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Time-resolved soft X-ray microscopy of magnetic nanostructures at the P04 beamline at PETRA III

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Abstract. We present first time-resolved measurements of a new mobile full-field transmission microscope [1] obtained at the soft X-ray beamline P04 at the high brilliance synchrotron radiation source PETRA III.

A nanostructured magnetic permalloy (Ni₈₀Fe₂₀) sample can be excited by the magnetic field of a 400 ps full width at half maximum (FWHM) long electric current pulse in a coplanar waveguide. The full-field soft X-ray microscope successively probes the time evolution of the sample magnetization via X-ray magnetic circular dichroism (XMCD) [2] spectromicroscopy in a pump-probe scheme by varying the delay between pump and probe pulses electronically. Static and transient magnetic fields of a permanent magnet and a coil are available in the sample plane to reset the system and to provide external offset fields.

The microscope generates a flat-top illumination field of 20 µm diameter by using a grating condenser [3] and the sample plane is directly imaged by a micro zone plate with 60 nm resolution onto a 2D gateable X-ray detector to select the particular bunch in the storage ring that contains the dynamic information. The setup is built into a mobile endstation vacuum system with in-house developed three-axis piezo motorized stages for high accuracy positioning of all microscopy-components inside the chambers.

1. Motivation and introduction

Besides the fascination of exploring magnetism at its fundamental time- and length-scales itself, research in this direction motivates many applications in modern data storage technology. The demand for smaller and faster storage devices has to be based on primal knowledge of magnetization processes happening at time scales from picoseconds to femtoseconds and length scales from nanometers [4–6] to single atoms [7] at once. The challenging investigation of magnetic systems influenced by femtosecond laser pulses [8–11] or short and confined magnetic field pulses [12–14] is of topical research interest.
In order to contribute to the research done in this direction we have set up a new mobile and flexible time-resolved magnetic transmission X-ray microscope (TR-MTXM) at the soft X-ray beamline P04 at PETRA III. The setup is able to investigate the magnetization dynamics of previously excited nanoscale objects by X-ray magnetic circular dichroism (XMCD) spectromicroscopy [2, 15, 16).

2. Experimental Setup

The schematic setup of the full-field transmission X-ray microscope (TXM) is outlined in figure 1 and also described in [1]. The circular polarized soft X-ray pulses of the P04 beamline at photon energies of the transition metal L edges are focused down to a flat-top spot of 20 µm diameter for Köhler-like illumination by a grating condenser [3] with included gold central stop and a focal length of \( f_c = 34 \text{ mm} \) at 852 eV photon energy. A micro zone plate of 80 µm diameter and outermost zone width of 50 nm images the sample plane located at a distance \( g = 2.75 \text{ mm} \) onto a 2D gateable pixel detector at \( b = 1930 \text{ mm} \) behind the zone plate corresponding to a magnification of \( M \approx 700 \).

The time resolution is implemented in a pump-probe approach where the sample is excited by a short magnetic field pulse emerging from current pulses in the conductor of a waveguide. The current pulses of 400 ps full width at half maximum (FWHM) duration are created by a picosecond pulse generator synchronized to an external clock of the storage ring. The delay \( \Delta t \) between pump and probe pulses is electronically tunable with a jitter of \( \sigma < 30 \text{ ps} \). In “timing mode” the storage ring PETRA III is filled with 40 electron bunches and delivers a light pulse every 192 ns for probing the sample. However, the sample can only be excited every 7.7 µs matching the revolution frequency of the synchrotron due to the high intensity of the pump pulses. In consequence, this means that only 1 of 40 bunches contains a transient signal and the unpumped bunches have to be filtered. For this purpose an in-house developed fast gateable X-ray detector is used: The microscope image is projected onto a P46 phosphor screen and imaged via a relay optic consisting of two fast camera lenses onto the photocathode of a commercial third generation image intensifier. The intensifier features a single microchannel plate (MCP).

![Figure 1. Setup of the TXM consisting of a grating condenser with gold central stop and a micro zone plate. Via a pulsed magnetic field \( \vec{H}_{\text{pulse}} \) of a current pulse in a coplanar waveguide (figure 2) the sample can be pumped in order to follow the time evolution subsequently.](image)

![Figure 2. Geometry of the 100 Ω impedance coplanar waveguide (CPW).](image)
amplification stage and a P46 output screen that is fiber-coupled to a Princeton Instruments PIXIS-XF back-illuminated charge-coupled device (CCD) camera with 1024 × 1024 pixel of 13 µm size. By applying a positive bias voltage of +30 V onto the photocathode of the image intensifier, the detector is closed by default as the generated negatively charged electrons cannot leave the cathode surface. A 30 ns long negative voltage gate pulse of -200 V amplitude synchronized to the storage ring trigger opens the detector to pass on the image information of the desired bunch containing the dynamic information by acceleration of the photoelectrons onto the MCP. The light output of the phosphor screen is collected at a fixed pump-probe delay Δt and integrated on the camera chip. The temporal overlap between pump, probe and gate pulses is achieved by moving a fast photodiode into the soft X-ray beam and monitoring all trigger signals by carefully taking into account electronic signal delays in cables and equipment.

Cobalt-platinum (Co/Pt) multilayers with perpendicular magnetic anisotropy or in-plane magnetized thin permalloy (Ni$_{80}$Fe$_{20}$) films of ≈30 nm thickness structured by electron-beam lithography and lift-off processing on a silicon substrate with a 200 nm thick silicon nitride membrane served as samples for the first measurements. Figure 2 shows the geometry of a 100 Ω impedance coplanar waveguide (CPW) [17] taking care of the current pulse transport to the nanomagnetic system and subsequent pulse termination in two microwave resistors. The conductor width is tapered down from 1 mm to 10 µm in order to increase the current density being related to the magnetic field amplitude. The CPW is directly manufactured on top of the nanostructured sample by photolithography, physical vapor deposition and lift-off processing and thus in direct contact with the magnetic layer. Proper overlap between magnetic and waveguide layer is ensured in a mask aligner by matching alignment structures written into both the magnetic layer and the waveguide lithography mask. The whole sample is tilted 70° with respect to the propagation direction of the photons to provide an XMCD sensitivity to the
in-plane and out-of-plane magnetization component.

Photographs of the setup and the sample stage are provided in figure 3 and 4. Additional magnetic fields in form of an adjustable offset field generated by a permanent magnet and a pulsed reset field of up to 2 µs duration generated by a water-cooled in-vacuum coil can be applied to the sample as well.

3. Results and discussion
The TR-MTXM has been set up successfully and features down to 60 nm are observable in good agreement with the theoretically achievable resolution in this case limited by the micro zone plate optic. Figure 5 shows a raw image of Co/Pt nanodots acquired with the full-field TXM by summation of 20 camera images exposed for 250 ms and only corrected by the subtraction of a detector dark image. Because of the high degree of spatial coherence of the P04 soft X-ray beam due to the high brilliance of the PETRA III storage ring, the illumination field shows a severe speckle pattern. It was necessary to stir the beam with the refocusing Kirkpatrick-Baez (KB) mirrors of the beamline to destroy this pattern and smoothen the illumination field like done in [3]. The smoothed background can then be removed completely by correction with a flat-field image.

A first static XMCD contrast image mapping the in-plane magnetization of a 2 µm × 2 µm permalloy square is depicted in figure 6 and shows the typical Landau flux-closure pattern consisting of four equally sized domains. Black and white absorption contrast corresponds to the magnetization components pointing along or against the photon beam direction. This XMCD information becomes visible by subtracting two images taken with the circular polarized beam of positive and negative helicity. Here, for each photon helicity 40 images with an exposure time of one second were recorded at the nickel L$_3$ edge (852 eV).

The achieved temporal overlap of pump and probe pulses is demonstrated in figure 7. The XMCD contrast magnetization maps are constructed by subtracting the sum of six images exposed for 60 s at each photon helicity. The image sequence shows modulated permalloy nanowires of 400 nm width. Because this is a time-resolved measurement, only one of the 40 bunches in the storage ring is usable due to the limited pumping rate of the excitation source. The exposure times are longer compared to the static images due to the reduced photon flux on the camera. The spatial resolution might be worse compared to the static images in figure 5.

Figure 5. TXM image of nanostructured magnetic Co/Pt multilayer system with a transmission of ≈ 75 % at the cobalt L$_3$ edge (778 eV). The illumination field suffers from a speckle pattern due to the high degree of spatial coherence of the P04 beam that can be removed by stirring the beam. The red labels indicate the dot diameter while the black labels specify the minimum distance between two dots. This figure represents the sum of 20 camera images taken at 250 ms exposure time (dark image subtracted).
Figure 6. XMCD image of the magnetization in a $2 \mu m \times 2 \mu m$ patterned permalloy element.

Figure 7. XMCD images of modulated permalloy nanowires of 400 nm width in an offset field before, during and after excitation by a magnetic field pulse via the CPW (visible in the left of each image).

and 6 owing to mechanical vibrations during the longer exposure times, the increased noise level due to less collected photons and because one XMCD image is generated out of the difference signal of two TXM images. Nonetheless, recent measurements show a discernibility of 40 nm features also in XMCD images (data not shown).

An offset field commonly aligns the magnetization of the nanowires in figure 7 along the field direction evident by a light contrast visible in the images before and after the magnetic pump pulse. During the presence of the pulse, however, the wire elements located in overlap with the waveguide (dark shaded area on the left) change the magnetization to the opposite direction appearing as dark contrast while the elements located next to the waveguide remain unchanged. Considering the in-plane component of the magnetic excitation pulse, this demonstrates the spatial confinement restricted mainly to the conductor width.

4. Summary and Outlook
We have set up and commissioned a new full-field TR-MTXM at the P04 beamline of PETRA III with 60 nm spatial resolution for time-resolved studies of magnetic nanostructures pumped by short magnetic field pulses and already demonstrated the time-resolved capabilities by acquiring XMCD images in temporal overlap of pump and probe pulses.

The intense magnetic field pulses producible by this setup enable a direct transient analysis of domain pattern destruction and recovery in magnetic nanostructures with possibilities to study interactions in magnetic chains and arrays. Element specific imaging of the magnetization may address questions concerning the redistribution of magnetic moments in alloys and multilayers after excitation by magnetic field pulses or femtosecond laser pulses.

A synchronized femtosecond laser system [1, 18] provides an alternative excitation source to tackle questions considering the dynamics of small magnetic systems triggered by optical pump pulses. In this case the time resolution would solely be limited by the PETRA III pulse duration of 90 ps FWHM [18] due to the already demonstrated synchronization with a residual jitter of $\sigma < 5$ ps [1, 18].

The TXM instrument will be extended to a mobile and flexible user endstation microscope with additional scanning, tomography and fluorescence imaging capabilities.

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