Cross-sectional AFM of GaAs-based multilayer

heterostructure with thin AlAs marks

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Introduction

Atomic force microscope (AFM) is widely used nowadays for study of the surface morphology of semiconductor structures. AFM is known as a simple device operating in air. Though the surface is oxidized, some significant features remain unaltered, for example, *GaAs*-monolayer steps are clearly distinguishable on a vicinal surface of *GaAs/InGaAs/GaAs* structure [1]. On the other hand, many elements of multilayer structure, such as layer thickness or interface quality, are hidden under the surface. A large set of alternative methods can also be utilized for heterostructure analysis. However, each of them has restricted field of application being more complex than AFM in air.

In this work we report the experimental results on AFM analysis of cleavage of multilayer *GaAs*-based heterostructure. Additional thin *AlAs* layer was introduced as a mark in every heterointerface between the main (thick) layers during the growth process in order to visualize the boundary.

AlAs is expected to be a suitable mark layer for GaAs-based structure. First, the lattice parameter of AlAs is very close to that of GaAs. Mark layer has therefore an epitaxial pseudomorphic growth mode, as well as the next layer grown over the mark. Second, natural oxidation is used as essential technological step. As previously reported in [2], oxidated AlGaAs layer on the cleavage of AlGaAs/GaAs structure is higher than neighboring GaAs layers. Hence, the boundary is seen as a wall in the cross-sectional AFM image. Finally, the sample preparation procedure is simpler in comparison with other techniques. An atomically flat cleavage surface of A^3B^5 crystal perpendicular to (001) crystal plane is due to a perfect (110) cleavage plane in the zinc-blend type crystals.

Two examples are presented below.

1. Layer thickness and growth rate measurements

Thickness is one of fundamental parameters of layer, but its value is difficult to measure precisely. Following example demonstrates advantage of the proposed method in solving this problem with a Solver-P4 atomic-force microscope (NT-MDT, Russia).

The structure S1 (see, Fig.1) consists of 16 *GaAs* layers grown on the *GaAs*(001) substrate by metal organic chemical vapor deposition (MOCVD) in a horizontal type low pressure reactor EPIQUIP VP-50RP. The precursors were trimethylgallium (TMG), arsine (AsH₃) and dimethylethylaminalan (DMEAA); hydrogen was a carrier gas. The growth temperature was 600°C for first 3 layers (growth time 4, 2 and 1 min, respectively). Then, 3 layers were grown at 550°C (4, 2 and 1 min), 2 layers - at 525°C (4 and 8 min) and 2 layers - at 500°C (4 and 8 min). Next 5 GaAs layers (600°C, 1 min) are included as a part of periodic (*AlAs/GaAs*) multilayer which serves as an inner thickness standard in the analyzed structure. The period value was accurately measured by x-ray diffraction in advance (DRON-4 diffractometer, CuK_{a1} radiation), that gives corrections to the cross-sectional AFM image scale. The topmost GaAs layer is a cap layer. 16 thin *AlAs* layers ("mark layers") were introduced during the epitaxial growth successively after each GaAs layer.

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The thin *AlAs* layers marking the interfaces on the cleavage are seen in Fig.1 as strips.







Fig.2. Dependence of GaAs growth rate on the growth temperature.

The thickness (h_{GaAs}) of the *GaAs* layer is

$$h_{GaAs} = D - h_{AlAs}$$

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where D is a distance between two neighbouring AlAs marks, and h_{AlAs} is a thickness of the mark. Each distance was measured in 5 different sections. When the scan step was equal to 2.5 nm mean-root-square error of D is around 2 nm because of sharp *AlAs* wall. The layer thickness and GaAs growth rates were obtained by the following procedure: the intermark distance dependence on the GaAs growth time was linear approximated for each growth temperature. The AlAs mark thickness is equal to the D value at the GaAs growth time t=0. The slope angle of this line gives the *GaAs* growth rate. The mean-root-square error about 1 nm/min was found. The GaAs growth rate vs. reciprocal absolute temperature dependence is plotted in Fig.2.

A consistent, general pattern for GaAs growth rate with TMG and AsH₂ sources vs. temperature has been reported in many studies, see, [3] for review. These results show that an examination of growth rate vs. temperature allows a general categorization of the process limiting the growth rate as either mass transport, surface kinetics, or thermodynamics. If the chemical reaction rates limit the growth rate (kinetically limited case) the growth rate increases with increasing temperature. As seen in Fig.2, this is a case for low-temperature region from 500 to 550°C in our experiment. The last point (600°C) deviates from the line indicating the beginning of the mass transport limited region.

This example shows that cross-sectional AFM of the MOCVD structure with *AlAs* mark layers enables us to determine the layer thickness and the growth rate dependence on temperature with high accuracy.

2. Analysis of interface planarity

The structure S2 (see, Fig.3) consists of 14 GaAs layers, 2 InGaAs layers and 16 thin AlAs marks. Distinctive feature of the growth process was an absence of GaAs buffer layer at the beginning of the growth.

The thin *AlAs* layers marking the growth front on the cleavage are seen in Fig.3 as white strips. The breakdown of the epitaxial growth took place at the very beginning of the process: thickness of few first layers dramatically varies. This implies a presence of some local impurities which remained on the growth surface. Epitaxy was suppressed on these local positions. Therefore the front becomes straight only after long overgrowth. The topmost



<u>Fig.3.</u> AFM image of the cleaved structure S2. The arrow indicates the growth direction.

InGaAs layer with overcritical thickness causes the formation of misfit dislocations; and the front undergoes a new distortion.

This example shows how cross-sectional AFM of the MOCVD structure with *AlAs* mark layers can be applied to visualization of the growth front evolution within one growth experiment.

Results

- The cross-sectional AFM of multilayer GaAs-based heterostructures with AlAs mark layers provide useful information on the growth front evolution and on the defects at interfaces.
- ➤ In the case the growth fronts are flat, the layer thickness and the growth rate can be determined from the AFM image with high accuracy.

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