ESRF	**Experiment title:** X-ray nano beam diffraction studies on complex magnetoelectric composites with defined microstructures	**Experiment number**: HC-1132
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Report:

Magnetoelectric (ME) composites, consisting of a piezoelectric and a magnetostrictive component, are of great interest for potential applications as highly sensitive magnetic sensors. Of central importance is the mechanical coupling at the interface of the two materials. A large ME response is only obtained if the lattice deformation induced by an external magnetic field in the magnetostrictive material can be transferred efficiently to the piezoelectric material.

We investigated complex ME microcomposites: Ni or Co filled pores (diameter/length: 200 nm / 270 µm) etched in single crystaline InP (Fig. 1a). In this experiment we wanted to see the influence of these magnetostrictive pores within the InP single crystal and how the InP is influenced indirectly by external magnetic fields applied along and perpendicular to the pore direction.

In the experiment the pores within the InP were aligned along the z direction. On applying a magnetic field the magnetostrictive (MS) component (Ni or Co) undergoes a change in lattice parameter. Our aim is to observe the degree of strain transferred to the InP surrounding the pores. Understanding this strain transfer is the goal of this experiment. To this end, Nanofocus X-ray transmission scanning diffraction was performed at the nanofocus endstation EH3 at ID13. The beamsize as measured with a gold wire was 180 x 140 nm^2 at an energy of 14.9 keV. Two external magnetic field directions were realized by motorized permanent magnets. The horizontal field (0-100±0.1 mT) was perpendicular to both the c-axis and the beam direction; the vertical field (0-70±0.1 mT) was parallel to the c-axis and perpendicular to the beam direction.

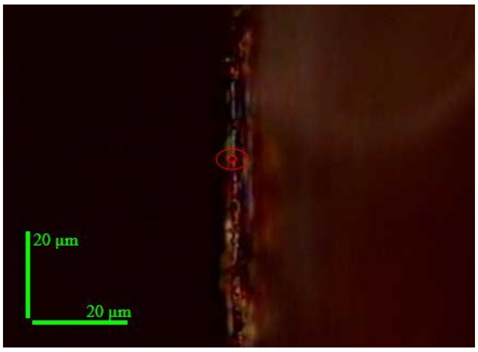
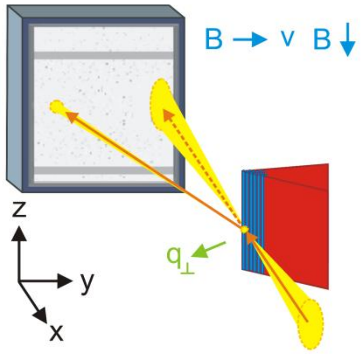


Figure 1 a) Microscope image of InP/Co. The pores are visible. The red eye shows the position of the beam.



b) Diffraction geometry. The pores are oriented in Z direction. The magnetic field can be applied to Z or Y direction.

The experimental geometry is shown in Fig. 1b. A Frelon 2D detector at 9.8 cm was used for initial alignment. Using a Maxipix detector with a pixel size of 55 x 55 µm² at a distance of 3.2 m from the sample a very high q-resolution ($\Delta q = 2 \cdot 10^{-4}$) was achieved. X-ray transmission diffraction measurements were performed in a series of line scans to map the InP sample at the InP (400) Bragg reflection (2Theta = 32.37°). Each line scan was repeated for every magnetic field increment in both horizontal and vertical field geometries. This procedure then has been repeated for different Theta values. It was necessary to perform these rocking scan as the lattice constant varies with applied magnetic field.

a)

b)

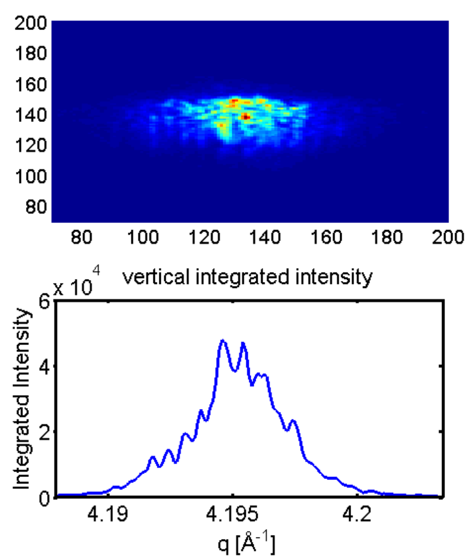


Figure 2 a) InP/Co, Maxipix image of InP (400) Bragg reflection. b) Integrated intensity of the InP (400) reflection from a). The corresponding position on the sample is 2 µm away from the edge.

A cross section of the InP (400) Bragg peak taken from the Maxipix image (Fig. 2a) is shown in Fig. 2b. As the peak has an unusual form due to coherence effects and overlapping InP domains, we follow the peak shift by observing the peak center of mass (COM). It represents the lattice changes within the illuminated area of the sample. Fig. 2b) shows the integrated intensity of the InP (400) Bragg reflection of the InP/Co microcomposite for a single point from the diffraction map. The COM is 4.1952 Å^{-1}, its full width at half maximum: 0.003 Å^{-1}.

First, we applied a magnetic field parallel and then in a second map perpendicular to the pore orientation for each sample. The initial results for the InP/Co sample are shown in this report. In Fig. 3a we present the COM profile of the InP (400) reflection along a line from bulk to edge at 0 mT. Fig. 3b shows the strain distribution in the sample when the magnetic field is applied. The lower line shows the strain distribution when the field is applied parallel to the pores in the InP/Co sample. There is hardly any change in strain in response to the applied field. As the magnetic domain are already oriented parallel to the long axis at 0 B field. This result is in good agreement with our predictions. One expects that the magnetic field would have a minimal influence on the magnetic domains in this case. In strong contrast a large influence of the horizontal magnetic field on the strain induced in InP is observed (Fig. 3b, upper line). This can be explained by the fact that the magnetic domains have to rotate by 90° to follow the applied field direction. We observe a change in the strain form compressive to tensile at different positions within that line profile. The local strain profiles, in the presence of an applied magnetic field, for two specific positions (y=124, y=126) are presented in Figure 4. In a) and c) we see the influence on the local lattice structure within the parallel field. The local strain fluctuates around $(0.1 \pm 2)\ 10^{-5}$. In contrast, when the perpendicular field is applied, as seen in b) and d), there is a striking effect, with the strain reaching a maximal value of $(8 \pm 1) \cdot 10^{-5}$ at 100 mT.

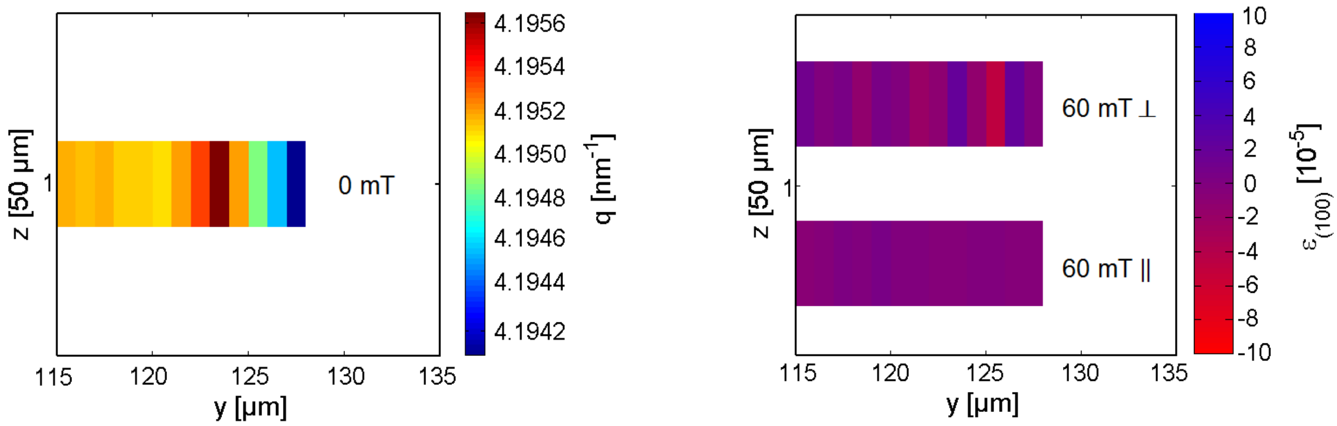


Figure 3a) COM profile of InP (400) across the InP/Co at 0 mT. The image is flipped compared to the microscope image

b) Local strain distribution at 60 mT. Both lines correspond to the line profile of a). Magnetic field is applied perpendicular (⊥, upper line) and parallel (||, bottom line) to the pore direction.

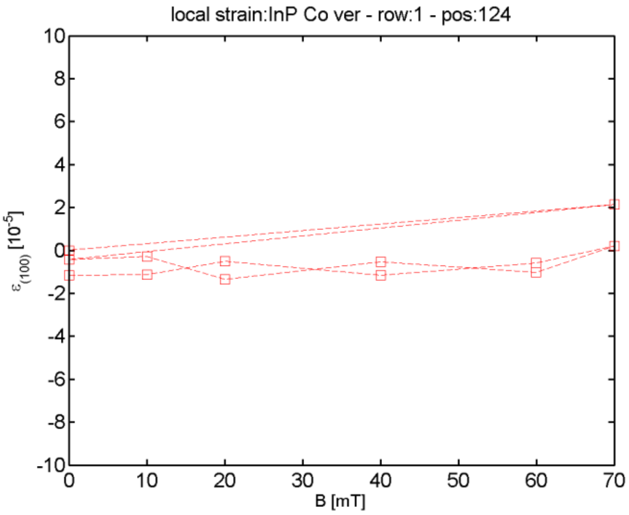
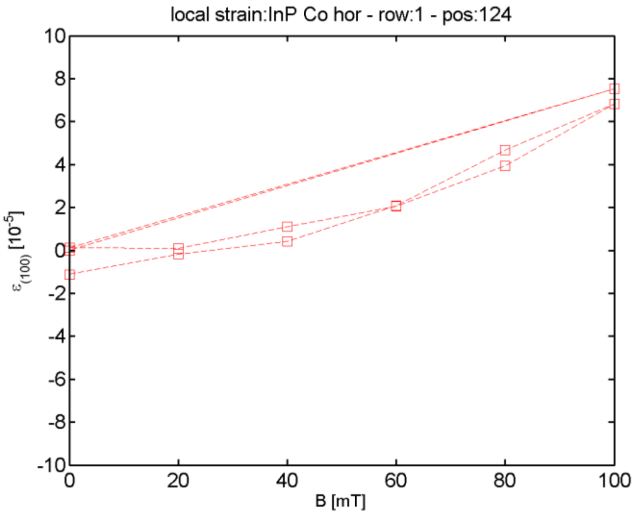
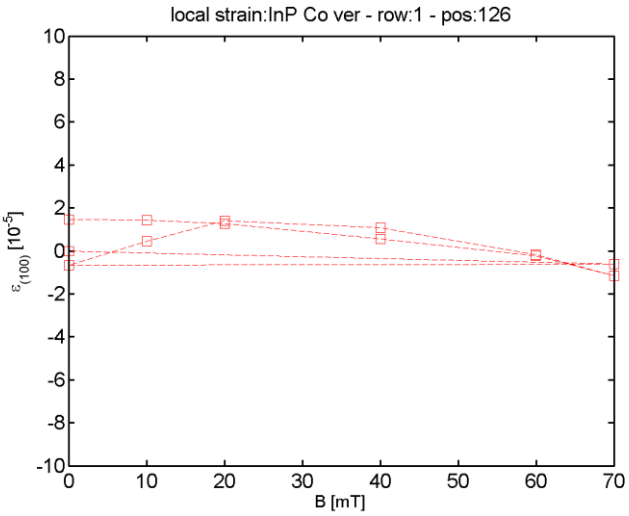


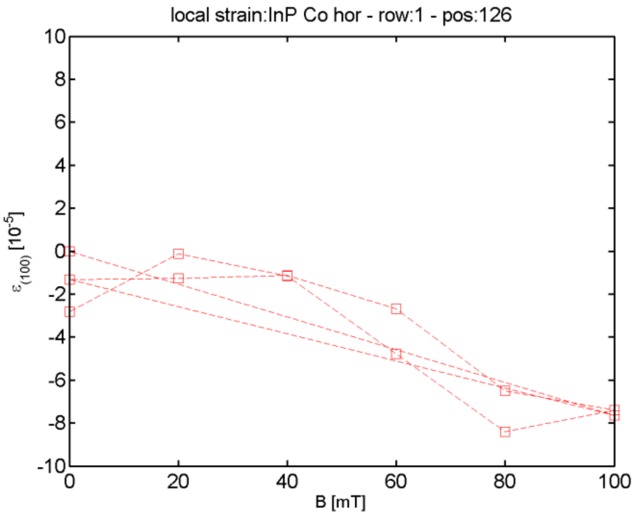
Figure 4a) local strain at a single position (124) in the InP as a function of applied parallel magnetic field (||)



b) local strain at a single position (124) in the InP as a function of applied perpendicular magnetic field (⊥)



c) local strain at a single position (126) in the InP as a function of applied parallel magnetic field(||)



d) local strain at a single position (126) in the InP as a function of applied perpendicular magnetic field (⊥)

In summary, we have shown that the magnetic domain orientation plays a major role in the strain behaviour of ME microcomposits. The strain effects observed are one order of magnitude when the magnetic field is applied in the direction perpendicular to the sample magnetic domain orientation without magnetic field. As the experiment ended 3 days ago, the analysis is still ongoing.

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