| ESRF | **Experiment title:** Large X-ray nano beam diffraction studies on complex magnetoelectric composites with defined microstructures | **Experiment number**: SI-2541 |
|---|---|---|
| **Beamline**: | **Date of experiment**: from: 02.03.2013 to: 05.03.2013 | **Date of report**: 05/03/2013 |
| **Shifts:** 9 | **Local contact(s)**: Manfred Burghammer | *Received at ESRF:* |

**Names and affiliations of applicants** (* indicates experimentalists):

S. B. Hrkac*****, M. Abes*****, 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:**

We investigated complex magnetoelectric (ME) microcomposites: piezoelectric ZnO microrods (width: 30 - 80 µm) coated with amorphous magnetostrictive $(Fe_{90}Co_{10})_{78}Si_{12}B_{10}$ (FeCoSiB, thickness: 500 nm). The coating was deposited in varied magnetic field directions to pre orientate the magnetic component. These were carried out in the presence of an external magnetic field by X-ray nanodiffraction using the transmission geometry as shown in Figure 1 at the ID13 beamline at an energy of 14.9 keV with a e beamsize of 150x150 nm². Using a Maxipix detector with a pixel size of 55 x 55 µm² at a distance of 2.6 m from the sample a very high q-resolution was obtained (resolution: $\Delta q = 2\cdot10^{-5}$). Additionally a Frelon 2D detector was used for SAXS and WAXS techniques. The external magnetic field was created by motorized permanent magnets and was applied perpendicular to the beam direction approximately parallel to the in-plane [100] direction with a 0.1 mT accurately. A series of X-ray diffraction measurements across the ZnO microrod at the ZnO (200) reflections were collected without and during the application of an external magnetic field. A separate uncoated ZnO sample was measured as a reference.

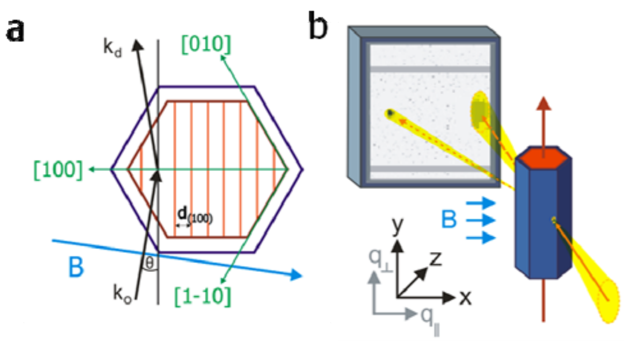


*Figure 1 a) Experimental geometry b) experimental setup in X-ray transmission diffraction*

Figure 2(a) shows the ZnO (200) Bragg reflection of a ZnO/FeCoSiB microcomposite with 80 µm diameter. The full width at half maximum of the (200) ZnO peak is about 0.04° demonstrating the very high crystalline quality of the ZnO substrate. The sample was scanned in 1µm steps in order to observe local strain behaviour. The reflection was measured at 5 µm from the edge of the samplealong the x-direction. The peak shifts show a dramatic dependence on the applied field. This is the first time, that the peak shift has ever been observed so clearly at this type of ME composite. The estimated strain induced

due to the applied B-field (20 mT) is $\epsilon_{100} = 5 \cdot 10^{-4}$. For the first time, due to the high intensity available at ID13 powder rings from the FeCoSiB (Fig. 2b) have been observed and even more interestingly SAXS is also clearly visible (Fig. 2b). Reference samples of pure ZnO show neither ring nor small angle reflection. confirming that the FeCoSiB is responsible. These data give us information about the structure in the amorphous FeCoSiB and will allow us to quantify the magnetic field induced shift and structural changes.

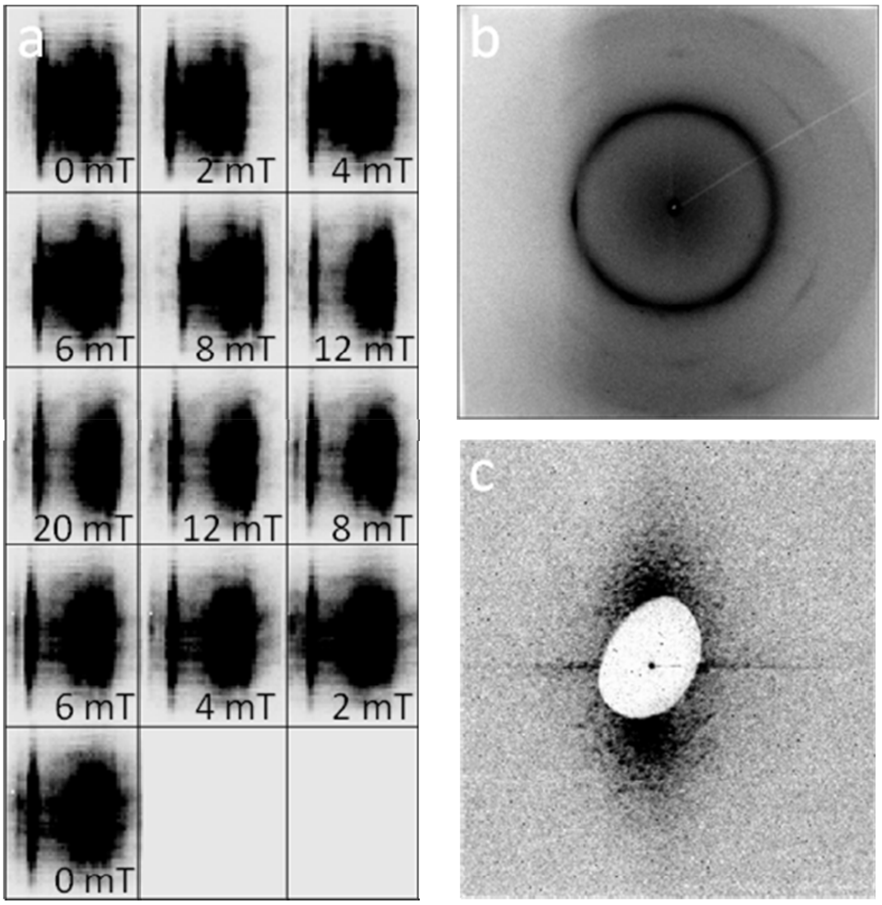


*Figure 2: (a) Image of ZnO (200) Bragg reflection inside different magnetic fields. A clear shift is visible. (b) Powder rings from FeCoSiB. (c) SAXS from FeCoSiB.*

In conclusion, we measured the magnetic field induced strain coupling in ZnO/FeCoSiB composites at ID13. A strong magnetic field dependence could be seen. The study also revealed powder diffraction and SAXS structures of the FeCoSiB giving us further understanding of the coupling in this magnetoelectric composit. This observation demonstrates that the local strain profile in micro-magnretoelectric microcomposits can be directly measured by nanofocus X-ray diffraction. The detailed analysis of the data will allow us to determine the magnetoelectric coupling factor in such composits containing crystalinne and amorphous components using the unique combination of high resolution nano-diffaraction nano-SAX and nano-WAX available on ID13.

In future work we plan to reproduce these promising results and perform systematic studies of related systems by this method.

We acknowledge financial support by the Deutsche Forschungsgemeinschaft via SFB 855 “Magne tolectric composites – future biomagnetic interfaces”.