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AFM and LFM Observations of Compound Semiconductor Surfaces and Heterointerfaces

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Atomic-level flatness of (100) GaAs surfaces was studied with atomic force microscope (AFM) and was found to be drastically improved with a HCl treatment. Remaining undulation of GaAs surfaces observed after dipping in a HCl solution was within one or two monolayers over a 1μm area. It is also demonstrated that heterointerfaces such as ZnSe/GaAs can be characterized using both vertical atomic force and lateral frictional force microscopes. The difference of semiconductors is distinguished by the lateral frictional force, while the difference of the atomic height is examined by the vertical atomic force.

1. Introduction

Atomic-level characterizations of semiconductor surfaces and heterointerfaces are getting more and more important for the precise control of quantum confined heterostructures. Scanning tunneling microscope (STM) is a nice tool for this purpose. However, it is not applicable to semi-insulating materials. Heterointerfaces characterized by STM gives informations overlapped by potential distributions determined by the impurity distributions and band offsets[1]. Therefore the interpretation of the images is not straightforward. Atomic force microscope, on the other hand, is applicable regardless of the substrate conductivities. It has a possibility to give informations complimentary to STM.

In this paper, two major findings related to AFM observations of compound semiconductor surfaces and heterointerfaces are reported. One is related to the characterization of GaAs surfaces. It is shown that (100) GaAs surfaces after dipping in a HCl solution gives flatness of one or two monolayers over 1-μm scale surface area.

The other finding is that heterointerfaces can be characterized using a lateral force microscope (LFM). This was motivated by the idea that the difference of electronegativity of each atom will result in different electrostatic force. The discrimination of atoms should be possible by measuring the difference on lateral frictional force, while the atomic-level flatness is monitored with the simultaneous AFM observations. The difference of semiconductors adjacent across the heterointerfaces is shown to be detectable by the lateral frictional force, while the atomic-level height across the heterointerface examined by the vertical atomic force was the same.

2. Characterization of Atomic-level Flatness of (100) GaAs Surfaces with AFM

GaAs surfaces are usually etched prior to epi-growth with etchants such as those including H_2SO_4 and H_2O_2. Surfaces before and after these chemical treatments were examined with AFM. GaAs substrates mainly studied were n-type with the electron concentration of ~$5x10^{17}cm^{-3}$. To reduce the atomic force during the AFM observations, GaAs surfaces were studied in pure water to reduce the surface tension from the moisture on the surfaces unless otherwise stated.

Figure 1 is the AFM image of a GaAs surface after cleaning with organic solvents. The surface had the undulation of 1~2 nm. Then the substrates were etched with a solution of $H_2SO_4 : H_2O_2 : H_2O = 4 : 1 : 1$ for 3 min. This resulted in etching of more than 10 μm in depth. The surfaces were then cleaned for 10 min. with flowing ultra-pure water with the resistivities of 18 MΩcm. Figure 2 shows the AFM image of the GaAs surface after the etching with the H_2SO_4 solution. The undulation of the surface still remained similar to the one prior to etching, and no improvements on the atomic surface flatness were observed.

Based on these observations, we tried a treatment in a HCl solution. After the etching in the H_2SO_4 solution, substrates were immersed in a conc. HCl solution for 3 min. and were rinsed in the flowing ultra-pure water for 10 min. The surface observed after this treatment is shown in Fig. 3. The undulation of the surface was found to be reduced to less than 0.5 nm. To confirm the contribution of the HCl treatment on the improvement of the surface flatness, the longer treatments in the HCl solution were examined. The improvement of the surface flatness was reproducibly observed, but no correlations to the time treated in the HCl solution were observed. The GaAs surface treated in the HCl solution for 20 min. showed the similar undulation to that shown in Fig. 3.

It is well known that GaAs surfaces slowly dissolve in water. Therefore the influence of the pure water rinse on the surface flatness was examined. After the HCl treatment, GaAs substrates were rinsed for just several seconds in a series of beakers containing pure water. AFM observation was performed in air in this case. Figure 4 shows the AFM image of the GaAs surface thus treated. The immersion into the HCl solution was 20 min. in this case. The overall flatness of the surface was drastically improved as is clearly seen in Fig. 4 in comparison to Fig. 3. The remaining slowly varying undulation on the surface was less than 0.5 nm. The quantitative information on the surface profile in the wider range of 1 μm is shown in Fig. 5. The vertical scale in Fig. 5 is ±0.5 nm, and therefore the most part of the surface undulation remained within ±1 monolayer fluctuation.

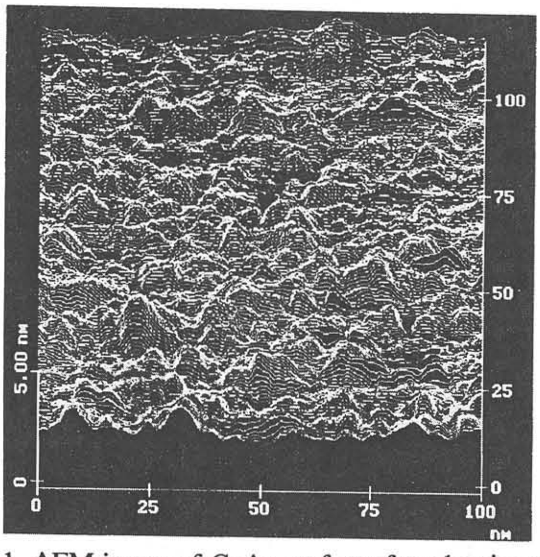

Fig. 1. AFM image of GaAs surface after cleaning with organic solvents.

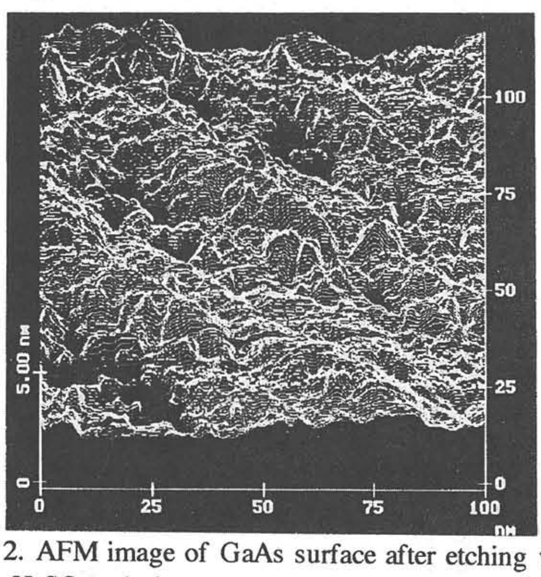

Fig. 2. AFM image of GaAs surface after etching with H_2SO_4 solution.

The above results clearly demonstrate that the atomic flatness of (100) GaAs surfaces is drastically improved with the HCl treatment. The mechanism to realize the atomically flat GaAs surfaces with the HCl treatment may be related to a selective etching on steps and kinks as was discussed in the case of a hydrogen plasma cleaning of GaAs surfaces[2].

X-ray photo-electron spectroscopic (XPS) studies of the GaAs surfaces treated with HCl solutions are now

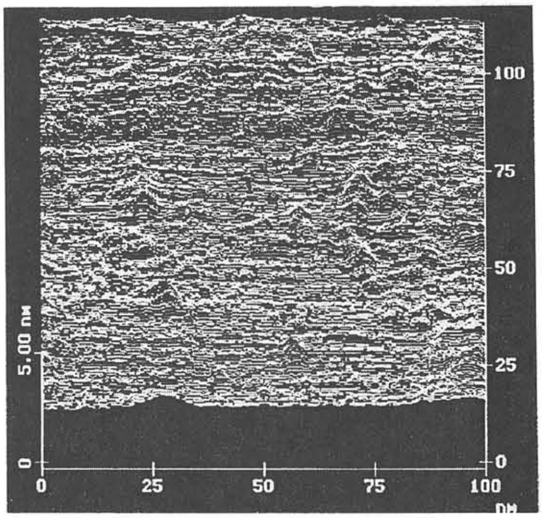

Fig. 3. AFM image of GaAs surface with HCl treatment after etching with H_2SO_4 solution.

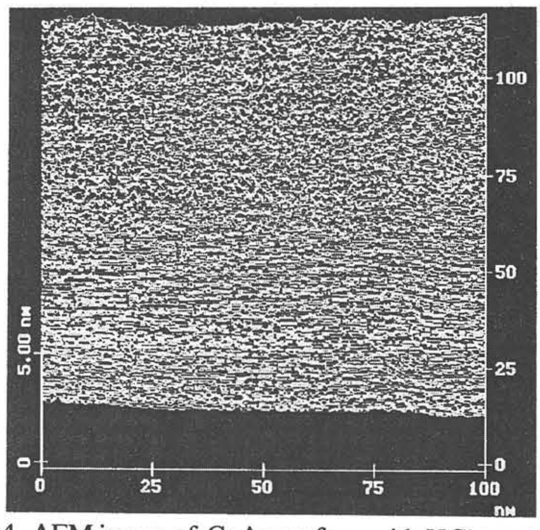

Fig. 4. AFM image of GaAs surface with HCl treatment after etching with H_2SO_4 solution (minimized water rinse).

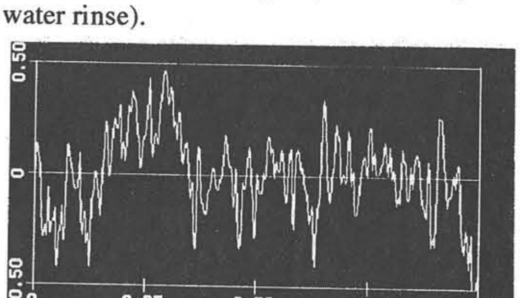

Fig. 5. Surface profile of the sample in Fig. 4 in 1-μm range.

under way, but the preliminary results suggest that Cl remains on the GaAs surfaces and that the Cl atoms are selectively bonded with surface Ga atoms. The details will be reported elsewhere. The atomic forces between the GaAs surfaces and the cantilever tips during the AFM measurements in water were reproducibly less than 1 nN in most cases when the HCl treatment was not included. However, the atomic forces were reproducibly increased to ~5 nN after the HCl treatment except for p-GaAs substrates. This may be the implication that the atomic force in the latter case mainly comes from the electrostatic force originating from the polarized Cl-adsorbed GaAs surfaces. The details are under study.

3. Characterization of ZnSe/GaAs Interfaces with AFM and LFM

Heteroepitaxial layers studied in this work were grown by metalorganic vapor-phase epitaxy at 515°C. ZnSe films or $ZnSe/ZnS_{0.18}Se_{0.82}$ superlattices of about 1 μm thicknesses were grown on (100) GaAs

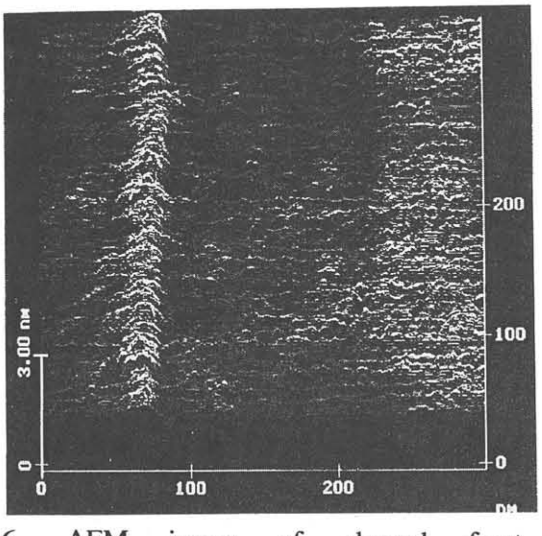

Fig.6. AFM image of cleaved facet of ZnSe(right)/GaAs(left) interface.

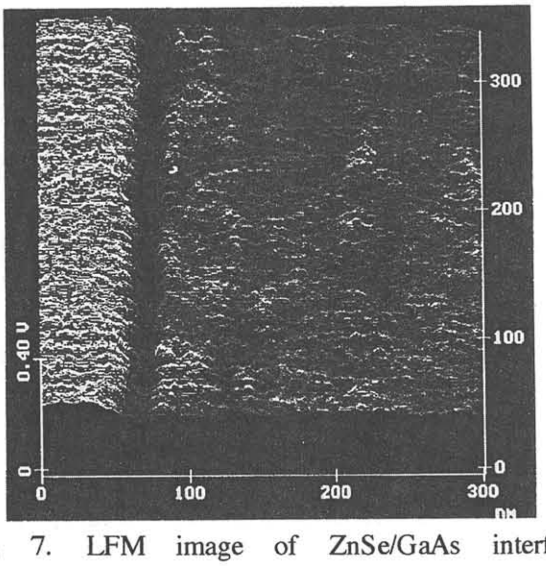

Fig. 7. LFM image of ZnSe/GaAs interface corresponding to Fig. 6.

substrates. Cleaved facets of the grown samples were observed in air with AFM and LFM simultaneously.

Figure 6 shows the AFM image of the cleaved facet of the ZnSe/GaAs interfaces. The right-hand side is the ZnSe film and the left-hand side is the GaAs substrate. The straight line with the width of ~20nm corresponds to the heterointerface. The width of the straight line corresponds to the intermixed layer that resulted in the ridge with the height of ~0.3nm during the cleavage. This significant diffusion at the heterointerface is caused by misfit dislocations due to the lattice mismatch between ZnSe and GaAs[3]. This interdiffusion is well suppressed by growing ZnSSe films lattice matched to GaAs substrates[4]. The superlattices are lattice matched in average and the heterointerface in this case observed with AFM was much sharper than that of Fig. 6.

Figure 7 shows the corresponding image of the lateral frictional force observed with LFM. The difference of the brightness on the GaAs and ZnSe surfaces shows the indication of the surface-dependent frictional forces. As was shown in Fig. 6, the surfaces of GaAs and ZnSe are located at the same horizontal level and will not be distinguishable without the ridge structure at the interface. These results indicate that the simultaneous observations of AFM and LFM gives complementary information on atomic height and difference of semiconductors across the heterointerfaces. It was found recently that the difference of the frictional force between the adjacent semiconductors was almost linearly proportional to the vertical atomic force.

4. Conclusions

The AFM observations of GaAs surfaces demonstrated that GaAs surfaces treated with HCl solutions result in almost complete Atomic-level flatness. This will offer the nicely-prepared surfaces to grow quantum confined heterostructures. Heterointerfaces were shown to be observed by the lateral frictional force even if the heights of the neighboring semiconductors are the same. This gives the possibility to distinguish the difference of atoms by improving the spatial resolution.

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Reference

[1] O. Albrektsen, D. J. Arent, H. P. Meier, and H. W. M. Salemink, Appl. Phys. Lett. **57**, 31 (1990).
[2] I. Suemune, A. Kishimoto, K. Hamaoka, Y. Honda, Y. Kan, and M. Yamanishi, Appl. Phys. Lett. **56**, 2393 (1990).
[3] T. Kanda, I. Suemune, K. Yamada, Y. Kan, and M. Yamanishi, J. Cryst. Growth **93**, 662 (1988).
[4] T. Ohmi, I. Suemune, T. Kanda, Y. Kan, M. Yamanishi, F. Nishiyama, and H. Hasai, J. Cryst. Growth **86**, 467 (1988).