



	<b>Experiment title:</b> Mechanical coupling in magnetoelectric composites	<b>Experiment number:</b> HS-4257
<b>Beamline:</b>	<b>Date of experiment:</b> from: 03.11.2010      to: 09.11.2010	<b>Date of report:</b> 01/03/2011
<b>Shifts:</b> 18	<b>Local contact(s):</b> Laurence Bouchenoire	<i>Received at ESRF:</i>
<b>Names and affiliations of applicants (* indicates experimentalists):</b> M. Abes*, M. Kasper*, S. Hrkac*, C. T. Koops*, O. M. Magnussen and B. M. Murphy* Institut für Experimentelle und Angewandte Physik (IEAP), Universität Kiel, Olshausenstr. 40, 24098 Kiel, Germany		

### Report:

In our experiments we studied the mechanical coupling at the interface of magnetoelectric (ME) composites by measuring the lattice deformation in the ZnO piezoelectric substrate, using the high-resolution and high intensity X-ray beam provided by BM28 at the ESRF. The ME samples were prepared in Kiel by sputter deposition of amorphous FeTb films of 100 nm thickness on the (001) surface of high quality ZnO single crystals. We investigated bulk reflections close to the interface, employing the scattering geometry shown in Figure 1a. A series of X-ray diffraction measurements on a pure ZnO substrate and a ZnO/FeTb sample at the (4 -4 0) specular and the (3 -3 1) forbidden reflections was collected, where the beam was impinging on the ZnO substrate at different distances  $d$  from the ZnO/FeTb interface or ZnO surface, respectively. In Figure 1b and c we present typical spectra collected in these experiments with a beam size in [001] direction of 10  $\mu\text{m}$  (for the (4 -4 0) peaks) or 50  $\mu\text{m}$  (for the low-intensity (3 -3 1) reflections). A Si (111) analyser was positioned between the detector and the sample in order to increase the resolution of the experiment. From the peak positions we determined the  $d_{4-40}$  interplanar spacing in the ZnO substrate and the corresponding strain  $\varepsilon_{1-10} = (d_{4-40} - d_{4-40}^0) / d_{4-40}^0$ , where  $d_{4-40}$  and  $d_{4-40}^0$  denote the interplanar spacing at different positions  $d$  of the beam relative to the interface and in the center of the ZnO sample, respectively. The results show that the ZnO substrate is under tensile epitaxial strain, reaching up to  $6 \cdot 10^{-3}$  at the ZnO/FeTb interface, and fully relaxes within the first 20  $\mu\text{m}$  from the interface (Figure 2a).

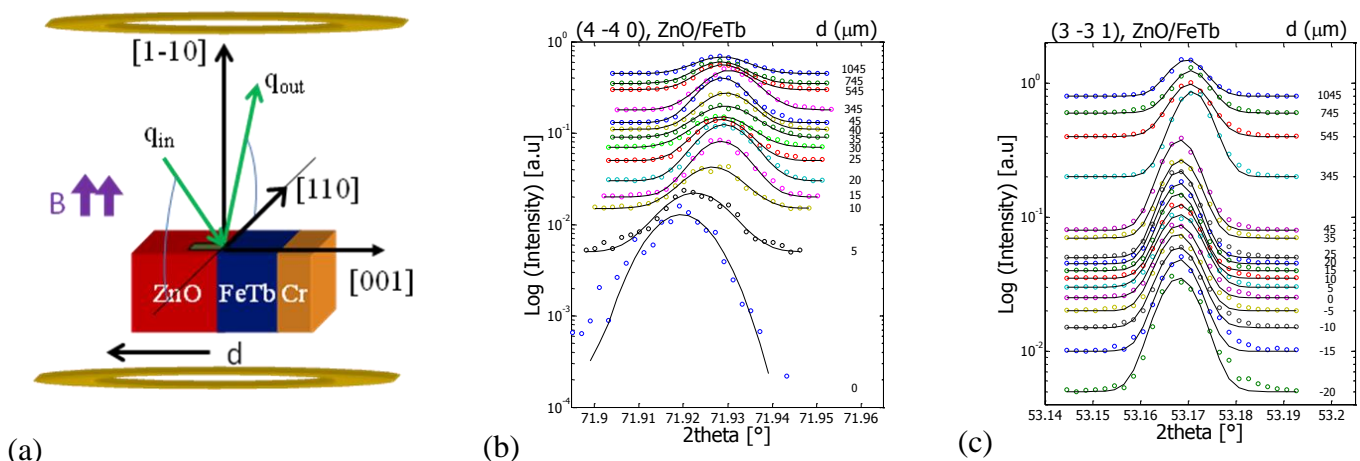


Figure 1: (a) Experimental geometry of the X-ray diffraction studies. (b) (4 -4 0) and (c) (3 -3 1) ZnO Bragg peaks, measured at different positions of the beam on the ZnO/FeTb sample (for clarity individual scans are offset with respect to each other).

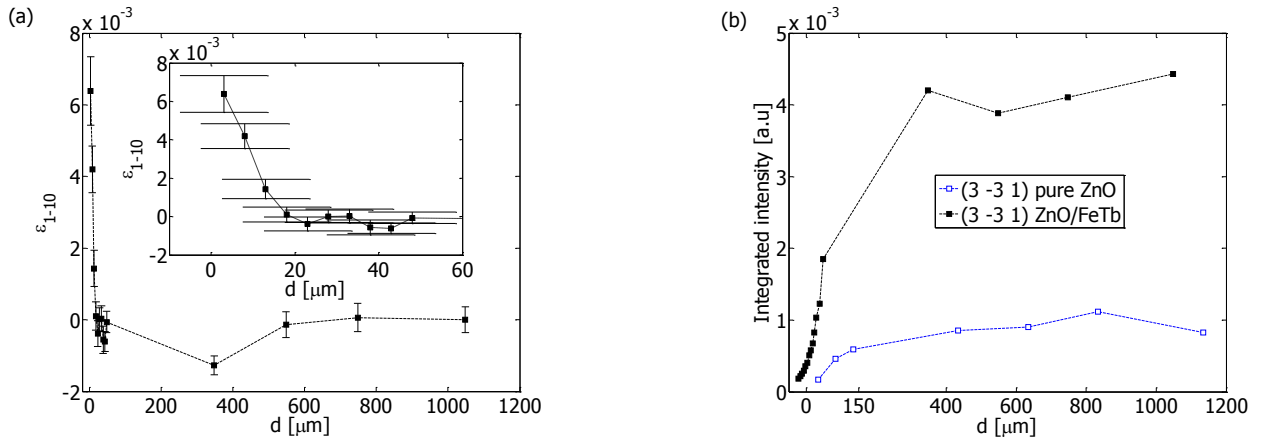


Figure 2: (a) Strain along the  $[1-10]$  direction and (b) integrated intensity of the  $(3 -3 1)$  forbidden reflection for ZnO/FeTb as a function of distance  $d$  to the ZnO/FeTb interface.

In addition, we performed similar measurements at the  $(3 -3 1)$  forbidden reflection for pure ZnO and ZnO/FeTb. Since the lattice deformation induced by interface strain removes the symmetry of the ZnO wurtzite structure, intensity is observed at the previously forbidden reflections, which is directly related to the magnitude of the strain in the sample. The results show that the integrated intensity measured on FeTb coated ZnO exceeds that of the pure ZnO by a factor of 4, confirming the presence of substantial epitaxial strain induced by the FeTb layer (the independence of the intensity on position  $d$  is probably caused by FeTb deposition on the sides of the sample). Further evidence of epitaxial strain is found in the rocking-curves of the  $(4 -4 0)$  and  $(3 -3 1)$  reflections, which exhibit peak splitting on the FeTb-coated samples.

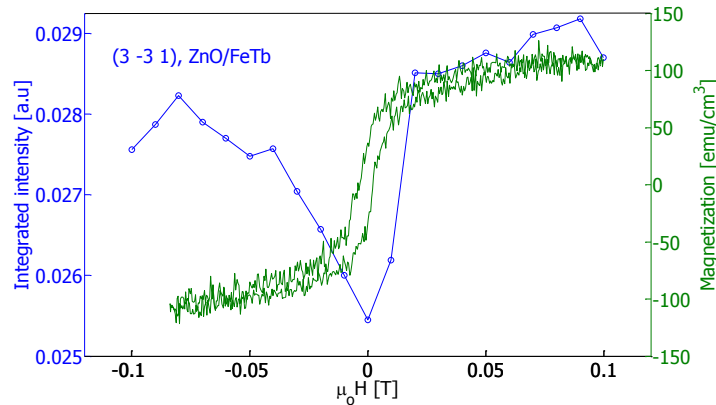


Figure 3: Intensity of the  $(3 -3 1)$  forbidden reflection at the ZnO/FeTb interface (blue) at different magnetic field strength and corresponding magnetization loop of the ZnO/FeTb sample (green).

To investigate the magnetoelectric coupling at the ZnO/FeTb interface, we also performed first X-ray diffraction scans close to the interface in the presence of an external magnetic field, which was provided by the 0.1 T magnet of BM28. The intensity of the  $(3 -3 1)$  forbidden reflection was found to increase with the increasing of the external magnetic field, reaching a nearly constant value at fields where the magnetization of the FeTb layer reached saturation (Figure 3). This observation gave first evidence that magnetic field induced strain at magnetoelectric interfaces can be directly measured by X-ray diffraction. Unfortunately, experimental problems related to the monochromator, which resulted in severe beam instability at the time of these measurements, prevented us from reproducing these results.

In conclusion, we succeeded in this first beam time in probing the strain profile at the interface of ZnO/TbFe magnetoelectric composites and obtained tentative results on the effects of the external magnetic field. In future work we plan to reproduce these promising results and study related systems by this method.

We acknowledge financial support by the Deutsche Forschungsgemeinschaft via SFB 855 ‘‘Magnetoelectric composites – future biomagnetic interfaces’’.