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Custom sample environments at the ALBA XPEEM

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ABSTRACT

A variety of custom-built sample holders offer users a wide range of non-standard measurements at the ALBA synchrotron PhotoEmission Electron Microscope (PEEM) experimental station. Some of the salient features are: an ultrahigh vacuum (UHV) suitcase compatible with many offline deposition and characterization systems, built-in electromagnets for uni- or biaxial in-plane (IP) and out-of-plane (OOP) fields, as well as the combination of magnetic fields with electric fields or current injection. Electronics providing a synchronized sinusoidal signal for sample excitation enable time-resolved measurements at the 500 MHz storage ring RF frequency.

1. Introduction

Photoemission electron microscopy (PEEM) combined with synchrotron x-ray radiation is a powerful non-invasive tool for micro- and nano-magnetism, nanoscience and technology, and surface science [1,2]. The tunable photon energy, high brilliance, full polarization control, and overall stability of modern undulator beamlines allow for obtaining element-specific magnetic contrast PEEM images using the XMCD and XMLD effects down to 20 nm lateral resolution [3]. Thanks to the extreme surface sensitivity (which is also an experimental constraint for the samples to be investigated), this method already works for sub nm sample thickness, i.e., very little magnetic material and/or moment is needed. In particular, the possibility to image not only ferromagnetic but also antiferromagnetic domains via the XMLD effect is an outstanding feature [4]. Many PEEM instruments are installed at soft x-ray beamlines at synchrotrons around the world with access to, e.g., the L absorption edges of magnetic 3d transition metals (e.g., Fe, Co, Ni) and are frequently used for research in magnetism, magnetic materials, and nanostructures. PEEM is an immersion lens microscopy technique, often combined with a low energy electron microscope (LEEM) [5,6]: high voltage is applied between the sample surface (i.e., the sample is part of the electron optics as one electrode for the accelerating electric field) and the first lens (objective) in order to accelerate photo-emitted electrons before passing the electron-optical imaging column. The high electric field, i.e., 10–20 kV within 2 mm distance, requires a careful design of the sample environment and good UHV conditions, in order to reduce the risk of electric arcs. In addition, the initially low electron kinetic energy implies a high sensitivity to magnetic fields or other perturbations, which needs to be taken into account for the sample holder design.

The LEEM-PEEM end station at the CIRCE beamline of the ALBA synchrotron is based on the commercial Elmitec LEEM III with imaging electron energy analyzer [3]. The standard sample holders of the instrument, mainly used in surface science, include electron bombardment heating of the sample (up to ca. 2000 K) and temperature readout through a W/Re thermocouple. For the ample X-PEEM user community working on micro- and nano-magnetism or on device-like prototype structures, a series of custom sample holders with enhanced functionalities have been built. In many experiments, an external magnetic field is required to either set the sample in a desired magnetic state (initialization or switching) or to measure its response with respect to the magnetic field. In other cases, electrical voltages, current pulses or other signals may be useful to excite the samples or patterned device-like structures.

In the following, we describe and characterize the custom sample holders and some auxiliary equipment, making use of the up to six electric contacts available in the ALBA PEEM sample stage. Some of the functionalities are unique so far, to the best of our knowledge.

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2. Overview, standard holder, electrical poling, and UHV transfer

Fig. 1 shows some of the custom holders currently in use at the CIRCE beamline. The functionalities are summarized in Table 1. The sample holders in the upper part of the table are explained below, while the versions of the lower second part of the table are described in more detail in Section 3.

The standard Elmitec sample holder with a filament for heating and a W/Re thermocouple is shown in Fig. 1(a). The sample itself is placed on top of the central tower and clamped down with a 'cap' with a central hole of variable diameter. It has four electric contacts (two for the filament and two for the thermocouple), insulated from the sample holder body or local ground (often called 'Start voltage', since it is the reference potential for the photoelectrons). Another two 'spare' contacts are available in the sample stage, the corresponding features of the sample holders can be seen e.g. in Fig. 1(a) as unused empty holes and in Fig. 1(d) as additional contact feet.

The modification for the application of a voltage between the sample top and bottom, for instance to polarize a piezoelectric substrate [7], is straightforward using Kapton-insulated UHV-compatible Cu wire connected to one of the spare contacts, and an insulating spacer below the sample, e.g. made from Macor. Thus, the sample surface is at the sample holder body potential while the backside of the sample can be kept at another voltage. Silver paint can be used to ensure good electrical contact, but should be degassed. The filament and thermocouple can still be used, keeping in mind that heating is limited by the Kapton wire and the insulating spacer material. The two spare contacts can also be used with a multimeter (floating inside the instrument high voltage rack) to measure in-situ the resistance of the sample, e.g., as a function of temperature, by placing the two wire ends at opposite sides of the sample and insulating them by small pieces of Kapton tape hidden under the cap.

An adapter for a UHV transfer chamber (suitcase) for sample platelets as used in many commercial systems (e.g. by Omicron or SPECS) is shown in Fig. 1(b). In order to stay as close as possible to the original electron optical layout and reduce the risk of arcing, compatible platelets with a top hat shape and thinned out inner edges are used.

3. Sample holders with electromagnets

Sample holders with integrated electromagnets for uniaxial, in-plane magnetic fields are in use at other PEEM end stations [e.g., at BESSY and SLS]. The equivalent model used at ALBA (Fig. 1(c)) is similar to these and uses a monolithic Armco or Permendur [8] yoke, with coils wound from 0.14 mm diameter insulated copper wire [9]. With 2×500 windings and 2.75 mm gap, 180 mT/A are measured at 27 Ω coil resistance. The maximum applicable field is limited on the one side by the current source (12 V compliance) and the heating of the wire (max 0.3–0.4 A can be applied for longer times, while up to 1 A could be applied for short pulses), but also by image distortions due to electron deflection by the field. As has been pointed out previously, [10] one main advantage of minimizing the yoke gap opening is, apart from yielding higher fields, the faster decrease of the field with distance from the sample surface, thereby reducing image distortions. A more systematic study of the effect of in-plane magnetic fields on the image resolution can be found in Ref. [11]. However limits on the minimum gap size may be imposed by requirements on field homogeneity (sample height) and practical considerations when it is needed to place a certain part of the sample over the gap.

However, these sample holders are restricted to a single magnetic axis so far while experiments with nano-patterned magnetic devices often require more than one field direction. We point out that sample rotation with respect to the XMCD contrast axis (along the direction of X-Ray incidence), as available at ALBA and several other PEEM instruments, is experimentally not equivalent to the in-plane rotation of the magnetic field vector with respect to the sample.

In Fig. 1(d) a novel sample holder for biaxial in-plane magnetic fields, using a quadrupole yoke, is shown. Two sets of two opposite coils are powered by a single current source (using the standard filament current supply) and the two additional spare electrical feed throughs. In order to provide a magnetic field in either one of

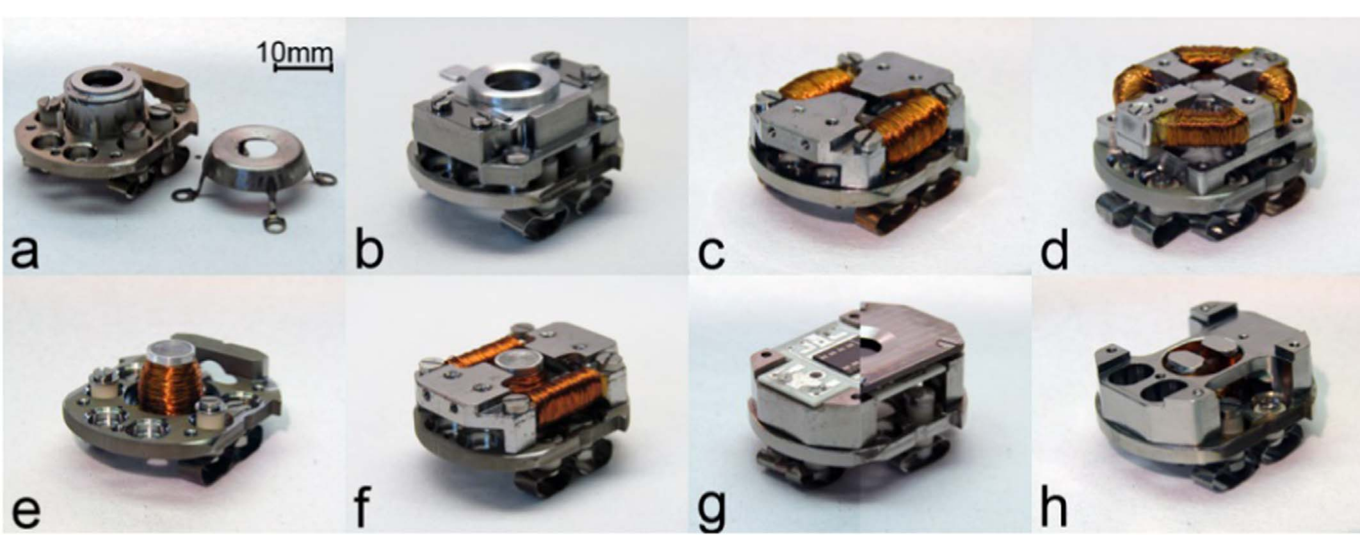

Fig. 1. (2 column): Photographs of some of the different sample holders in use at the ALBA PEEM: (a) Standard Elmitec sample holder for high temperature (cap on the side). Note the two holes in front for the two spare contacts. (b) Sample holder with adapter piece for platelets as used in many deposition and characterization systems. (c) Sample holder with uniaxial in-plane electromagnet. (d) Sample holder with quadrupole in-plane electromagnet (more details in Fig. 2). The two extra feet in front of the spare contacts are used for one coil set. (e) Sample holder with out-of-plane electromagnet. (f) Sample holder combing the out-of-plane electromagnet with a wide gap in-plane electromagnet. (g) Sample holder with adapter piece for a printed circuit board (PCB) for electrical contacts, e.g., by bonded wires. The cap is mounted floating, to avoid short-circuiting the contact wires. To show more details, this image is composed of two separate pictures, with and without cap, respectively. (i) Sample holder with adapter piece for PCB and a mini in-plane electromagnet (also out-of-plane magnets can be mounted in the central free space).