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Report:

We performed x-ray diffraction (XRD) experiments in order to retrieve the composition and strain distribution of small ensembles or even single semiconductor quantum dots (QDs). The technique of obtaining the QD strain and composition distributions from reciprocal space maps has been established for large QD ensembles (typically 10^5 to 10^6 QDs), where good statistical averages of the properties are obtained. On the other hand, considerable progress has been made in focusing hard x-rays (10 keV energy range) to sub-μm beam sizes. We show here the first attempt to combine both approaches, i.e., use a very small beam to perform a ***diffraction*** experiment on a limited number of QDs.

Originally we intended to use for focusing a pair of linear crossed zone plates, which has been demonstrated to achieve focus sizes in the range of 100 nm. Preliminary experiments on ID01 showed that vibrations of the monochromator, the stage carrying the focusing element, and the sample stage will in any case limit the achievable focus size to a few μm. We thus used circular Fresnel zone plates. The resulting beam profile consisted of a central focal spot of about $3.5\times5\ \mu m^2$ (vertical × horizontal), surrounded by a "halo" (higher order focusing) roughly 20 μm in diameter, which contained about 10% of the x-ray flux on the sample. More details can be found in the submitted reports for BLC-2175 and IH-HC 766 (June 2005), reports added for the MI-744 experiment.

As the scattered intensity scales with scattering volume squared, we used large pyramid-shaped semiconductor QDs (1-3.2 μm base size, 0.5-1 μm height), and 2-10 μm laterally spaced (samples supplied by M. Schmidbauer). The substrate is Si(100). These QDs serve as a model to prove the feasibility of scattering experiments from individual islands, and of identifying a particular island in a diffraction experiment, in order to investigate **the same** island at

different Bragg reflections, and to correlate the XRD results with those from other locally resolving methods.

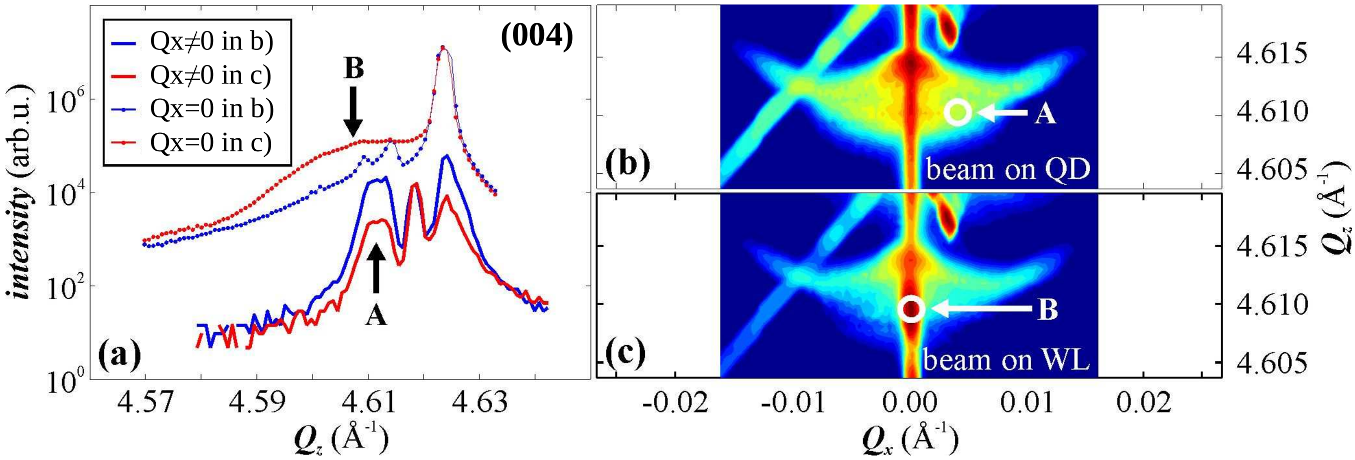

Fig. 1: (a) radial scans along the coherent truncation rod (Q_x=0) and in the diffuse scattering position (Q_x≠0). Beam on an island (blue curves) and on the wetting layer (WL) (red curves). **(b)** reciprocal space map (RSM) around (004) Bragg reflection with beam on a QD. **(c)** the same RSM with the beam on the WL.

The samples were mounted on the diffractometer and pre-aligned in the center or rotation (where the focused beam was) using an optical microscope (islands are large enough to be observed; in future experiments, markers could also be used). Radial scans and reciprocal space maps - (004) and (511) reflections - for different lateral sample positions were recorded (Fig. 1). To fine-tune the beam position on the sample, we used two different ***Q***-values in reciprocal space [Fig. 1(b,c)]: position B is on the coherent truncation rod, which is strong if mostly the pseudomorphic wetting layer is illuminated, while position A is aside the truncation rod in the diffuse scattering, which is strong if mostly an elastically relaxed island is illuminated. This reversed contrast is very well highlighted in Fig. 2

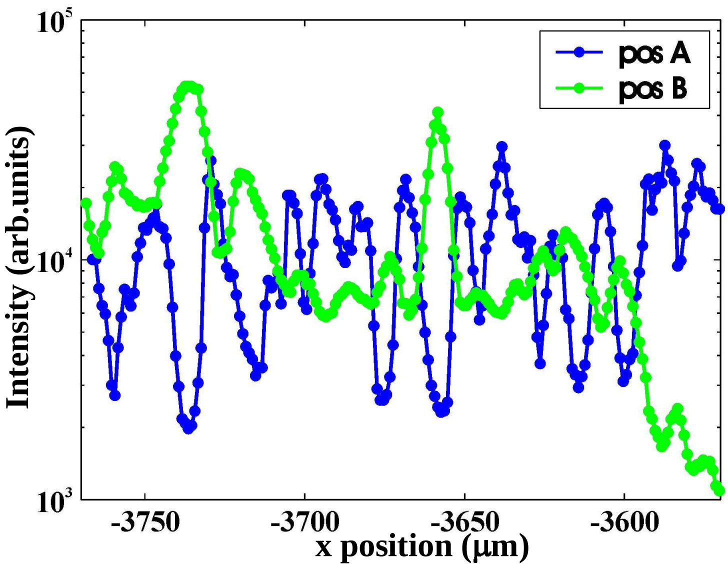

Fig. 2: Line scans with the momentum transfer fixed at different reciprocal space positions: pos.A (Q_x≠0: sensitive to QDs, blue) and pos.B (Q_x=0: sensitive to WL, green) as indicated in Fig. 1(b,c). Note the perfectly reversed contrast.

Thus, it is possible to measure the spatial island distribution (Fig. 3). We can clearly identify individual islands in the x-ray scattering pattern. For the indicated islands, we recorded symmetrical (around (004) Bragg peak position) and asymmetrical (around (511) Bragg peak position) RSMs in order to obtain the chemical

composition and strain profile. Reference RSMs were also measured in a spot where only the WL was illuminated (labeled as “EMPTY”, see also fig.1c).

In all, 3 different samples (pyramid sizes of 3.2, 1.8 and 1 μm) were mapped and measured, at- and near-QD positions, in symmetric and asymmetric conditions.

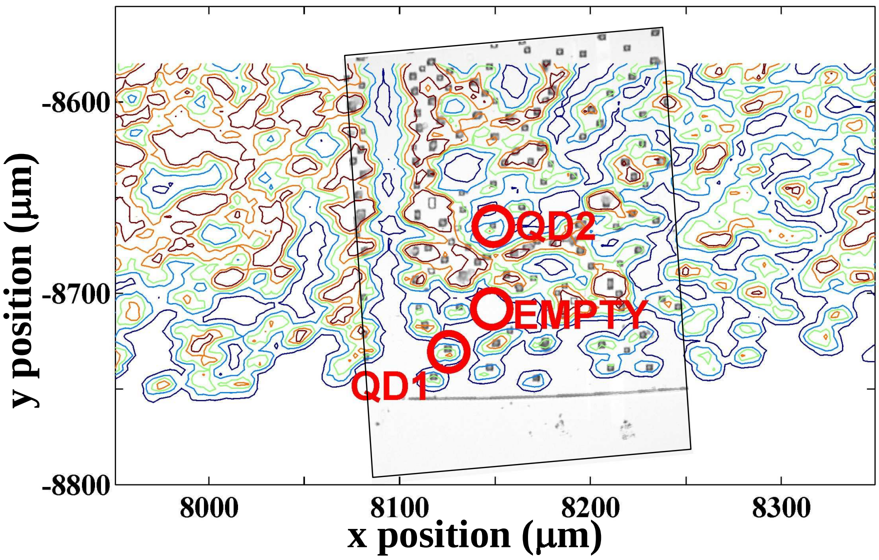

Fig. 3: Optical microscope image of the 3.2 μm-islands sample surface (inset, in gray) overlaid with the spatial intensity distribution (colored contour lines) measured by translating the sample at fixed momentum transfer in position A as indicated in Fig. 1(b). Some positions (rather well separated QDs as well as an area “EMPTY” with only WL) are indicated, where RSMs have been recorded.

In summary, we have demonstrated that:

- x-ray diffraction on single semiconductor QDs is feasible and results in a count rate of about 10^4-10^5 cps at the Bragg peak for the larger-size islands
- it is possible to identify, analyse and compare individual islands. A repetition of reciprocal space maps (after ~1 day) showed a long-term stability of the setup (drifts of the sample smaller than 2 μm), in spite the fact that we used conventional stepping-motor driven stages. Note also the relatively fast procedure to pre-align and then identify areas of interest in samples.

Our experiment also identifies several improvements that have to be made in the future:

- the most important improvement is expected by a reduction of the focused size of the spot. This can be done either by using different focusing optics, but definitely the most important aspect is the reduction of vibrations in the monchromator and goniometer setup (see also the above mentioned setup reports). For the goniometer, we suggest to build a stage as compact as possible, carrying both the focussing element and the sample, so that mutual vibrations are minimized. The design has to enable the goniometer movements of the sample relative to the focussing element in order to perform diffraction experiments, and must minimize the distance between focussing elements (collimating/cleaning slits) and sample.