Effects and applications of oxidation, nitridation and oxi-nitridation on GaAs and InAlAs

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Abstract

Oxidation by UV and Ozone process, nitridation by N_2 plasma process and combination of these (oxi-nitridation) are applied on GaAs and InAlAs wafer to investigate the nm thin insulating film. These are characterized by different experimental means such as roughness by atomic force microscope (AFM), cross sectional image by transmission electron microscope (TEM), compositional analysis by X-ray photoelectron spectroscopy (XPS), photoluminescence, electrical characteristics etc. Obtained results are applied in field effect transistors. UV and ozone oxidation on GaAs forms nm thin oxide layer with surface state density. The thickness of oxide layer is proportional to square root of process time. Long time oxidation realizes the atomic flat interface. The main composition of the oxide layer is Ga_2O_3 and As_2O_3. Nitridation on bare GaAs deteriorates the surface/interface quality. On the other hand, nitridation on oxidized wafer shows the good performance than the nitridated and oxidated sample. Nitridation on oxidized wafer improves the interface quality. XPS data indicates that nitrogen plasma incorporates the nitrogen atoms and removes out the arsenic atoms from the oxide layer. Nitridation changes the Ga_2O_3/As_2O_3 composition to GaON layer. Cross sectional image indicates that the crystal disorder is disappeared by nitridation on oxidized wafer. Nitridation on oxidized wafer increases the photoluminescence intensity. It implies that nitridation reduces the non-radiative recombination centers in the interface region. Nitridation decreases the leakage current and implies the reduction on surface damage. The frequency independent capacitance-voltage characteristics are observed in oxi-nitride sample. Using the GaON layer as gate insulator, GaAs MISFET has been demonstrated with good performance. Hysteresis loop is not observed in the MISFET devices. The maximum obtained transcondutance is 110 mS/mm.

By the similar process to that on GaAs, nitridation, oxidation and oxi-nitridation effect on InAlAs are applied. Nitridation on bare and oxidized InAlAs wafer gradually and drastically decreases the photoluminescence intensity It indicates the deterioration of surface/interface quality. On the other hand, oxidation on InAlAs has recovery effect by increasing the photoluminescence intensity. InAlAs/InGaAs-MOSHEMT has been demonstrated with transconductance of 200 mS/mm in enhancement mode operation. Due to not perfect recovery the hysteresis loop is observed in DC curve.

1. Introduction

In field-effect type semiconductor devices, metal-oxide-semiconductor (MOS) or metal-insulator-semiconductor (MIS) junction is better than the metal-semiconductor (Schottky) junction to suppress the gate leakage current which is desirable for ultra high speed device operation. However, MIS junction is not well established in compound semiconductor devices due to lack of high quality nm thin insulating film with reduced surface state density. There are so many insulator-semiconductor interface related problems such as high surface state density, crystal disorder, surface defects etc. which leads large leakage current. Recently many researchers have investigated the insulating film such as Si_3N_4, SiO_2, Al_2O_3, Ga_2O_3 etc. and reported on the MOSFET or the MISFET with good performance. M. P. Houng et al [1] reported a liquid-phase deposition method of SiO_2 to form a good SiO_2/GaAs junction, which was achieved by controlling composition of liquid. M. A. Khan et al [2] demonstrated the AlGaN/GaN metal-oxide-semiconductor heterostructure field effect transistors on SiC substrate where SiO_2 is used as the gate oxide layer formed by deposition method. N. Maeda et al [3] reported high performance of AlGaN/GaN heterostructure field effect transistors with Al_2O_3/Si_3N_4 gate insulator. The obtained transconductance is 168 mS/mm. P. D. Ye et al [4] demonstrated GaAs MOSFET where Al_2O_3 is used as gate insulator. Their MOS diode shows the comparable low leakage current than the Schottky diode. M. Passlack et al [5] realized excellent capacitance-voltage characteristics of metal/Ga_2O_3/n-GaAs junction. F. Ren et al [6] demonstrated enhancement mode p- and n-channel GaAs MOSFET with $Ga_2O_3(Gd_2O_3)$ as the gate insulator. J. K. Yang et al [7] reported the Gd_2O_3/GaAs interface with reduced surface state density and it exhibits the improved electrical characteristics. These insulating films are formed by deposition methods. Very few efforts have been reported in conversion method by means of conversion of semiconductor surface to insulator. S. Wada et al [8] reported improved capacitance - voltage characteristics in nitrogen - oxygen - argon plasma treated sample with post thermal annealing. M. Losurdo et al [9] demonstrated that the photoluminescence intensity is increased in remote N_2-H_2 (a mixture of 97% N_2 and 3%H_2) plasma treated sample than the untreated sample. R. Nakamura et al [10] reported the good performance of plasma oxidation on GaAs surface. The capacitance - voltage characteristics show the small frequency dependant without hysteresis loop. The main composition of the oxide film is As_2O_3 and Ga_2O_3. But these are not sufficient to realize the reliable MIS structure for commercial products. More investigation is essential to establish the compound semiconductor MOS/MIS devices as commercial products. Here I investigated the insulating film which being formed by means of conversion of semiconductor surface to insulator by oxidation, nitridation and oxi-nitridation on GaAs and InAlAs. Respectable results were successfully applied to GaAs MISFETs and InAlAs/InGaAs MOSHEMTs.

2. Oxidation, Nitridation and Oxi-nitridation of GaAs

The GaAs wafers used here are n(1.7-3×10^{17} cm^{-3}, 0.4μm)/semi-insulating substrate and n($2\times10^{18}cm^{-3}$, 1μm)/p-substrate epitaxial ones. Five groups (A, B, C, D and E) of wafers were used for this experiment. The conditions of all wafers are shown in Table-I and II. All wafers were cleaned by acetone and etched by buffered hydrofluoric acid to remove the native oxide. C and D group wafers were oxidized by UV & ozone process at room temperature for 8hrs. The concentration of ozone gas is 5000 ppm. Then A, B, C and D group wafers were nitridated by N_2 plasma at room temperature. RF power for the plasma is 250/280 W. The flow rate of the N_2 gas is 10 sccm. A and C group wafers were used for compositional analysis by X-ray photoelectron spectroscopy (XPS) and photoluminescence analysis. Ni and AuGe are evaporated on B and D group wafers for fabricating Schottky and MIS diode respectively. E group wafers were separately oxidized by same process at 300^0C for different period and used for thickness and roughness analysis.

Table-I Nitridated/Oxi-nitridated samples

Nitridation \ Oxidation	Nitridation time					Used for
	0hr	1hr	2hr	4hr	8hr	
No oxidation	A1	A2	A3	A4	-	XPS
	B1	B2	B3	B4	-	Diode
8hr oxidation	C1	C2	C3	C4	C5	XPS
	D1	D2	D3	D4	D5	Diode

Table-II Oxidated samples

Samples	Oxidation time (hour)	Used for
E1	0	AFM
E2	1	
E3	3	
E4	6	
E5	10	

Characterization

The properties of insulating film and interface quality are investigated by different experimental means such as roughness by atomic force microscope (AFM), compositional analysis by X-ray photoelectron spectroscopy (XPS), cross sectional analysis by transmission electron microscope (TEM), photoluminescence, and electrical characteristics etc.

Thickness and roughness (AFM)

E-group wafers were oxidized by UV & ozone process. Partially removing the oxide layer by buffered hydrofluoric acid, the thickness of oxide film is measured by an atomic force microscope (AFM). Figure 1 shows the thickness of oxide layer with respect to process time. The thickness of oxide film is nearly proportional to the square root of process time. The roughness of oxide surface and interface (after removing the oxide layer by buffered hydrofluoric acid) were measured by AFM. The roughness vs process time is plotted in Fig.2. The roughness of the bare surface is 0.4 nm. The roughness of the oxidized surfaces is increased to 0.9 nm after 10 hr oxidation. On the other hand, interface roughness is decreased to 0.15 nm after 10 hr oxidation. It indicates that long time oxidation forms the atomic flat interface because the spacing between the Ga and As layer is 0.14nm.

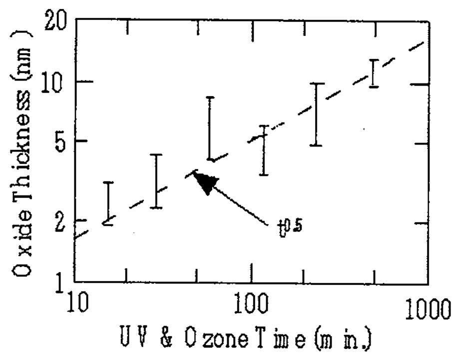

Fig.1 Oxide thickness vs UV & ozone time

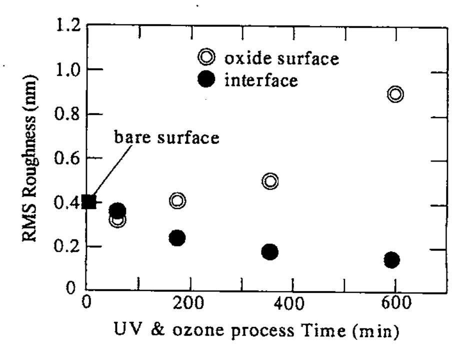

Fig. 2 Roughness vs UV & ozone time

Cross sectional image (TEM)

The cross section of metal-oxide-semiconductor (MOS) structure was measured by transmission electron microscope (TEM). Figure 3 shows the cross sectional image of 2 hr oxidized sample. The thickness of oxide layer is 2~4 nm thick amorphous type. The thickness is not uniform over the surface. The crystal disorder and interface roughness is found in interface region. On the other hand 8hr nitridation after 8hr oxidation form approximately 8nm thick insulating film. The cross section of MIS junction is shown in Fig. 4. The thickness is uniform over the surface. The interface is nearly atomic flat due to long time oxidation. The crystal disorder is hardly found in the interface layer.

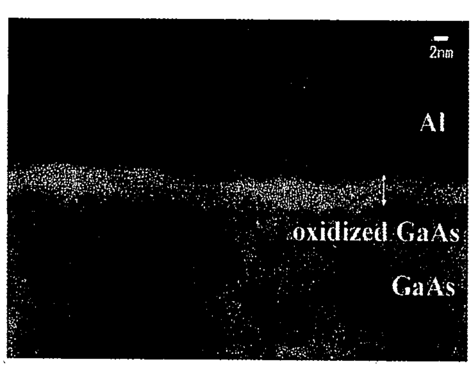

Fig. 3 Cross sectional image of MOS diode (2hr oxidation)

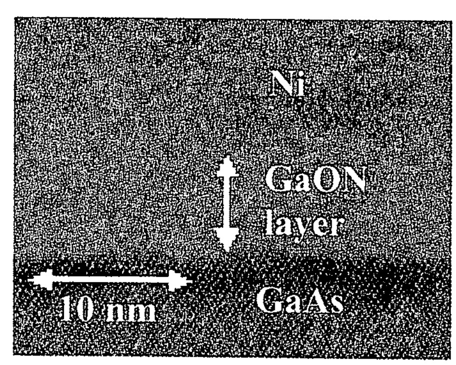

Fig.4 Cross section of MIS diode (8hr oxidation + 8hr nitridation)

Photoluminescence

The spectral shape and intensity of photoluminescence of A- and C-group samples are measured using Ar laser excitation. It is noted that photoluminescence intensity is closely related with the surface/interface state density. The photoluminescence spectra and normalized photoluminescence intensity with respective to nitridation time are shown in Fig. 5(a) and Fig. 5(b) respectively.

Nitridation on bare GaAs surface decrease the photoluminescence intensity although 1hr nitridated sample shows small increased intensity. It implies that nitridation on bare GaAs increases the nonradiative recombination centers in the interface region. On the other hand, nitridation on oxidized GaAs wafer increases the photoluminescence intensity up to 2hr then slightly decreases by long time oxidation. It implies that nitridation after oxidation decreases non-radiative recombination centers and improve the interface quality. This result well coincides with the cross sectional TEM image of oxi-nitridated wafer.

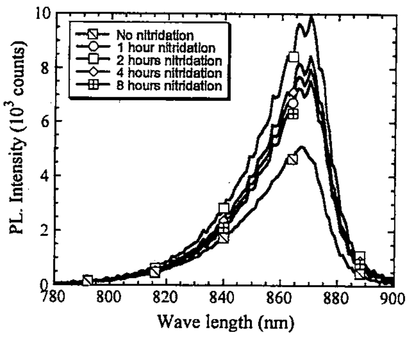

Fig. 5(a) Photoluminescence spectra of oxi-nitridated sample

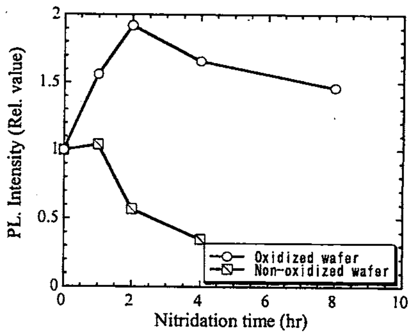

Fig. 5(b) Normalized photoluminescence intensity vs nitridation time

Current-voltage characteristics

I fabricated the MIS diode using B-group (nitridation) and D-group (oxi-nitridation) wafers. The specification of the wafers are n($1.8\times10^{17}cm^{-3}$, 0.75μm)/semi-insulating GaAs and n($1.9\times10^{17}cm^{-3}$, 0.7μm)/semi-insulating GaAs for nitridated and oxi-nitridated samples respectively. Nitridated and oxi-nitridated layers were selectively removed by buffered hydrofluoric acid for ohmic contact. Then Ni and AuGe were evaporated on wafer surface for MIS and ohmic contact respectively. The current-voltage characteristics of these samples are shown in the Fig. 6 and Fig.7 respectively. Nitridation on bare GaAs surface increases the reverse leakage current 3 ~5 order of magnitude. It implies that nitridation on bare GaAs strongly damage the wafer surface. On the other hand nitridation on oxidized sample has good influence. Nitridation decreases the leakage current until 4 hr then increases with longer nitridation. It indicate that optimal nitridation on oxidized GaAs decreases the surface and interface state density.

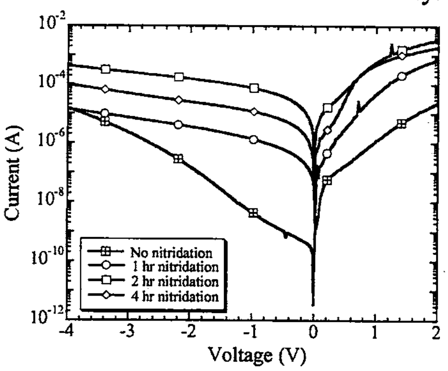

Fig.6 Current voltage characteristics of nitridated wafer

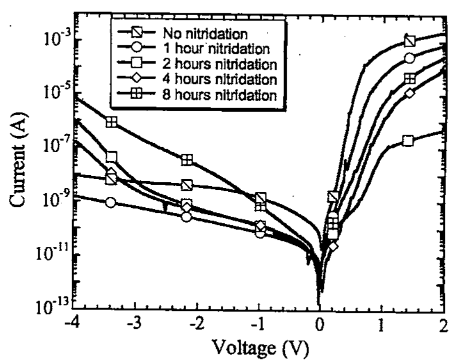

Fig.7 Current-voltage curve of oxi-nitridated wafer

Capacitance-voltage characteristics

The nitridation effect on capacitance - voltage characteristics at 1, 10, 100, 1000 kHz are investigated. Basically $1/C^2$-V relation is applicable in case of Schottky diode to measure the barrier height. However, when thickness of insulating film is negligible compared to thickness of depletion layer, this relation can also be applied in MIS diode. Nitridation on bare GaAs (B-group wafers) makes the capacitance-voltage curves strongly dependent on frequency and very weakly dependent on voltage. It implies that nitridation increases the surface/interface state density. The $1/C^2$-V characteristics of oxidized and oxi-nitridated (D-group) samples are shown in Fig. 8(a) and 8(b) respectively. The reverse bias curves are almost frequency independent. The barrier height of MIS diode measured from $1/C^2$-V curve is plotted in Fig. 9. The barrier height of 0.5eV is observed in only 8 hr oxidized sample and it increases to 1.1eV by 4hr nitridation. Nitridation on oxidized GaAs wafers has good influence and increases the Schottky barrier height. It indicates that nitridation on oxidized wafer creates the insulating film with reduced surface/interface positive charge density.

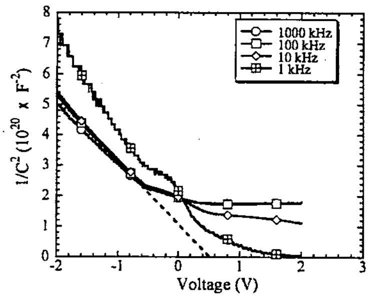

Fig. 8(a) $1/C^2$-V curve (8hr oxidation)

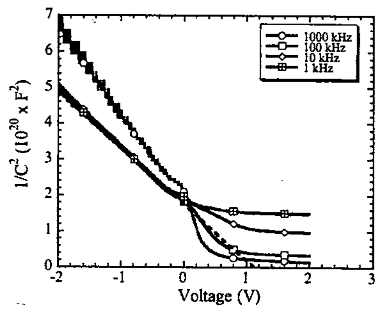

Fig. 8(b) $1/C^2$-V curve (8hr oxidation & 4hr nitridation)

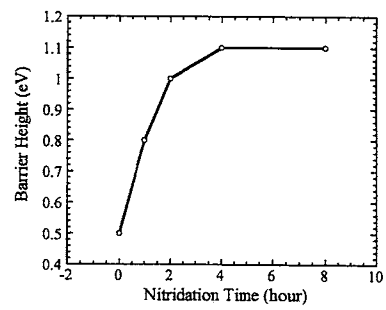

Fig.9 Schottky barrier height vs nitridation time

3. GaAs-MISFET

From the above discussion, it is clear that nitridation on oxidized GaAs creates the nm thin insulating film with good interface performance. Using the oxi-nitride insulating film, GaAs MISFET with good performance are fabricated. The thickness and donor density of epi-layer is 0.3 μm and 3×10^{17} cm^{-3} respectively. The specification and structure of the fabricated GaAs MISFET is shown in Fig.10. After pretreatment by acetone and hydrofluoric acid, the wafers were rinsed by deionized water. AuGe/Ni are evaporated on the wafer surface to form source and drain contact. After removing the suitable epi-layer by recess etching (H_3PO_4:H_2O:H_2O_2=4:90:1), the insulating layer is formed in gate area by oxidation and oxi-nitridation. Al is evaporated in gate area for gate electrode.

The DC characteristics of the MOSFET (4hr oxidation) and the MISFET are shown in Fig.11(a) and Fig.11(b) respectively. The drain current pinch off is not good in the MOSFET. The low transconductance is observed in the gate voltage of +1V region. On the other hand 2 hr nitridation after 4hr oxidation sample (MISFET) shows much better pinch-off than the MOSFET.

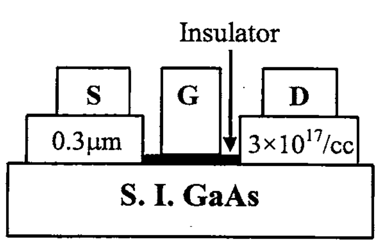

Fig.10 Specification and structure of MISFET

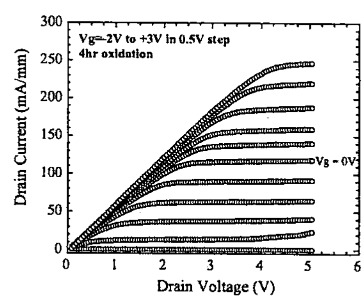

Fig.11 (a) DC curve of MOSFET (Lg=1μm)

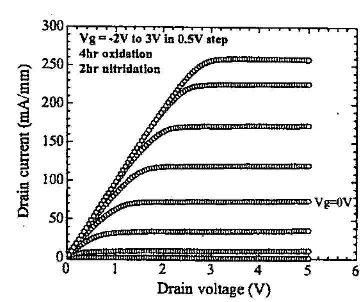

Fig.11 (b) DC curve of MISFET (Lg=1μm)

Current-voltage curves of the MOSFET and the MISFET during the drain voltage swing-up and swing-down are shown in Fig. 12(a) and Fig.12(b) respectively. Large hysteresis loop is observed in the MOSFET. On the other hand, there is no hysteresis loop in MISFET. It implies that nitridation on oxidized wafer improves the interface quality with reducing the surface/interface state density.

The transconductance of 1μm gate length were measured at drain voltage of 0.5V as shown in Fig. 13. With increase of nitridation time on oxidized wafer, the peak value of transconductance is increased. The maximum transconductance of 60 mS/mm is observed in 4 hr oxidized sample. On the other hand, 2 hr nitridation on 4hr oxidized sample shows the transconductance of 110 mS/mm at gate voltage of 1.1V. So nitridation on oxidized wafer improves the interface quality and increases the transconductance.

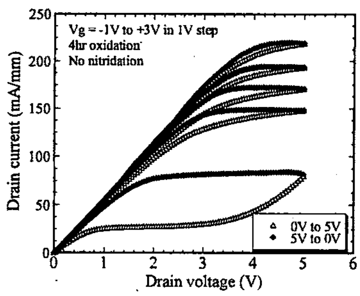

Fig. 12(a) Hysteresis loop of MOSFET

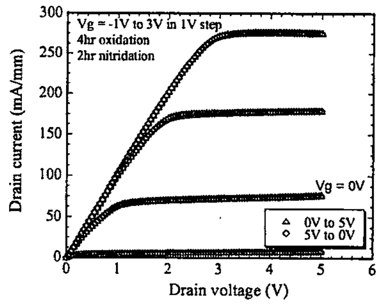

Fig. 12(b) Hysteresis loop of MISFET

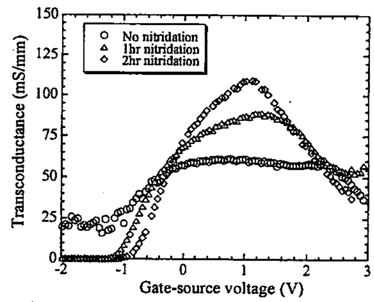

Fig.13 Transconductance with respect to gate voltage

4. Oxidation, Nitridation, Oxi-nitridation of InAlAs

The effect of oxidation, nitridation and oxi-nitridation on InAlAs was investigated by the similar way to that on the GaAs wafers. The results were analyzed by means such as photoluminescence, roughness, cross sectional TEM image, electrical characteristics etc.

Photoluminescence

The oxidation, nitridation and oxi-nitridation of InAlAs are initially investigated by photoluminescence. After ultrasonic cleaning, the wafers were etched by buffered hydrofluoric acid to remove the native oxide. Oxidation by UV & ozone, nitridation by N_2 plasma and combination of both are applied to four groups (A, B, C and D) of InAlAs wafer. The conditions of all wafers are shown in Table-III. A & B-, C- and D-groups wafers are used for oxidation, nitridation and oxi-nitridation process respectively. The photoluminescence spectra of oxidated, nitridated and oxi-nitridated samples were measured. The photoluminescence spectra and relative intensity of oxidized samples (A-group) are shown in Fig. 14 (a) and Fig. 14(b) respectively. The wavelength of the spectral peak of unprocessed wafer is observed at 810 nm and that peak moves to 800 nm by short time oxidation of 1 hr. With increase of oxidation this peak come back to near the 810 nm. The intensity is markedly reduced by a short time oxidation (1^{st} experiment). With increase of oxidation time the intensity shows recovery. It indicates that long time oxidation recovers the surface damage. In order to check the reproducibility of this result, I did same experiment using B-group wafers (C-group) again. The 2^{nd} time data reproduced the 1^{st} time data until 2 hr then decreases the intensity.

Table-III Conditions of InAlAs sample

Minute	0	15	30	60	120	240	480	960
Oxi.	A1	-	-	A2	A3	A4	A5	-
	B1	B2	B3	B4	B5	B6	B7	B8
Nitri.	C1	-	-	-	C2	C3	C4	-
Nitri. (8 Oxi.)	D1	-	-	D2	D3	D4	D5	-

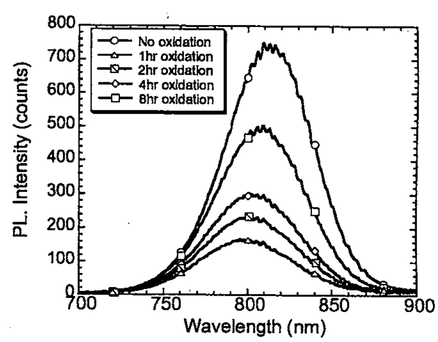

Fig. 14(a) Photoluminescence spectra of oxidized wafer

The normalized photoluminescence intensity of nitridated (C-group) and oxi-nitridated (D-group) sample with respect to nitridation time are shown in Fig 15 and Fig.16 respectively. Nitridation on bare and oxidized InAlAs wafers gradually and drastically decreases the intensity. It indicate that nitridation effect on bare and oxidized InAlAs increases the surface/interface damage.

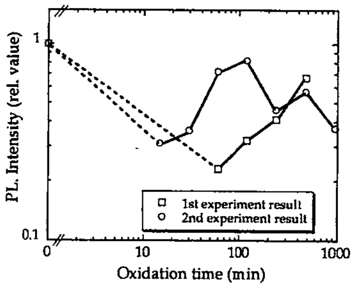

Fig. 14(b) Normalized Pl. intensity of oxidized wafer

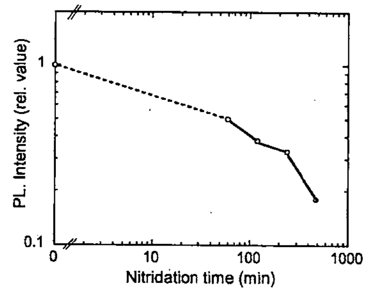

Fig.15 Normalized Pl. intensity of nitridated wafer

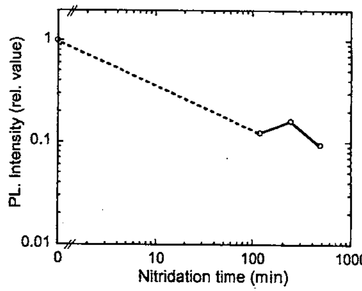

Fig.16 Normalized Pl. intensity of oxi-nitridated wafer

Cross sectional image (TEM)

Considering the recovery properties (photoluminescence intensity) of oxidation on InAlAs, more investigation on oxidized sample was performed by the experimental means of cross sectional structure of interface, electrical characteristics etc. The cross sectional image of oxide-semiconductor interface was measured by transmission electron microscope (TEM) as shown in Fig. 17. Very thin 4~5nm thick oxide layer is formed at 1hr oxidation. The thickness of oxide layer is not uniform type through the surface. The oxide/semiconductor interface is not smooth. The crystal disorder and high roughness is strongly found in the interface layer. One of crystal disorder is indicated by arrow mark. The 4 hr oxidation (not shown here) shows improved interface quality than the 1 hr oxidized sample.

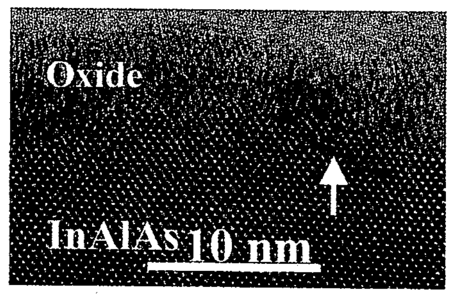

Fig.17 Cross section of oxide/InAlAs interface

Current-Voltage characteristics

The MOS diode was fabricated using n-InAlAs/InP wafer to investigate the leakage current. After ultrasonic cleaning, the wafers were oxidized by UV & ozone process. The density of the ozone gas is 5000 ppm. The flow rate of the O_2 gas is 1 Lt/min. The oxide layer is selectively removed by buffered hydrofluoric acid for ohmic contact. Then AuGe and Ni electrode were formed on the wafer surface for ohmic and MOS contact respectively. The current-voltage characteristics of MOS diode are shown in Fig. 18. The leakage current is gradually decreased with increase of oxidation time. The oxide thickness vs current suppression is shown in Fig.19.

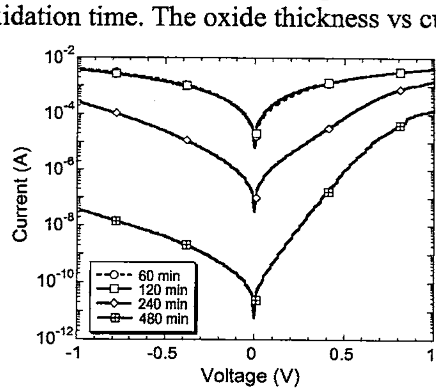

Fig. 18 Current-voltage curve of MOS diode

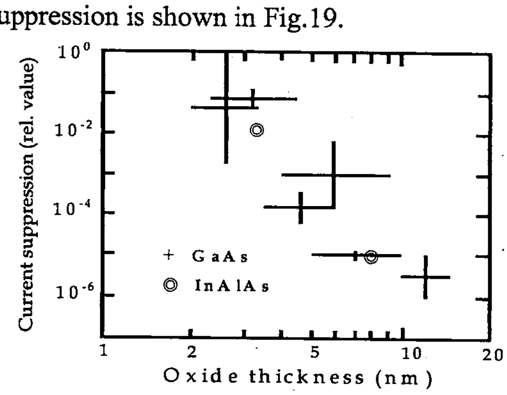

Fig.19 Current suppression by oxide thickness

5. InAlAs/InGaAs - MOSHEMT

Based upon the photoluminescence results, the InAlAs/InGaAs-MOSHEMT were fabricated using 4 hr ozone oxidation. The structure and top view of the MOSHEMT are shown in the Fig.20(a) and Fig.20(b) respectively. After ultrasonic cleaning the wafers, AuGe/Ni were evaporated on the wafer surface to form the source and drain electrode. The epitaxial layer was mesa etched for device isolation. By photolithography process, the gate pattern was formed on the wafer surface. Then suitable epitaxial layer was removed by recess etching in the gate area. The recess etching is performed in three steps. In the first recess etching, the n-InGaAs and InAlAs layer were removed by H_3PO_4:H_2O:H_2O_2 = 4:90:1 at 1^0C. The n-InP layer was etched by HCl:H_3PO4:CH_3COOH:H_2O = 1:1:2.5:1 at room temperature. Then n-InAlAs layer was partially or fully removed by H_3PO_4:H_2O:H_2O_2 = 4:90:1 (3^{rd} etching) for different periods. Oxide layer was formed in the recessed gate area by 4 hr ozone oxidation at room temperature. Then Al was evaporated in the gate area to form the gate electrode. The gate lengths are 0.5μm, 0.7μm, 1μm, 1.5μm and 2μm respectively. The gate width is 40μm (40μm × 2 fingers).

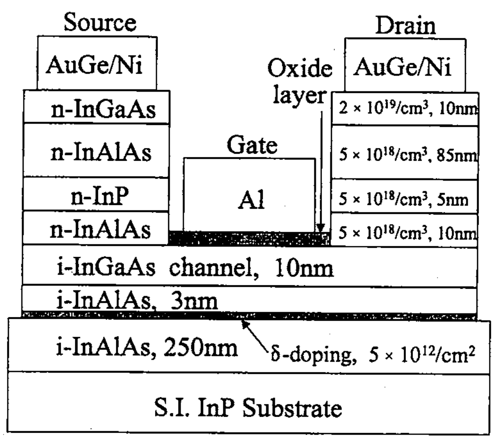

Fig. 20 (a) Wafer specification and structure of MOSHEMT

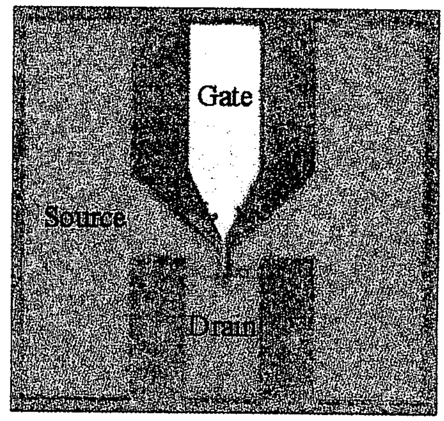

Fig. 20(b) top view of MOSHEMT

The DC characteristic of 25 sec 3^{rd} etching sample is shown in Fig. 21(a). The gate voltage is changed from −3V to +2V in 0.5V step. At zero gate voltage, large drain current of 68mA/mm flows through the channel indicating the depletion mode operation. It also suggests that upper n-InAlAs layer still remains thick and not fully converted into insulating layer. The maximum transconductance of 35 mS/mm is observed at negative gate voltage of -2.5V ~ -3V. The drain current pinch-off is not good due to the remained thickness and so high doping concentration in the upper layer.

The DC characteristics of 35 sec 3^{rd} etching sample is shown in the Fig. 21(b). This data suggest that the upper n-InAlAs layer is mostly removed or converted into oxide layer. Large drain current is observed in the positive gate voltage of +1V to +2.5V and indicates the enhancement mode operation. The drain current pinch-off is better than the previous short time recess etching sample. The small g_m is observed in negative gate voltage up to +1V and increased to 200 mS/mm in positive gate voltage of 2V. The hysteresis curve of MOSHEMT was measured at drain voltage swing up and down as shown in Fig. 22. Due to not perfect recovery the hysteresis loop exist in MOSHEMT.

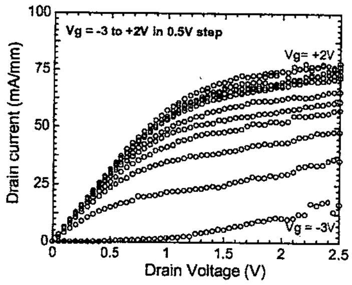

Fig. 21(a) DC curve of depletion mode MOSHEMT (25 sec 3rd etching)

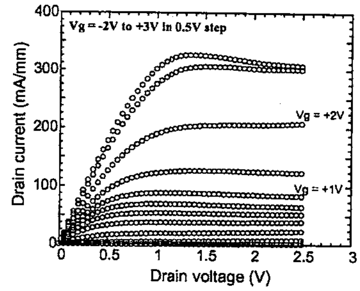

Fig. 21(b) DC curve of enhancement mode MOSHEMT (35sec 3rd etching)

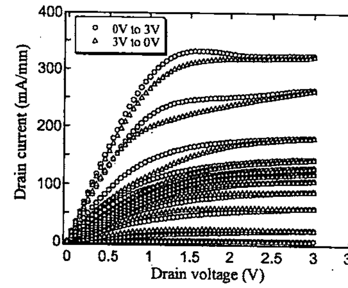

Fig.22 Hysteresis curve of MOSHEMT

6. Conclusion

UV & ozone oxidation on GaAs forms oxide layer with crystal disorder. Nitridation on bare GaAs strongly deteriorate the surface. On the other hand nitridation on oxidized GaAs form nm thin oxi-nitride layer with reduced surfaceinterface states density. Obtained transconductance from the MISFET (oxi-nitride sample) is 110 mS/mm.

Long time oxidation on InAlAs recovers the surface defects with increasing the photoluminescence intensity. The MOSHEMT shows the maximum transconductance of 200mS/mm in enhancement mode operation. On the other hand nitridation on bare and oxidized InAlAs deteriorates the InAlAs surface/interface quality.

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学位論文審査結果の要旨

平成18年1月31日に第一回学位論文審査委員会を開催し、提出された学位論文及び関係資料について検討を行い、同2月2日の口頭発表、第二回学位論文審査委員会において協議の結果、以下の通り判定した。

金属/絶縁体/半導体（MIS）構造のゲートを有する電界効果トランジスタ（FET）は金属/半導体ゲートのトランジスタと比較して様々な利点があるが、化合物半導体では絶縁体/半導体界面が劣化しやすいため良好なものが実現されていない。本論文では、紫外線とオゾンによる酸化とプラズマによる窒化を用いて、GaAsやInAlAs表面への酸化・窒化・酸窒化の効果を実験的に調査し、処理条件や組合わせによっては界面近傍の結晶性の劣化が回復し、電気特性や光電特性が良化することを見出している。また、これを空乏型やエンハンスメント型のGaAs MIS-FETやMIS型InAlAs/InGaAs高電子移動度トランジスタのゲート絶縁膜形成に適用して、良好且つ安定な特性が実現できることを実証している。更に、微細化によるトランジスタ高速化の限界を高める手段にもなり得ることを明らかにしている。

本研究の成果はMISゲート化合物半導体トランジスタの実用化に向けた貴重な新技術であり、本論文は博士（工学）の学位を受けるに値するものと判定する。