Characterization of GaAs/Al_xGa_{1-x}As selective reactive ion etching in $SiCl_4$/SiF_4 plasmas

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Selective reactive ion etching of GaAs over Al_xGa_{1-x}As using SiF_4/$SiCl_4$ gas mixtures is studied. Etch rates of GaAs and Al_xGa_{1-x}As are measured versus pressure, etch gas ratio, and plasma self-bias voltage. A selectivity ratio of 500:1 has been obtained for GaAs over $Al_{0.35}Ga_{0.65}$As at a gas composition ratio of $SiCl_4/SiF_4 = 0.25$, a pressure of 60 mTorr, and a self-bias voltage of -60 V. The etch stop mechanism is studied using Auger electron spectroscopy. Auger surface scan spectra indicate a high concentration of fluorine at the surface of etched AlGaAs samples suggesting the formation of an AlF_3 etch stop layer.[1] It is also shown that the AlF_3 layer is easily removed with a very short dip in HCl/H_2O or NH_4OH/H_2O. Hall effect measurements at 300 and 77 K are made on etched GaAs/$Al_{0.3}Ga_{0.7}$As modulation doped heterostructures to determine the effect of low-power etching on two-dimensional electron gas (2-DEG) sheet concentrations and carrier mobility. Slight reduction in both 2-DEG sheet carrier concentration and mobility are observed after the RIE process. This may be an indication of ion-beam induced damage to the AlGaAs/GaAs heterostructure. Finally, the effects of extended etching on the dc and microwave device characteristics of short gate length GaAs/AlGaAs/InGaAs MODFETs are reported.

I. INTRODUCTION

Selective removal of GaAs over AlGaAs is essential for defining the gate recess in GaAs/AlGaAs and GaAs/AlGaAs/InGaAs modulation-doped field effect transistors (MODFETs). It has been shown[2] that performing the gate recess using selective reactive ion etching (SRIE) produces more uniform dc device characteristics in comparison to wet chemical etching. The improvement in dc device characteristics is attributed to the high degree of selectivity attainable with RIE, and is necessary if high-performance MODFET-based integrated circuits are to be obtained. In our previous work,[2] we reported the development of a selective etch process which employs $SiCl_4$/SiF_4 gas mixtures. Preliminary results were reported which showed that devices fabricated with the dry-etch gate recess process produced significantly more uniform threshold voltages than devices fabricated with the wet-etched gate recess method. Microwave characteristics of wet versus dry etched devices were nearly identical indicating that no deleterious effects were generated by the SRIE process.

The $SiCl_4$/SiF_4 etch chemistry has several advantages over prior approaches,[1,3-6] which rely on CCl_2F_2-based plasmas. Because the chlorine and fluorine are contained in separate gases in the $SiCl_4$/SiF_4 mixture, higher selectivities can be achieved by controling the process gas ratios. Also, because the $SiCl_4$/SiF_4 gas does not contain fluorocarbons, there is no polymer formation during the etch process.

In this paper, we present further results of our work on characterization of the $SiCl_4$/SiF_4 selective etch. Etch selectivity is studied as a function of aluminum mole fraction, $SiCl_4$ to SiF_4 gas ratio, chamber pressure, and plasma self-bias voltage. Selectivity of greater than 500:1 is demonstrated at optimal etch conditions for high Al mole fractions. The etch stop mechanism is studied using Auger electron spectroscopy (AES). Results of Auger spectral scans after exposure to the selective etch and subsequent wet chemical etching are presented and discussed. Hall effect measurements on etched GaAs/$Al_{0.3}Ga_{0.7}$As modulation-doped heterostructures are also presented and show decreases in the two-dimensional electron gas (2-DEG) sheet concentration and mobility at both 300 and 77 K. Finally, dc and microwave measurement results on overetched short gate-length pseudomorphic GaAs/AlGaAs/InGaAs MODFETs are reported.

II. EXPERIMENTAL PROCEDURE

The samples used in this study were grown by molecular-beam epitaxy. The etch characterization material consists of 1 μm thick layers of GaAs and varying compositions of AlGaAs doped with silicon ($n = 5\times10^{17}$ cm^{-3}) grown on semi-insulating (100) GaAs substrates. Hall mobility measurements are made on a GaAs/AlGaAs MODFET structure consisting of a 20 nm n^+ GaAs cap, a 35 nm n^+ $Al_{0.3}Ga_{0.7}$As donor layer, a 4 nm undoped $Al_{0.3}Ga_{0.7}$As spacer layer, and a 1 μm GaAs buffer layer. Auger samples consist of 1 μm layers of undoped $Al_{0.3}Ga_{0.7}$As grown on semi-insulating (100) GaAs substrates.

Reactive ion etching is performed in a Plasma Technology RIE system with a 30-cm diam chamber. A load-locked nitrogen-purged glove box encloses the chamber to prevent atmospheric contaminants from reacting with residual gases. The 17-cm diam cathode is made of alumina-coated aluminum, rf powered at 13.56 MHz and is covered with a 1/4-in. thick quartz plate. The cathode and anode are spaced 5 cm apart and watercooled to maintain a nominal cathode temperature of 24 °C. For all experiments, the chamber is initially pumped to about 100 mTorr by a Roots blower/mechanical pump combination. A turbomolecular pump is then used to pump the system to a base pressure of 5×10^{-6} Torr. During etching only the mechanical pump is used to pump process gases.

Etch characterization data was obtained by simultaneously etching GaAs and $Al_xGa_{1-x}As$ samples and comparing the resulting etch rates. The selective etch was characterized over a wide range of process conditions. $SiCl_4/SiF_4$ mixtures of 1:9, 1:4, 3:7, and 1:1, plasma self-bias voltage levels of -60, -100, and -140 V, and chamber pressures of 30 and 60 mTorr were all investigated. Etch rates and the corresponding selectivity ratios were determined by measuring etch steps on a Tencor Instruments Alpha-Step 100.

III. RESULTS AND DISCUSSION

A. Etch characterization

Figure 1 shows the results obtained from etching GaAs and $Al_xGa_{1-x}As$ samples at 60 mTorr, 20% $SiCl_4$, for three different dc bias levels. It is seen from examination of Fig. 1 that the AlGaAs etch rate is dependent on both aluminum mole fraction and self-bias voltage. The figure also shows that the GaAs etch rate is not as sensitive to changes in self-bias voltage as $Al_xGa_{1-x}As$ etch rates. $Al_xGa_{1-x}As$ etch rates decrease by 70%–80% with a dc bias reduction from -140 to -60 V, while GaAs etch rates decrease only by 16% (i.e., 370 nm/min to 309 nm/min). It is known from previous studies[1] that the formation of AlF_3 provides the etch stopping mechanism in plasmas containing fluorine. It is also known that AlGaAs and GaAs etch at about the same rate in pure $SiCl_4$ plasmas.[7] Thus, it can be concluded from the data in Fig. 1, that the AlF_3 etch inhibiting layer forms faster when more aluminum is present in the substrate, which explains the reduction in etch rate with increasing aluminum mole fraction. These results suggest, from the large shift in AlGaAs etch rate with changes in self-bias voltage, that there is competition between the formation of AlF_3 and the chemical etching of AlGaAs by $SiCl_4$. At higher self-bias voltages, the AlF_3 is sputtered at a faster rate, decreasing the effectiveness of the etch stop mechanism, thereby allowing the chemical etching of AlGaAs to increase. A plot of etch rate versus material composition at 30 mTorr (not shown here) display the same trends as that in Fig. 1 but with slightly lower etch rates. The etch rate of GaAs at 30 mTorr is about 35% lower than the 60 mTorr rate and the etch rate of AlGaAs samples etched at 30 mTorr is about 26% lower than samples etched at 60 mTorr. Thus, the selectivity ratio at 30 mTorr is slightly less than 60 mTorr selectivity ratio.

Figure 2 shows selectivity versus aluminum mole fraction and is derived by dividing the GaAs etch rates by the $Al_xGa_{1-x}As$ etch rates given in Fig. 1. Figure 2 shows that selectivity improves with higher aluminum mole fraction and lower dc bias. The -60-V dc bias curve produces a maximum selectivity of 490:1 derived from a GaAs etch rate of 309 nm/min compared to an $Al_{0.35}Ga_{0.65}As$ etch rate of 0.63 nm/min at the conditions given (20% $SiCl_4$, 60 mTorr). Examination of the etched surfaces of these samples using both an optical microscope and scanning electron microscope show smooth AlGaAs surfaces under these conditions, while GaAs surfaces appear slightly rough. For the gate recess etch, the GaAs surface morphology is not important since all the GaAs must be etched away to expose the AlGaAs surface for Schottky gate contact.

Figure 3 shows selectivity versus etch gas ratio at a pressure of 60 mTorr for two different mole fractions of AlGaAs ($x = 0.07$ and 0.35). A constant self-bias voltage of -60 V was maintained by changing the rf power level accordingly for the different gas ratios. A constant gas flow rate of 10 sccm was also maintained for all experiments. As expected, the figure shows decreasing selectivity ratios with increasing $SiCl_4$ in the gas mixture. Selectivity is also seen to decrease with reduction in aluminum mole fraction, which agrees with the data presented in Fig. 2.

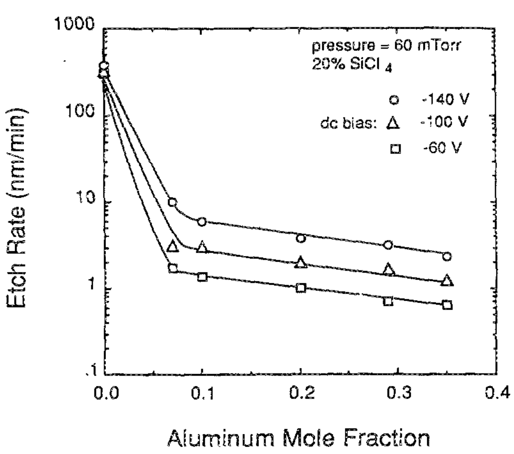

FIG. 1. Etch rate vs aluminum mole fraction for different dc bias, at 60 mTorr, 20% $SiCl_4$.

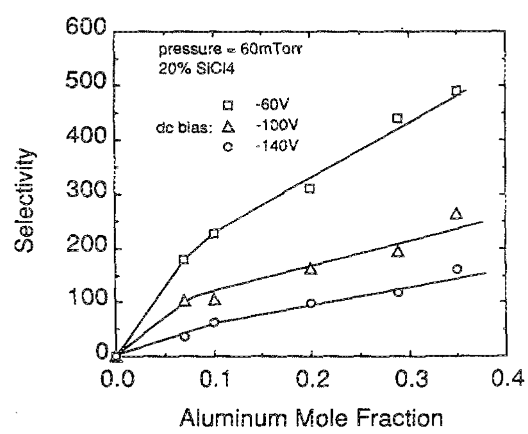

FIG. 2. Selectivity vs aluminum mole fraction for different dc bias, at 60 mTorr, 20% $SiCl_4$.

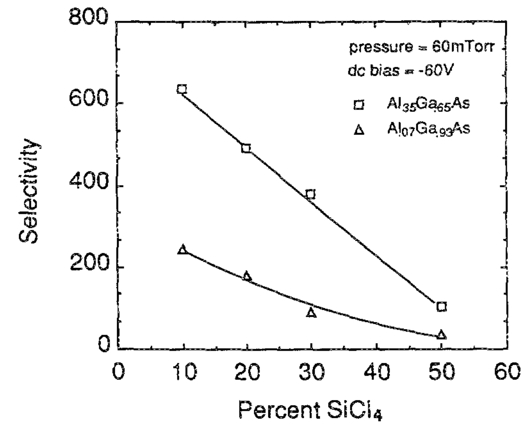

FIG. 3. Selectivity vs percentage of $SiCl_4$ for two different mole fractions of AlGaAs, at 60 mTorr, − 60-V dc bias.

B. Surface analysis

To investigate the etch mechanism, and check for possible surface contamination, AES analysis is performed. Figure 4 shows AES spectra of $Al_{0.3}Ga_{0.7}As$ samples processed under varying conditions. Sample (1) is a spectra of $Al_{0.3}Ga_{0.7}As$ after etching in pure $SiCl_4$ at 30 mTorr, − 60 V dc bias for 2 min. Large oxygen and carbon peaks are detected on the surface due to environmental contamination. Residual silicon and chlorine are also observed on the surface and are attributed to low energy implantation and gallium halide incorporation in the etch stop layer during the etch process. Sample (2) is a spectra of an $Al_{0.3}Ga_{0.7}As$ sample which has been etched in $SiCl_4/SiF_4$ (1:4), at 60 mTorr, − 60 V, for 2 min. These spectra indicate a large fluorine concentration on the sample surface, which is attributed to the formation of the AlF_3 etch stop layer. The presence of the etch stop layer is further supported by the reduction in the gallium and arsenic peak heights in comparison to sample (1). Auger electron escape depths are of the order of 2–3 nm and thus, due to the presence of the Ga and As peaks along with the F and Al peaks, it can be concluded that the AlF_3 layer is not thicker than 3 nm. The aluminum peak appears at a kinetic energy which suggests that both metallic and oxidized aluminum are present on the sample surface. Sample (3) has been etched simultaneously with sample (2) but followed by exposure to a low power (5 W, − 60 V) oxygen plasma. The O_2 plasma does not seem to alter the magnitude of the spectra peaks in comparison to the peaks of sample (2) and thus it seems likely that the O_2 plasma does not aid in removing the etch stop layer or any other surface contamination. Sample (4) has been processed simultaneously with samples (2) and (3) but then followed by a wet-chemical etch in $HCl:H_2O$ (1:2) for 30 s. These spectra show that the fluorine peak disappears after the wet chemical etch. The Ga and As peaks increase to control sample values (not shown) and thus it is concluded that the HCl dip removes the AlF_3 layer and restores the stoichiometry of the AlGaAs surface. Similar results have been observed for wet chemical etching in $NH_4OH:H_2O$(1:2) for 30 s (spectra not shown).

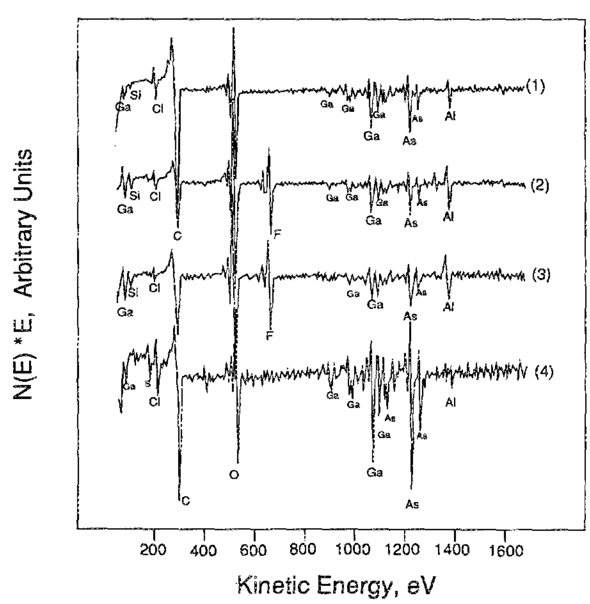

FIG. 4. Auger spectra of $Al_{0.3}Ga_{0.7}As$ (1) etched in pure $SiCl_4$, (2) etched in $SiCl_4/SiF_4$, (3) same as 2 followed by O_2 plasma, (4) same as 3 followed by $HCl:H_2O$ wet etch.

C. Electrical characterization

In order to determine the effects of the low-power selective etch on the thin AlGaAs donor layer and underlying channel, Hall effect measurements are made on a GaAs/$Al_{0.3}Ga_{0.7}As$ modulation-doped heterostructure sample. After selectively wet etching the GaAs cap layer, the 2-DEG sheet concentration and channel electron mobility at temperatures of 300 and 77 K are determined by Hall measurements. The sample is then etched in $SiCl_4/SiF_4$ (1:4), 60 mTorr, − 60 V dc bias, for 1 min and the Hall measurements repeated. The etch process is repeated three times with sheet concentration and mobility measured after each etch. For the etch process given, the etch rate of $Al_{0.3}Ga_{0.7}As$ is 0.7 nm/min. An etch initiation delay time of about 30 s results in a total of 0.35 nm of $Al_{0.3}Ga_{0.7}As$ being etched during the 1 min process. For the 3 min etch measurement a total of about 1 nm has been etched from the 35 nm $Al_{0.3}Ga_{0.7}As$ donor layer. Thus, it is assumed that all changes in the Hall measurements are due to low energy ion beam damage and not etching of the donor layer. The results of the Hall study described above are shown in Fig. 5. It is seen from this figure that electron mobilities at 300 K are only slightly changed (3.2%) from the control value after 3 min of etching. Sheet concentration at 300 K decreases by 17% after 1 min of etching and decreases a total of 22% from the control value through three 1-min etch processes. Low-temperature (77 K) measurements indicate a 32% decrease in mobility and 10.4% decrease in sheet concentration from the control values through three 1-min etch processes. Most of the decrease at low temperature, like the room-temperature data, occurs in the first minute of etching. Thus, it can be concluded from these data that 1 min of etching does

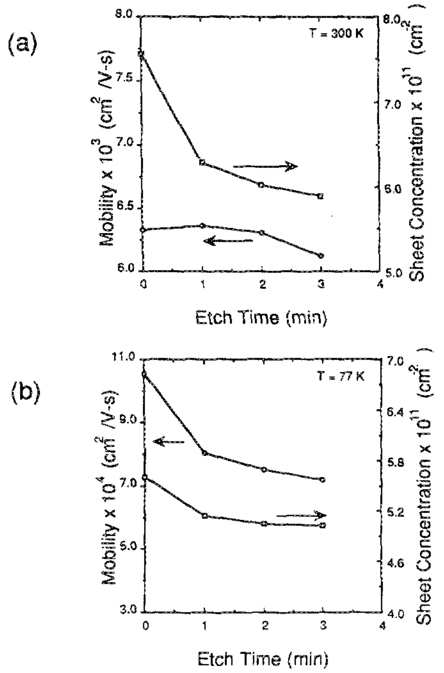

FIG. 5. Hall mobility and sheet carrier concentrations vs etch time for (a) 300 K and (b) 77 K.

introduce some ion bombardment damage to the heterostructure, but that further etching causes only slight changes to the heterostructure properties.

D. Device results

MODFETs were fabricated on pseudomorphic AlGaAs/InGaAs/GaAs heterostructures grown by molecular-beam epitaxy (MBE) on semi-insulating GaAs substrates. The structure consists of a 17-nm undoped $In_{0.2}Ga_{0.8}As$ channel, a 50-Å undoped $Al_{0.25}Ga_{0.75}As$ spacer layer, a 35 nm uniformly doped (Si, 2×10^{18} cm^{-3}) $Al_{0.25}Ga_{0.75}As$ barrier layer and a 20-nm doped (Si, 2.7×10^{18} cm^{-3}) GaAs ohmic contact layer. Hall measurements indicate an electron sheet charge density of 2.2×10^{12} cm^{-2} at 300 K with mobilities of 5500 cm^2/V s at 300 K and 18 200 cm^2/V s at 77 K. Devices with gate lengths between 0.4 and 2.5 μm were fabricated with an all electron beam lithography process.[8]

An SRIE MODFET gate recess process was studied in order to determine the effects of overetching on device characteristics. Devices were recess etched in 20% $SiCl_4$ at 60 mTorr and a plasma self-bias of −60 V for 2 and 4 min. It has been determined that a 2-min etch time assures complete removal of the GaAs cap in the narrow gate region with minimal overetching. The 4-min etch time, therefore, represents a 100% overetch.

Devices etched for 4 min showed positive threshold voltage shifts ranging from 25 to 200 mV, increasing with gate length, over 2 min etched devices. Similarly the 4 min etched devices showed a decrease in transconductance of 10%–20%. On-wafer microwave measurements were performed on select devices and indicated similar or slightly lower unity current gain cut-off frequencies (f_t) for the 4 min devices compared to the 2 min etched devices. Clearly overetching can have an adverse effect on the MODFET device characteristics and we are presently investigating this phenomenon.

IV. CONCLUSIONS

The selective reactive ion etching of GaAs over $Al_xGa_{1-x}As$ in $SiCl_4/SiF_4$ plasmas has been characterized. Selectivities of greater than 500:1 are demonstrated at low $SiCl_4/SiF_4$ gas ratio, high pressure, and low self-bias voltage. AES spectra show high concentrations of fluorine on etched surfaces which has been attributed to an AlF_3 etch stop layer. Removal of the etch stop layer is achieved with HCl:H_2O or NH_4OH:H_2O wet chemical etching. Hall mobility studies show ion bombardment damage to heterostructure samples occurs after 1 min of etching at −60-V dc bias. dc and microwave device characteristics of various gate length pseudomorphic MODFETs etched for 2 and 4 min show increases in threshold voltage and a decrease in transconductance with extended etch times. The evidence suggests that device properties are altered by the selective dry etch and warrants further study of this phenomenon.

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