

Novel, Organic Acid-Based Etchants for InGaAlAs / InP Heterostructure Devices with AlAs Etch-Stop Layers

To cite this article: Tom P. E. Broekaert and Clifton G. Fonstad 1992 *J. Electrochem. Soc.* **139** 2306

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probably due to the additional annealing after the wafer bonding and it shows that the bonding procedure does not degrade a thermally grown Si-SiO₂ interface located approximately 250 Å from the bonded interface. Considerable generation of interface states during injection was found for both types of samples. The pronounced peak of interface states at approximately 0.75 eV above the valence bandedge in the reference samples was not as clear in the bonded samples. Generation of interface states during charge carrier injection is extensively described in the literature¹⁴⁻¹⁶. The small differences in the generation of interface states found between the bonded and the reference samples indicates a possible influence of the bonding procedure or the bonded interface on the Si-SiO₂ interface state generation.

The oxide charges introduced by the bonding procedure and the charging phenomena during Fowler-Nordheim injection may be important for the performance of SOI materials prepared by wafer bonding. The reported charging of the oxides during wafer bonding is rather small. The present magnitudes ($\leq 10^{11}$ cm⁻²) will probably not result in an inverted back-channel. The large oxide field used during the injection procedures reported in this paper is normally not present in the buried oxide of SOI materials. The charging of the oxides during the charge injection raises, however, questions about the performance of such wafer-bonded SOI materials in radiation environments.

Conclusions

A more pronounced charging of the oxide was found in wafer-bonded MOS capacitors during injection of hot electrons (Fowler-Nordheim tunneling) as compared to reference capacitors. The centroid of the trapped negative charge in the bonded SiO₂-SiO₂ samples was found to be located close to the bonded interface. A high temperature (1200°C) annealing reduced the negative oxide charging but still the charging was considerably larger than in the reference samples. The electron injection caused generation of interface states in the bonded structures comparable to the reference structures. A difference between bonded and bulk oxides was found in the spectral structure of the interface state energy distribution.

Acknowledgments

Dr. Anders T. Dejenfelt is gratefully acknowledged for valuable discussions and for sharing software for the Fowler-Nordheim injection procedure. Thanks are also due to Dr. Sten Norrman and the technology laboratory staff for parts of the device fabrication. This project was supported by the Swedish National Board for Technical Development (NUTEK).

Manuscript submitted Oct. 3, 1991; revised manuscript received April 6, 1992. This was Paper 489 presented at the Phoenix, AZ, Meeting of the Society, Oct. 13-17, 1991.

Chalmers University of Technology assisted in meeting the publication costs of this article.

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Novel, Organic Acid-Based Etchants for InGaAlAs/InP Heterostructure Devices with AlAs Etch-Stop Layers

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ABSTRACT

Several organic acids have been identified that enable the etching of indium compounds, while maintaining selectivity with respect to AlAs, and these acids have been used to develop InGaAlAs etching solutions that allow the selective etching of InP lattice-matched InGaAlAs heterostructures using thin pseudomorphic AlAs layers as etch stops. Of the organic acids tested, the nonaromatic, polycarboxylic acids have been found to be most effective, and some of the best results have been obtained for etchants consisting of succinic acid, ammonia, and hydrogen peroxide. It is found that the etch rate of In_{0.53}Ga_{0.47}As with this solution can be as much as 1000 times the etch rate of AlAs, while the etch rate of In_{0.52}Al_{0.48}As can be as much as 500 times that of the AlAs.

Selective wet etchants for various III-V compounds have been known for quite some time and have found applications in the fabrication of numerous heterostructure devices¹⁻³. More specifically, the (hydrogen) peroxide-ammonia system for etching GaAs preferentially over AlGaAs is well known^{1,2}. A similar etchant that etches the InP-substrate-based quaternary InGaAlAs preferentially over a thin strained AlAs etch stop layer could prove equally use-

ful, but has heretofore not been available. It is well known, for example, that the peroxide-ammonia system does not form any soluble complexes with indium, and thus cannot be used for etching InGaAlAs compounds⁴. We have shown^{5,6} that this limitation can be alleviated by the addition of organic acids, some of which readily form soluble complexes with indium⁷. In addition, it is found that the etching solutions thus obtained can be made selective, by regulating their pH, to etch In_{0.53}Ga_{0.47}As and In_{0.52}Al_{0.48}As at much higher rates than those at which they etch AlAs.

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Table I. Concentration and pH of the acid ammonia solutions used.
 Acids marked with an asterisk must have ammonia added to dissolve all the acid, upon which the given pH is reached.
 The acids which are not marked with an asterisk are very soluble in water and can therefore also be tested at lower pH.
 pK_a values of the acids used are also indicated (Ref. 12).

Label	Acid added per 1 H ₂ O	pH, by adding ammonia	pK_1	pK_2
OA	15 g oxalic · 2 H ₂ O	6.3	1.27	4.27
OCA	25 g oxalic · 2H ₂ O, 100 g citric	6.3		
MA	75 g malonic	6.1	2.83	5.70
SA	200 g succinic*	4.2	4.21	5.64
GA	200 g adipic*	4.3	4.31	5.41
AA	200 g methylsuccinic*	5.2	4.42	5.41
MSA	250 g methylsuccinic*	3.8	4.13	5.64
DSA	100 g 2,2 dimethyl-succinic* (rac)	4.0	3.93	6.20
FA	50 g fumaric*	4.2	3.10	4.60
MEA	200 g maleic	4.0	1.83	6.07
CA	200 g citric ($pK_3 = 6.40$)	4.0	3.13	4.76
PTA	200 g 1, 2, 3 propane tricarboxylic ($pK_3 = 6.38$)	1.75	3.67	4.87
BTA	200 g 1,2,3,4 butane tetracarboxylic ($pK_3 = 6.38$, $pK_4 = 7.16$)	1.85	3.43	4.58
AcA	130 ml acetic	4.75	4.76	

The results presented here extend this concept to a much wider range of organic acids than has previously been reported⁵.

The following acids were found useful for the etching of indium containing compounds: oxalic (ethanedioic)⁶, malonic (propanedioic)⁶, succinic (butanedioic)⁶, glutaric (pentanedioic), adipic (hexanedioic), methylsuccinic, 2,2-dimethylsuccinic, fumaric (*trans*-butenedioic), maleic (*cis*-butenedioic), citric (2-hydroxy-1,2,3-propane tricarboxylic), tricarballic (1,2,3-propane tricarboxylic), 1,2,3,4-butane tetracarboxylic, acetic, L-malic (L-2-hydroxy butanedioic), tartaric (2,3-dihydroxy butanedioic). Acids that were also tested but that did not result in etching or had impractically low etch rates are: pimelic (heptanedioic), terephthalic (1,3-benzene dicarboxylic), isophthalic (1,4-benzene dicarboxylic), and L-glutamic (2-aminopentanedioic).

These etchants have already found applications in the elimination of mesa-sidewall gate leakage current in HFETs⁸ and for uniform and reliable control of etch depth in ridge laser structures⁹. Other devices that could be improved by use of these etchants to facilitate their fabrication are devices, like the resonant tunneling hot electron transistor¹⁰, where electrical contact to a layer, typically In_{0.53}Ga_{0.47}As, adjacent to an AlAs layer is needed (see Ref. 11 and references therein for a collection of devices of this type that could benefit from the selective etch discussed here).

Procedures and Results

Test samples for the etchants were grown by molecular beam epitaxy (MBE) and typically consisted of a buffer layer of 400 nm of In_{0.53}Ga_{0.47}As or In_{0.52}Al_{0.48}As followed by a strained 10 monolayer (m.l.) thick AlAs layer, and a top 100 nm layer of In_{0.53}Ga_{0.47}As or In_{0.52}Al_{0.48}As. Several test samples were also grown in which the thicknesses of the strained AlAs layer was varied down to 3 m.l. Prior to testing the etch rate of the etchant solutions, the samples were degreased and briefly etched in 10:1:1 H₂O:H₃PO₄:H₂O₂ to obtain reproducible surface conditions. The samples were then partially covered with black wax to enable the measurement of step profiles. The etch rate was determined by measuring the etch depth as a function of immersion time into the etchant solution. The selectivity of the etchant was determined by measuring the time required for the etchant solution to break through the AlAs stop layer. The breakthrough of the AlAs stop layer was observed by inspection of the surface by optical microscopy, and resulted in a discolored surface for the low selectivity (less than 100:1) etchants, due to surface roughening as a consequence of local masking; and resulted in local etch pits for the highly selective (greater than 400:1) etchants.

Table I describes the etchant solutions that have been studied. They typically consist of an aqueous solution of an organic acid to which ammonia is added to regulate the pH. Not listed in Table I are etchants with L-malic and tartaric acid which were also tested but were not selective with respect to AlAs. Table II gives a summary of the etch rates and selectivity obtained with these solutions when peroxide is added as the oxidizing agent. The etch rate of the AlAs, required to calculate the etch rate selectivity, is obtained from the same required to break through the AlAs stop layer, and the thickness of the AlAs stop layer, which is taken to be 0.273 nm per monolayer.

It should be noted that the selectivities listed in Table II are based on the time required to break through a 10 m.l. AlAs stop layer. However, this break through time was only monitored for a maximum of 25 min. Consequently the selectivities listed for the etchants with low etch rates may only be lower bounds; the actual selectivity may be somewhat higher. For such high selectivity etchants, the time required to break through a 5 m.l. AlAs stop layer would be a better measure of selectivity relative to the other etchants.

The etch rate dependence of In_{0.53}Ga_{0.47}As and In_{0.52}Al_{0.48}As on pH and peroxide concentration for the succinic acid based etchant has been studied in more detail. The results are shown in Fig. 1a and b, respectively⁶. After 25 min of exposure to the etchant, the AlAs stop layer was still mostly intact, with the exception of a few square-shaped etch pits, mostly occurring at the edges of the sample⁶. The square shape of the etch pits is most likely due to stress relief cracking of the strained AlAs layer along the

Table II. In_{0.53}Ga_{0.47}As and In_{0.52}Al_{0.48}As etch rates and selectivity of the acid solutions studied.

Acid:H ₂ O ₂ ratio (pH)	InGaAs etch rate (nm/min)	InAlAs etch rate (nm/min)	10 m.l. AlAs break time (min)	5 m.l. AlAs break time (min)	InGaAs to AlAs selectivity	InAlAs to AlAs selectivity	InGaAs to InAlAs selectivity
OA 20:1	40	20	5	2	70	35	2
OCA 25:1	75	5	15	—	410	25	15
MA 25:1	100	6	6	—	220	13	17
SA 25:1	76	40	>25	—	>700	>365	2
GA 25:1	56	11	>25	3	>500	>100	5
AA 25:1	30	0.4	>25	1.5	>640	>8	75
MSA 25:1	40	0.5	>25	3	>360	>4	80
DSA 25:1	25	<0.2	>25	15	>270	>4	>125
FA 25:1	50	6.5	20	2	370	45	8
MEA 25:1	65	13	10	0	240	45	5
CA(2) 25:1	55	30	5	0	100	55	2
CA(4) 25:1	70	5	>25	2	>640	>45	14
PTA(1.75) 25:1	50	20	12	2	220	85	2.5
PTA(4) 25:1	50	1	>25	3	>450	>9	50
BTA(1.85) 25:1	50	3.6	17	3	310	22	14
BTA(4) 25:1	50	<0.5	>25	10	>450	>4	>100
AcA 25:1	150	60	2	0	110	44	2.5
SA 15:1	120	60	>25	0.25	>1100	>550	2

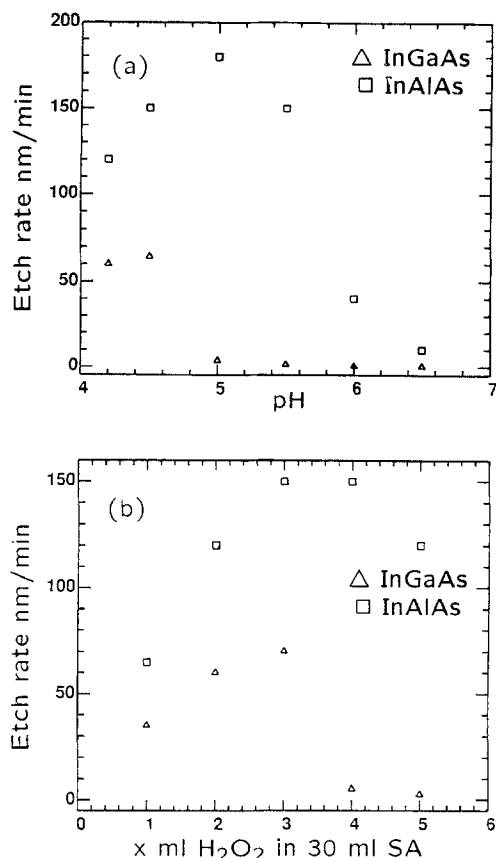


Fig. 1. $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ (\square) and $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ (Δ) etch rate as a function of (a) pH and (b) added m.l. peroxide in 30 ml SA.

$\langle 110 \rangle$ and $\langle \bar{1}\bar{1}0 \rangle$ directions as a result of undercutting of the AlAs layer at an initial breakthrough point.

All the etchants described thus far have also been used in device processing^{5,8,9}. It is found that they do not noticeably affect standard positive photoresists, nor do they attack Cr/Au contact metallization patterns and can thus be used in self-aligned structures.

Discussion

A clear improvement in $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ to AlAs selectivity can be seen in going from oxalic to malonic to succinic acid. The mechanism for this improvement is not well understood. However, it should be noted that for the oxalic acid-based etchant, the $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ to AlAs selectivity improved dramatically, and the $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etch rate dropped, by buffering through the addition of citric acid. Since these etchants are typically used in the pH 4 to pH 7 range in order to obtain selective behavior, the improvement in selectivity with the use of malonic and succinic acid seems due in part to the fact that they are better buffered as such, as their pK_1 values are closer to the pH values used in the selective etchants. Table I also gives the pK values of the acids used¹².

The succinic acid-based etch, having the highest selectivity, is most useful for device fabrication, and is relatively insensitive to pH and peroxide concentration for etching $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$. However, for $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ the etch rate drops dramatically at a pH of 5 and higher, and a peroxide concentration of 30:3 SA: H_2O_2 and larger. Therefore, if $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ is to be etched, the pH and peroxide concentration must be kept low. On the other hand, by using an SA: H_2O_2 solution at high peroxide concentration and at a pH of about, 5.5, $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ can be etched selectively over $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$, with a selectivity better than 50:1. The best selectivity of $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ to $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etching rates has been obtained with dimethylsuccinic acid, *i.e.*, better than 125:1 as shown in Table I.

In an effort to understand some of the reaction mechanisms, a calculation was performed of the concentrations

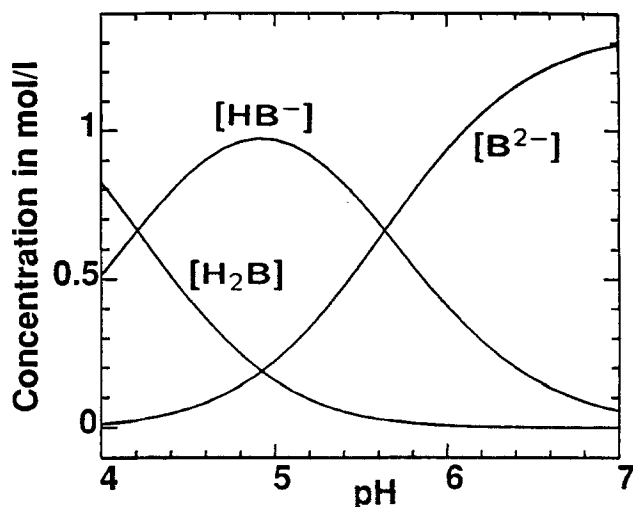


Fig. 2. Calculated concentration profiles as a function of pH for the succinic acid based etchant. H_2B represents succinic acid.

$[\text{H}_2\text{B}]$, $[\text{HB}^-]$, $[\text{B}^{2-}]$ as a function of pH, where H_2B represents succinic acid. The result is shown in Fig. 2, which can be compared with Fig. 1 which showed the $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ and $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etch rates as a function of pH. The similarity between the $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ etch rate and the $[\text{HB}^-]$ profile implies that the etch-rate limiting reaction step is likely to be a reaction with the HB^- ion. Note that this means that the maximum etch rate is obtained at $\text{pH} = (\text{pK}_1 + \text{pK}_2)/2$ for which the HB^- ion concentration reaches a maximum. For the $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etch rate, however, no such similarity occurs. The sudden drop in the etch rate of the $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ for higher peroxide concentrations, suggests that the etching of the Al compound material becomes inhibited due to passivation by Al oxidation products as a result of the higher Al oxidation rates. The sudden drop in $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etch rate with increasing pH can then be explained to be due to the lower solubility of Al oxidation products with increasing pH. (The solubility of aluminum oxidation products decreases up to a pH of ≈ 5 , after which the solubility rises again¹².)

It should be stressed that the other acids tested do not necessarily follow exactly the same pattern in etch rate as a function of pH. For example, the $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ and $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etch rate in maleic acid ($(\text{pK}_1 + \text{pK}_2)/2 \approx 4$) peak at a pH of 3 and 2, respectively, indicating that a reaction with HB^- is not the rate limiting step in this etch. Other observations that can be made follow.

1. Succinic acid is the only acid of those tested that can be made to etch $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ and maintain high selectivity to AlAs. The pH must be less than 4.5 to achieve this, and the etchant cannot stop on very thin (5 m.l.) AlAs stop layers under these conditions.

2. Variations to succinic acid, like the addition of a methyl group or the introduction of a double bond, result in a significantly lower $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ etch rate.

3. Acids with a small pK_1 (oxalic, malonic, maleic) have lower selectivity.

4. The tri- and tetracarboxylic acids tested have good selectivity, even at very low pH. The monocarboxylic acid tested (acetic) has low selectivity.

5. Addition of an hydroxyl group results in lower, or loss of, selectivity (citric, L-malic, tartaric).

Conclusion

A technique for the selective etching of InGaAlAs compounds with the use of a strained AlAs layer as an etch stop has been described. Applications for these etchants can be found in the fabrication of a variety of novel, high-performance heterostructure devices, in particular, applications toward devices that include resonant tunneling diodes that have strained AlAs tunnel barriers seem promising. For purposes of a better understanding of the reaction mechan-

isms, a systematic study of the etch rate dependence on pH, and acid and peroxide concentration is needed. It was shown that for the succinic acid the likely $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ etch-rate limiting step is a reaction with the once dissociated succinic acid molecule, whereas for $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$, Al oxidation products can limit or prevent the etching. However, a more general picture of why certain acids are capable of etching, e.g., $\text{In}_{0.53}\text{Ga}_{0.47}\text{As}$ and not $\text{In}_{0.52}\text{Al}_{0.48}\text{As}$ is lacking.

Acknowledgments

This work has been funded in part by the Joint Services Electronics Program through the MIT Research Laboratory of Electronics Contract No. DAAL 03-89-C-0001, by the Army Research Office through Contract No. DAAL 03-91-G-0051, and by the National Science Foundation through Grant No. ECS 9008485.

Manuscript submitted Sept. 17, 1991; revised manuscript received April 3, 1992.

Massachusetts Institute of Technology assisted in meeting the publication costs of this article.

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Etch Stop Barriers in Silicon Produced by Ion Implantation of Electrically Non-Active Species

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ABSTRACT

Silicon layers implanted with silicon, germanium, and carbon ions at doses between $1\text{E}14$ and $3\text{E}16$ ions/cm² and energies between 35 and 200 keV were tested as etch stop barriers in an ethylenediamine-pyrocatechol-water solution. The decrease in the etch stop effect with annealing temperature was measured. The results obtained indicate that the effectiveness of the etch stop is influenced by both the implantation damage and the chemical interaction between the implanted ions and the defective crystal.

Thin single-crystal silicon layers have found a variety of applications in electronic and micromechanical devices, and different techniques have been developed in order to obtain them¹. The production of submicron silicon-on-insulator (SOI) for electronic devices is currently an active field of development due to the technological relevance of this material. At present, separation by implantation with oxygen (SIMOX) appears to be the best technique available for whole wafers. However, the high defect densities, caused by the necessary implantation doses ($>1\text{E}18$ ions/cm²), the poor quality of the oxide, and the difficulties involved in using larger wafer diameters have encouraged work on other approaches.

A promising alternative is direct bonding of oxidized wafers and subsequent thinning of the seed wafer to the desired thickness (see Ref. 2 for a recent overview). So far, no convincing solution has been found for the thinning step. Efforts currently being made to develop an appropriate polish-back process have shown progress, and a thickness of 500 nm with good thickness uniformity has been

reported³. Nevertheless, the requirement of thickness uniformity could pose limitations to this method, thus making it difficult to reach the ultimate desired thickness in the range of tens of nanometers over the whole wafer by polishing.

Mechanically or chemically thinning the seed wafer to a few microns and then chemically etching back the rest (BE-SOI: bonding-and-etchback-SOI) is another approach. A buried etch stop in the seed wafer preserves the thin silicon layer close to the bonding interface. The surface roughness caused by the mechanical thinning is compensated and a good thickness uniformity can be obtained. Basically, three different types of etch stops have been proposed based on their reduced etching as compared to silicon in specific etchants [mainly anisotropic alkaline etchants like KOH or ethylenediamine-pyrocatechol (EDP)]: highly p-doped silicon, a continuous layer of a silicon compound and silicon implanted with different electrically non-active species.

Silicon heavily doped with boron ($>10^{19}$ cm⁻³) is a very effective etch stop⁴. The main disadvantage of this approach is that the deposited or implanted boron diffuses easily and affects the doping of the surrounding silicon. Additional steps are necessary if the doping is not expressly desired: the growth of an additional silicon epilayer, an MeV ion implantation or double ion implantations have been reported^{5,6}. Also, these steps may reduce the

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