ESRF	**Experiment title:** Imaging dynamics of plastic relaxation of InGaN films under thermal annealing.	**Experiment number**: MA-5373
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Names and affiliations of applicants (* indicates experimentalists):

Carsten RICHTER (Leibniz Institute for Crystal Growth)*

Julita SMALC-KOZIOROWSKA (Institute of High Pressure Physics PAS WARSAW)*

Tobias SCHULZ (Leibniz Institute for Crystal Growth)*

Joanna MONETA (Institute of High Pressure Physics PAS WARSAW)*

Robert KERNKE (Leibniz Institute for Crystal Growth)*

Report:

In this experiment we studied the plastic relaxation that occurs at the (In,Ga)N/GaN interface. We study films with an indium concentration of nominally 20% and thickness in the range of 40-60 nm. These films are fully strained (no misfit dislocations observed), but plastic relaxation and thus dislocation nucleation and glide may be triggered through thermal annealing of these samples.

We followed these thermally activated dislocation kinetics in real-time by full field X-ray diffraction microscopy (FFXDM) at ID01 during *in-situ* annealing. To increase the time-resolution, we used a coplanar grazing incidence geometry and optimized the beam shaping optics (see Fig. 1). To achieve this, we selected the 013 reflection of GaN and tuned the X-ray energy to approx. 7.6 keV leading to an incidence angle close to the critical angle of GaN (0.3°). The exact incidence angle could be fine-tuned by changing the X-ray energy in a small range or by selecting the direction of the sample miscut which was 0.5 °. This way, we could switch between the film and substrate peaks, tune the penetration depth and the footprint size. The scattering angle (2θ) for this reflection and this energy was around 65.4°, which represents a compromise of low image distortion (minimal at 90°) and high elastic scattering intensity (proportional to cos 2θ). An image of the diffracting regions of the samples was obtained by an objective lens combined with a CCT camera at a large distance of ~6 m.

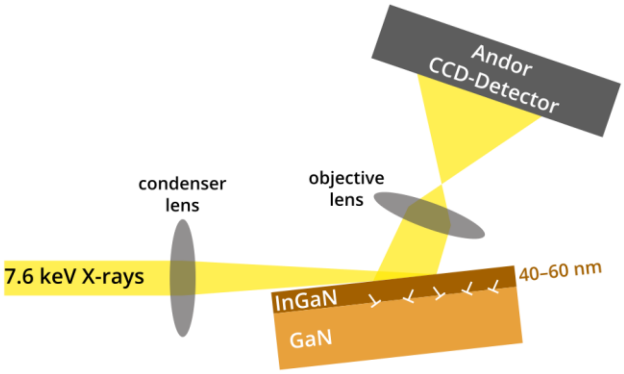

Fig. 1: Concept of grazing incidence FFXDM. Dislocations at the interface are indicaed by a ⊥ symbol.

The field of view of FFXDM at this conditions is approximately 100 μm in both vertical and horizontal directions. To assure that most of the incident flux was used we matched the footprint size of the incident beam on the sample surface to this field of view. This was achieved by pre-focusing the incident beam to a spot size of 5 μm × 100 μm in the horizontal and vertical direction, respectively. These conditions give us a diffraction contrast image of a 100 μm × 100 μm region in within ~2 s of exposure time. We can choose either substrate or film peak sind the dislocations at the interface cause strain in both lattices. The small angle of incidence provides sensitivity to surface-near regions. The ideal imaging conditions have been obtained when incidence angle was detuned ~ 0.015° from the substrate peak, which leads to maxima of intensity from strained regions close to dislocation lines (weak beam contrast).

We annealed 4 samples with $In_{0.20}Ga_{0.80}N$ layers of various thickness and deposited on substrates with different threading dislocation density (TDD). The first annealed sample with 60 nm thick $In_{0.20}Ga_{0.80}N$ layer deposited on GaN bulk substrate with TDD in the range of $10^6 cm^{-2}$ already exhibited a quite high density of misfit dislocations. We performed several annealing steps with a duration of 30 minutes in the temperature range from 300 - 600 °C, but we did not observe formation of any new dislocations. Consecutively, we studied a sample with thinner 50 nm layer deposited on the same kind of a GaN bulk substrate with TDD in the range of $10^6 cm^{-2}$ and we choose for the observation an area with low initial density of misfit (a+c) dislocations (Fig. 2a).

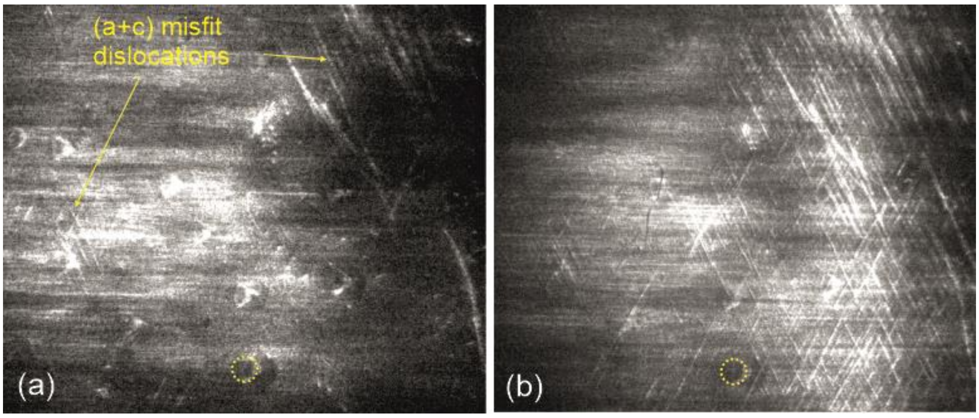

Fig. 2: *FFXDM images of the structure with a 50 nm $In_{0.20}Ga_{0.80}N$ layer deposited on a GaN substrate with $10^6 cm^{-2}$ TDD. (a) region of interest with low initial density of (a+c) misfit dislocations, (b) the same area after consecutive annealing at the temperatures in the range 350-650 °C. The yellow circle marks the same defect in both images, which was caused by the presence of an In droplet at the surface during the growth of the InGaN layer.*

Fig. 2 exemplarily shows the evolution of the misfit dislocation network due to annealing. During the annealing of the following sample with 40 nm layer deposited on a bulk GaN substrate with TDD in the range of $10^4 cm^{-2}$, we were able to observe nucleation of single dislocations and their glide through the crystal. Important question of our experiment were about dislocations glide velocities and mechanisms of 120° turns of dislocations, which we could successfully observe. Fig. 3 shows selected images of a moving dislocation acquired during annealing at 420 °C. The dislocation is stopped for several seconds along its way several times. Finally, at some point it changes its propagation direction. Since it is not possible for 1/3{11-23} dislocations to cross-glide to another {11-22} plane, such a change of the propagation direction must involve a reaction with another dislocation. In-situ studies of moving 1/3{11-23} dislocations in nitrides were never carried out before, and such observations were done here for the first time.

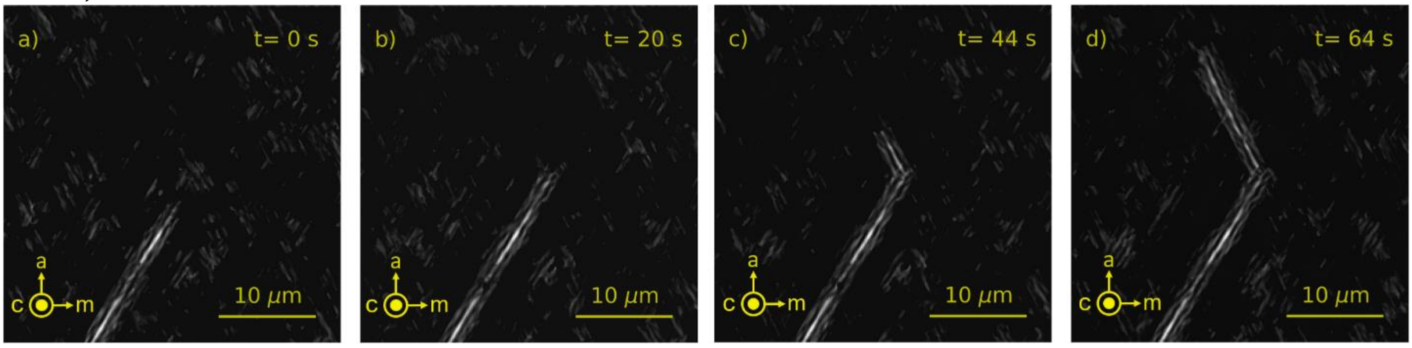

Fig. 3: Images (a) to (d) are filtered FFXDM images at a sample temperature of 420 °C showing in-situ glide and the change of line direction from (b) to (c). The images are processed to enhance the visibility of the MD lines.

The results also provide new insight into strain relaxation of epitaxial nitride layers and merit publication. In the future, this may open ways to produce highly relaxed InGaN buffers as substrates for growing layers with In content >30%, which is one of the major challenges in the development of efficient optoelectronic devices.