AFM study of dry etched cleavages of $Al_xGa_{1-x}As$/GaAs heterostructures

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The cleavages of the planar $Al_XGa_{1-X}As$/GaAs multilayer structures underwent dry etching with various set-ups. Obtained arrays were then studied by means of atomic force microscopy (AFM).

Introduction. Dry processing techniques, such as reactive ion etching (RIE) or planar plasma etching (PPE) can be successfully applied to the patterning of III-V semiconductor surfaces [1-3]. Almost ever the masking is compulsory, that in turn implies the sophisticated treatment of an overlay and use of auxiliary materials [1,2,4].

The ion etching of $Al_XGa_{1-X}As$/GaAs heterostructures in CCl_2F_2 containing chemistries is well studied during last two decades [1-8]. The effects of the plasma composition, the operating pressure and the self-bias voltage were described in sufficient detail [6-8]; the etch-stop reactions and the processes occurring upon exposure to air are examined as well [5]. Under certain conditions, the selectivity $>10^3$ is reported for x=0.4 [7]. Even if x as low as 0.13, the selectivity and etch rate are retained above 10^2 and 7 nm/min, respectively [8]. Addition of an inert gas (He) is effective for removing of the contaminated layer, enhancement of surface chemical reactions and providing of ion assisted anisotropic etch of GaAs with high rates and vertical sidewalls [6]. On the other hand, the better selectivity and less sputtering of $Al_XGa_{1-X}As$ are achieved by isotropic etching in the gas mixtures enriched with the CCl_2F_2 agent [7]. A minor rule of photomask was assumed from the substantial difference in the etch rates of components [6].

As known [9], the accuracy of AFM measurements on the sub-micron level depends essentially on the availability of suitable calibration standard. Therefore, fabrication of the test structures is an important task, where both inorganic and organic materials may serve as precursors [10]. In the former case, the low-dimensional calibration array are formed using recent progress in VLSI technology [11].

In this work, a simple technique, which allows to form the nano-sized microstrip structure without mask is described. The prepared arrays may be utilized as an article for the AFM tip characterization or as a template for the fabrication other microstructures [12].

Experimental. The sample was grown by means of metallorganic vapor phase epitaxy (MOVPE) in a planar type reactor. The process pressure was 50 mbar at the temperature 650°C. Trimethylgallium, trimethylamine alane and arsine were sources of Ga, Al and As, correspondingly; the carrier gas was hydrogen. Such type of heterostructures was assigned to electrical measurements. The details of the sample composition and conducting properties are described elsewhere [13].

Then, the sample was investigated by the X-ray diffraction with DRON-4 diffractometer. The overall period was found approximately at 53 nm with 20% fluctuation along the sample surface. The latter was caused by some inhomogeneity of the growth process in the reactor.

The depth profiling and quantitative elemental analysis were carried out by means of Auger electron spectroscopy (AES) in the ESO-3 apparatus [13]. The AES data showed the entire period (***p***), that is the sum of thickness of $Al_XGa_{1-X}As$ and GaAs layers, about 60 nm. There were totally 15 periods in array upper the buffer GaAs layer. The x-value was equal to 0.6. Besides, the $Al_XGa_{1-X}As$ layers are usually somewhat narrower than neighboring GaAs layers.

Dry etching was carried out in the conventional Secon XPL-26 semiautomatic installation. The process parameters were as follows: gas flow composition CCl_2F_2 (18 sccm) / He (72 sccm) or CCl_2F_2 (44 sccm) / He (44 sccm), operating pressure – 350 mbar, thermostat setting – 85±5°C, rf power – 500 W. Two etching modes were used: the bottom electrode is rf-driven, whereas top electrode is grounded for RIE; and the bottom electrode is grounded, top electrode is powered for the PPE. In both cases the sample was located on the bottom electrode. Prior to the carrying in of the gas mixture, the chamber was pumped down to the vacuum below 10 mbar and kept half an hour for degassing. Originally, the process conditions corresponded

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to the previously described in works [6,7]. Further, they altered depending on the subproducts.

The AFM measurements were carried out in the SOLVER P-4 (NT-MDT) microscope equipped with the standard silicon cantilever operating in the tapping mode. Electron micrographs were obtained in the JEM 2000EXII (JEOL) microscope at 200 keV.

Results and discussion. In Fig.1a the surface of the fresh cleavage is shown. The air-induced oxidation of Al-containing layer gives a lateral array with contrast of 1.5 nm in Z-direction (see, also, Fig.2a). As follows from Fig.1,2a, the double distance between the top of oxidized $Al_XGa_{1-X}As$ layer (clear strips) and the floor of GaAs layers (dark strips), *i.e.*, ***p***, corresponds to 65±5 nm. This is in a reasonable accordance with the data obtained by other techniques (see, Experimental). The $Al_XGa_{1-X}As$/GaAs thickness ratio appears to be far higher than that measured by AES. It may be explained by the mushroom-like cross-section of oxidized layer.

RIE with various regimes modifies the cleavage surface – Fig.1b,c. The short time exposure results in formation of narrow troughs, whose depth approaches 8 nm by AFM (Fig.2b). At this step, the actual parameters of etched cleavage were additionally controlled by electron microscopy – Fig.3a. Comparing Fig.1b and 3a, one can derive that there is no remarkable distinction between the surface profile taken with these two methods. As expected, an increase of etching time increases the vertical contrast of the array for all regimes used (Fig.1b,c and Fig.2b,c), which is accompanied by a change in the trough profile (see, below).

According to [7], use of equal gas proportion will lead to a more isotropic etch, whereas the mixture lacking active etchant produces a vertical profile. Our experiments yield the contrary results. The surface etched in plasma with higher He content is apparently more coarse and irregular (Fig.1c). Moreover, no significant progress in the trough formation was detected (Fig.2c). The cross-sections of the etched samples in both regimes are waved rather than rectangular. One of the reasons consists in possible reciprocal diffusion atoms and nonabrupt change of composition x at interfaces.

Next, an effect of PPE with 1/1 gas ratio was examined. In Fig.1d the images of $Al_xGa_{1-x}As$/GaAs cleavages exposed to plasma for 20,40 and 80 s are presented. The fall of height between $Al_XGa_{1-X}As$ and GaAs layers, *i.e.*, the depth of trough, becomes 2.8, 6.3 and 10 times greater, respectively.

The extended reaction time results not only in the development of array, but also in the change of its profile . At the shortest etching time, small intermediate bars are getting seen onto the GaAs layer (Fig.1,2d). Their visualization means that the AFM tip has reached the bottom of the trough without sidewall restrictions. The reason why this sub-array arises is not yet clear.

Two competitive processes run in etched sample: deepening of the GaAs troughs and overgrowth of $Al_XGa_{1-X}As$ strips. Consequently, prolonged etching will not facilitate the developing of the array.

Figs.1-2 display the higher quality of PPE etched cleavages in comparison with RIE. Typical for RIE fine sidewalls were not revealed here. Moreover, in contrast to the commonly observed trend [1-3], anisotropic chemical etching is more effective than RIE of self-biased sample. Both mismatches may be due to the absence of thick mask layer. An influence of the critical dimensions cannot be also ruled out. It is worth noticing, that we did not succeed in getting such arrays on the similar $Al_XGa_{1-X}As$/GaAs heterostructure with smaller period, which experienced the same treatment.

A difference between the AFM images and electronic micrographs of the 80 s etched cleavage is caught by bringing into confrontation Fig.1d and Fig.3b. The depth of the trough measured by AFM does not exceed 13 nm. It is fairly less than in the cross-section micrograph (Fig.3b), where average trough depth is 18 nm. Furthermore, the sidewall profile in Fig.3b is close to a sine, whilst in the AFM scans it is rather parabolic (Fig.2d) .

The above said let us to believe that the lower curve in Fig.1d represents a convolution of the AFM tip shape on the etched $Al_XGa_{1-X}As$/GaAs cleavage. More work on the quantification of results is in progress.

Conclusion. In fact, fabrication of the AFM tip grading based on III-V compounds with precisely shaped pattern is a complicated multi-step procedure [1-12]. An ideal (rectangular or triangle) alignment on the atomic scale remains very desirable. Alternative objects (*e.g.*, polystherene spheres, large biomolecules) with well-known geometry can be tested instead [10-14]. The selection is made by taking into account several important features, such as quality of a standard, its accessibility, simple technology, durability and cost basis. An attempt to combine these features is demonstrated in the present study.

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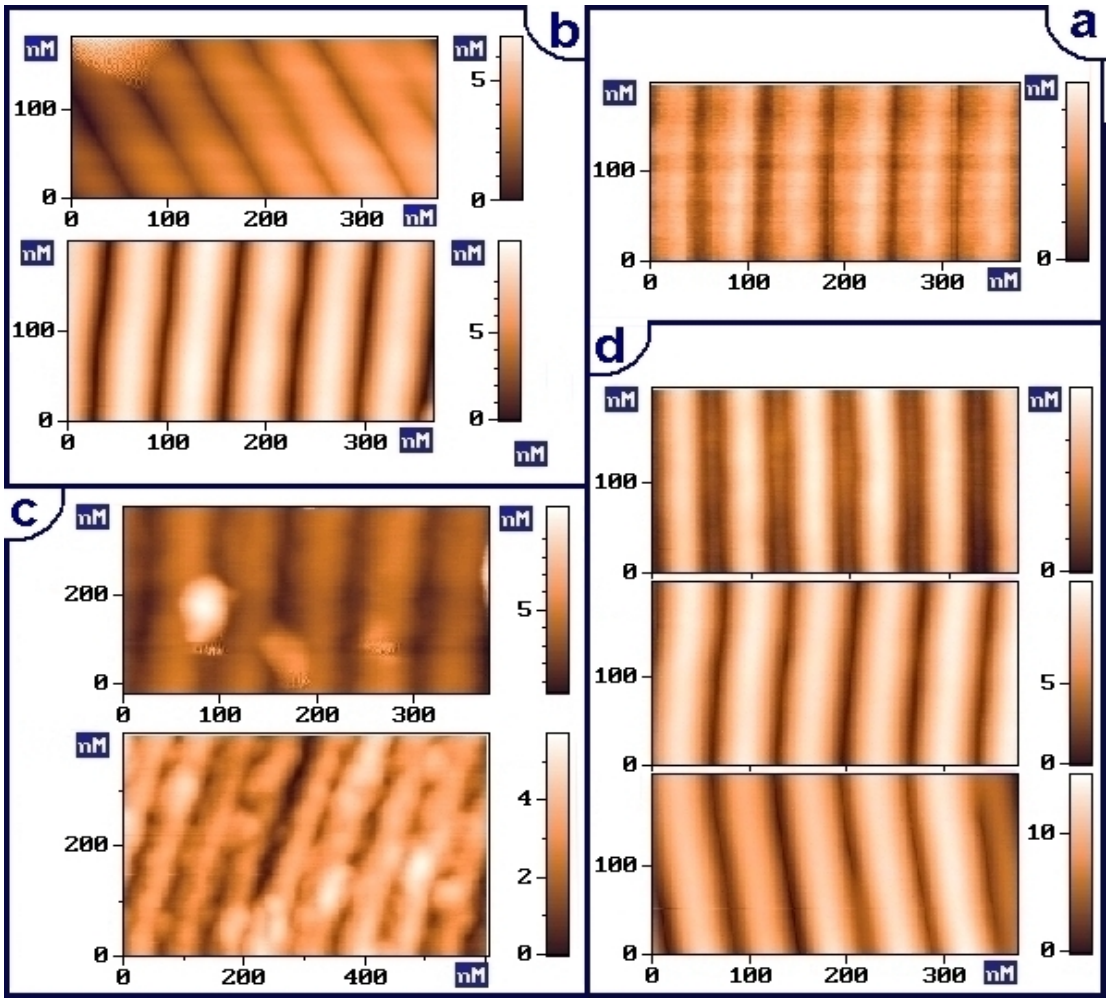

FIGURE 1 The AFM images of of $Al_XGa_{1-X}As$/GaAs cleavage etched in various regimes: (a) – non-etched, (b) – RIE in 1/4 mixture, (c) – RIE in 1/1 mixture, (d) – PPE in1/1 mixture – see, Experimental. Etch time, top to bottom: (a)-(c) 20 and 40 s, (d) 20, 40 and 80 s.

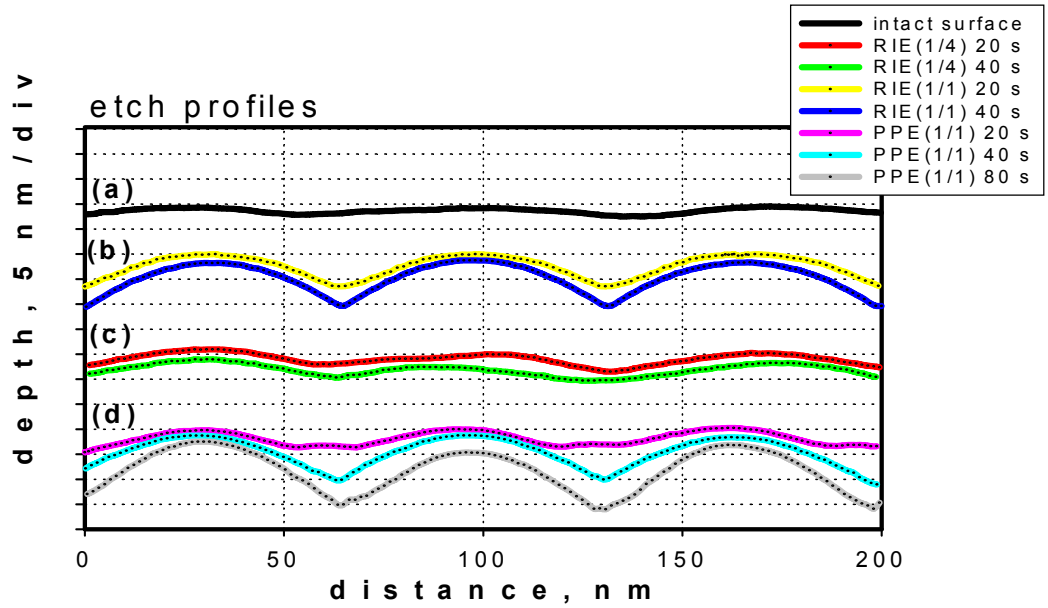

FIGURE 2 The AFM profiles corresponding to the images in Fig.1.

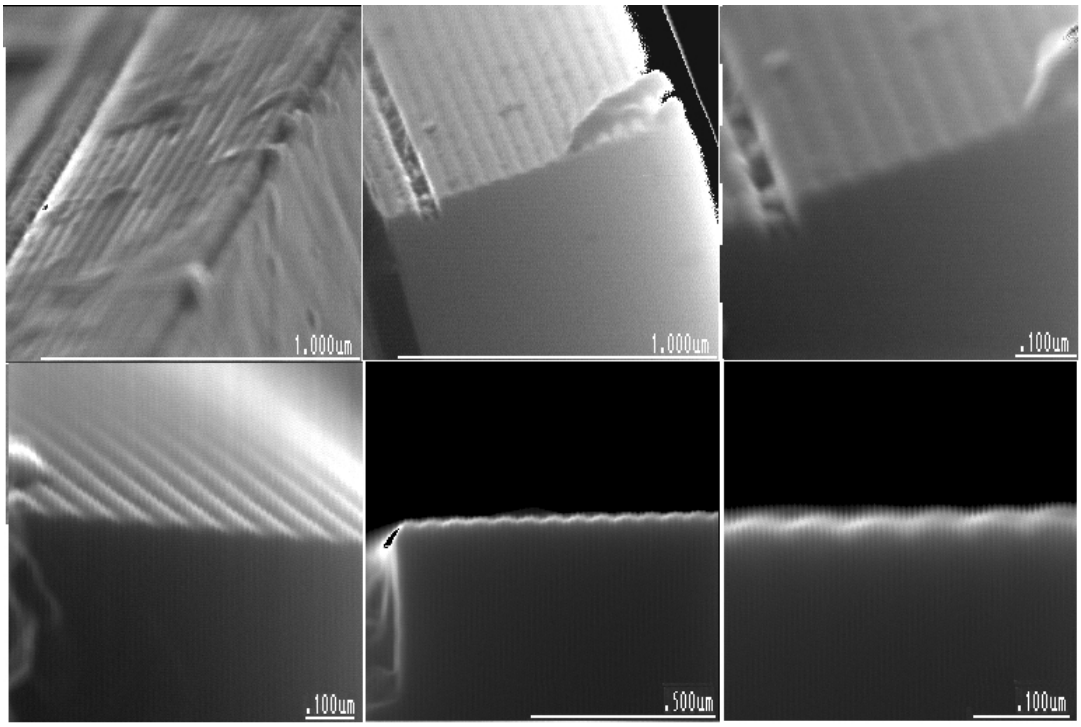

FIGURE 3 Electron micrographs with various resolution duplicating the AFM images shown in Fig.1b-1 (upper row) and Fig.1d-3 (lower row), respectively.

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