# A new approach to AFM investigation of buried Al/In<sub>x</sub>Ga<sub>1-x</sub>As/GaAs interfaces and quantum dots

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We performed an investigation of InAs self-assembled quantum dots (QD) and  $In_xGa_{1-x}As/GaAs$  heterostructures with Al caps by means of Auger depth profiling, XRD and AFM in combination with the preliminary selective chemical etching. We observed that properties of surface and buried QD are identical and did not detect existance of wetting layer in samples with QD. It was found for the first time that intensive interdiffusion of In and Ga resulted in 1.6 nm gradient layer at the base of QD.

#### 1. Introduction

Atomic force microscopy (AFM) is known as an informative method for investigation of quantum dots arranged on semiconductor surfaces. QD array buried under a semiconductor or a metal layer may alter their properties. The initial lattice strain may be partly relaxed and previously formed QD may be partly dissolved. In this work a new approach recently proposed in [1-3] is developed for buried QD investigation by AFM. It is based on a preliminary removal of the upper layer by a selective chemical etch which does not attack the bottom layer – etching is stopped at interface with monolayer uncertainty. The results of the QD investigation of Al/In<sub>x</sub>Ga<sub>1-x</sub>As/GaAs structures by means of AFM combined with XRD and Auger depth profiling are presented.

#### 2. Experimental

We investigated Al/In<sub>x</sub>Ga<sub>1-x</sub>As/GaAs structures prepared by metal organic chemical vapour deposition (MOCVD). The In<sub>x</sub>Ga<sub>1-x</sub>As layer or the array of QD on the surface of GaAs are buried under an aluminium layer in the *in-situ* growth process. The thickness of Al layer was about 100 nm. The indium concentration determined by X-ray diffraction (XRD) is shown in Table 1. In samples N577 and N578 the InGaAs layer had a thickness nearly 3 nm. These samples were used as test structures to define the details of a selective chemical etching process with a control of the results. Instead of the InAs layers in the samples N579, 618a,b QD arrays were formed. The buffer GaAs layer and QD layer in the samples N618a and 618b were grown at identical growth conditions, but the Al cap layer in the sample N618b was absent. The AFM investigation was performed with the SPM MDT "Solver". For Auger depth profiling and XRD investigations we used the ESO-3 spectrometer and DRON-4, respectively.

### A. <u>Selective chemical etching</u>

Fig.1a shows the AFM image of the Al surface in sample N578. This image is typical for all samples under investigation. The height of its relief reaches 50 nm. According to XRD data, the Al layer grown by MOCVD was (111)- textured grains. Fig.1b shows the results of Auger depth profiling of the sample N578. The chemical etching of the Al layer was performed in a 0.5% solution of KOH at the room temperature. Fig.2a shows the AFM image of the InGaAs surface in sample N578 after the etching of Al. As we can see, the relief is radically altered from Fig.1a and roughness decreases to value below 2 nm, which is usual for AFM images of epitaxial films in the air ambient. It is important

that a surface relief of etched structures was not altered after additional over etching time equals 10 minutes. It proves that Al etching had a selective character. Besides, an indirect evidence of this follows from the results of Auger depth profiling of sample N578 shown in Figs.1b and 2b. Depth profiling which was started from the Al layer shows gradual Al/GaAs junction up to 50 nm width. This value is about the roughness of the initial Al surface. The In Auger peak is absent here due to the low depth resolution as a result of the initial Al surface roughness. Auger depth profiling of sample N578 with a preliminary removed Al layer gives absolutely different results. Fig.2b shows that the InGaAs/GaAs heterojunction width was below 1.5- 1.7 nm, which is determined by the fundamental limitations of the Auger depth profiling method. The Al Auger peak was not observed on the In<sub>x</sub>Ga<sub>1-x</sub>As surface independently on "x":  $0 \le x \le 1$  (samples N577, 578, 579 and 580). In our opinion, these data conveniently show that the method of selective chemical etching does not alter the relief of internal Al/In<sub>x</sub>Ga<sub>1-x</sub>As interface with any value of "x".

### B. Analysis of InAs QD

Fig.3a shows the AFM image of sample N579 with a removed Al layer. It allows us to analyse the main properties of the InAs QD array. They are typical for the used MOCVD growth parameters. For example, the mean height is about 5-7 nm. In order to investigate the presence of an In<sub>x</sub>Ga<sub>1-x</sub>As wetting layer on the surface of InAs QD array, we used the method of Auger depth profiling. Fig.3b shows the In distribution in sample N579 with the removed Al layer. It has a linear decreasing with depth of the In contents without any stepped behaviour near its maximum. It proves the absence of a thin wetting layer on the surface of GaAs. The value of the In profile width in Fig.3b is near 10 nm, which is in a good agreement with the AFM data (Fig.3a). Figs.4a and 4b show the AFM image of sample N618a with the removed Al layer and N618b, where the Al layer was not grown. The QD arrays in these samples are similar. It proves that growth of an Al layer on the surface of QD layer didn't alter the properties of QD array.

The absence of thin wetting layer on the surface of QD layer is confirmed by the results of additional selective chemical etching of sample N579 with the removed Al layer in concentrated HCl solution at 75°C during 2 minutes. The AFM image of the resulting surface is shown in Fig.5a. The HCl solution removes the semiconductor  $In_xGa_{1-x}As$  with a large value  $x \approx 1$ . The layer with a low value of "x" was not etched. Some part of the sample N579 was capped by the photoresist, then it was etched in HCl. The step along the boundary in Fig.5a is not clearly defined, which also indicates the absence of a wetting InAs layer. Fig.5b shows the AFM image of sample N579 after the etching of InAs QD in HCl in more detail. The height histogram demonstrates that after the QD etching their plane bases still remained at the GaAs surface with a constant height of about 1.6 nm. In our opinion, it may be connected with a diffusion or segregation of the Ga atoms into the InAs QD during their growth.

#### 3. Conclusion

We performed an investigation of InAs self-assembled quantum dots (QD) and  $In_xGa_{1-x}As/GaAs$  heterostructures with Al caps by means of Auger depth profiling, XRD and AFM in combination with the preliminary selective chemical etching. We observed that properties of QD arrays placed on the surface and buried under an Al layer are identical and did not detect existance of wetting layer in samples with QD. It was found for the first time that a gradient  $In_xGa_{1-x}As$  layer with a thickness near to 1.6 nm exists in the base of InAs QD. An occurrence of a gradient layer is connected with intensive interdiffusion of In and Ga between QD and GaAs underlayer.

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# References

- [1] R.Rettig, W.Stolz, Physica E, 2 (1998) 277.
- [2] Georg Bernatz, Siegfried Nau, Rasmus Rettig, Heinz Jansch, and Wolfgang Stolz, J. Appl. Phys., 86 (1999) 6752.
- [3] Georg Bernatz, Siegfried Nau, Rasmus Rettig, and Wolfgang Stolz, J. Electron. Mater., 29 (2000) 129.

Ν	Х
577	0.28
578	0.38
579	1
580	0
618a,b	1

Table 1.



Figure 1. (a) AFM image and (b) Auger depth profile of the sample N578



Figure 2. (a) AFM image and (b) Auger depth profile of the sample N578 with a removed Al layer.



Figure 3. (a) AFM image and (b) Auger depth profile of the sample N579 with a removed Al layer.



Figure 4. (a) AFM image of the sample N618a with removed Al layer; (b) AFM image of the sample N618b, where Al layer was not grown initially.





Figure 5. (a) AFM image of the sample N579 etched in HCl (the left part of the structure was closed by photoresist); (b) AFM image of the sample N579 etched in HCl (in more detail).