Spatially resolved measurements of the ferromagnetic phase transition by ac-susceptibility investigations with x-ray photoelectron emission microscope

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Spatially resolved ac susceptibility measurements on epitaxial Fe films are performed as a function of temperature using a conventional soft-x-ray photoelectron emission microscope. A magnetic contrast is observed at sample locations where the magnetic film undergoes a para/ferromagnetic phase transition. Due to the wedge structure of the Fe film and the thickness dependence of the Curie temperature the spatial extend of the phase transition region and the correlation length can be estimated.

X-ray magnetic circular dichroism^{1–3} (XMCD) is a very powerful tool to investigate magnetic properties in an element specific way. The combination of XMCD with a photoelectron emission microscope (PEEM), the so called X-PEEM (Ref. 4) provides a lateral resolution which allows to determine magnetic properties with a spatial resolution down to 15 nm. Even time^{5,6} and depth resolution⁷ is possible. On the other hand, integral ac-XMCD has been performed to measure the element-specific ac-susceptibility at the para- to ferromagnetic phase transition in magnetic monolayers. Here, we combine both approaches to record the element-specific ac-susceptibility on a Fe wedge film with a spatial resolution of the order of less than 100 nm. The determination of the Curie temperature (T_C) was carried out using complementary ac susceptibility (χ_{ac}) combined with integral XMCD measurements for instance by Aspelmeier *et al.*⁸

In ferromagnetic structures with nanoscale dimensions below the characteristic exchange length, finite size effects may change the effective magnetization and shift T_C to lower values. E.g., nanoparticles become paramagnetic at room temperature,⁹ or become superparamagnetic¹⁰ depending on the composition. To study those changes of T_C of a single nanoparticle interacting with other nanomagnets in an ensemble is of increasing interest for the stability of data storage and spintronic applications.

The correlation length diverges at T_C , as demonstrated in Ref. 11. We have realized spatially resolved χ_{ac} measurements with an X-PEEM by triggering its imaging unit to the different directions of an alternating magnetic field and measuring the XMCD contrast, to show for instance the divergence of the correlation length.

In this letter, we show how a χ_{ac} measurements performed in combination with PEEM provide a unique tool to measure (element-specific) T_C in nanostructures with spatial resolutions of smaller than 100 nm. The results obtained on a prototype Fe thin film directly show the applicability of the method to nanomagnetism in general.

Experiments were performed at the Helmholtz-Zentrum Bessy II/UE49 using the spin-resolved photoelectron emission microscope (SPEEM) setup. A detailed sketch of the setup is shown in Fig. 1. To measure χ_{ac} we used a sample holder implementing alternating magnetic fields and temperature control. With the integrated magnetic yoke we applied a magnetic field of ± 0.2 mT alternating with a frequency of 250 Hz during imaging. The current driving the field coils (4 in Fig. 1) is indicated by I_{B+} (3 in Fig. 1). Image shifts between the two field directions due to the Lorentz force appeared to be very small (few pixels only) and could easily be compensated by a drift correction using a structural defect as a reference. The temperature was controlled by resistively heating the magnetic yoke, which can also be cooled with liquid N_2 down to 150 K. We monitored the temperature with a Pt100 thermometer. Absolute values of T may differ by 10 K, while the relative accuracy is about 0.2 K.

We studied the χ_{ac} of an epitaxial Fe wedge grown on InAs(001) (Ref. 12) and capped with 3 nm Al. Due to the wedge shape with a maximum thickness of 10 atomic monolayers T_C varied in our sample from below 1N₂ temperature

FIG. 1. (Color online) Schematic setup of the measurement. The sample (1) is fixed on top of the magnet yokes and is illuminated with x-rays, while the produced free electrons pass the SPEEM optics. At the channel plate (2) they will only be multiplied if a high voltage is applied. This voltage is triggered to the phase (3) of the magnetic field, which is produced with an inductive coil (4). The temperature (T) is varied by resistive heating in the magnet yoke (5), while a base temperature is set with a liquid nitrogen cooled sample holder (6). T is measured by a resistance thermometer (rt).

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FIG. 2. (Color online) χ_{ac} images of 10 ML Fe/InAs(001) at the L_3 edge. The darker gray band seen in the center of the grayscale images corresponds to the χ_{ac} signal at different T . For higher temperatures the signal moves to the left side, where the film becomes thicker. The inset on the left hand is the normal XMCD contrast of the same region where lighter gray is the ferromagnetic and thicker part (see wedge structure over images). The inset on the right hand side is χ_{ac} signal for $T \gg T_C$, where no χ_{ac} is observable. (a)–(d) mark the corresponding thickness in Fig. 3. See text for details.

to 290 K, as confirmed by integral superconducting quantum interference device (SQUID) measurements. The rather low T_C at the maximum thickness is most likely due to Fe-Al interdiffusion which is known to result in reduced Curie temperatures.¹³ Prior to the χ_{ac} measurements we recorded static XMCD images at the Fe L_3 edge as a function of T to identify the T_C phase transition on our wedge. Starting from below room temperature we aligned the PEEM on the ferromagnetic (i.e., thick) part of the Fe wedge. Then we slowly increased the temperature until we could observe the ferromagnetic paramagnetic phase-transition within our field of view as shown in the upper left inset in Fig. 2.

While in XMCD measurements we obtained the magnetic contrast by subtracting two images recorded with opposite x-ray helicities at the Fe L_3 resonance, in the χ_{ac} we kept the helicity constant and evaluated the difference of two images taken with opposite directions of the applied magnetic field. This required a synchronization of the SPEEM imaging unit with the alternating magnetic field. To achieve this we gated the channel plate voltage with a voltage offset of 400 V indicated by U_{CP} in Fig. 1. This enhances the image amplification during the gate pulse by three orders of magnitude.¹⁴ Since the charge coupled device camera we used for image acquisition could not be operated at 250 Hz, we had to integrate the signal for one field direction over many switching cycles of $I_{B\pm}$.

With a somewhat slower frequency of about 1 Hz we shifted the phase of U_{CP} by 180° , to record images for both field directions. To obtain an image of the spins while oscillating the magnetic field we recorded about 50 images, each with one second exposure time, shifting the phase of U_{CP} by 180° after each image. Finally we performed a drift correction and added images with the same direction of the applied magnetic field. As χ_{ac} signal we display the difference between two images recorded with the same helicity but opposite directions of applied magnetic field divided by their sum. This magnetic contrast is proportional to the susceptibility signal.

Results of the χ_{ac} measurements are shown in Fig. 2, where a series of four typical χ_{ac} images at the Fe L_3 edge and at different temperatures is shown. Dark gray corresponds to a larger χ_{ac} contrast, while light gray is equal to zero χ_{ac} contrast. We started at lower temperature (left image) where the T_C phase transition could be observed in the center of our field of view (see inset). In the corresponding χ_{ac} contrast we find a thin line at the wedge position where

T_C is reached. The signal is due to the change in a hysteresis with the temperature: the closer the temperature to the Curie point, the smaller the coercive field of the hysteresis of a ferromagnetic sample.¹⁵ When measuring with a given fixed amplitude of the alternating magnetic field, at a certain temperature the ac field is sufficiently strong to switch the magnetization, so that the magnetic contrast of the spins oscillating with the external field appears in our image. The width of this line indicates the region in which the coercivity is sufficiently low and this width will depend on the thickness slope of the wedge, the amplitude of the magnetic field, and the material of the wedge itself. With increasing temperature we observe the dark line corresponding to a susceptibility signal propagating from the right toward the left where the thickness of the Fe wedge is larger. The full width half maximum (FWHM) of this χ_{ac} signal changes from 0.7 to about $2 \mu\text{m}$ with temperature but is not strictly proportional to it.

To explain this behavior, a conventional PEEM measurement at the Fe L_3 edge (707 eV) and at the pre-edge (703 eV) were performed. They are subtracted from each other and result in a contrast which is proportional to the amount of Fe. In Fig. 3, the averaged line profile of this difference picture is shown, measured along the direction perpendicular to the susceptibility signal of Fig. 2 (compare with inset). It clearly indicates a varying film thickness with a peak at $8 \mu\text{m}$ which corresponds to the broad susceptibility signal at

FIG. 3. (Color online) Height profile of the measured area. Shown is a line scan (averaged over the marked area in the inset) of a X-PEEM image of the difference of the edge/pre-edge of Fe (the inset shows the χ_{ac} and not the X-PEEM picture). The signal is proportional to the Fe thickness, which determines the Curie temperature in this case. (a)–(d) marks the corresponding χ_{ac} signal in Fig. 2.

FIG. 4. (Color online) Comparison of XMCD and χ_{ac} signal. The upper part shows the χ_{ac} signal, the lower part the XMCD image. The transition area has in XMCD and χ_{ac} contrast the same width of about $0.7 \pm 0.1 \mu\text{m}$.

$T = 258.3 \text{ K}$ in Fig. 2. The susceptibility signal propagates as expected parallel to the edge gradient.

As reported in Ref. 16, the thickness dependence of T_C can be described as $[T_C(\infty) - T_C(d)]/T_C(\infty) = Cd^{-1/\nu}$, where $T_C(\infty)$ is the bulk Curie temperature, d the thickness, and ν the critical exponent. This thickness dependence qualitatively accounts for the spatially propagating susceptibility signal via the change in d .

The large FWHM in the two pictures on the right in Fig. 2 can be explained by the peak in the height profile. In Fig. 3 in the range of 6.2 to $8.8 \mu\text{m}$ the change in d and the resulting T_C is in the same limits as the high ac field allows to separate. Therefore that area is only visible as one susceptibility signal with $\text{FWHM} = 2 \mu\text{m}$.

The width of the phase transition measured by XMCD and χ_{ac} is in the same range of $0.7 \pm 0.1 \mu\text{m}$, like demonstrated in Fig. 4. These different measurements show, that the correlation length at T_C seems to be in the range of $0.7 \mu\text{m}$.

In conclusion, we use an X-PEEM setup that allows for ac susceptibility measurements to study the spatially resolved T_C in an Fe wedge-like film. We find that the conventional X-PEEM setup is expanded to allow ac susceptibility mea-

surements. Its functionality is demonstrated and we observed the thickness dependent T_C of an epitaxial thin Fe film with a narrowest FWHM of about $0.7 \pm 0.1 \mu\text{m}$, which is explained by the divergence of the correlation length at T_C . The measurements clearly reveal that X-PEEM is an extremely suitable method to perform laterally resolved χ_{ac} measurements combined with element specificity. Future work is expected to yield measurements of T_C of single nanoparticles.

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