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DESY NanoLab

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Abstract: The DESY NanoLab is a facility providing access to nano-characterization, nano-structuring and nano-synthesis techniques which are complementary to the advanced X-ray techniques available at DESY's light sources. It comprises state-of-the art scanning probe microscopy and focused ion beam manufacturing, as well as surface sensitive spectroscopy techniques for chemical analysis. Specialized laboratory X-ray diffraction setups are available for a successful sample pre-characterization before the precious synchrotron beamtimes. Future upgrades will include as well instrumentation to characterize magnetic properties.

1 Introduction

Today's third generation and future diffraction limited synchrotron radiation facilities allow experiments with nano-focused X-ray beams such as single object nano diffraction and imaging (Hoppe et al., 2013; Pfeifer et al., 2006). These demanding experiments require involved pre- and post-experimental sample preparation and characterization with complementary techniques. The DESY Nanolaboratory (DESY NanoLab) is a facility providing photon science user access to advanced nano-characterization, nano-structuring and nano-synthesis techniques which are complementary to the advanced X-ray techniques available at DESY's light sources. A special focus is on the reproducible transfer of individual nano objects from the DESY NanoLab microscopes to the beamlines and vice versa. The scientific instrumentation of the DESY NanoLab is being continuously extended. Currently, an ultrahigh

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vacuum (UHV) setup for surface preparation and nanoparticle growth is available that combines surface science techniques such as X-ray photoemission spectroscopy (XPS), reflection-absorption infrared spectroscopy (UHV-RAIRS) and ultra high vacuum scanning tunneling and atomic force microscopy (STM/AFM). In addition, an X-ray diffraction laboratory is operational, which allows specular reflectivity and grazing incidence X-ray diffraction measurements on a routine basis using sealed tube Mo and Cu X-ray sources and parabolic multilayer optics. A high resolution field emission scanning electron microscope (FE-SEM) is available for users, as well as a dual electron and focused ion beam (FIB) for high precision sample structuring. Magnetic sample characterization will be possible in the future by a Kerr microscope and a physical properties measurement system (PPMS), as well as magnetic force microscopy (MFM). Access to DESY NanoLab instrumentation is possible via submission of a research proposal for a combined access to the DESY NanoLab and the DESY light sources PETRA III or FLASH via DOOR (DESY online office for research with photons: https://door.desy.de/door/). Within this framework, DESY NanoLab currently offers the instrumentation presented here in the areas of surface spectroscopy, X-ray diffraction, high resolution microscopy and magnetic characterization.

2 Surface spectroscopy and sample preparation

2.1 Ultra-high vacuum system for sample preparation

Our ultra-high vacuum (UHV) apparatus consists of a tunnel chamber connected to the load-lock, growth or preparation chamber, reflection-absorption infrared spectroscopy (RAIRS), X-ray photoelectron spectroscopy (XPS), and scanning tunneling and atomic force microscopy (STM). This allows carrying out sample cleaning, modification, metal (oxide) deposition and characterization under UHV chamber without exposing it into air. The preparation chamber has the following characteristics:

- Base pressure of 10^{-11} mbar, ion getter pump, turbo molecular pump and titanium sublimation pump
- Sample heating up to 1500 K by thermal and e-beam heating
- Combined low energy electron diffraction (LEED)- Auger system
- Electron beam evaporators
- Sputter gun for sample surface cleaning
- Thermal cracker for oxygen and hydrogen
- 1-inch molybdenum sample holder with an optimal sample size of 10 x 10 mm^2
- Back-pack sample holders for transferring diverse sample holders to different systems in the UHV lab (STM/AFM, XPS)
- Gas dosing system (Ar, O_2, C_2H_4, CO, H_2, ...)

2.2 Ultra-high vacuum-reflection absorption IR spectroscopy (UHV-RAIRS)

We operate an ultrahigh vacuum (UHV) apparatus to which a state of the art vacuum IR spectrometer is connected (Bruker, VERTEX 80v). The powerful design allows carrying out reflection-absorption infrared spectroscopy (RAIRS) experiments at grazing incidence on well-defined metal and oxide single crystal surfaces in a wide pressure range from UHV to ambient condition and temperature range from 110 K to 1000 K. The unique feature of our IR apparatus is the entirely evacuated optical path to avoid background signals from gas phase species.

2.2.1 Specifications:

- External MCT detector for RAIRS measurements on well-ordered solid surfaces.
- Internal DTG detector for Fourier-transform IR spectroscopy (FTIRS) on powder samples.
- In-situ measurements at variable pressure range: 10^{-10} mbar < p < 1 bar.
- In-situ measurements at variable temperature range: 110 K (liquid N_2 cooling). < T < 1000 K
- Gas dosing system

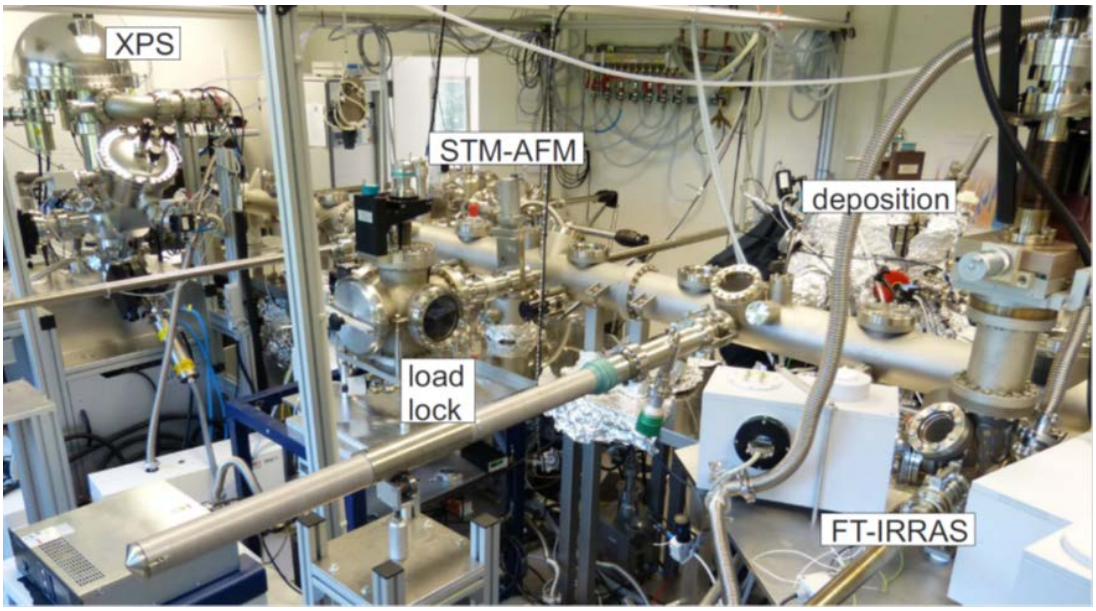

Figure 1: Multi method UHV lab as part of the DESY Nanolab.

2.3 X-ray photoelectron spectroscopy (XPS)

XPS is performed by bombarding a sample with mono-energetic X-rays, causing core-level photoelectrons to be ejected from the sample. The binding energy and intensity of a photoelectron peak provide information about elemental identity, chemical state, and concentration within the probed volume. The XPS signal arises from two to about twenty atomic layers in depth from the surface, depending on the material, the energy of the photoelectrons concerned, and the electron exit angle with respect to the surface.

2.3.1 Information obtained by XPS

- Quantitative chemical elemental analysis of surfaces
- Chemical or electronic state of each element in the surface
- Surface contaminations and adsorbates
- Surface core level shifts
- Homogeneity of elemental composition across the top surface
- Homogeneity of elemental composition as a function of depth (profiling by ion beam etching)

2.3.2 Specifications:

- Variable temperature range: 100 K < T < 1000 K (liquid N_2 cooling).
- Variable pressure range: 10^{-10} mbar < p < 10^{-4} mbar.
- PHOIBOS 150 2D-DLD Elevated Pressure Energy Analyzer equipped with differential pumping system
- Laser pointer for sample positioning and alignment
- Monochromatic X-ray source FOCUS 500 equipped with differential pumping system.
- Flood gun FG 15/40.
- Mono-energetic Al Ka
- Ion source for sample surface cleaning
- Programmed depth profiling sputter gun
- Fast entry load lock

3 X-ray Diffraction

The Desy NanoLab operates two independent X-ray scattering set-ups. One is dedicated to X-ray reflectivity measurements, the other to surface sensitive X-ray diffraction in a variety of geometries. Each measurement station is located inside a radiation proof lead hutch equipped with a door interlock, thereby resembling a typical synchrotron beamline. Both diffractometers can handle relatively large sample environments, which can be as large as 600 mm diameter and weigh up to 50 kg. In this way, the X-ray diffraction stations allow for carrying out experiments, which are very similar to those done at the synchrotron beamline. The symbiosis of experiment control, sample environment and appropriate access to reciprocal space, results in the ability to perform in-situ and operando X-ray scattering studies in the lab. This can be used for complementary studies or to optimally prepare for precious beamtimes at the synchrotron and in some special cases even replace those experiments. Additionally, the X-ray stations serve as excellent training sites for students, who get accustomed with setting up and controlling dedicated experiments exactly in the same way as they will have to do it at the synchrotron.

	Reflectometer	Diffractometer
Anode material	Mo	Cu
Max. power (kW)	3.0	0.05
Size e-beam spot (mm^2)	12 x 0.4	0.04 x 0.04
Focusing	1D	2D
X-ray beam size on sample (HxV, mm^2)	10 x 0.6*	0.25 x 0.25
X-ray beam divergence (HxV, $mrad^2$)	14 x 0.4*	5 x 5
Distance optics-sample position (mm)	1000	650
Photon flux at the sample position	10^7	3 x 10^8

Table 1: Characteristics of the two X-ray setups.

*Horizontally the beam is unfocused and its size and divergence are determined by a slit between the source and sample.

3.1 Reflectometer

The reflectometer uses a vertical scattering geometry, i.e. the sample surface lies horizontal in the lab frame. Five motions, of which 3 rotations and 2 translations are used to align the surface normal in the lab frame. Typical θ-2θ scans are performed. Two pairs of tungsten slits provide a collimator system on the detector arm, thereby allowing to optimizing the resolution and reducing the background to a minimum. Table 1 shows the beam characteristics. Figure 2 (left) shows a typical result of a reflectivity measurement performed on an approximately 15 nm thin iridium film grown on a sapphire substrate. The dynamical range of the setup is 7-8 orders of magnitude.

3.2 Diffractometer

A large 6-circle diffractometer allows for several scattering geometries. The most common one used is for samples with a well-defined face, such as polished single crystal substrates or thin films, with their surface mounted horizontally in the lab frame (Lohmeier & Vlieg, 1993). In particular, the diffractometer is suited for measurements with a fixed angle of incidence with respect to the sample surface, which is beneficial for the signal-to-background ratio when measuring surface sensitive crystal truncation rods. There are two rotations and 3 translations available to align the surface normal of the sample parallel to the omega rotation axis. In Figure 2 (right) (1,0) crystal truncation rod data (solid points) of an Ir(111) single crystal surface are shown.

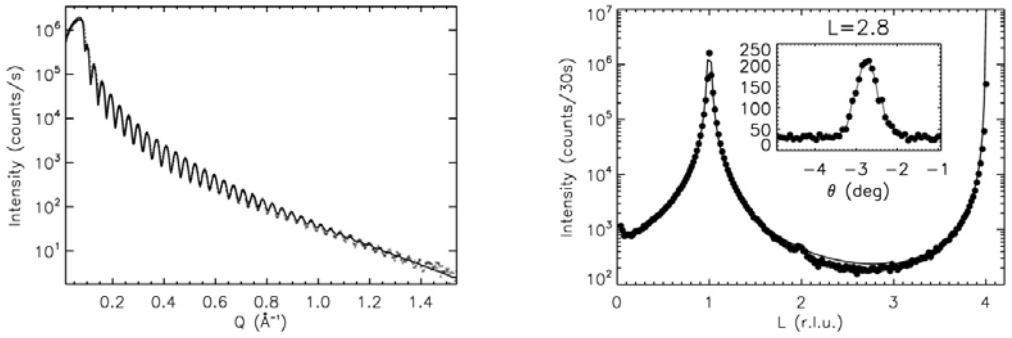

Figure 2: **Left:** X-ray reflectivity data (open symbols) and fit (solid line) of an approximately 15nm thin Ir film on a Al_2O_3(0001) substrate using Mo Kα radiation. **Right:**(1,0) CTR of an Ir(111) single crystal obtained in air (filled circles) and theoretical fit (solid line) (Vlieg, 2000). Inset: Rocking scan at L=2.8.

A simulation of the experimental intensity variation with diffraction index is shown as well (solid curve). The inset shows a rocking curve close to the minimum of the CTR at L=2.8 and indicates that the diffraction signal from the surface is significantly higher than the background.

3.3 Control software and detectors

Both diffraction set-ups are controlled by the program SPEC, which is a software distribution coming with different solutions regarding different scattering geometries and angle calculations (www.certif.com). Since this program is widespread among the different synchrotron communities, there are many possibilities for hardware integration and control. In parallel, a Tango device server is used, which offers a great deal of flexibility towards hardware control of apparatus that is not directly supported by SPEC. Since the detector arms are relatively large and the software allows for the implementation of many different hardware solutions, many different types of detectors can be used. Currently, the reflectometer is equipped with a point detector (Cyberstar, NaI(Tl) scintillator) and is foreseen to be also run with a strip detector (Dectris, Mythen). Through the Desy photon science detector loan pool, it is possible to obtain other systems, like a 2D pixel photon counter (Dectris, Pilatus 100K).

4 Microscopy & Nanostructuring

The DESY NanoLab provides state of the art microscopy instrumentation for real space imaging of materials surfaces adjusted to meet the demands of potential users of the X-ray light sources on the campus. Several types of microscopes are available, including a high resolution field emission scanning electron microscope (HR FE-SEM), a variable temperature ultra-high vacuum scanning tunneling/atomic force microscope (UHV STM/AFM) permitting a UHV sample transfer within the UHV cluster at DESY NanoLab, and a high resolution AFM for operation under ambient conditions. This equipment is complemented by a dual beam (FIB) instrument combining a focused ion beam and an electron beam. The FIB permits a sample nano-structuring as, e.g., slicing for transmission analysis using both, X-rays and electrons, and also iterative milling and imaging to obtain 3D tomographic structural and elemental information. Furthermore, an optical microscope is available.

4.1 Scanning electron microscopy (SEM)

SEM instrument:
The HR FE-SEM is a FEI Nova Nano SEM 450 instrument. Its compact shape permits highest lateral resolution in various imaging modes. Several detectors sensitive to topographic and chemical contrast

permit to obtain complementary information from the sample surfaces. While standard operation is in reflection, thin sections of a sample, membranes, or small objects on membrane carriers can be analyzed using a retractable scanning transmission electron microscopy (STEM) detector. Chemical contrast can be obtained by electron dispersive spectroscopy (EDS). Fig. 3 shows a view inside the SEM chamber with the pole shoe and sample holders on top of the sample translation stage. The gas injection needle used for electron beam assisted deposition of metalorganic precursors facilitates to write markers close to regions of interest, which in a subsequent step can be used for re-localization at other nano-instruments. An arbitrary marker shape is possible via a bitmap import, see, e.g., Figure 3.

Figure 3: Chamber-view inside the SEM with pole shoe, gas injection needle and sample table. IR-CCD camera permits to track the sample position.

4.1.1 Specifications:

- Field emission gun with Schottky field emitter
- High voltage 200 V - 30 kV, landing energy 20 V - 30 kV
- Beam current up to 200 nA
- Lateral resolution 0.8 nm at 30 kV, with the STEM detector; 1.0 nm at 15 kV and 1.4 nm at 1 kV using the secondary electron trough lens detector, (TLD–SE) and 3.5 nm at 100 V (directional back scatter detector, DBS)
- Imaging in high-vacuum and low-vacuum is possible
- Translation stage permitting a lateral sample movement of 110 mm × 110 mm
- Navigation and pattering software
- Beam deceleration option to analyze isolating sample surfaces
- Dynamical tilt
- Gas injection system to write Pt based markers on sample surfaces via electron assisted deposition
- Plasma cleaner

4.1.2 Detectors:

- Secondary electron (SE) Everhart-Thornley detector (ETD)
- High resolution through-lens detector for tunable SE and BE ratio (TLD)

- Lens-mounted concentric backscatter detector (CBSD)
- High resolution STEM detector for transmission analysis of thin sample slices, membranes, and membrane supported micro- and nano-objects
- X-Max 150 EDS silicon drift detector for elemental analysis, energy resolution 127 eV @ Mn Kα (Oxford Instruments)
- Low vacuum backscatter detector (LVD)

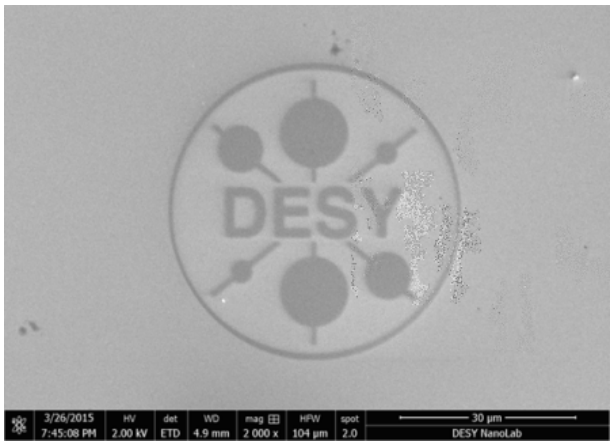

Figure 4: DESY logo written by electron beam assisted deposition of platinum on a silicon wafer surface.

4.2 Variable temperature ultra-high vacuum scanning tunneling (STM) / atomic force microscope (AFM)

1. UHV AFM/STM instrument:
   The variable temperature ultra-high vacuum STM/AFM is an Omicron VT instrument connected to the UHV cluster at DESY NanoLab, permitting a direct sample transfer under UHV conditions throughout the cluster with its preparation, deposition and characterization tools (see section 2.1). It provides atomic resolution in the lateral and vertical dimension.
2. Specifications:
   - Variable temperature range: 100 K < T < 500 K (liquid N_2, option for He cooling)
   - Resolution in vertical z-direction (as specified by the manufacturer): < 0.01 nm
   - Range of tunneling current: 1 pA – 330 nA. Gap voltage: ± 5 mV – 10 V
   - True pA current STM
   - dI/dV spectroscopy
   - Beam deflection AFM
   - QPlus sensor based AFM using a modified quartz tuning fork
   - Range of piezoelectric stage (x/y/z): 10 µm × 10 µm × 1.5 µm
   - Range of coarse movement of translation stage (x/y/z): 10 mm × 10 mm ×10 mm
   - Optical microscope for probe navigation, resolution < 10 µm)
   - Base pressure: 5×10^{-11} mbar
   - Load lock
   - In-situ tip exchange
   - Possibility of in-situ evaporation.
3. Modes of operation:
   - STM/AFM mode
   - STM tunneling spectroscopy
   - High resolution quartz tuning fork AFM
   - Magnetic force microscopy (MFM) option

- Electrostatic force microscopy option
- Kelvin probe microscopy option.

4.3 Atomic microscope (AFM)

1. AFM instrument:
   The AFM is a CP-II instrument from Digital Instruments. It provides easy access to nano- and microscale surface topography of conducting and non-conducting materials. Fig. 5 shows the instrument inside the home-made acoustic isolation chamber. Varies modes of operations permit to obtain additional local surface information as, e.g., mechanical properties, friction forces or the electrical conductivity.
2. Specifications:
   - High resolution piezoelectric scanner (lateral scan range 5 µm × 5 µm, height range 2.5 µm), with a lateral / vertical resolution of 0.0013 Å / 0.009 Å, (digital analogue conversion, DAC, as specified by the manufacturer)
   - Large area piezoelectric scanner (lateral scan range 90 µm × 90 µm, height range 7.5 µm)
   - Laser diode and position-sensitive photodetector
   - Optical microscope for laser and sample alignment
   - Microscope stage with translation stage permitting a coarse sample alignment (8 mm × 8 mm)
   - Home-built acoustic isolation chamber
   - Anti-vibration system
   - ProScan data acquisition and image processing software
3. Modes of operation:
   - Contact mode
   - Non-contact / intermittent (tapping) mode
   - Lateral-force / friction mode
   - STM mode.

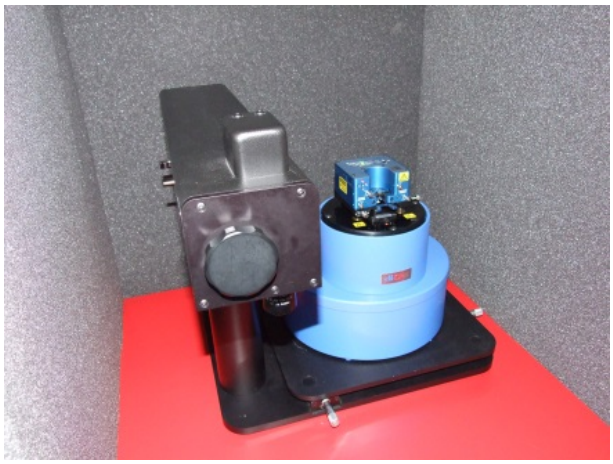

Figure 5: AFM instrument at DESY NanoLab.

5 Instrumentation for Micro- and Nanoscale Magnetism

The past decades have witnessed an enormous progress in the preparation of magnetic nanostructures, together with the development of methods for their characterization. Highly brilliant X-ray sources continue to provide a significant impact in this field as they allow to probe spin dynamics and electronic correlations on relevant temporal and spatial scales with elemental specifity. It is the goal of the instrumentation provided by the DESY NanoLab to facilitate a thorough understanding of magnetic properties from atomic to macroscopic length scales by offering advanced lab-based methods for magnetic characterization in concert with the high-resolution X-ray scattering and spectroscopic methods at the DESY photon sources. To realize this, it is planned to equip the DESY NanoLab with two versatile instruments:
A system for precision measurement of physical properties constitutes an advanced and widely-used toolbox for magnetic and transport measurements in modern condensed matter laboratories. It provides options for magnetometry like vibrating sample magnetometry (VSM) and AC-susceptibility, thermal measurements like heat capacity and thermotransport as well as electro-transport measurements like DC resistivity. All these measurements can be performed in external fields up to 14 T and within a temperature range of 1.9 K – 400 K, thus matching the experimental conditions provided by cryomagnet systems available at PETRA III beamlines.
An ultra-high sensitivity magneto-optical Kerr effect magnetometer shall provide laser-based Kerr magnetometry and near video-rate Kerr microscopy in a single instrument. Such an instrument will be a powerful tool for studies in spintronics, magnetoelectronics, GMR/TMR structures, and thin film magnetism with spotsizes/spatial resolution as small as 2 μm which matches the X-ray spot sizes provided by many of the KB mirror systems at PETRA III. Combined with the coordinate transfer system established in the DESY NanoLab, it will be possible to address the same sample spots with the magneto-optical techniques of this instrument and the X-ray methods at PETRA III.

6 Summary

In summary, the DESY NanoLab provides a versatile platform for DESY photon science users for in depth sample nano-characterization by spectroscopy, microscopy, magnetism and X-ray diffraction around beamtimes, thereby providing methods complementary to the techniques available at the DESY photon science facilities. For the future operation of fourth generation synchrotron radiation sources with highly improved nano-focusing capabilities such a complementary approach will be highly beneficial.

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