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Cross-sectional observation of NaClO stain-etched $Al_{0.5}Ga_{0.5}As/GaAs$ multilayer by atomic force microscopy

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The cross-section of multilayered $Al_{0.5}Ga_{0.5}As/GaAs$ epitaxial structure was investigated by atomic force microscopy (AFM). For the first time, a 5% NaClO etchant was employed to discern each layer and a clear cross-sectional image of the multilayered epitaxial structure was obtained in less than 3 s etching time. The AFM image using 0.1 M HCl was poorer than that using 5% NaClO; this is attributed to the difference in etching selectivity between HCl and NaClO solution.

1. Introduction

Recently, the multilayered epitaxial structure, which consists of alternately grown thin epitaxial layers of two semiconductor materials, has been attracting much attention for electronic and optoelectronic device application. There has been a growing interest in the evaluation technology of the nanoscale epitaxial structure. This interest has led to the development of an observational methods enabling the study of microfabricated features. For instance, scanning electron microscopy (SEM) and transmission electron microscopy (TEM) [1] are commonly used to characterize multilayered epitaxial structures. Furthermore, many groups reported the cross-sectional observation of a multiquantum well by scanning tunnelling microscopy (STM) [2–3]. However, there are several limitations, like complex sample preparation procedures and the requirement of an ultra-high vaccum environment.

However, an ambient-type AFM, which obtains the morphology of the surface from the measurement of the contact force between tip and the sample surface, does not require special care for sample preparation. AFM has sufficient resolution in the evaluation of microfabricated features, for it to be suitable for cross-sectional observation. The cross-sectional observation using AFM has already been reported by several groups [1, 4]. Chalmers *et al.* [4] reported the observation of a tilted superlattice structure (GaAs/AlAs) and Friedbacher *et al.* [1] investigated a multilayered structure (AlGaAs/GaAs). They discerned the cross-sections by using 0.1M HCl. However, the AFM images were relatively poor. This is attributed to poor etching selectivity of the HCl solution because the latter dissolves both arsenic and gallium oxide. Therefore, the selection of an etchant having good etching selectivity is very important for the examination of the cross-section.

In this paper, we report the cross-sectional observation of multilayered $Al_{0.5}Ga_{0.5}As/GaAs$ by AFM using both HCl and NaClO solution; the resolution of the AFM compares with that of the SEM, and the optimum etching conditions for observation are ascertained.

2. Experimental procedure

We used an ambient-type AFM (SFM-BD2, Park Scientific Instruments) which is based on the optical lever deflection mechanism [5–8], and a pyramedical Si_3N_4 cantilever whose length is 200 μm and force constant is 0.064 $N\,m^{-1}$. Typically we measured the AFM image at 2 Hz scan rate and with a contact force between the tip and the surface of around 10^{-6}–10^{-8}N.

The multilayered epitaxial structure ($Al_{0.5}Ga_{0.5}As$/GaAs) was formed on semi-insulating GaAs (100) wafer by metallorganic chemical vapour deposition (MOCVD). Fig. 1 shows a schematic cross-sectional view of $Al_{0.5}Ga_{0.5}As/GaAs$. It consists of three parts (A, B, C) as shown in Fig. 1. The first part (A) consists of a 100 nm GaAs and 1 μm AlGaAs layers serving as a buffer layer. The second part (B) consists of three types of multilayered AlGaAs/GaAs structure which are 30, 100, and 300 nm thick and prepared by depositing five layers of alternate AlGaAs and GaAs. The third part (C) consists of a 10 nm thick GaAs capping layer and a 3 μm thick AlGaAs layer which is grown in order to avoid difficulty in approaching the tip to the sample near the edge.

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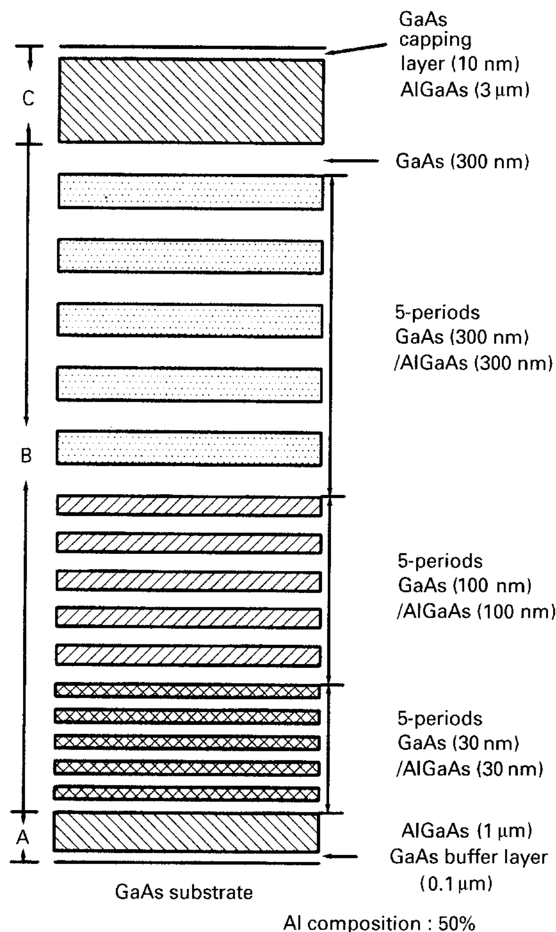

Figure 1 Schematic illustration of the structure of an $Al_{0.5}Ga_{0.5}As$/GaAs multilayer grown by an MOCVD.

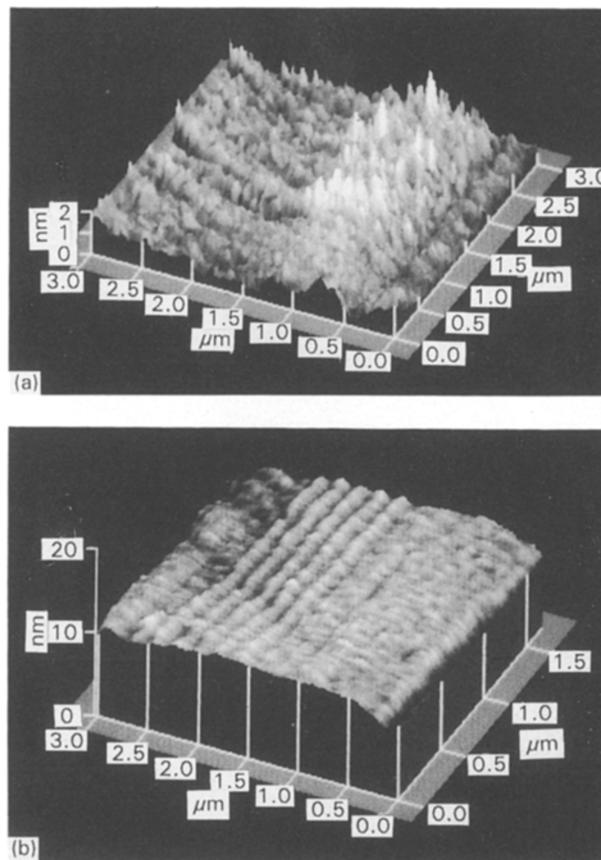

Figure 2 Three-dimensional images of stain-etched cross-section by using 0.1 M HCl for (a) 1 min and (b) 5 min. Note that very irregular corrugations result when using HCl etching.

After the sample was cleaved, AFM measurement was done in the direction perpendicular to the cross-sectional surface which is stain-etched. For the first time, we have employed 5% NaClO as an etchant and have determined the optimum etching conditions for AFM measurements of the sample. NaClO was chosen as an etchant because it was a strong oxidizer and its etching selectivity was good. For comparison, we studied a surface etched in 0.1 M HCl solution within the range of etching time from 1–5 min. Also we observed the AFM image of the unetched cross-section of the sample and studied the etching effect of NaClO and HCl solutions.

3. Results and discussion

We investigated the cross-section of multilayered epitaxial structures etched at different etching times (1, 3, and 5 min) in the HCl etchant as other groups did. Fig. 2 shows the three-dimensional AFM pictures of a sample etched in 0.1 M HCl. It is observed that the AlGaAs layers formed grooves and the GaAs layers formed ridges. It is expected that oxidation will proceed in the deeper region for the AlGaAs layer due to the high reactivity of aluminium. Because HCl acts as an oxide remover, the etched AlGaAs layers are deeper than the etched GaAs layers. Fig. 2a shows that the cross-sectional surface is not smooth and the boundary of each layer is not clearly distinguished. After 3 min etching, it is possible to discriminate between the GaAs and AlGaAs layers (not shown here). This fact is similar to the result reported by Friedbacher *et al.* [1]. Fig. 2b is the image of the sample etched for 5 min and shows the boundary between GaAs layers and AlGaAs layers, but the morphology of the cross-sectional surface is very poor. The corrugation depth between the 100 nm GaAs buffer layer and the 1 μm AlGaAs layer is about 0.6 nm and that of the 30 nm layers is 0.3 nm. The corrugation depth is the difference in the heights of the AlGaAs layer and the GaAs layer and the measurements were done in the middle of each layer. The idea of etching the cross-sectional surface to obtain enhanced resolution of the cross-sectional image, is based on the difference in etching rate for different layers. Therefore, the selection of the etchant with good etching selectivity is essential. We employed the 5% NaClO solution, which has a good etching selectivity, in order to obtain clearer AFM images.

Fig. 3 shows two cross-sectional images. The former is observed by AFM after 5 s etching and the latter is observed by SEM after 80 s etching in NaClO. We found the AlGaAs layers forming the ridges and the GaAs layers forming the grooves. In NaClO, AlGaAs is oxidized faster than GaAs, so the AlGaAs layer is higher than the GaAs layer. The etching morphology at the GaAs layer in the AFM image is also shown, but there is no such morphology in the SEM image. The 100 and 300 nm layers are easily

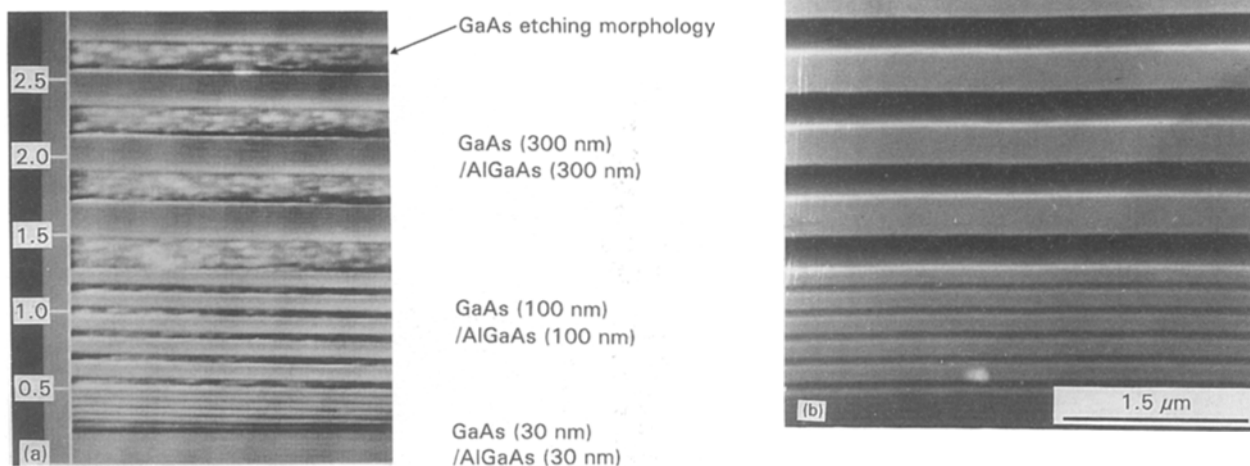

Figure 3 (a) AFM and (b) SEM images. Only features whose dimensions are smaller than 100 nm are distinguished by AFM.

observed in both cases, but the 30 nm layers are not well resolved in the SEM image.

We investigated the AFM image for different etching times in NaClO. Fig. 4a shows the AFM image of the cross-sectional surface after 1 s etching in 5% NaClO solution. The edges of the AlGaAs layers are very sharp and the boundary between AlGaAs and GaAs layers is clear. In the 30 nm layer region, the corrugation depth is 2 nm. The corrugation depth between the 100 nm GaAs buffer layer and the 1 μm AlGaAs layer is 9 nm. We noted that different corrugation depths were observed for different parts of the multilayer. Corrugation depths for 30, 100, and 300 nm regions are 1, 10, and 13 nm, respectively. The different corrugation depths are caused by the pyramidal shape of the tip. This tip shape limits its probing depth because of the difference in length between ridges. One other possible cause is the difference in the area exposed to the etchant. For a thicker layer, the exposed area is wider and it is etched more efficiently, giving rise to higher corrugation depth. After 5 s etching, the edge becomes round and unclear. The corrugation depth between the GaAs 100 nm layer and the AlGaAs 1 μm layer increases to 100 nm. Fig. 4b shows a three-dimensional display of an AFM image after 30 s etching. A much rounder edge is seen than that shown in Fig. 4a. The edge became rounded owing to the expansion of the oxide layers in all directions. The corrugation depth between the 100 nm GaAs layer and the 1 μm AlGaAs layer became large and reached 114 nm. As etching time increased, the rate of change in the corrugation depth decreased remarkably especially on thin layers. Fig. 5 shows a plot of the corrugation depth between the 100 nm buffer layer and the 1 μm AlGaAs layer as a function of etching time. The decrease in the rate of change in the corrugation depth is attributed to two factors: that the etching effect is reduced as the etchant becomes saturated, and the formed oxide layer acts as the membrance.

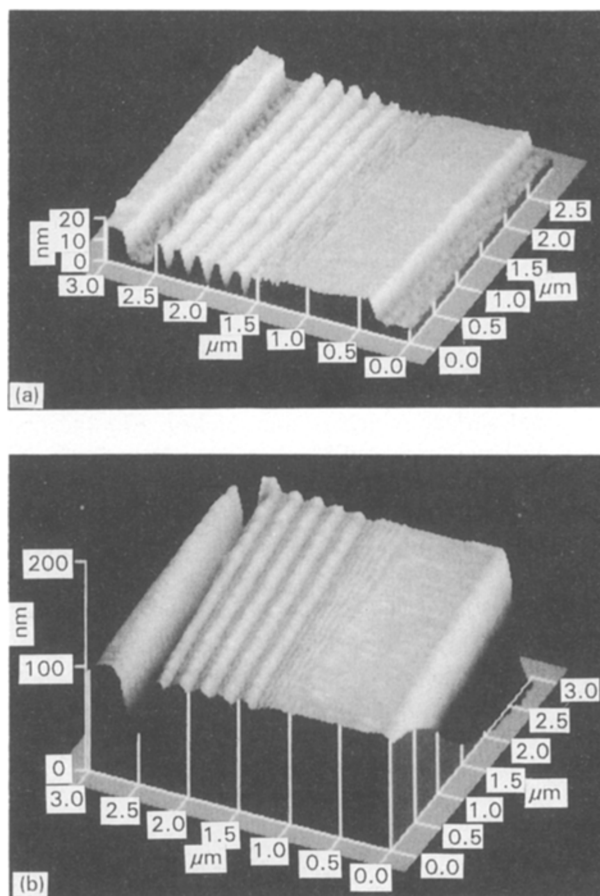

Figure 4 Three-dimensional display of NaClO stain-etched AlGaAs/GaAs multilayer with etching times of (a) 1 s and (b) 30 s. Ridges and grooves correspond to AlGaAs and GaAs layers, respectively. Note that even the etching morphology of the GaAs layers is clearly observed in the 1 s etched cross-section.

In Fig. 6a, the same sample is scanned in the 30 nm layer region with an area of 0.5 μm by 0.5 μm, especially after 1 s etching in 5% NaClO. The corrugation depth of the 30 nm layer region is 6.6 nm. The AlGaAs and GaAs layers are sharply distinguished,

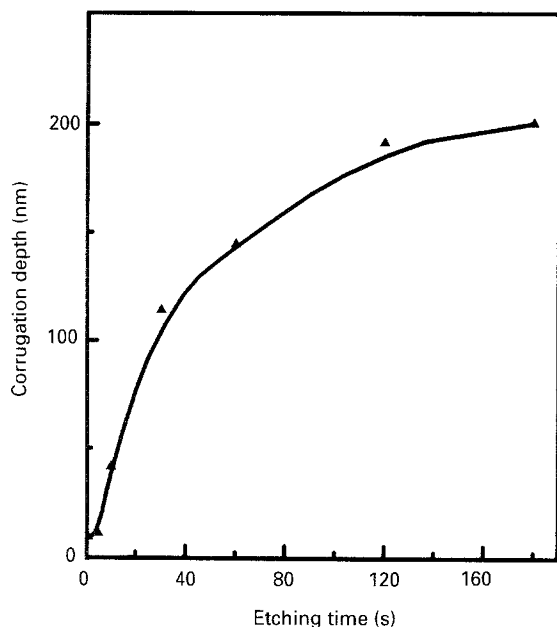

Figure 5 The corrugation depth between a 100 nm GaAs buffer layer and 1μm AlGaAs layer as a function of etching time when using 5% NaClO. The line is drawn as a guide.

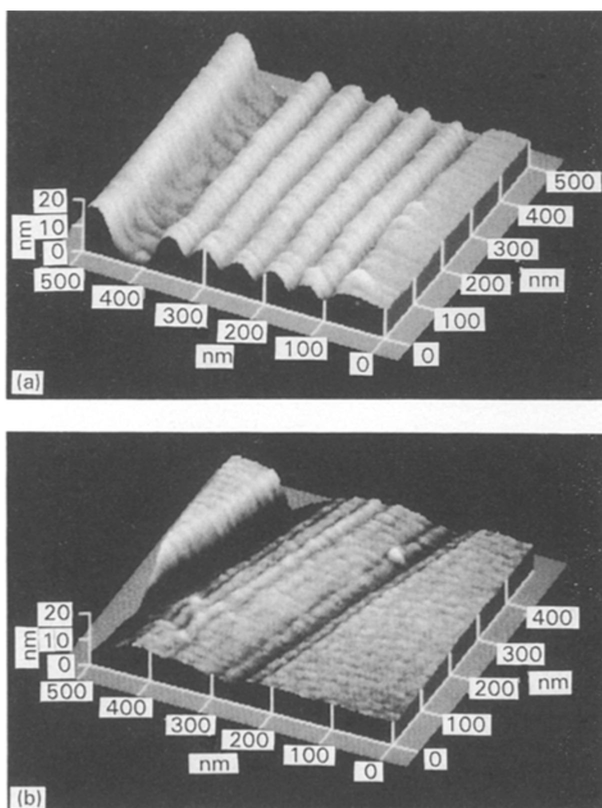

Figure 6 Three-dimensional displays of 30 nm thick structure with a 0.5 μm by 0.5 μm scan area after (a) 1 s and (b) 5 min NaClO etching, respectively. Note that 30 nm thick layers are degraded after 3 min etching.

but it becomes less easy to observe the edge of the 30 nm AlGaAs layers as the etching time increases. For instance, after etching for 5 min, as shown in Fig. 6b, it is impossible to distinguish each layer of 30 nm thickness. Thus it is possible, that if the sample was etched for a long time, for example, over 1 min, the real cross-sectional structure could be distorted. Therefore, an etching time below 3 s is desirable to obtain undistorted AFM images using 5% NaClO solution.

Even without etching, we could obtain an AFM image as a result of natural oxidation of the AlGaAs layers in air. However, this method is not suitable because it is necessary to wait for a long time, more than 1 day after cleavage, before results can be obtained. The cross-section can be easily damaged and contaminated during this period. When we observed the AFM image of the unetched sample which was exposed to air for 6 days after cleavage, the corrugation depth between the 100 nm GaAs buffer layer and the 1 μm AlGaAs layer was 0.7 nm and that of the 30 nm layers was about 0.3 nm. The AlGaAs layers formed ridges. The AFM image of an unetched surface is similar to the image of the etched surface in the 5% NaClO. By using NaClO, the etching morphology of grooved GaAs layer could be observed. In contrast, this morphology was not observed for the unetched sample. In addition, we observed the morphology of two facing surfaces just after cleavage, Tanaka *et al.* [4] suggested the possibility of a geometrical effect which is the corrugation formed mechanically by cleaving, but they could not confirm it in the air STM. Although we observed the two freshly cleaved cross-sectional surfaces, we could not find any difference in the two cross-sections of two facing surfaces. Because we used the same experimental conditions as Tanaka *et al.* [4], that is, the AlGaAs layer is oxidized in air immediately after cleavage, the mechanical morphology of the cross-section by cleavage cannot be observed in air.

4. Conclusion

5% NaClO etchant has been employed for the first time to observe the cross-section of multilayered epitaxial structure ($Al_{0.5}Ga_{0.5}As/GaAs$). By applying NaClO, a clear image, e.g. the sharp boundary between AlGaAs layers, could be observed. The AlGaAs layer forms a ridge due to the oxidation of the AlGaAs layer. In contrast, the etching morphology of the GaAs reveals a grooved layer. It was confirmed that more uniform and clearer AFM images were obtainable than those etched in HCl. An etching time below 3 s in 5% NaClO was sufficient to observe the cross-section of the multilayered epitaxial structure by AFM. In this case, the corrugation depth between the 100 nm GaAs buffer layer and the 1 μm AlGaAs layer was 9 nm and that of the 30 nm layers was 2 nm. The usefulness of cross-sectional observation by AFM using 5% NaClO etchant is attributed to the simple procedure for sample preparation, the feasibility in air, and the short observation time.

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