advanced Photon Source, the emphasis is on spectroscopic applications of X-rays – using X-ray Absorption Fine Structure (XAFS). At PNC/XOR, we have implemented a surface science system for epitaxially-grown films to be characterized primarily by polarization-dependent XAFS, but also by reflectivity and X-ray Standing Wave (XSW) techniques. Designated MBE1, this system is available for General Users wishing to undertake in-situ surface studies. We describe this system below.

## OVERVIEW

MBE1 exists to perform *in-situ* X-ray investigations of epitaxially-grown thin films using the techniques of Surface XAFS, X-ray Standing Wave and small angle Reflectivity under UHV conditions. Primarily intended for deposition of metal films on metal or semi-conductor substrates, the system can also be used for mineral surfaces or other in-situ surface studies. The caveat being (apart from the need to collaborate with the beamline staff member responsible) that no experiments that would contaminate the system for metal film growth, beyond what a bake-out could cure, are permitted.

At the core of the system is its sample positioner. A custom manipulator based on the GB-16 goniometer from Thermionics Northwest provides 3 translations, 3 rotations, cooling (indirect, copper braid) to -100°C and continuous heating to 800°C (flash to 1200°C). A carousel, load-lock and transfer system off the main chamber permits up to four samples to be stored or interchanged. A schematic of the main chamber identifying the ports and their application is shown in Fig. 1. Pumping on the main chamber is done by a scroll-pump-backed 360L/s turbopump and a 240L/s ion pump. A titanium sublimation pump is also used. The base pressure in the system is better than $2\times10^{-10}$ Torr after bakeout. Small gate valves separate the main chamber from the carousel and carousel from load-lock entry system. The carousel has a 30L/s ion pump and is evacuated with a scroll-pump-backed 250L/s load-lock turbopump, to a pressure better than $3\times10^{-9}$ Torr. The entire system was assembled on a custom table with vertical and horizontal travel and tilt capability for positioning in the X-ray beam with or without a toroidal mirror in use. MBE1 is situated in the last hutch on the 20-ID undulator beamline at the APS. With appropriate scheduling, samples can be prepared in advance without

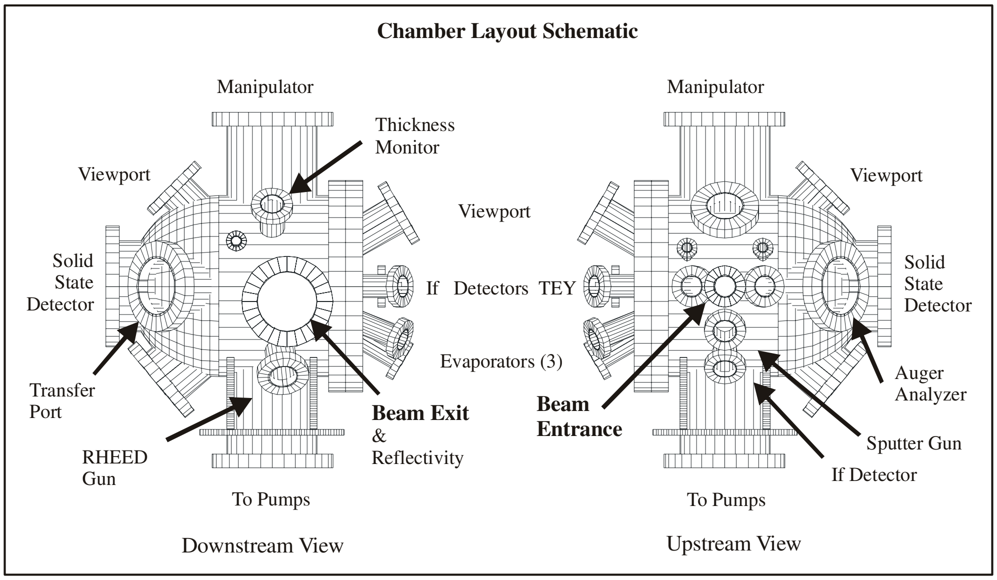

**FIGURE 1.** Schematic drawing of the MBE1 chamber identifying ports assigned to major components of the system. Views are as on 20-ID with beam entering from right (upstream) and exiting left (downstream).

interfering with experiments being conducted in a hutch upstream.

## SAMPLE PREPARATION

Samples are mounted on molybdenum platens and introduced through the load-lock chamber. Although wafers 0.4mm thick are preferred, samples, typically 12mm square, can be up to 6mm thick. Pre-sputtering heat-treatment can be done using the heater incorporated in the manipulator. Sputtering is done using a PHI 04-161 sputter gun. The main chamber is back-filled with ultra-pure argon gas, further purified using a NuPure getter system. For sputtering gallium arsenide, the backfill pressure is $2x10^{-5}$ Torr, and is done for 2.5 to 3 hours with continuous azimuthal rotation of the sample. The removal of surface contaminants can be confirmed by Auger spectroscopy using a single-pass analyzer (TFA-200, RBD Enterprises). To obtain a reconstructed surface, samples can be annealed, with the anneal treatment monitored by Reflection High Energy Electron Diffraction (RHEED) using an EMG-14 electron gun with small-spot option (Kimball Physics). With the desired surface prepared, films can be deposited using one or more of three Omicron EFM-3 evaporators. A recent upgrade in RHEED monitoring from custom software to a commercial package from K-Space Associates readily permits observation of oscillations of RHEED spot intensitie as films are deposited epitaxially. For non-epitaxial growth, a thickness monitor (Leybold-Inficon XTM/2) is also available, but, due to the collimated nature of the material flux from the EFM-3 evaporators, this cannot be done simultaneously. One must calibrate the flux, then deposit the film.

## X-RAY CHARACTERIZATION

MBE1 was designed first and foremost as a surface XAFS facility. A sample can be oriented with respect to the X-ray k-vector anywhere from grazing angle to normal incidence. It can be oriented with respect to the X-ray electric field (polarization) from out-of-plane to any in-plane orientation relative to some crystallographic axes of the sample. Detector options include Total Electron Yield (TEY), custom fluorescence ionization chambers [7] – housings for which also permit use of a Oxford Cyberstar scintillator. A rear port extending into the chamber with a 50mm beryllium window is available for a solid state detector. PNC/XOR has single-element, 7-element and 13-element Ge(Li) detectors available for insertion into this port for fluorescence detection. A large (95mm diameter) Be exit window permits reflectance to be measured. We have used the system to prepare the 4x6-reconstructed surface of GaAs(001) with subsequent epitaxial growth of Fe and Pd. This was followed by extensive polarization-dependent XAFS studies to characterize the tetragonal distortion of the Fe film [8,9], search for in-plane structural anisotropy in the iron [10], and probe the interfaces of Fe/Pd/Fe trilayers [11].

The second technique regularly used in the system is low-angle Reflectivity. The large beryllium exit window is offset. This allows reflectivity measurements with the X-ray incident angle varying from (less than) 0 to 7 degrees for both in-plane and out-of-plane polarization orientations. All detection options are available for these measurements – fluorescence, TEY and reflectance.

The third technique for which MBE1 was designed is X-ray Standing Wave. The back-reflection geometry, involving exciting a Bragg peak with the X-ray beam near 90 degree incidence, is readily achieved. Select

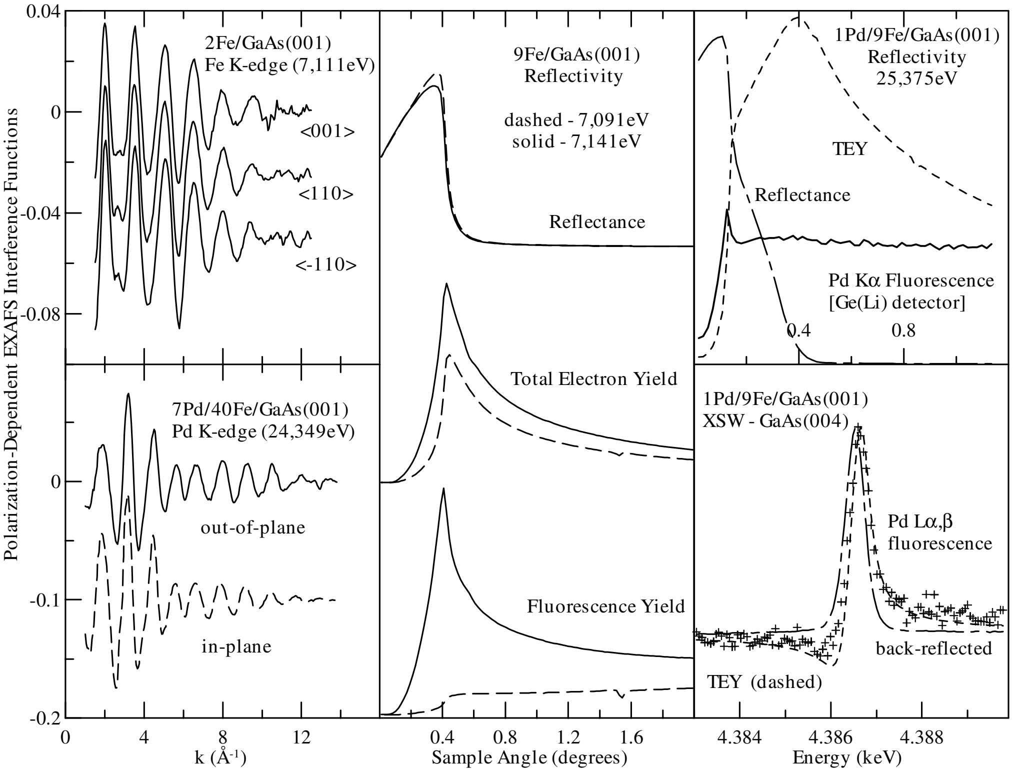

**FIGURE 2.** Example data from the MBE1 system: in-situ measurements of surface XAFS, Reflectivity and XSW.

other geometries are available, given the locations of the other beryllium windows for reflectance or fluorescence measurements (upstream $I_F$ 45° below beam, downstream $I_F$ 60° off beam/30° off polarization, exit window for low-angle incident X-rays). All three techniques are illustrated in Fig. 2. X-rays were provided by the ID-20 Si(111) double crystal monochromator. A set of Si(311) crystals are also available.

## SUMMARY

The MBE1 end-station is a versatile surface science facility located at the PNC/XOR beamline, Sector 20 of the Advanced Photon Source. It possesses the ability to prepare and examine thin films and surfaces in-situ using the methods of XAFS, XSW and Reflectivity. The system is available for General Users on a collaborative basis.

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