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Experiment title: "X-ray strain analysis of periodically
patterned GaAs nanorods VLS- grown by vapour-
liquid-gas mode onto GaAs[111B]''

Experiment number:

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Report: The aim of this proposal was to measure periodic nanorod (NR) arrays grown by seed-free MOVPE growth through a pre-patternd SiN_x mask. The samples have been grown by Hendrick Paetzelt for Solid Chemistry department of Leipzig University. In particular we have measured GaAs Nanorods [NR] grown on GaAs[111]B and on Ge[111]. Whereas homoepitaxial seed-free VLS growth is known from literature, expitaxy onto Ge[111] was new. Patterning of the SiN_x mask with lateral spacing of 1 μ m < D < 5 μ m was performed within an area of 600 x 600 μ m². The NR array was visible by bare eye and could be precharacterized by high-resolution X-ray diffraction in home laboratory. Fig.1 show a SEM pictures of a GaAs NR sample grown on GaAs[111]B substrate and the respective GaAs(111) reciprocal space map. The hexagonal shape of NR and the hexagonal arrangement of the pattern are clearly visible in the SEM image. The HRXRD map shows periodic side peaks along q_x-direction with distance Δ q_x = 2 π /D. Unexpected we found a second row with same period at Δ q_z /q_z = -2,6 10⁻³ with respect to GaAs(111) which could not be identified so far.

The experiment at ID1 was performed using the nanofocus setup defined by compound refractive lenses focusing the incident beam to a spot size of about 500x1500 nm² which was sufficiently small in order to separate individual NR's with D>3µm in coplanar symmetric scattering geometry. The use of quasiforbidden 222 reflection revealed most promising because its intensity varies strongly with changing defect density. In order to identify the patterned area we screened the whole sample at fixed angle position of about 1 degree off the exact Bragg peak position. The strength of diffuse scattering varied by more then a factor of 5 inside and outside the patterned area. In addition, we found a change in diffuse scattering by factor 2 at a position exactly on a NR compared to a position in between. This characteristic change of intensity allowed us to scan a limited area of the patterned sample and identify individual nanorods. The mapping for a GaAs NR sample grown on GaAs[111] is shown in Fig.2a. One clearly can identify regularly patterned scattering maxima which we have associated with certain NR positions.

Next, reciprocal space maps in the vicinity of the (222) reflection have been recorded at selected positions inside and outside the patterned area. Two examples are shown in Fig.2b, one taken outside the patterned area (left), the other at a certain NR's position (right), showing an additional peak along the vertical crystal truncation rod. This strain feature is visible at the NR position only and is probably related to a strain field imposed by the epitaxy process, The crossed streaks are not related to the sample and are originating from the experimental setup (Monochromater and PSD, respectively).

In order clarify the origin of the strain feature we measure a GaAs NR pattern grown on Ge[111]. Because the Ge(222) reflection is forbidden, substrate intensity is very low. Nevertheless, we found this reflection easily caused by the defects within the substrate material or due to the influence of strain between SiN_x mask and the substrate. Therefore, we were able to identify the patterned area by same procedure as described above. Here the gain in diffuse scattering was factor of 10 inside and outside the area. Because of the non-polar

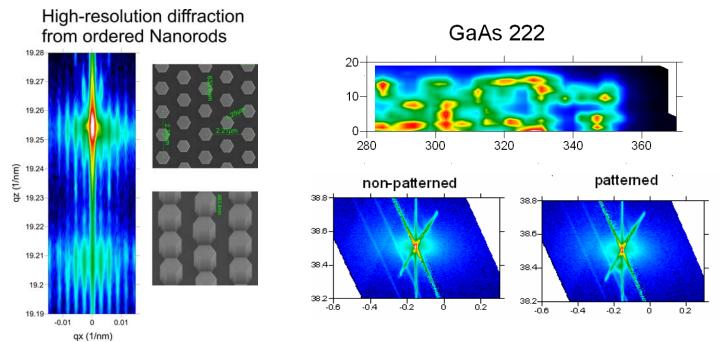


Figure 1 HRXRD RSM taken in vicinity of 111(GaAs) (left), the SEM pictures display the regular hexagonal shape of NRs. **Figure 2**: Intensity pattern of diffuse scattering recorded inside the NR pattern. (top). RSMs taken outside and inside the patterned area.

surface of the substrate the NRs did grow non uniform. The SEM picture shown in Fig.3 shows hexagonally and trigonally shaped NRs grown on regular positions. RSMs taken at different positions inside the NR patteren displayed different shape information. Because of the use of a PSD we could record 2-dimensional slices through the Fourier transform of a certain NR. The measuremnts (fig. 4) show side peaks differently spaced in q_x measuring different NRs.

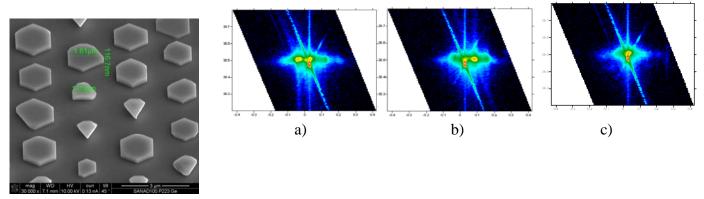


Figure 3 SEM picture of GaAs NRs grown seed-free onto Ge[111] substrate

Figure 4: RSMs taken at different positions within the NR pattern. The vertical axis is q_z , the horizontal Δq_x both given in \mathring{A}^{-1} . a) – coresponding to hexagonal rod; b) – tetragonal rod; c) – poor substrate

Out of these RSMs we could identify both types NR shapes by the different Δq_x distance measured at $q_z = 30.5 \text{ Å}^{-1}$. In addition, all RSMs do not display the strain feature found in the sample grown on GaAs substrate. Therefore we suggest that the strain field is caused by alloying of SiN_x with GaAs substrate in the beginning of NR growth.

Our experimens provides the following important results:

- 1. The nanofocus setup provided at ID1 is sufficient to select individual NRs of a patterned NR array.
- 2. By recording RSMs of individual NR one is able to obtain selective information of shape and strain. In next experiment we plan to use the method to investigate the shape variation of NR as function of the NR position within the patterened area and to analyse the interplay between neighboured Nrs during VLS growth.

References

- [1] H. Peatzelt et al. J. Cryst. Growth (2008) in press;
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