***In situ* membrane bending setup for strain-dependent scanning transmission x-ray microscopy investigations**

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We present a setup that allows for the *in situ* generation of tensile strains by bending x-ray transparent Si_3N_4 membranes with the application of a pressure difference between the two sides of the membrane, enabling the possibility to employ high resolution space- and time-resolved scanning transmission x-ray microscopy for the investigation of the magneto-elastic coupling. *Published by AIP Publishing.* [<http://dx.doi.org/10.1063/1.4971849>]

INTRODUCTION

The magneto-elastic (or inverse magnetostrictive) coupling allows one to introduce many different effects by applying a mechanical strain to a magnetostrictive material, ranging from changes in the saturation magnetization and in the Curie temperature, to the control of the magnetic anisotropy, and even to the possibility to induce phase transitions in the magnetostrictive material.^{1–8} In particular, the possibility to manipulate the magnetic anisotropy by strain allows for the magnetic field-free control of the magnetization.^{1–6} Furthermore, a rich magneto-dynamic behavior in magnetostrictive materials as a function of the applied strain is predicted from micromagnetic simulations,³ making the experimental investigation of the magneto-elastic effect at the ns time scale also of interest.

The traditional methods employed for the investigation of the magneto-elastic coupling in magnetostrictive materials involve the use of piezoelectric crystals or thin films to generate the required strain.^{1–6} For micro- and nanostructured magnetostrictive elements, the magnetic configuration is investigated by spatially resolved magnetic microscopy techniques such as magnetic force microscopy,^{4,5} and x-ray photoemission electron microscopy.^{1–3} However, both techniques have shortcomings. Magnetic force microscopy does not allow for time-resolved measurements, and extra care has to be taken that the stray field generated by the magnetized tip does not involuntarily influence the magnetic configuration of the sample. Instead, photoemission electron microscopy requires the samples to have a conducting surface, and its probing depth is limited to only a few nm from the top surface.⁹ Finally, the use of piezoelectric crystals as substrates further limits the possibilities of time-resolved investigations due to the challenges in the fabrication of impedance matched structures (e.g., Oersted lines) on top of these materials.

Scanning transmission x-ray microscopy (STXM) is a non-invasive microscopy technique that can be employed for the space- and time-resolved investigation of micro- and nanostructured magnetic materials without the depth and

conductivity limitations of photoemission electron microscopy. However, STXM requires samples fabricated on x-ray transparent substrates, which has severely limited the application of this technique for the study of the magneto-elastic coupling. In this work, we present a setup that allows for the straining of micro- and nanostructured magnetic materials fabricated on x-ray transparent Si_3N_4 membranes. In particular, static tensile strains of magnitudes comparable to those achievable with piezoelectric crystals can be applied, whilst still preserving the experimental conditions for quasi-static and time-resolved STXM imaging. Furthermore, thanks to the use of silicon as the substrate frame, the fabrication of impedance matched structures is simpler if compared to piezoelectric crystal substrates, therefore simplifying the procedure for time-resolved measurements.

Finally, we demonstrate the performance of our setup in both the quasi-static and time-resolved imaging regimes by investigating the magneto-elastic anisotropy in microstructured Ni elements.

DESIGN OF INSTRUMENTATION

In a STXM imaging experiment, schematically illustrated in Fig. 1, the samples, typically fabricated on x-ray transparent Si_3N_4 membranes, are positioned at the focal point of a focused x-ray beam. The transmitted photon intensity is recorded with a suitable detector (typically, an avalanche photodiode—APD—or a photomultiplier tube). To form an image, the sample is raster scanned with a piezoelectric stage, and the transmitted x-ray intensity is recorded at each point of the scan. The x-rays are focused on a sub- μm spot (for soft x-rays on the order of 10–20 nm) on the sample by a Fresnel zone plate. For magnetic materials, it is possible to directly image the magnetic configuration of the sample by illuminating it with circularly polarized x-rays, employing the x-ray magnetic circular dichroism (XMCD) effect.^{10,11} Depending on the relative orientation of the sample with respect to the wave vector of the incoming circularly polarized x-ray beam, magnetic images with in-plane and out-of-plane contrast can be obtained with XMCD-STXM imaging.

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FIG. 1. Sketch of the operating principle of STXM imaging. The monochromatic x-rays are focused onto a sub- μm spot on the sample with a Fresnel zone plate, combined with an order selecting aperture (OSA), which allows only the first-order light (i.e., the focused beam) to illuminate the sample. The photons transmitted through the sample (usually fabricated on an x-ray transparent Si_3N_4 membrane) are then detected with a suitable detector.

For experiments aimed at the analysis of the magneto-elastic effect with XMCD-STXM imaging, the requirement of x-ray transparent samples severely limits the possibility to employ piezoelectric crystal substrates or thin film piezoelectrics which are grown on single crystalline substrates. Here, we describe a setup, currently installed at the PolLux (X07DA) beamline of the Swiss Light Source, that allows us to overcome this issue by employing thin Si_3N_4 membranes to generate the strains necessary for the investigation of the magneto-elastic effect, whilst still preserving the experimental conditions for both quasi-static and time-resolved STXM imagings.

The setup reported here employs, for the generation of tensile strains up to about 10^3 ppm, the mechanical bending of thin Si_3N_4 membranes when applying a pressure difference between the two sides of the membrane. This is achieved by installing the Si_3N_4 membrane in a sealed gas cell, where N_2 gas can be injected to the desired pressure, while the rest of the STXM chamber is kept in vacuum. This module is based on an environmental gas cell design previously developed for the PolLux beamline,¹² with some additional elements that allow the integration of the membrane bending setup with the other modules employed for the investigation of the magnetization dynamics. In particular, the design of the membrane bending setup described here employs one of the sealing elements of the setup described in Ref. 12 and utilizes newly designed front sealing element as well as gas connections outside of the vacuum chamber, which were specifically designed for the purpose of bending membranes. The setup is schematically shown in Fig. 2(a) where two Si_3N_4 membranes are used to create a sealed environment inside the vacuum chamber of the STXM.

The gas cell is connected to the outside of the vacuum chamber, where different gases can be injected, causing the bending of both the front sample and the back sealing membrane. Note here that the back membrane is simply employed to maintain the pressurization inside the gas cell, and the bending of the second membrane does not have any influence on the strain generated by the first membrane. The gas cell system here described allows for the use of different gases to pressurize the gas cell. Here, N_2 was selected as the pressurizing gas out of practical considerations, as the PolLux beamline is equipped with a centralized N_2 gas line, which simplifies the connections to the gas cell. This does not hinder the possibility of employing different pressurizing gases (e.g.,

FIG. 2. (a) Schematic design of the gas cell setup employed to generate strain by membrane bending. Two Si_3N_4 membranes are used to create a sealed environment, where gaseous N_2 can be injected at a desired pressure by operating a needle valve. The pressure difference between the two sides of the membranes is monitored with two capacitance pressure meters (G_1 and G_2), one installed directly in the STXM vacuum chamber and the other connected to the sealed environment of the gas cell. The gas cell can be pumped by opening a valve that connects the gas cell directly with the STXM vacuum chamber. To avoid the accidental damaging of the Si_3N_4 membranes, an overpressure valve (0.2 barg) is installed on the pressurizing line. The setup allows for operation both in the static and flow regimes; (b) photograph of the PCB employed for the injection of electrical currents. The PCB has the double function of providing a sealed environment, whilst allowing at the same time for the injection of electrical currents; (c) photograph of the mounted gas cell setup, showing how the PCB integrates with the gas cell. RF stands for Radio Frequency.

lighter gases such as He, with a lower x-ray absorption at the energies typically employed for XMCD-STXM imaging experiments), should the need arise.

The setup can be operated to generate a pressure difference between the two sides of the Si_3N_4 membrane. As schematically shown in Fig. 2(a), the connections outside of the vacuum chamber are limited to a single valve that allows the connection of the gas cell with the STXM vacuum chamber (therefore allowing for the depressurization of the cell). The cell is pressurized by operating a needle valve that connects a N_2 gas line to the cell itself. To avoid the accidental damaging of the Si_3N_4 membranes by an overpressure (the membranes employed for the proof-of-principle measurements described later in this manuscript were damaged by pressure differences higher than 1.2 bar), the gas line is equipped with an overpressure valve, limiting the pressure to 1.2 bar absolute (0.2 barg). The pressure difference between the two sides of the Si_3N_4 membrane is measured with two capacitance pressure meters (Pfeiffer CMR 361), one installed on the outer connections of the gas cell and the other installed directly on the STXM vacuum chamber. The pressure measurement system is integrated with the experimental physics and industrial control system (EPICS) of the PolLux beamline, allowing for the automatic measurement of both pressures.

To allow for time-resolved investigations, it is necessary to provide a mechanism that enables the excitation of the dynamical processes under analysis. Here, we will focus on magneto-dynamic processes, which are typically excited either with a fast magnetic field excitation (see, e.g., Refs. 13–16) or by injecting electrical currents in the magnetic material (see, e.g., Ref. 17). Both of these excitation methods require the injection of electrical current pulses on ns-time scales on either a stripline/coplanar waveguide (that act as Oersted lines) or across the magnetic material itself. It is thus necessary to provide an impedance matched electrical connection inside the gas cell, whilst guaranteeing a proper sealing at the same time.

To allow for the injection of electrical pulses on the sample installed in the gas cell, a printed circuit board (PCB) combined with a sealing element (see Fig. 2(b)) was employed as the front sealing element of the gas cell. The PCB was fabricated using a low-degassing PTFE laminate (Rogers RO-4003C), and the electrical vias in the board were sealed with a vacuum-tight epoxy (EPO-TEK 353ND), which was also employed to attach the sealing element to the PCB. The Si_3N_4 membranes can then be attached to the PCB with a vacuum-tight wax (Crystalbond 509 Amber), with their top surface facing to the inside of the gas cell. As shown in Fig. 2(c), the PCB sealing element is designed to integrate with the other components of the gas cell. The whole setup can then be integrated with the sample holder setup of the PolLux beamline, as shown in Fig. 3.

BENDING OF THIN MEMBRANES

The application of a pressure difference between the two sides of a thin Si_3N_4 membrane causes, as shown in Fig. 4, an outward bending of the membrane, associated with

FIG. 3. Photograph of the mounted gas cell (including the PCB for the injection of electrical current pulses) installed on the 30° holder of the STXM at the PolLux beamline of the Swiss Light Source. The gas cell can also be mounted on the 0° holder for the investigation of perpendicularly magnetized materials.

the generation of a tensile strain on the membrane and on the materials grown on top of it. The bending of a thin membrane (i.e., thickness of the membrane much smaller than the maximum deformation resulting from the bending) due to a uniform pressure can be described according to the relations given in Refs. 18 and 19. In particular, in the elastic regime, the membrane exhibits a parabolic bending, with the largest displacement at its geometrical center. In the simple case of a circular membrane, under the assumption that the bending of the membrane only gives rise to radial strain (i.e., the tangential strain is zero¹⁹) the displacement at the center of the membrane (see Fig. 5(a)) can be determined analytically as follows:¹⁹

$$\Delta p = 4 \frac{ht}{r^2} \left(\sigma_0 + \frac{2}{3} \frac{h^2}{r^2} \frac{Y}{1-\nu^2} \right), \quad (1)$$

where Δp is the pressure difference between the two sides of the membrane, h is the displacement along the z -direction at the center of the membrane, t is the thickness of the membrane, σ_0 is the residual stress of the membrane, r is the radius of the circular membrane, and Y and ν the Young modulus and Poisson ratio of the Si_3N_4 , respectively. Solving Eq. (1) for the membrane displacement h as a function of the pressure difference will allow us to estimate the displacement

FIG. 4. Optical microscopy images of a $1.5 \times 0.25 \text{ mm}^2$ Si_3N_4 membrane (50 nm thick) mounted on the gas cell setup, showing the bending of the membrane at different applied pressures inside the gas cell.

FIG. 5. (a) Schematic illustration of the definitions of the variables used in Eq. (1), showing a simplified circular membrane model (where it is assumed that the thickness of the membrane is much smaller than its radius). The red line shows the section of the circular membrane employed for the estimation of the radial strain; (b) and (c) calculation, according to Eqs. (1) and (2), of the magnitude of (b) the membrane deflection and (c) the radial strain obtainable for two different radii of a circular membrane as a function of the pressure difference between the two sides of the membrane. Here, it was assumed that the residual stress of the Si_3N_4 membrane is negligible, and a Young modulus and Poisson ratio of 310 GPa and 0.25 were utilized, respectively. The thickness of the membrane was assumed to be 50 nm.

at the geometrical center of the membrane at a given pressure difference. From this value, it is then possible to estimate the radial strain, given by the following relation:¹⁹

$$\epsilon_r \approx \frac{2}{3} \frac{h^2}{r^2}. \quad (2)$$

In Figs. 5(b) and 5(c), the membrane deflections and radial strains calculated with Eqs. (1) and (2), with realistic values for the pressure difference and membrane radius are shown.

Here, it is possible to observe that strains up to 1000 ppm can be generated.

In the more realistic case of a square or rectangular membrane, which are the typical Si_3N_4 membrane geometries employed for STXM investigations, the dependence of the bending upon the application of a pressure difference needs to be estimated either experimentally or simulated with finite element method simulations.^{18,19} Furthermore, for rectangular membranes, the different aspect ratio will give rise to a different magnitude of the strain generated along the edges of the membrane, thus allowing, by judiciously selecting the geometry of the membrane, to tune the direction of the applied strain. One possible example, which will be shown in the proof-of-principle measurements reported later in this article, would be the generation of a quasi-uniaxial tensile strain by choosing a rectangular Si_3N_4 membrane with a large aspect ratio.

Eq. (2) can be employed, once the displacement of the geometrical center of the membrane is known, to estimate the magnitude of the radial strain generated by the bending. This is a directly measurable quantity in a STXM imaging experiment: when the membrane bends, its surface no longer finds itself in the focal point of the x-ray beam, resulting in the acquisition of a de-focused image. To obtain, once again, a focused image, it is necessary to retract the zone plate by a distance equal to the z-displacement of the membrane, therefore allowing, from the reading of the zone plate position, the *in situ* determination of the membrane displacement and, from Eq. (2), the magnitude of the applied strain. An example of the membrane displacements that can be measured by determining the position of the zone plate as a function of the applied pressure difference is shown in Fig. 6(a), and the estimated magnitude of the tensile strain generated by such bending (using Eq. (2)) is shown in Fig. 6(b).

Note that tensile strains on the order of 10^2 – 10^3 ppm can be generated with the setup reported here. For comparison, piezoelectric crystal substrates also allow the generation of strains up to 10^3 ppm (see, e.g., Ref. 20 for the strain-vs.-electric field curves for $[\text{Pb}(\text{Mg}_{1/3}\text{Nb}_{2/3})\text{O}_3]_{(1-x)}-[\text{PbTiO}_3]_x$). To generate tensile strains of magnitudes comparable to those achievable with piezoelectric crystals, it is necessary to generate pressure differences on the order of 10^2 mbar. As the x-rays also cross the pressurized volume of the gas cell, the injection of N_2 gas at high pressures will lead to a reduction of the transmitted intensity and to an increase of the measurement time (under otherwise equal conditions). The transmittivity of the gas cell was verified up to pressure differences of 1 bar and, for this extreme case, the count rates halved in comparison to the depressurized condition. This reduction of the transmitted intensity is comparable to the predicted reduction of transmittivity of soft x-ray photons (energies in the range between 700 and 850 eV, covering the L_3 edges of the most important 3d magnetic elements) in a 1 mm long section containing 1 bar of N_2 , according to the tables reported in Ref. 21. Therefore, STXM imaging experiments are possible even at the highest pressure differences allowable by the employed membranes.

As pressure differences of the order of 10^2 mbar are necessary to achieve the desired strain magnitudes, the

FIG. 6. (a) Displacement along the z -direction of the central point of a $1 \times 0.5 \text{ mm}^2$ Si_3N_4 membrane (thickness of 50 nm) as a function of the pressure difference between the two sides of the membrane. The displacement was measured by determining the retracting distance of the Fresnel zone plate that focuses the x-ray beam onto the sample surface. The error in the measurements is given by the resolution of the focus scan employed, and is eventually limited by the depth of focus of the zone plate (in the case shown here, the depth of focus of the zone plate at the considered x-ray energy is of about $1 \mu\text{m}$); (b) estimation of the tensile strain along the directions parallel to the edges of the membrane (x being the 1 mm long edge, and y the 0.5 mm long edge) determined by applying Eq. (2), showing that the choice of the aspect ratio of the membrane can be employed to modulate the direction and magnitude of the tensile strain caused by the bending of the membrane.

pressure stability of the gas cell is a critical point for the success of the measurements. To achieve such pressure stability, the sealing of the gas cell is verified prior to its mounting, by applying an overpressure with respect to the atmospheric pressure and monitoring the pressure variation. The typical points where a leakage was observed were in the sealing of the electrical vias of the PCB (which is easily amended by repeating the sealing procedure described earlier), or on the wax platform employed to attach the sample to the PCB (in this case, the leakage can be sealed by re-melting the wax and allowing it to solidify once more whilst applying a small pressure on the silicon frame of the sample to guarantee a good contact with the wax). Once the correct sealing of the gas cell is verified, the pressure of the gas inside the gas cell was observed to be stable over periods of several days,

therefore providing the necessary experimental conditions for the measurements.

PROOF-OF-PRINCIPLE MEASUREMENTS

To verify the performances of the setup reported here, proof-of-principle measurements, both in the quasi-static and in the time-resolved regimes, were carried out. The experimental setup consisted of 20 nm thick microstructured Ni squares (with an edge length of $2 \mu\text{m}$) lithographically patterned on rectangular, 50 nm thick, $1 \times 0.5 \text{ mm}^2$ Si_3N_4 membranes. The magnetic configuration of the Ni microstructures was investigated both quasi-statically and dynamically as a function of the applied pressure. As the Ni microstructures exhibit an in-plane magnetization configuration, the samples were mounted at a 30° angle with respect to the incident x-ray beam (see Fig. 3 for a photograph of the sample setup).

In the quasi-static proof-of-principle measurements, the magnetic configuration of the Ni microstructured squares was investigated as a function of the applied tensile strain, as shown in Fig. 7. At no applied strain (depressurized gas cell), the Ni squares exhibit a symmetric Landau flux closure pattern.²² When the Ni squares are strained, a strain-induced uniaxial anisotropy arises, causing the growth of the domains with the magnetization pointing along the anisotropy axis (in the case shown in Fig. 7, the domains with the magnetization along the x axis grow at the expense of the domains pointing along the y axis). The changes in the magnetic configuration of the Ni squares caused by this additional uniaxial anisotropy are comparable to those obtainable when generating the strain with a piezoelectric crystal (see Ref. 1).

This additional, strain-induced, magneto-elastic anisotropy term (K_{ME}) can be described, under the assumption of a thin film magnetostrictive material where shear strain is negligible, by the following relation:^{1,2,4,6}

$$K_{\text{ME}} = -\frac{3}{2}\lambda_s Y |\epsilon_{xx} - \epsilon_{yy}|, \quad (3)$$

where λ_s denotes the magnetostrictive constant (for Ni, $\lambda_s \approx -32 \text{ ppm}$ ²³), and ϵ_{ii} the applied strain along the $i = \{x, y\}$ direction.

By comparing the measured magnetic configuration of the Ni microstructured squares with micromagnetic simulations, it is possible to reliably estimate the magnitude of the magneto-elastic anisotropy.¹ These simulations were carried out with the MicroMagnum framework,²⁴ using the same parameters as employed in Ref. 1. This provides a testing ground for verifying the determination of the applied strain through the measurement of the zone plate displacement described above, the magneto-elastic anisotropy estimation through the micromagnetic simulations being independent from the membrane displacement. Fig. 8 shows the comparison between the values of the magneto-elastic anisotropy obtained from the comparison with micromagnetic simulations (black dots) and from the determination of the position of the zone plate as a function of the applied pressure (red squares). A good agreement between the two methods can be observed, demonstrating that Eq. (3) provides a reasonable estimation of the applied strain also for rectangular membranes.

FIG. 7. XMCD-STXM images of the in-plane magnetic configuration of a $2\ \mu\text{m}$ wide Ni microstructured square as a function of the applied strain (pressure difference between the two sides of the Si_3N_4 membrane). As the applied strain is increased, an additional, strain-induced uniaxial magnetic anisotropy is generated, causing the growth of the domains pointing along the x axis (black and white domains in the images) at the expense of the domains pointing along the y axis. The red arrows indicate the orientation of the magnetization in the square.

FIG. 8. Magnitude of the uniaxial anisotropy generated by the application of a pressure difference between the two sides of a rectangular Si_3N_4 membrane. The measured values were determined by comparing the magnetic configuration of the microstructured Ni elements, measured by XMCD-STXM imaging, with micromagnetic simulations of the Ni microstructures with different uniaxial magnetic anisotropies applied, similarly to the method reported in Ref. 1. The measured anisotropy values were then compared with the anisotropy calculated from the measured membrane displacement values, using Eq. (3) to then estimate the magnitude of the uniaxial anisotropy. A good agreement between the measured and calculated magnitudes of the uniaxial anisotropy can be observed.

Finally, the performances of the setup reported here were verified also for time-resolved measurements. For this purpose, a $10\ \mu\text{m}$ wide, $100\ \text{nm}$ thick Cu stripline was fabricated on top of the microstructured Ni squares. The magnetic configuration of the microstructured Ni squares was excited by generating a $40\ \text{ns}$ long in-plane magnetic field pulse (by injecting a current pulse across the Cu stripline), with a field magnitude of about $2\text{--}4\ \text{mT}$ at the employed pulse amplitudes. The dynamical response of the Ni microstructures was then recorded with time-resolved STXM imaging in the pump-probe scheme. The experiments were repeated for various applied pressure differences and, as shown in Fig. 9, profound changes in the dynamical behavior of the magnetization in the microstructured Ni elements were observed upon changing the applied strain. These changes in the dynamical behavior of the magnetization were attributed to the influence of the additional magneto-elastic anisotropy term described statically in Eq. (3). In particular, we observe that the dynamical response of the system is strongly reduced

FIG. 9. Proof-of-principle time-resolved measurement of a $2\ \mu\text{m}$ wide Ni microstructure excited by a $40\ \text{ns}$ long magnetic field pulse as a function of the pressure difference between the two sides of the Si_3N_4 membrane on which the structures are fabricated. The graph shows the XMCD signal in the area of the Ni square marked by the blue circle (inset), where it is possible to observe profound changes in the magnetization dynamics, caused by the applied strain. The red arrows in the inset indicate the direction of the magnetization in the Ni square.

when a high pressure difference between the two sides of the membrane is applied, which could be interpreted by the higher energy cost necessary to expand a domain with the magnetization perpendicular to the easy axis of the magneto-elastic anisotropy term at higher pressures (i.e., higher magnitude of the magneto-elastic anisotropy).

These first proof-of-principle measurements demonstrate that the membrane bending setup reported here is suited for time-resolved STXM imaging experiments which are aimed at analyzing the magneto-elastic effect at the ns and sub-ns time scales.

CONCLUSIONS

We report here an experimental setup that enables the *in situ* tensile straining of samples by applying a pressure difference between the two sides of a thin Si_3N_4 membrane. This enables the straining of materials without the need to employ piezoelectric materials, whilst maintaining the possibility to execute both quasi-static and time-resolved STXM

imaging experiments. The capabilities of the instrumentation described here have been demonstrated by investigating the magneto-elastic coupling in microstructured Ni elements both in the quasi-static and time-resolved configurations.

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