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The soft X-rays spectroscopy beamline at the National Synchrotron Radiation Centre Solaris

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The recently commissioned national synchrotron source in Poland has opened the possibility for comprehensive sample characterization in the soft X-ray photon energy regime. The PEEM/XAS beamline dedicated both for spectroscopy and microscopy offers two well-equipped end stations in the UHV standard that enable advanced sample preparation and synchrotron experiments in divers sample environments.

Introduction

The bending magnet (04BM) PEEM/XAS beamline has been constructed and operates at the National Synchrotron Radiation Centre Solaris. It is dedicated to microscopy and spectroscopy in the absorption of soft X-rays. The beamline is equipped with two end stations: a photoemission electron microscope (PEEM), and a universal station for X-ray absorption spectroscopy (XAS)[1]. The beamline is designed to study chemical and electronic, structural and magnetic properties exploiting X-ray absorption spectroscopy (XAS) with polarized X-ray: X-ray natural linear dichroism (XNLD), X-ray magnetic circular dichroism (XMCD) and X-ray magnetic linear dichroism (XMLD). The methods are suitable for probing element-specific properties of surfaces, interfaces, thin films and nanomaterials. The photon energy range (200–2000 eV) covers the absorption K edges for light elements, from carbon to silicon, L edges of elements with Z between 20 and 40, including 3d elements, and also M edges of many heavier atoms, including 4f elements.

The beamline parameters

The optical design of the beamline based on the plane grating monochromator working in the collimated light (cPGM) has been optimized for the soft X-ray photon energy range. The optical concept used is common at other facilities and gives the opportunity to reduce the contribution from the higher-order radiation and to optimize flux versus energy resolution during the experiment. The cPGM is equipped with two gratings to obtain energy resolution ($\Delta E/E$) in the order of 2.5×10^{-4} or better in the accessible energy range and for the available linear horizontal and elliptical left and right polarizations. A summary of the beamline parameters is shown in Table 1.

The PEEM/XAS beamline is operated under the cooperation of Solaris with the Jerzy Haber Institute of Catalysis and Surface Chemistry, Polish Academy of Sciences and the AGH University of Science and Technology. The consortium partners are involved in the beamline operation and take care of the beamline development.

Source	Bending magnet (1.31 T)
Available (optimal) energy range	150–2000 eV (250–1700 eV)
Energy resolution ΔE/E	2.5 x 10^{-4} and better
Beam size at sample (H x V) [mm x mm]	At PEEM: 0.200 x 0.050 At XAS: 2.5 x 2.5
Photon flux at sample	10^{9} – 10^{10} [ph/s/0.1 A]
Polarization	Linear (horizontal) and elliptical
The photoemission electron microscope	XPEEM, μ-XAS, μ-XPS, variable sample temperature (100 - 1200 K)
The X-ray absorption spectroscopy chamber	The XAS, XMCD, XMLD in applied field (200 mT) and variable sample temperature (20 - 660 K)

Table 1. The summarized main beamline parameters, as determined during commissioning.

The PEEM end station

The PEEM instrument (PEEM III with energy analyzer from ELMITEC Elektronenmikroskopie GmbH) is an electron microscope that uses low energy electrons emitted from a sample after excitation with photons to form an image with a spatial (lateral) resolution of a few dozen nanometres. With a tunable X-ray source (XPEEM), elemental sensitivity is accomplished by tuning of the exciting photon energy to the absorption edge of the studied element. In this case, secondary electrons are efficiently used to form an image. Alternatively, with the energy analyzer the excited photoelectrons can be energy-selected, which gives the additional image contrast (also chemical) resulting from characteristic electron binding energies, in analogy to X-ray photoelectron spectroscopy (XPS). Magnetic domain structure is accessible with polarized photons using XMCD or XMLD effects. By taking image series as a function of energy, spectroscopic information can be retrieved with a spatial resolution of the image (micro-spectroscopy: μ-XAS and μ-XPS). Another way to accomplish μ-XPS is to use the dispersive properties of the energy analyzer, so that a photoemission spectrum in the energy bandwidth of approximately 10 eV can be taken in a single shot from a preselected micrometre-sized sample area.

The PEEM end station is a fully equipped "surface science laboratory". It includes a load-lock and an entrance chamber for a fast sample transfer from air into the ultrahigh vacuum (UHV) environment, a preparation chamber, the main microscopic chamber. The preparation chamber includes LEED and Auger spectrometers, several evaporation sources, an ion sputtering source, and a gas dosing system. An additional evaporation source is mounted in the main chamber for real-time microscopy during the deposition.

When planning your XPEEM experiment, consider that:

- Samples must be UHV compatible, flat and should not charge under illumination;
- Samples must fit a sample holder that limits their dimensions to 14 mm in diameter and 3 mm in height;
- Typical information depth is a few nm;
- Typical image acquisition time can vary from the video rate to hours;
- Accessible field of view is from 1.5 to 150 μm;
- Available temperature is from 120 K to 1200 K (imaging) or from 300 K to 2200 K (preparation).

An example of the XPEEM application is shown in Fig. 1. An epitaxial (111)-oriented magnetite film, 10 nm thick, was grown in the preparation chamber by reactive deposition of iron on a Pt(111) single crystal. Then the sample kept at the preparation temperature of 250°C was exposed for half an hour to molecular oxygen at 8*10^{-6} mbar. The following XPEEM analysis revealed a chemically inhomogeneous surface with dendritically spreading hematite embedded in the magnetite matrix. The both phases are distinct in respect of magnetic properties, as shown in the XMCD image in Fig. 2c; sub-micrometre magnetic domains are characteristic for ferromagnetic magnetite, whereas antiferromagnetic hematite does not reveal any XMCD contrast.

The XAS end station

The XAS in the soft X-ray energy range is often termed NEXAFS (Near Edge X-Ray Absorption Fine Structure). This spectroscopy provides element and chemical specificity, as well as sensitivity to polarization effects related to

magnetic and crystal structure of the materials by means of XMCD and XMLD effects. The main signal detection mode is total electron yield (TEY) realized by measurement of the sample drain current. For low-conductive samples two other modes are available - the partial electron yield detection using electron multiplier (channeltron) and a fluorescence detection realized with the use of silicon drift detector type. The XAS end station is a two-chamber UHV system equipped one chamber for spectroscopy and another one for preparation. It features:

- Sample (powder or bulk) stations fitting a standard flag-style sample holder (so-called "Omicron plate");
- Beam spot on the sample (h x v): 2.5x2.5 mm^2; may be reduced using slits;
- High vacuum or UHV sample environment;
- Spectroscopic measurements in a broad temperature range of 20–660 K and external magnetic field of 0–2 kOe; sample rotation about the vertical axis;
- Fully equipped preparation chamber (comparable to that of the PEEM end station).

Results of three accomplished experiments is presented in this section. The recently explained antiferromagnet (AFM)/ferromagnet (FM) magnetic moment structure in an exchange bias CoO/Fe(110) system[2] is the first example. It was proved that the FM layer with strong uniaxial magnetic anisotropy determines the interfacial spin orientations of the neighbouring AFM layer and rotates its easy axis. The following successful study aims at determining the structure of conduction band of a TiO_2 based system. Figure 2 shows the Ti L-edge and O K-edge XAS spectra collected for metal titanium disc (before and after sputtering of Ti disc) and Ti disc thermally oxidized at 800°C. In the measured Ti XAS spectra, two main regions attributed to the dipolar excitations of $2p_{3/2}$ electron (L_3-edge) and $2p_{1/2}$ electron (L_2-edge) into unoccupied Ti 3d states are observed. The difference between the spectra of metallic titanium and oxidized Ti-species lies in the edge shift and spectral shape change. As the metal surfaces relatively easily undergo oxidation in an ambient environment with the depth of the oxide layer in the range of few to tens of nanometers, the ability of surface sputtering is of importance for measurements of reference metal materials. The unprocessed sample shows a clear Ti-oxide signal that vanishes after 3h of sample sputtering.

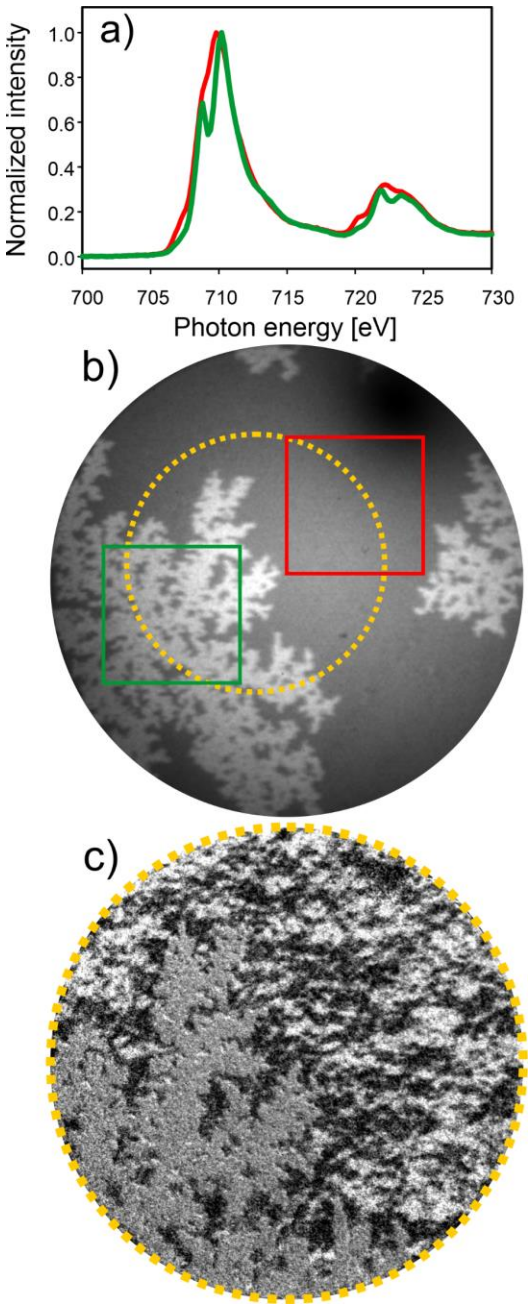

Fig. 1. XPEEM analysis of an epitaxial magnetite 10-nm Fe_3O_4(111) film on Pt(111) that was partially oxidized by exposing *in situ* to an oxygen dose of app. 10^4 langmuir at 250°C: a) selected-area XAS spectra that expose magnetite (red) and hematite (green) stoichiometry. The spectra were collected on the colour-corresponding squared areas marked in an XPEEM image (b), FoV 20 μm, taken at an X-ray energy of 710 eV. c) XMCD image at 708.5 eV of a selected area (FoV 10 μm), marked yellow in (b).

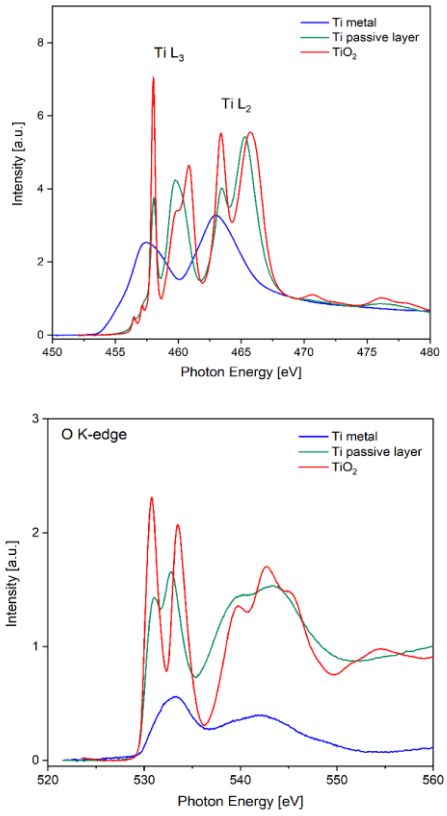

Fig. 2. Example of application. The Ti L-edge and O K-edge XAS spectra collected for TiO_2 based system (courtesy of Dr A. Wach, INP PAN, Kraków).

Multiplet effects revealed in XAS is often used as a fingerprint of the local structure of ions in semiconductors. For instance, it shows that Mn dopants in InSb nanowires produced by pulse electrodeposition, have a strong tendency to clustering, which results in ferromagnetic response at room temperature[3]. Even more subtle structural effects can be probed if polarization analysis is involved, for instance in the experiments performed on anisotropic monocrystals. The difference between XAS probed at two distinct orientations between linearly polarized incident photons and main crystal axes, which is often termed as XNLD (X-ray natural linear dichroism), may be used to assess the evolution of structural distortions. Such an experiment performed on a single crystal of Bi_2Te_3 shows the evolution of surface atomic structure *in-situ* upon Au deposition (Fig. 3). Local anisotropy of surface Te ions, revealed in strong XNLD oscillation beyond Te M_{45} edge, is enhanced at, a low thickness (up to 5 monolayers) and relaxed at thicker Au cap layers. Robustness of the surface structure is a prerequisite for the preservation of topological insulator properties of Bi_2Te_3.

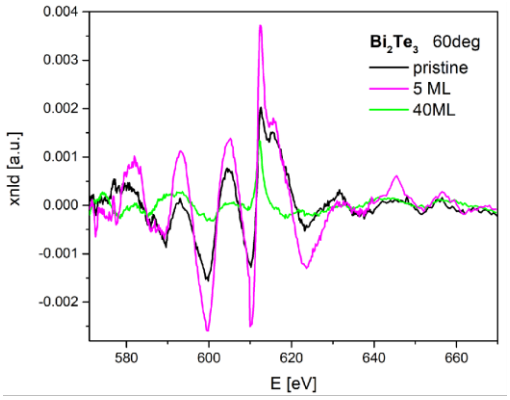

Fig. 3. XNLD at Te M_{45} edge collected from pristine and Au covered Bi_2Te_3 single crystal. The amplitude of the oscillations is a measure of anisotropy in the local structure of surface Te.

Summary

The PEEM/XAS beamline is fully operational and available for users at the National Synchrotron Radiation Centre Solaris. The experimental setup is dedicated for soft X-ray spectroscopy and microscopy. Moreover, the two available end stations provide separate chambers for the state-of-the-art surface preparation and characteri-zation. The access to the Solaris infrastructure is open for the users via Solaris' or CERIC's call for proposals[4].

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