ESRF	**Experiment title:** Finite size effect on the alpha//beta phase transition in MnAs patterned thin films.	**Experiment number**: HS-3632
Beamline: ID-01	**Date of experiment**: from: Nov. 5th, 2008 to: Nov. 11th, 2008	**Date of report**: Jan 2009
Shifts: 18	**Local contact(s)**: P.Boesecke	*Received at ESRF:*

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Report:

Ferromagnetic MnAs is a promising candidate for electrical spin injection into GaAs and Si based semiconductors [1]. It exhibits a large carrier spin polarization, small coercive field and relatively high saturation magnetization and Curie temperature. Bulk MnAs is ferromagnetic (FM) at room temperature (RT) (α phase, hexagonal NiAs-type structure (P63/mmc)) and shows close to 40°C a first order transition to the paramagnetic β phase (orthorhombic, MnP-type structure (Pnma)). Epitaxial MnAs films on GaAs substrates, show at RT the coexistence of both phases [2] resulting in the formation of self-organized stripes of alternating α and β phases due to anisotropic strain applied by the GaAs substrate [4]. We aimed at investigating the finite size and confinement effects on the strain release on the α/β coexistence in MnAs thin films since one cannot simply extrapolate the thin films results to patterned objects [5].

Specular X-ray diffraction allows probing the crystalline structure of the full film thickness and allows to address the possible relaxations providing new insights in the understanding of the correlations between relaxation and striped phase formation for low-lateral dimension objects. A 300 nm thick MnAs film epitaxially deposited on GaAs single crystalline substrate and was subsequently electron-beam lithographed to obtain patterns with samples of lateral dimensions from sub-micron (0.75 µm) to 12 µm as shown in **Fig. 1**; the expected orientation of the α / β stripes in the thin film is also shown. The chosen shapes enables the study of the size effect with respect to the orientation of the confinement dimension with respect to the stripes. Intuitively, effects are expected to appear for dimensions reaching the 1.5 µm (~5 × film thickness).

In order to probe individual structures by diffraction, a micron-size beam (E = 9.5 keV) was produced using a set of 36 Be parabolic lenses placed ~750 mm upstream the sample providing a beam of 1.7 × 4.5 μm^2 (full width half maximum FWHM, vert. × horiz.) and ~10^{11} ph./s (3 undulators). For a vertical diffracting plane geometry and the typical Bragg angles characteristic to the two crystalline phases at these energy (24° and 24.3° respectively), the footprint of the beam on the sample can be assimilated to a disc of 5 µm, small enough to ensure the illumination of single structures[1].

[1] In the case of the big objects, it characterizes the center of the object, but measurements on a "grid" fully describing the object were performed as well, but not reported here

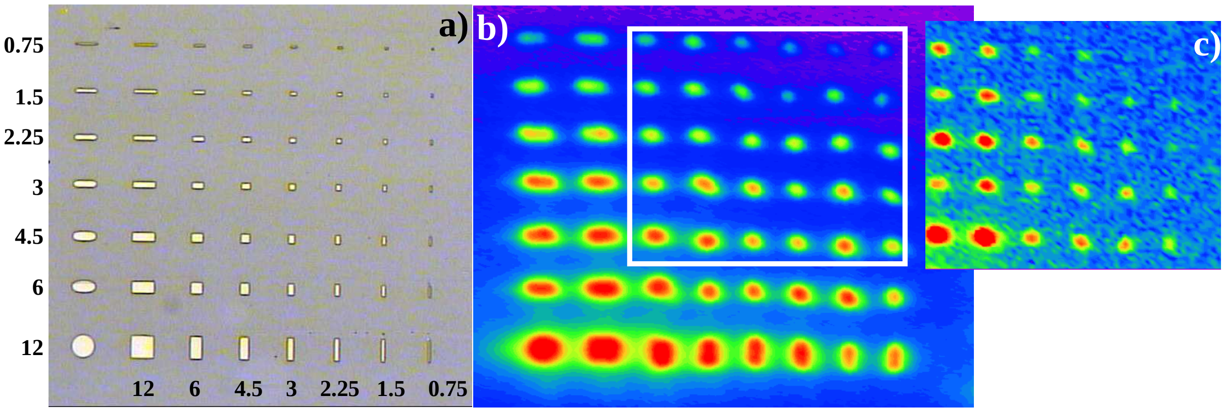

Figure 1: (a) Optical image of the patterned sample: rectangular structures with lateral sizes from 0.75 to 12 µm are produced (the sizes are marked on the figure). The hatched area consists of a column of elliptic structures that were not investigated in this project. The average spacing between structures is ~30 µm in both dimensions. (b) Raster image obtained with x-rays (see text), showing the sample (characteristic position for the α phase, 2θ=48°). The ellipse structures are as well identified (hatched area). The white rectangle shows the area corresponding to the β-phase image depicted in c); (c) Raster image showing part of the patterned area, characteristic position of the β phase (2θ=48.6°).

First we had to localize and identify the different structures / shapes. The approach used (for details see [6]) is the following: the angles of the diffractometer are set such that, in reciprocal space, the detector is recording the signal corresponding to the MnAs layer (α or β phase, *i.e.* θ-2θ geometry, θ=24° and 24.8° respectively). Then, a raster image is obtained by scanning laterally the sample and recording in each point the detector signal (**Fig. 1b**). It is then possible to laterally place the sample such that the micro-focused x-ray beam hits the desired object. In this way the diffraction experiment is done locally on the individual object:

i) firstly, radial scans (θ-2θ) are done for each object; the position of the Bragg peaks give access to the lattice parameter of each of the 2 phases respectively;

ii) then, for each phase, the integrated intensity (rocking scans) give access to the relative quantity of each phase present in the investigated object;

iii) for both crystalline phases, the FWHM gives hints about the presence of defects (due eventually to the reorganization of the phases in the small object).

Figure 2 depicts the 3 results mentioned above, for an "as prepared" sample measured at RT. All the dots with the small dimension ≤ 2.25 µm (corresponding to the red bars in the lower part of the panel) show only the α crystalline phase[2] (we could have expected that some show the α, some the β phase). It is clear that the alignment parallel or perpendicular to the stripes is driving this effect, which seems to appear for sizes which are (by a factor ~3) above the expected stripes width of the continuous film. Local measurements with sub-micron beams near the edges of such small objects, coupled with finite element method (FEM) simulations and measurements at different temperatures[3] (continuation of this project), should allow to understand how the relaxation of the film is happening and why the α phase is preferred.

The shift of the Bragg position of the α peak with respect to the reference measured on the thin film (without lenses, $2\theta_{\alpha}$-film =47.974°) is reported in the central panel – it is always towards larger Bragg angle (smaller lattice parameter). The biggest shift (0.144°) seems to happen for the dot which has a rectangular shape with the long dimension perpendicular to the α/β stripes, *i.e.* it will contain a lot of these stripes on its length – the relaxation is then different. The biggest dot (12×12 µm) does also exhibit a shift (0.024°), suggesting that it is not fully relaxed. The smallest shift (0.004°, *i.e.* basically no shift) is exhibited by the dot which is elongated along the α/β stripes, and which contains only α-phase (see left panel). It seems that alone, in confined geometry, this phase can relax to the same value as in the continuous (reference) film.

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[2] Although the 2 smallest dots seem to show some traces of ~5% of α-phase, a detailed analysis shows that the signal identified as β-phase is in fact just background coming from the extended width of the α-phase peak.

[3] Some preliminary tests were performed during this measurement campaign: the sample was un-mounted, cooled down to ~ -5°C then re-measured at RT (only a few dots on the diagonal of the pattern, *i.e.* square sizes). The ratio α/β was found to be modified (increase of α quantity for the large dots).

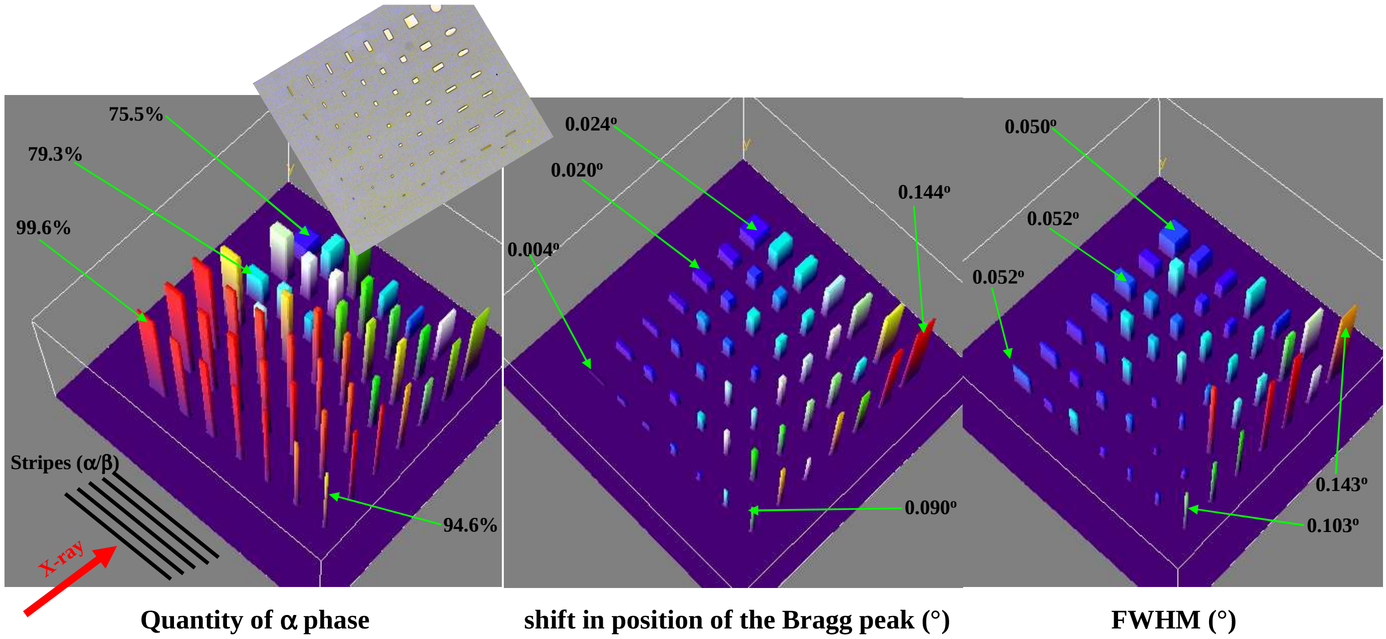

Figure 2: The case of the α phase: quantitative measure of the presence of the α phase (%), shift in the position of the Bragg peak with respect to the flat (non-patterned) film and FWHM for each of the objects. The values are shown as colored bars; their height and color are directly related (proportional) to the amplitude of the reported signal in each case. Some numerical values are shown as well. The geometry of the measurements (x-rays orientation with respect to the stripes) and a sketch of the patterned area are also shown.

The presence of the stripes (contact lines between α and β phase) is not found only to change the relaxation parameter in the α-phase, but is found to disturb the crystalline quality of each phase as well, which is of course expected to happen at the contact area. Again, a trend is easily seen, as an increased FWHM for the dots having the long dimension perpendicular to the stripes, and even larger if a confinement along the stripe is happening (if a slight increase for the smallest dot can be attributed to the poor signal/noise ratio, for the other dots, the trend is obvious).

Using the local x-ray diffraction for such system can quantify the presence of the different phases with the advantage to access non-destructively the crystallinity of the different phases in order to validate elasticity models predicting the properties of such systems at small scales.

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