

Selective Wet and Dry Etching of NiO over β -Ga₂O₃

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ABSTRACT

Patterning of NiO/Ga₂O₃ heterojunctions requires development of selective wet and dry etch processes. Solutions of 1:4 HNO₃:H₂O exhibited measurable etch rates for NiO above 40 °C and activation energy for wet etching of 172.9 kJ.mol⁻¹ (41.3 kCal.mol⁻¹, 1.8 eV/atom), which is firmly in the reaction-limited regime. The selectivity over β -Ga₂O₃ was infinite for temperatures up to 55°C. The strong negative enthalpy for producing the etch product Ga(OH)₃ suggests HNO₃-based wet etching of NiO occurs via formation and dissolution of hydroxides. For dry etching, Cl₂/Ar Inductively Coupled Plasmas produced etch rates for NiO up to 800 Å.min⁻¹, with maximum selectivities of < 1 over β -Ga₂O₃. The ion energy threshold for initiation of etching of NiO was ~55 eV and the etch mechanism was ion-driven, as determined the linear dependence of etch rate on the square root of ion energy incident on the surface.

Introduction

The absence of conventional p-type dopants for β -Ga₂O₃ has intensified interest in using p-type oxides in heterojunctions with n-type Ga₂O₃. The most common of these has been NiO⁽¹⁻¹⁶⁾, in which the p-type doping level can be controlled by the O₂ partial pressure during sputter deposition. A variety of impressive device demonstrations involving p-NiO/n-Ga₂O₃ heterojunctions⁽¹⁻¹⁶⁾. The NiO can also be used as an edge termination material and this requires the ability to pattern the NiO⁽¹⁷⁾. The integration to devices requires the development of the high- resolution pattern transfer processes. To develop a fully optimized pattern transfer process for NiO, both wet chemical etching and dry etching processes are needed⁽¹⁷⁻¹⁹⁾. The former typically has high etch rate, low damage and excellent selectivity, but is generally isotropic (poor directionality) and etch rates are sensitive on temperature and light irradiation. By contrast, dry etching has excellent anisotropy (directionality) but low etch rate, high ion damage and poor selectivity. To this point there has been little investigation of these processes for NiO on Ga₂O₃⁽²⁰⁻²²⁾.

In terms of wet etching, oxidizing solutions are known to create a passivating oxide layer on Ni, which prevents further etching unless the initially present NiO and constantly forming oxide, can be dissolved. The dissolution of the oxide is the basis for Ni etching using H₂O₂ (for the oxidation of Ni) and HF to respectively create and dissolve the NiO⁽²²⁻²⁹⁾. It is less obvious how to select wet etch solutions than dry etch chemistries, which depend on etch product volatility⁽³⁰⁻³²⁾. An alternative oxidizer is nitric acid and dissolver is HCl^(33,34). Ga₂O₃ can be slowly etched ($< 1\text{nm}\cdot\text{min}^{-1}$) in HF at room temperature⁽³⁴⁾. Metal-assisted chemical etching at $< 2\text{nm}\cdot\text{min}^{-1}$ was achieved in HF/ K₂S₂O₈ solutions using Pt as a metal catalyst⁽³³⁾. At temperatures $> 100^\circ\text{C}$, HNO₃,²⁸ H₂SO₄,³⁰ and H₃PO₄³⁰ are effective reactant-limited etchants, with rates up to $0.15\text{ }\mu\text{m}\cdot\text{min}^{-1}$ at 200°C ^{27,28}. In hot KOH solutions, photo-enhanced chemical etching with ultraviolet illumination increases the etch rate to 30 nm min^{-1} for (010) plane and 150 nm min^{-1} for (201) orientation⁽²⁹⁾.

Generally, dry etch rates of NiO are relatively slow under conventional dry etching conditions but high-density plasmas can produce higher rates. Inductively Coupled Plasmas (ICP) in Cl₂/Ar or BCl₃/Ar chemistries produces rates of $\sim 100 \text{ nm} \cdot \text{min}^{-1}$ ⁽¹⁷⁾. Single crystal Ga₂O₃ is also etched by these chlorine-based discharges ^(20,21,32), so it is expected that it will be difficult to achieve high selectivity for dry etching NiO over Ga₂O₃.

In this paper we report the wet and dry etching of sputtered NiO and the resultant selectivity to β -Ga₂O₃. A wet solution based on HNO₃ produced reaction-limited etching of NiO, with complete selectivity over β -Ga₂O₃. The influence of the ion energy and density on the dry etch rate of NiO in Cl₂/Ar plasmas was examined by varying at various RF cathode and ICP source powers. There was a threshold ion energy of $\sim 55 \text{ eV}$ for initiation of dry etching ⁽³⁵⁻³⁷⁾, and the etch rates increased monotonically with both source and chuck powers.

Experimental

NiO was deposited by magnetron sputtering on glass slides at 3mTorr and 100W of 13.56 MHz power using two targets to achieve a deposition rate around $0.2 \text{ \AA} \cdot \text{sec}^{-1}$. The Ar/O₂ ratio was used to control the doping in the NiO in the range $5\text{-}6 \times 10^{18} \text{ cm}^{-3}$, with mobility $< 1 \text{ cm}^2 \cdot \text{V}^{-1} \text{ s}^{-1}$. The β -Ga₂O₃ was (100) bulk, Sn-doped substrates.

Glass slides with 90 nm thick sputtered NiO with surface patterns were used for all the wet etch conditions. The choice of etchant was guided by a literature search, which suggested that nitric-acid-based solutions might etch NiO. 50mL of Ni Etchant TFB was heated without dilution in a beaker to 40, 45, 50, and 55°C respectively. This etchant has the composition 1:4 HNO₃ H₂O, with addition of a proprietary surfactant and was obtained from Transcene Company, Inc. Samples were first cleaned with 10% HCl and treated by an ozone cleaner for 15 minutes. Afterward, four samples were submerged under the etchant surface at each temperature until the NiO pattern

visually disappeared to determine the maximum available etching time. Subsequently, four more samples were etched with half of the maximum available etching time. After the experiment, the Tencor profilometer measurements were used to calculate the etch rate.

190 nm thick sputtered NiO on glass slides with patterned PR1818 photoresist were used for all the dry etch experiments in a PlasmaTherm 790 reactor. We chose the plasma chemistry based on the expected higher volatility of nickel chloride etch products compared to fluorine or any other etchant. Discharges with 15 sccm of Chlorine and 5 sccm of Argon at a fixed pressure of 5mTorr were used to etch all the samples. Two sets of conditions were applied with regards to the ICP power, RF power and etching time: 1. Fixed 200W RF power with 100, 200, 600, and 800W ICP power; etched for 2, 1.5, 1, and 1 minute respectively. 2. Fixed 400W ICP power with 50, 100, 200, and 400W RF power; etched for 1.5, 1.5, 1, and 1 minute respectively. After the dry etch process, 5 minutes of oxygen reactive ion etching was performed for the following three conditions due to severe carbonization of the photoresist: RF 200W with ICP 600W, RF 200W with ICP 800W, and RF 400W with ICP 400W. The photoresist could be removed with the help of an acetone spray gun. The Tencor profilometer was then used to calculate the etch depth and corresponding etch rate.

Results and Discussion

Figure 1 shows the etch rates of NiO as a function of temperature in the $\text{HNO}_3:\text{H}_2\text{O}$ solution. There was no etching of the Ga_2O_3 under these same conditions, so the etch selectivity was infinite since the selectivity is the NiO etch rate divided by the Ga_2O_3 etch rate, with the latter being zero. An Arrhenius plot of NiO etch rate in 1:4 $\text{HNO}_3:\text{H}_2\text{O}$ is shown in Figure 2. The large activation energy of $41.3 \text{ kCal.mol}^{-1}$ ($172.9 \text{ kJ.mol}^{-1}$ or 1.8 eV/atom), indicates the etching is reactant-limited⁽²⁷⁻²⁹⁾. This means the rate-limiting step is reaction of the etchant with the NiO surface, rather than diffusion of the etchant species through the wet etch solution. The etching also

followed the general characteristics of reaction-limited etching, namely that the etch depth was linearly dependent on etch time, the rate was independent of stirring or agitation of the liquid etchant and the rate was exponentially dependent on temperature with activation energy > 6 kCal.mol⁻¹. This type of etching is preferred for manufacturing in contrast to diffusion-limited etching where the rates are strongly dependent on stirring and agitation rate of the solution ⁽²⁷⁻²⁹⁾.

It has been suggested previously that the etch mechanism of NiO involves formation of the hydroxide [Ga(OH)₄]⁻ ^(27-29,33,34). The calculation of standard enthalpy of reaction ($\Delta H_{\text{rxn}}^\ominus$) from standard heats of formation ($\Delta H_f^\ominus$) for the reaction $\text{Ga}_2\text{O}_3 + 2\text{OH}^- + 3\text{H}_2\text{O} \rightarrow 2[\text{Ga}(\text{OH})_4]^-$ can be obtained using the respective values for the components, ie. $\Delta H_f^\ominus\{\text{Ga}(\text{OH})_4^-\} = -289.82$ kcal/mol = -1213.42 kJ/mol, $\Delta H_f^\ominus\{\text{H}_2\text{O}\} = -285.8$ kJ/mol, $\Delta H_f^\ominus\{\text{OH}^-\} = -139.056$ kJ/mol, $\Delta H_f^\ominus\{\text{Ga}_2\text{O}_3\} = -1089.095$ kJ/mol. Then $\Delta H_{\text{rxn}}^\ominus = \sum \Delta H_f^\ominus\{\text{products}\} - \sum \Delta H_f^\ominus\{\text{reactants}\} = (2) \Delta H_f^\ominus\{\text{Ga}(\text{OH})_4^-\} - (3) \Delta H_f^\ominus\{\text{H}_2\text{O}\} - (2) \Delta H_f^\ominus\{\text{OH}^-\} - (1) \Delta H_f^\ominus\{\text{Ga}_2\text{O}_3\} = (2)(-1213.42) - (3)(-285.8) - (2)(-139.056) - (1)(-1089.095) = -202.233$ kJ/mol ⁽³⁸⁻⁴⁰⁾. With this strong negative enthalpy, HNO₃ based wet etching of NiO is consistent with it occurring via formation and dissolution of hydroxides. A caveat is that negative enthalpy of the NiO reaction would not explain the lack of etching of the Ga₂O₃ in the same solution, and thus the enthalpy of reaction can only be used as a possible indicator of positive etch rates.

Turning to the dry etching, Figure 3 shows the NiO and Ga₂O₃ etch rates in the Cl₂/Ar ICP discharges as a function of either (top) rf chuck power at fixed source power or (bottom) ICP source power at fixed rf chuck power. The etch rates increase monotonically with both powers. The former controls the self-bias on the sample electrode and hence the incident positive ion energies incident on the sample surface, while the source power controls the ion density. Note that the etch rate of NiO is lower than that of Ga₂O₃ under all conditions investigated.

Since NiCl_x etch products have relatively low volatilities, it would be expected that the

etch mechanism is ion-driven. For such an ion assisted etching mechanism, the etch rate (ER) is given by ⁽³⁵⁻³⁷⁾

$$ER = (J_+ Y_{sat} \Theta) / N_t$$

where Θ is the surface coverage by reactive neutral species ($0 < \Theta < 1$), J_+ is the positive ion flux, Y_{sat} is the ion assisted chemical etch yield on a saturated surface and N_t is the atomic density ⁽³⁷⁾. Then it follows that

$$\Theta = 1 / \left(1 + \beta J_+ \frac{Y_{sat}}{S_n J_n} \right)$$

where J_n is the flux of reactive neutral species S_n is the reaction probability, β is the number of reactive atoms desorbed per reaction product and Y_{sat} is the ion-assisted chemical etch yield on a saturated surface, given by (in the ion energy range for plasma etching) ⁽³⁵⁻³⁷⁾

$$Y_{sat} = A_{sat} (\sqrt{E} - \sqrt{E_{th}})$$

A_{sat} is a proportionality constant that depends on the specific plasma-material combination, E is the ion energy, and E_{th} is the threshold energy for initiation of etching. The etch rate should therefore increase linearly with $\sqrt{E}$ provided etching is ion-flux limited ⁽³⁵⁻³⁷⁾, i.e. $\beta J_+ Y_{sat} / S_n J_n \ll$

1

Then

$$ER = J_+ Y_{sat} / N_t$$

$$ER = (J_+ A_{sat} (\sqrt{E} - \sqrt{E_{th}})) / N_t$$

Thus a plot of etch rate versus the square root of ion energy should yield a straight line whose intercept is the threshold ion energy ⁽³⁷⁾. In general, an increase in ICP source power leads to an increase of both the reactive neutral density (through an increase of the dissociation degree of the reactive molecular species) and the positive ion density. Moreover, the ion energy which is given by the sum of DC self-bias voltage and sheath potential (about 25 V for ICP sources) also depends

on the ICP source power ⁽⁴¹⁾. Figure 4 shows a plot of etch rate versus this approximate ion energy. Above some threshold, the etch rate increases linearly with E , with E_{th} being ~ 55 eV for Cl_2/Ar in our system. These results clearly indicate that etching is driven by the same ion-assisted mechanism over the whole range of ion energies investigated. This specific energy dependence corresponds to the ion-flux-limited regime.

While our simple model suggests that the etch rate is independent of the reactive neutral flux in the ion flux limited regime, a decrease of the ion energy with increasing ICP source power would lead to a decrease of the etch rate. One therefore concludes that the increase of the NiO etch rates with ICP source power presented in Figure 3(bottom) essentially results from an increase of the positive ion density.

The selectivity for etching Ga_2O_3 relative to NiO is defined as the etch rate of the former divided by the etch rate of the latter. Selectivities for dry etching of Ga_2O_3 over NiO are shown in Figure 5 as a function of either (top) rf power or (bottom) ICP source power. A rule of thumb in industry is that a minimum selectivity of 10 is needed and the results are actually much less than this under all conditions and are <1 for NiO over Ga_2O_3 . ^(41,42). Therefore, a likely approach is to partially remove the NiO using dry etching and finish with the completely selective wet etch process. An interesting sidelight would be the effect of an initial dry etch on the subsequent wet rate. The fact that ion-induced damage to the NiO would likely facilitate the subsequent wet rate, but from a practical viewpoint, the NiO thickness is so small^(43,44) that it may not be an easily observed experimentally.

Summary and Conclusions

The use of p-type oxides with n-type Ga_2O_3 shows promising device results. The development of selective patterning processes generally requires having both anisotropic plasma etching and damage-free wet etch approaches, which can be used in combination. The results

presented here show that dilute HNO_3 provides selective removal of NiO from single crystal Ga_2O_3 , while dry etching in Cl_2/Ar has selectivity <1 for NiO over Ga_2O_3 . The wet etch follows a general procedure of the surface oxidizing in solution followed by the dissolving of the produced hydroxide. We determined the activation energy for the HNO_3 wet process and found it to be reaction-limited. Similarly, the dry etch process was ion-driven, as expected from the low volatility of the etch products.

Acknowledgments

The work at UF was performed as part of Interaction of Ionizing Radiation with Matter University Research Alliance (IIRM-URA), sponsored by the Department of the Defense, Defense Threat Reduction Agency under award HDTRA1-20-2-0002. The content of the information does not necessarily reflect the position or the policy of the federal government, and no official endorsement should be inferred. The work at UF was also supported by NSF DMR 1856662 (James Edgar).

Data Availability

The data that supports the findings of this study are available within the article and its supplementary material.

Declarations

The authors have no conflicts to disclose.

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Figure Captions

Figure 1. Wet etch rate of NiO in 1:4 HNO₃:H₂O as a function of solution temperature.

Figure 2. Arrhenius plot of NiO etch rate in 1:4 HNO₃:H₂O. The activation energy indicates the etching is reaction-limited.

Figure 3. ICP dry etch rates of NiO and Ga₂O₃ in 15Cl₂/5 Ar discharges as a function of (top) rf power at a fixed ICP source power of 400W (bottom) ICP source power at a fixed rf power of 200W. The dc self-bias is indicated in both cases.

Figure 4. NiO etch rate plotted as a function of $\sqrt{25 + \text{self-bias}}$, indicating the etching is ion-driven.

Figure 5. Selectivity for dry etching of Ga₂O₃ over NiO in 15Cl₂/5 Ar discharges as a function of either (top) rf power or (bottom) ICP source power.









