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Highly Selective Wet-Etching Using Adipic Acid for Uniform Damage-Free Process of InAlAs/InGaAs HEMTs

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Highly selective wet-etching of InGaAs on InAlAs was demonstrated using pH-controlled adipic acid, NH_4OH, and H_2O_2 solutions. Maximum selectivity of 250 was obtained by controlling the etching mechanism of InGaAs and InAlAs. By investigating the rate-determining step for the etching of InAlAs and InGaAs, we found that the high selectivity is due to the difference in dissolvability between the oxide of InAlAs and that of InGaAs in the adipic acid solution. This highly selective etching was applied to the fabrication of InAlAs/InGaAs HEMTs. The standard deviations of the threshold voltage and transconductance were as low as 38 mV and 11 mS/mm, respectively, in a 3-inch diameter wafer.

1. Introduction

InAlAs/InGaAs high electron mobility transistors (HEMTs) are considered the most promising devices for millimeter-wave and optical communications because of their excellent high-frequency and low-noise characteristics.[1), 2)] These are due to the high mobility, high saturation velocity, and high sheet carrier density of the InAlAs/InGaAs /InP system. Better uniformity is one of the most important requirements for the HEMTs in order to fabricate MMICs. Since forming a recess for the Schottky gate is the most critical procedure in the fabrication of HEMTs, the selective etching of InGaAs on InAlAs is useful technique for obtaining high uniformity. C. Lauterbatch et al.[3)] reported the selective dry-etching of InGaAs on InAlAs using a CH_4 and H_2 gas mixture. Application to device fabrication, however, was not acceptable due to the poor selectivity of only 6 and the high self-bias, which would cause damage in the InGaAs channel. Although a damage-free selective etching process using photo-chemical etching has been reported, the selectivity is still only 25[4)]. A dry etching technique using a gas mixture containing fluorine compounds was also reported to have higher selectivity[5)], but the inactivation of the donor by the fluorine contained in the etching gas prevented high-yield production of InAlAs/InGaAs HEMTs[6)]. On the other hand, selective wet-etching techniques have also been reported because they have some advantages over dry-etching, for examples, no damage, low-cost, and good reproducibility. Selective wet-etching using a solution of citric acid and hydrogen peroxide has been widely used for the gate-recess and mesa-sidewall etching due to the relatively high selectivity of 60 and its simplicity[7)]. Other wet-etching techniques have also been reported, for example, using oxalic acid[8)], succinic acid[9)], malonic acid[10)] and adipic acid.[10)] However, the selectivities of these etchants are still as low as about 100.

This paper investigates the mechanisms of etching of InGaAs and InAlAs by the adipic acid based etchant. By controlling the etching mechanism, we obtained high selectivity of 250. InAlAs/InGaAs HEMTs fabricated using the adipic acid based etchant exhibited high uniformity.

2. Experiments

The adipic acid based etchant was prepared as follows. The adipic acid solution was prepared by adding 5 ml of deionized water to 1 g of adipic acid with the addition of ammonia hydroxide. The amount of added ammonium hydroxide was controlled so that the pH of the solution was between 5.3 and 7.0. Hydrogen peroxide (30%), which is an oxidizer of InGaAs and InAlAs, was added to the pH-adjusted solution at volume ratios between 0.013 and 0.12.

Examined samples of undoped $In_{0.53}Ga_{0.47}As$ and $In_{0.52}Al_{0.48}As$ layers for selective etching were grown by molecular beam epitaxy (MBE) on (100) InP substrates. They were patterned by the photoresist (AZ1350J) which is not eroded by the etchant. After cleaning the surfaces of the samples with ammonia hydroxide solution, we etched them at 25 Åé with stirring. Etching was stopped by rinsing the samples in flowing deionized water for approximately 3 minutes. The photoresist was removed with acetone after the etching. The etching rates were obtained from the gradient of the dependence of etched depth on the etching time since the depth linearly increased with the etching time for all the etchants in this study.

InAlAs/InGaAs HEMTs were fabricated by using adipic acid based etchant for the gate-recess processing. The epitaxial layers were grown by MBE lattice-matched on InP. The thickness of the Si-doped (3×10^{19} cm^{-3}) InGaAs cap layer, which was selectively removed by the adipic based etchant, was 50 nm. The thicknesses of the undoped InAlAs barrier layer, the Si-doped (5×10^{18} cm^{-3}) InAlAs carrier-supplying layer, and the undoped InAlAs spacing layer were 10, 12, and 2 nm, respectively. Non-alloyed ohmic contacts consisting of Ti/Au were used for the source and drain electrodes. The gate was defined by photo-lithography. The gate metal of Mo/Au was formed by a lift-off technique after gate recessing was done by using adipic based etchant.

3. Results and discussion

3-1. Etching mechanism

III-V semiconductor materials are usually etched by an oxidation-reduction reaction at the semiconductor surface followed by dissolution of the oxide materials. All etchants in this study contained hydrogen peroxide as the oxidizer with the pH-adjusted solution dissolving the resultant oxide materials because no etching occurred when the samples were soaked in pH-adjusted solution containing no hydrogen peroxide. The dissolution process is considered to be divided into two steps. One is the reaction of the oxide materials by an acid or ions in an etchant, and the other is the desorption of the resultant materials from the surface. As a result, the etching rate is determined by the rates of three processes, that is, 1) the oxidation-reduction reaction at the semiconductor surface, 2) the reaction of the oxidized materials by an acid or ions in the etchant, and 3) the

desorption of resultant materials from the surface. The step having the slowest rate becomes the rate-determining step for the etching. Since the depth linearly increased with the etching time for all the etchants in this study, step 3 was faster than steps 1 and 2 for InGaAs and InAlAs because the depth would have been proportional to the square root of the etching time if step 3 is the rate-determining step[7].

3-2. Dependence of etching rate on volume ratio of H_2O_2

Figure 1 shows the etching rates for InGaAs and InAlAs plotted against the volume ratio of added 30% hydrogen peroxide. For the InGaAs layers, etching rates at both the edge and the center of patterns are given in fig. 1 because an abnormal etched profile, which has different etching depths at the pattern edges and center, was obtained as shown in fig. 2. The etching rates of InAlAs were almost constant while the hydrogen peroxide ratio increased. This can be understood if the rate-determining step for InAlAs is step 2. On the other hand, the etching rates of InGaAs for the edge and center increased monotonically with the ratio of hydrogen peroxide up to 4%, whereas from 4% to 12%, the rates decreased with the ratio. This means that the etching

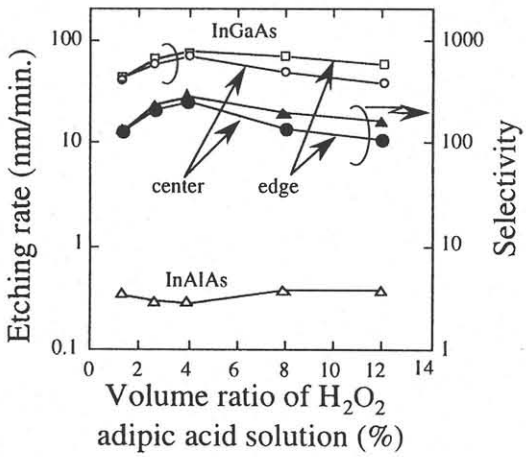

Fig. 1. Rates of etching InGaAs and InAlAs by etchant of pH=5.5 and the selectivity as a function of volume ratio of hydrogen peroxide to the pH-adjusted adipic acid solution.

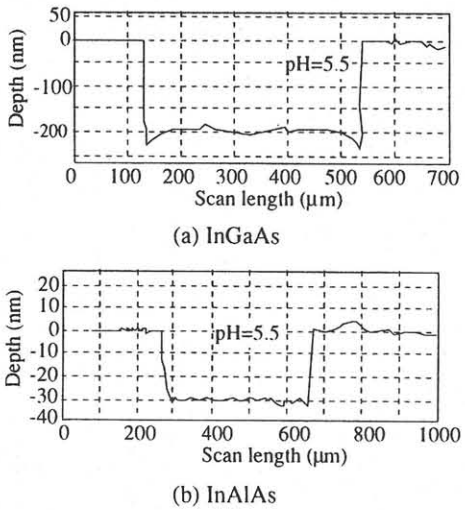

Fig. 2. Profiles of (a) InGaAs and (b) InAlAs etched by etchant of pH=5.5. Abnormal etching was observed at the edge of a profile for InGaAs while that for InAlAs was flat.

rate is limited by step 1 rather than by step 2 for etchants of pH=5.5 containing hydrogen peroxide up to 4%. For the etchant with more than 4% hydrogen peroxide, the rates decreased with ratio because thicker oxide was formed by hydrogen peroxide. Thus, the etching rate tended to be limited by step 2 with the ratio of hydrogen peroxide.

3-3. Dependence of etching rate on pH

Figure 3 shows the dependence of the etching rates of InGaAs and InAlAs and the selectivity on pH of the etchant with hydrogen peroxide of 4%. The etching rates of InGaAs decreased rapidly with pH while those of InAlAs decreased slowly with pH. In order to understand these tendencies, we calculated the concentrations of adipic acid and the related ions. Figure 4 shows the calculated concentrations of H_2B, HB^- (an ion that has dissociated one proton), and B^{2-} (an ion that has dissociated two protons) as a function of pH, where H_2B represents adipic acid. The dissociation constants of adipic acid at room temperature, pK1=4.42 and pK2=5.41, were used for the calculation. Comparing fig. 3 with fig. 4, we can see that the dependence of the etching rate for InGaAs on pH is similar to that of HB^-. This means that the oxide of InGaAs was dissolved by HB^-, and the etching rate was limited by step 2 rather than by step 1. Considering the discussion in the previous subsection, we can conclude that the etching rate of InGaAs is limited by both step 1 and step 2 for the etchant with 4% added hydrogen peroxide. No similarity was found for the etching rate of InAlAs, which indicates that step 2 of InAlAs is not related to only a single ion or to adipic acid but several ions and/or adipic acid.

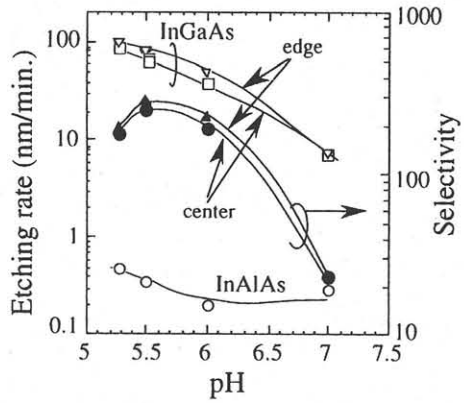

Fig. 3. Rates of etching of InGaAs and InAlAs by etchant containing 4% hydrogen peroxide as a function of pH.

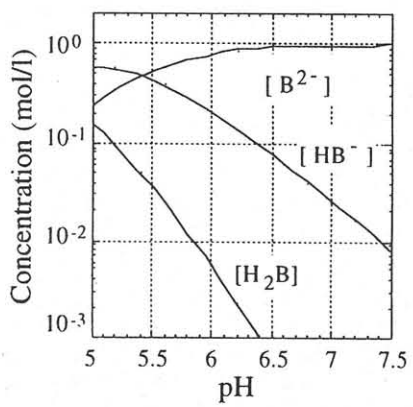

Fig. 4. Calculated concentrations [H_2B], [HB^-], and [B^{2-}] as a function of pH. Here, H_2B represents adipic acid.

3-4. High selectivity

The selectivity of 250 is the highest ever reported for selective wet-etching of InGaAs on InAlAs. At the condition resulting in the highest selectivity, since the etching rate of InAlAs was limited by step 2, the rate of step 2 was less than that of step 1 for InAlAs. On the other hand, the etching rate of InGaAs was limited by both steps 1 and 2, so the rate of step 1 was almost equal to that of step 2. Furthermore, the rates of steps 1 and 2 for InGaAs were faster than the rate of step 2 for InAlAs because of the high selectivity. Accordingly, the reaction rate of the oxide (step 2) of InGaAs was faster than that of InAlAs. This is the reason for the high selectivity. In other words, the high selectivity is caused by the difference in dissolvability between the oxide of InAlAs and that of InGaAs by the adipic acid solution.

3-5. Device performance

Figure 5 shows typical DC characteristics of InAlAs/InGaAs HEMTs fabricated by adipic acid based etchant. The gate-recess was formed by the etchant that gave the highest selectivity of 250. Good pinch-off characteristics were obtained with the threshold voltage of -0.96 V, and there were no abnormal kinks which would be caused by abnormal etching at the edge as shown in fig. 2. The difference in the etching rates between the edge and center of the gate-recess would be expected to be 10 nm/min. from fig. 3. Therefore, if the thickness of an InGaAs cap layer is 50 nm, the edge of the gate-recess will be more etched than the center in only about 7 sec. The excess time of 7 corresponds about 0.05-nm etching of InAlAs, which is thinner than one mono-layer of InAlAs. Thus, the abnormal etching profile of InGaAs (fig. 2) causes no problems in application to the gate recess process.

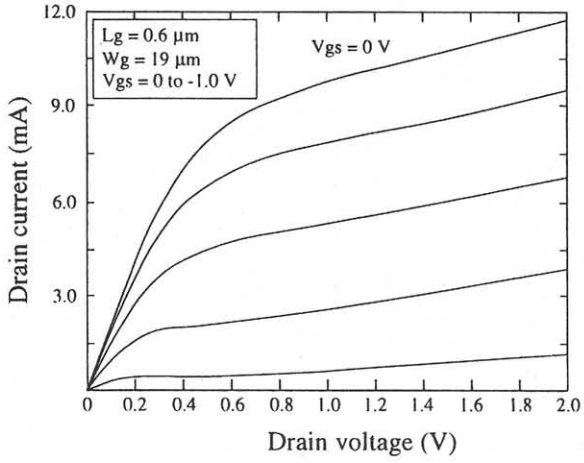

Fig. 5. Typical I-V characteristics of the InAlAs/InGaAs HEMT fabricated using adipic acid based etchant for the gate recess formation.

Figure 6 shows histograms of (a) the threshold voltage (V_{th}) and (b) the extrinsic transconductance (gm) of InAlAs/InGaAs HEMTs with a gate length of 0.6 μm. The measurements were made on 33 devices across a 3-inch wafer. Excellent standard deviations of 38 mV and 11 mS/mm for the threshold voltage and transconductance were obtained with average values of -0.96 V and 726 mS/mm, respectively. These standard deviations are low enough to fabricate MMICs.

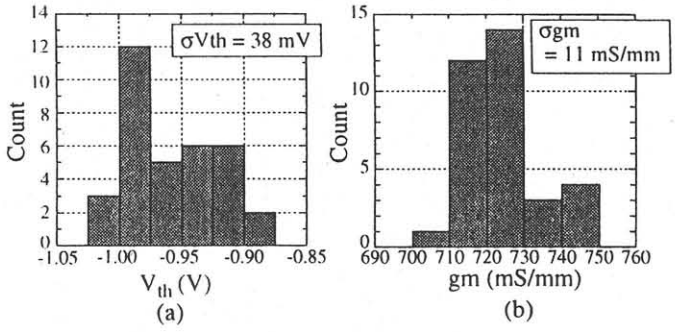

Fig. 6. Histograms of (a) the threshold voltage (V_{th}) and (b) the extrinsic transconductance (gm) of InAlAs/InGaAs HEMTs with a gate length of 0.6 μm in a 3-inch wafer.

4. Conclusion

By investigating the rate-determining steps for adipic acid based etching of InAlAs and InGaAs, we obtained highly selective wet-etching of InGaAs on InAlAs, with maximum selectivity of 250. The high selectivity was caused by the difference in the dissolvability between the oxide of InAlAs and that of InGaAs in the adipic acid solution. The selective wet-etching technique was applied to the gate-recess formation of a InAlAs/InGaAs HEMT. The standard deviations of the threshold voltage and transconductance were as low as 38 mV and 11 mS/mm, respectively, in a 3-inch diameter wafer.

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