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To cite this article: M Suzuki et al 2013 J. Phys.: Conf. Ser. 430 012017

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A hard X-ray nanospectroscopy station at SPring-8 BL39XU

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Abstract. We upgraded the hard X-ray spectroscopy beamline BL39XU at SPring-8 by installing Kirkpatrick-Baez mirrors to equip it with submicron-focusing capability. At the new experimental end-station located 74 m from the undulator source, an X-ray nanoprobe with a size of 100 nm has become available at 5–16 keV. This upgrade is fully compatible with the X-ray polarization tunability and helicity-switching between right- and left-circular polarizations; these are unique features of the beamline, which is equipped with a diamond X-ray phase retarder. A spatial resolution of ≈100 nm for X-ray absorption fine structure and X-ray magnetic circular dichroism (XMCD) spectroscopy has been achieved. We show a magnetization reversal process of an individual magnetic dot in bit-patterned perpendicular recording media using element-specific XMCD magnetization measurements to demonstrate the performance of the new station.

1. Introduction

X-ray spectroscopic techniques, X-ray absorption fine structure (XAFS), and X-ray magnetic circular dichroism (XMCD) are useful tools for characterizing functional materials. These techniques allow chemical, structural, and magnetic characterization in both element-specific and site-specific ways. The techniques use hard X-rays and offer bulk sensitivity, a variety of sample environments, and easy combination with an external field or optical excitations. Our goal is to combine a micro/nanoprobe capability with hard X-ray spectroscopy using a focused X-ray beam. We are aiming at XAFS and XMCD at 100-nm spatial resolution. With this capability, one can perform two-dimensional XAFS mapping to obtain a spatial distribution of chemical and bonding states and local symmetries of a sample with nanostructures. Local XANES/EXAFS measurements allow us to investigate particular parts of nanostructures or very
small samples, such as single catalyst particles [1]. An XMCD technique using a nanofocusing beam also allows scanning element-specific magnetic domain imaging and XMCD spectroscopy and element-specific magnetization (ESM) curve measurements in a local area of samples with magnetic domains or magnetic nanostructures [2]. Single magnetic device elements or small magnetic particles of submicron dimensions can be studied in this manner. In this paper, we describe the upgrade of an existing beamline BL39XU at SPring-8 to include features for hard X-ray nanospectroscopy. Early results of XMCD magnetometry in CoPt perpendicular magnetic dots are presented to demonstrate the performance of the new experimental station.

2. Upgrade of BL39XU with a new experimental station

We upgraded BL39XU at SPring-8, which has been used for XMCD spectroscopy in the hard X-ray region. The beamline is equipped with a Si 111 double-crystal monochromator and a diamond X-ray phase plate to generate circularly polarized X-rays. The existing experimental hutch is located 45 m from the source and equipped with several apparatus for XMCD experiments, including an electromagnet, a 10-T superconducting magnet for high-field experiments, and diamond anvil cells for XMCD studies at high pressures of up to 170 GPa [3]. In 2011, we extended the beamline and constructed a new experimental hutch dedicated to nanospectroscopy applications, located 74 m from the source, as shown in Fig. 1. We installed Kirkpatrick-Baez mirrors in the hutch to focus the X-ray beam down to 100 nm [4].

![Figure 1. Layout of the nanospectroscopy station at the SPring-8 BL39XU beamline.](image)

Table 1 summarizes the parameters of the focused X-ray beams. The available X-ray energy ranges from 5 to 15 keV, which covers the absorption edges of most 3d transition metals, rare earth elements, and 5d noble metals, such as Pt, a vital element in some catalysis reactions and magnetic materials for data storage. One can choose two focusing modes—“high-resolution” and “high-flux” modes—by changing the opening of the virtual source slit 36 m upstream of the focusing mirror. In the high-resolution mode with the small opening of the virtual source slit [8 (V) × 18 (H) μm²], the typical beam size is 120 (V) × 100 (H) nm². The available photon flux is on the order of 10⁹ photons/s in the spot. When higher flux is needed, a flux higher than 10¹² photons/s in a spot of 300 × 250 nm² is obtained with a virtual source slit of 2000
(V) × 54 (H) μm² in size, i.e., fully open in the vertical direction. This photon flux value is incredibly high compared with the total flux at a typical undulator beamline of SPring-8, which is on the order of 10¹³ photons/s and that at a standard bending magnet beamline, which is with 10¹⁰–10¹¹ photons/s.

One of the most important features of the beamline is the control of the X-ray polarization state using the diamond phase plate. We have confirmed that the phase plate has no influence on the focused beam size by comparing focused beam profiles of ≈100 nm obtained under three different conditions: without a phase plate and with a 0.73-mm-thick phase plate in the 220 Laue geometry that was tuned for left- and right-circular polarizations. No significant increase in the beam size was observed within the reproducibility of the measurement (≈±10 nm) by a knife-edge scan. The current upgrade was shown to be fully compatible with polarization control using a diamond phase plate, and it would directly lead to XMCD applications using a focused beam.

### Table 1. Parameters of the focused X-ray beams.

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<thead>
<tr>
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<th>High-resolution mode</th>
<th>High-flux mode</th>
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<tbody>
<tr>
<td>X-ray energy (keV)</td>
<td>5–15</td>
<td>5–15</td>
</tr>
<tr>
<td>beam size (nm²)</td>
<td>120 (V) × 100 (H)</td>
<td>300 (V) × 250 (H)</td>
</tr>
<tr>
<td>photon flux (photons/s)</td>
<td>6.2 × 10⁹</td>
<td>1.7 × 10¹²</td>
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3. **XMCD magnetometry in a single magnetic dot**

To demonstrate the performance of our hard X-ray nanoprobe, we performed XMCD magnetometry in a single magnetic dot. We studied a prototype of bit-patterned media, which is a candidate for ultra-high-density recording media in the near future. The sample consisted of CoPt magnetic dots of 200-nm diameter arranged on a matrix with a 1-μm spacing. A magnetic thin film with a structure of Co₈₀Pt₂₀(15 nm)/Ru(15 nm)/NiW(5 nm)/C(3 nm)/Si substrate was deposited by dc magnetron sputtering, and dots were patterned by electron beam lithography and Ar beam milling [2].

We focused an X-ray beam of size 320 (V) × 290 (H) nm² onto a single magnetic dot and successfully recorded XAS and XMCD spectra at the Pt L₃ edge for the single dot (see Fig. 2). In the XAS spectrum, the W L₂ (11.542 keV) absorption from the NiW underlayer overlaps that of the Pt L₃ (11.565 keV), which demonstrates the bulk sensitivity of our hard X-ray nanoprobe. The XMCD signal was observed only at the Pt L₃ edge because Pt has a net magnetic moment due to hybridization with Co, but W does not. We recorded ESM curves of a single dot by monitoring the XMCD amplitude at the Pt L₃ edge as a function of the external magnetic field. In Fig. 3, the solid circles show a hysteresis loop measured for a single magnetic dot. The magnetization of the dot switches very rapidly at the coercive field, demonstrating that such a small dot has a small number of (less than a few) magnetic domains and that the magnetization reversal process is nearly coherent. For comparison, a hysteresis loop recorded using our previous focusing setup with a 2-μm beam is shown [5], as denoted by the line in the figure. With a larger X-ray beam spot, about 100 dots were included in the footprint of the focused beam, and the measured signal was an average for these dots. The resulting hysteresis loop, therefore, changes more slowly compared with that for a single dot. Coercivity fields are not exactly the same for many dots, and there is some distribution in the switching field. Using the 300-nm focused beam, we were able to characterize the switching field for 100 individual dots by recording each ESM curve for these dots [6].
4. Summary
In summary, we upgraded the SPring-8 BL39XU beamline by equipping it with submicron-focused capability and achieved a focused X-ray beam of extremely high flux for a beam size of 300 nm and a reasonable flux for a beam size of 100 nm. Ongoing studies using the submicron-focused X-ray beam include (i) XAFS analysis of single particles of micro/nanocatalysts [1], (ii) in situ and time-resolved XAFS in phase-change memory devices, and (iii) two-dimensional mapping of chemical, structural, and magnetic states in Nd$_2$Fe$_{14}$B sintered permanent magnets. We have a number of instrumentation issues that need to be addressed: we hope to achieve higher stability of the focused beam in the short and long term and expand the variety of sample environments; we are also considering the use of a fast and large-area X-ray detector to accept strong X-ray fluorescence signals from the high input photon flux on the sample that has been achieved by this upgrade.

Acknowledgments
This work was performed with the approval of SPring-8 (Nos. 2011A1808, 2011A2058, 2011A2065, and 2011A2066) and was supported by the Research-Network Building Program for Reduction of Carbon-dioxide Emission and a Grant-in-Aid for Scientific Research (No. 23360016) from the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan.

References

Figure 2. (a) X-ray magnetic circular dichroism and (b) X-ray absorption spectra of a Co$_{80}$Pt$_{20}$ magnetic dot of 200-nm diameter, measured at the Pt L$_3$ edge.

Figure 3. Element-specific magnetization curves of Co$_{80}$Pt$_{20}$ magnetic dots, recorded with a 300-nm focused X-ray beam (solid circles) and 2-μm beam (line).