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## Indium segregation effects during GaAs cap-layer growth on InAs-quantum dots monitored by reflectance anisotropy spectroscopy

E. Steimetz, T. Wehnert, T. Trepk, J.-T. Zettler and *W. Richter*  
Institut für Festkörperphysik, TU Berlin, Germany

**Abstract.** Performing Reflectance Anisotropy Spectroscopy (RAS) measurements during GaAs cap-layer growth on InAs quantum dots (QD) Indium segregation effects were monitored in-situ. Segregation during growth was found to be enhanced for elevated growth temperatures (775 K) and low cap-layer growth rates. A later intermixing of the islands with the GaAs cap-layer was observed during post-growth thermal annealing. For thin caps (10 nm) the RAS spectra became more and more InAs-like ( $2\times 4$ -like) while for 20 nm GaAs-cap-layers a smoothening of the surface but no indium related structure was found.

### Introduction

The real-time monitoring of quantum dot (QD) formation in Stranski-Krastanov growth mode by Reflectance Anisotropy Spectroscopy (RAS) in MOVPE allows the determination of the growth mode transition from 2D to 3D-growth and the later development of uncovered InAs islands [1]. The effect of surface segregation during GaAs cap-layer growth on top of the islands and later intermixing of the islands with the surrounding material are the topics of this study. Temperature dependent indium segregation effects during GaAs cap-layer growth and post-growth annealing have been monitored already for MBE growth of InGaAs-quantum wells [2], [3] and via TEM also for InAs-QDs [4]. STM studies of uncovered InGaAs islands showed that the top of the islands contains only InAs [5]. For postgrowth thermal annealing of covered InGaAs-QDs a strong blue-shift in Photoluminescence was observed, indicating an intermixing of the InGaAs layer with the surrounding GaAs [6] The suppression of indium segregation during cap-layer growth and subsequent intermixing are the remaining challenges for a routinely InAs-QD-based laser production, where elevated temperatures for the growth of the aluminium containing mirrors are necessary. We investigated the influence of different growth conditions (temperature, growth rate and total pressure) on segregation and intermixing with the GaAs cap during MOVPE growth by optical in-situ measurements. Post-growth annealing of samples with different cap-layer thickness, which showed no segregation effects during growth, allowed us to determine the minimum cap-layer thickness for a complete coverage of the islands.

### 1 Experiment

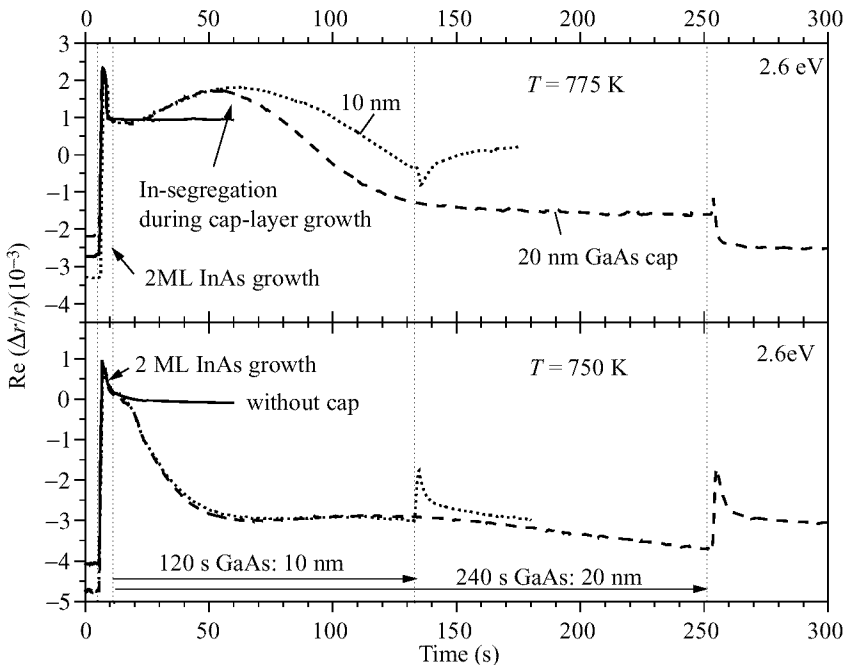
All measurements were performed in a low-pressure, horizontal MOVPE reactor. The RAS set-up [7] was attached to the MOVPE reactor via a purged, low-strain quartz window on top of the reactor. We were able to obtain spectra for photon energies ranging from 1.5 eV to 5.5 eV and to perform time-resolved measurements with a resolution better than 1 s. The shortest time for measuring one spectrum at a reasonable signal-to-noise ratio was 5 min.

## 2 In-segregation effects during cap-layer growth

In a first series of experiments the whole deposition sequence (2ML InAs/ GaAs, 5 s growth interruption and GaAs-cap-layer growth) is monitored at the As-dimer related energy of 2.6 eV. This was done for different growth temperatures. The transient measurements for 750 K and 775 K are given in Fig. 1.

Immediately after starting the InAs-deposition the reconstruction changes from  $c(4\times 4)$  for GaAs via  $(1\times 3)$  towards  $(2\times 4)$  for InAs and this causes a strong signal change in RAS [8]. When the growth mode transition takes place, the RAS-signal reduces, caused by a thickness reduction of the 2-dimensional InAs-wetting-layer. The islands themselves do not have any influence on the RAS-signal as long as they are small and isotropic. The overgrowth of the InAs islands with GaAs should lead to an As-rich  $c(4\times 4)$  reconstructed surface again, causing a reduction of the RAS-signal. For the lower growth temperature of 750 K this is really the case, but for  $T = 775$  K a strong increase of the RAS signal with the onset of GaAs overgrowth was found. Since at 2.6 eV RAS is sensitive to the As-coverage of the surface [9] and the thickness of the growing In(Ga)As [10], this increase can be interpreted by a more In-rich conditions during GaAs-growth due to In-segregation.

The influence of the cap-layer thickness on the development of the surface after stopping the GaAs deposition is also shown in Fig. 1. Transients were taken for a nominal deposition of 10 nm and a 20 nm GaAs. The growth rate was determined by RAS oscillations on GaAs before. At 775 K a GaAs layer of nominally 10 nm is

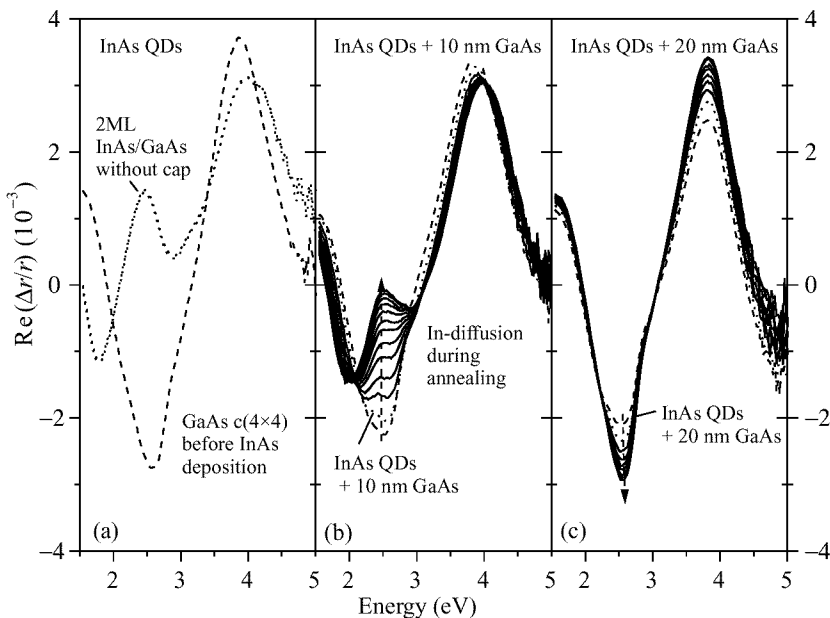


**Fig 1.** Time resolved RAS measurements during InAs-QD deposition and cap-layer growth for different growth temperatures.

obviously not sufficient to overgrow the islanded surface. After stopping the deposition the RAS transient shows an increase again, indicating that the surface still contains islanded material that is now rearranging at the surface. Ex-situ AFM measurements still showed islands at the surface for a 10 nm thick GaAs cap-layer. For the 20 nm deposition at 775 K this effect in RAS was not found, indicating that 20 nm are sufficient to cover the islands completely. In AFM a smooth surface was observed. The different slope in the RAS transients during GaAs overgrowth at 775 K might be attributed to a slightly reduced GaAs growth rate in the 10 nm experiment or might be caused by a different roughness of the initial GaAs-surface leading to a different island density. For the lower growth temperature of 750 K, no In-segregation effects during cap-layer growth were monitored. In this case even 10 nm of GaAs seem to be enough to cover the surface. But the starting RAS level for GaAs is not reached again even for 20 nm GaAs. This might be caused by a very rough surface after the whole deposition process due to the low growth temperature for GaAs. In AFM islands up to 6 nm height were found.

### 3 Post growth annealing of InAs-QDs with different cap-layers

In the second experiment InAs QDs were annealed after growth as long a changes of the surface stoichiometry, indicated by changes in the RAS signal around 2.6 eV, were found. Two monolayers InAs deposited at 750 K (no segregation during growth) were covered by GaAs at 750 K. The samples were prepared with varying cap-layer thickness (10 nm and 20 nm). In Fig. 2 the results of the annealing procedure after growth at deposition temperature are given. Fig. 2a shows the spectra before and after QD deposition with no GaAs cap-layer for reference. The InAs QD spectrum is typical



**Fig 2.** RAS spectra of uncovered, partly covered and completely covered InAs QDs and their evolution during annealing at 750 K.

for an InAs deposition slightly above the critical layer thickness. If there were clusters at the surface they would cause an additional structure in the high energy range of the RAS spectra [1]. For a QD deposition covered by 10 nm GaAs the RAS spectra after deposition show a  $c(4\times 4)$  reconstructed surface again, but the minimum at 2.6 eV is not as steep as it was before QD growth (see also Fig. 2a). Taking spectra every 5 minutes after the deposition a continuous change of the surface towards a more In-rich  $2\times 4$ -like reconstructed surface was found. STM investigations of uncovered InGaAs islands have shown that the topmost layer of flat islands is containing only InAs [5]. We believe that the In-content in the uppermost layers is increasing during annealing due to segregation of indium atoms from the only partly covered islands to the top. For a GaAs cap of 20 nm this effect could not be observed. In contrast, here the surface seems to become smoother by annealing, indicated by a sharpening of the minimum at 2.6 eV, typical for a well ordered  $c(4\times 4)$  reconstruction. AFM measurements indeed showed a smooth surface.

#### 4 Summary

By RAS measurements during GaAs cap-layer growth on InAs quantum dots (QD) indium segregation effects were observed in real-time. Segregation during growth was found to be enhanced for elevated growth temperatures. Post-growth annealing of samples at growth temperature resulted in an intermixing of the islands with the cap-layer material. The effect of intermixing on the surface stoichiometry is strongly dependent on the cap-layer thickness. For thin caps (10 nm) the RAS spectra became more and more InAs-like, indicating an In enrichment of the surface. For 20 nm GaAs cap-layers no indium related structure could be observed and annealing led to a smoothening of the  $c(4\times 4)$  GaAs surface.

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