ESRF

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Three dimensional imaging of dopant atoms in non perfect crystals with kinematical X-ray standing waves	HS-3481

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Report:

The aim of our experiment was to investigate the structure of epitaxially grown thin films using the novel kinematical X-ray standing waves (KXSW) method in symmetrical Bragg reflection and grazing incidence diffraction geometry. As samples we used (001) oriented Cr doped GaN films grown on a sapphire substrate and multiferroic $BiFeO_3$ films grown on a $SrTiO_3$ substrate. To perform KXSW measurements on $BiFeO_3$, it is necessary that only a single domain is illuminated. We intended to achieve this by polarizing the film by applying an electric field. Unfortunately this was not possible due to technical problems during sample preparation.

The GaN samples were produced by MOVPE (metal organic chemical vapor phase epitaxy). They consist of a sapphire substrate on which an undoped GaN buffer layer is grown. On top of this layer a 400 nm thick Cr doped layer was deposited. GaN has wurtzite structure and the position of the Cr dopant atoms is unknown so far. Due to the low crystal quality conventional XSW measurements are cumbersome and require special techniques like normal incidence XSW (NIXSW) or KXSW. Since thin films are to be investigated, also the influence of the substrate must be taken into account. To overcome this problem, i. e. to reduce the influence of the substrate, we used the KXSW method in grazing incidence geometry.

In the second part we planned to measure the secondary signal within a large solid angle similar to multiple energy x-ray holography (MEXH). For the measurement we used an improved version of the high speed diffractometer, which we tested already in an earlier beamtime (HS-2937).

Results:
In the first part of the experiment, we measured the (110) Bragg reflection at 6.5 keV in order to determine the in plane component of the Cr atom position. This was repeated at different angles of incidence to gain depth resolution. As secondary signal we recorded the Cr K fluorescence. The measured secondary yield curves showed only a small variation around the Bragg angle, which is an evidence of a low coherent fraction. At larger deviations from the Bragg angle some curves showed a strong dependence of the Cr fluorescence on the azimuthal angle. Sometimes even a jump in the secondary signal was observed. An explanation for this could be a non uniform Cr concentration or a large amount of disordered Cr, which is arbitrarily distributed on the surface.
Since it turned out that the GaN samples were not suitable for a XSW measurement, we decided to use a different sample for the second part and measured the Ti K fluorescence of a rutile (TiO_2) single crystal instead. With the improved setup we could increase the speed of the measurement and we also removed some mechanical problems, which lead to spurious ripple structure on the data in the last experiment. Fig. 1 shows a preliminary analyzed dataset (90° by 54°, Ti K fluorescence signal measured with a single channel analyzer). In fact the raw data consists of many 360° azimuthal scans, in order to increase the image quality we made use of the four fold symmetry of rutile.

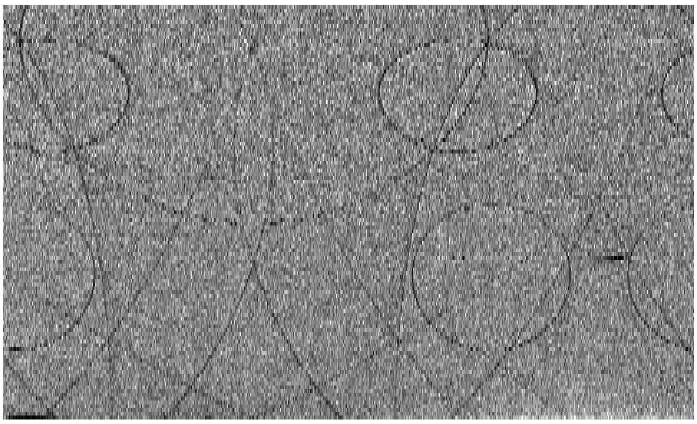

Figure 1: Ti fluorescence measured in a 90° by 54° solid angle region

In comparison to our previous experiment (HS-2937) the quality of the data is much better and also a broader angular range could be covered. This means, that the improved setup, which uses a better azimuthal rotation stage and an improved data taking software, is very well suited for this type of measurements.
Prospects:
During our beam time we have for the first time applied the KXSW method in grazing incidence geometry. Unfortunately we experienced different problems related to the preparation of the samples. In the second part of the experiment, we could measure for the first time a large solid angle region with sufficient high resolution for KXSW evaluation. A thorough data analysis is ongoing.
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