Solid State Phenomena Vol. 219 (2015) pp 47-51 Online available since 2014/Sep/26 at www.scientific.net © (2015) Trans Tech Publications, Switzerland doi:10.4028/www.scientific.net/SSP.219.47

InGaAs (110) surface cleaning using atomic hydrogen

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Keywords: InGaAs (110) surface, atomic hydrogen, native oxide removal, scanning tunneling microscopy

Introduction

A major obstacle facing III-V semiconductor based metal oxide semiconductor field effect transistors (MOSFETs) is the large density of trap states that exist at the semiconductor/oxide interface.[1] These trap states can pin the Fermi level preventing the MOSFET from acting as a switch in logic devices. Several sources of Fermi level pinning have been proposed including oxidation of the III-V substrate. [2, 3] In order to minimize the presence of III-V oxides it is crucial to employ either an ex-situ etch or to use an in-situ method such as atomic hydrogen cleaning.[4, 5] Although atomic H cleaning of III-V surfaces is well known, it has never been demonstrated on InGaAs (110) crystallographic faces. Furthermore, tri-gate field effect transistors (finFETs) have recently been employed in commercially available logic chips.[6] This unique device architecture allows for a reduction in short channel effects, minimization of the subthreshold swing, and a higher transconductance.[7] The InGaAs (110) surface would be the sidewalls of a vertically aligned (001) based finFETs.[8] Therefore, it is essential to find an in-situ method to efficiently remove any oxides or contamination from the (110) surfaces that is also compatible with the (001) surface. In this study. STM was employed to determine if atomic hydrogen can be used to remove the native oxide from air exposed InGaAs (110) samples. A post clean anneal was used to restore the surface to molecular beam epitaxy (MBE) levels of cleanliness.

Experimental



Figure 1. (Left) $30x30 \text{ nm}^2$ filled state STM image showing the decapped InGaAs (110) surface. Rows are oriented in ($\overline{110}$) direction. Surface has low density of bright sites (green circle) and dark features (red box). (Right) Model of clean InGaAs (110) surface showing buckled surface atoms and dangling bonds (red oval)

100 nm of In_{0.53}Ga_{0.47}As (110) was grown by MBE on InP (110) substrates and doped with 1×10^{18} cm⁻³ Si. All samples were capped with 50 nm of As₂ and shipped/stored under vacuum. The samples were loaded into an Omicron variable temperature UHV chamber with base pressure of 1×10^{-10} torr. Samples were decapped at 360 °C to expose the bare InGaAs (110) surface. The order and defect density of the decapped InGaAs (110) surface were determined using scanning tunneling microscopy (STM) in an analysis chamber with base pressure of 1×10^{-11} torr. The images were obtained in constant current mode with a current set point of 100 pA and a sample bias of -3 V, i.e. filled state images were obtained. Subsequently, the samples were transferred to the load lock and exposed to air for 30 min to allow formation of an oxide layer. An Oxford Applied Research TC-50 thermal gas cracker was employed to clean the InGaAs samples with atomic hydrogen after air exposure. A variety of cleaning temperatures were used with a H_2 pressure of 1×10^{-6} torr and operating power of 45 W. Subsequently, the samples were annealed to various temperatures, and STM was used to quantify the defect density and determine the quality of the surface. The quantifications in table 1 were obtained by manually counting the number of islands and dark sites in the corresponding 100 x 100 nm² STM images and extrapolating these numbers to 1 μ m². The surface islands are defined as any group of atoms on a terrace greater than 3 nm² while the dark sites are surface vacancies ranging from 0.01-3 nm². The density of dark sites for the air exposed samples were not obtained since atomic resolution was difficult to achieve due to the large number of atomic steps.



Figure 2. (a) 100 x100 nm² STM image of decapped InGaAs (110) surface. (b) surface after 600 L of atomic H exposure at 100 °C for 30 min. Surface appears amorphous. (c) Sample with subsequent anneal to 200 °C for 30 min. Terrace formation and step edges (red arrow) are observed and surface begins to flatten.
(d) Sample after anneal to 386 °C for 30 min. Atomic terraces well defined but with an increased density of surface islands compared to decapped surface

Fig. 1 shows an STM image of the decapped InGaAs (110) surface. Ordered rows of InGaAs are observed in the ($\overline{110}$) direction. The surface atoms are buckled and composed of heterobonds as shown in the model. This relaxed configuration results in the As surface atoms adopting an sp³ geometry with a filled dangling bonds while each In/Ga atom is in an sp² geometry with an empty dangling bond. There are two types of intrinsic defects on this surface, bright sites (green circle) and dark sites (red box). The defect density was manually quantified and is shown in Table 1. The dark site defects are several orders of magnitude greater than the bright site defects on the decapped non-annealed sample.



Figure 3. (a) InGaAs (110) decapped sample after 30 min air exposure. An amorphous oxide layer has formed. **(b)** Air exposed sample after 600 L atomic H clean at 100 °C. Surface remains disordered but terrace formation is observed. **(c)** A subsequent anneal to a maximum temperature of 472 °C for 30 min results in large atomic terraces and a smooth surface. The bright features (blue circle) are hypothesized to be surface atoms which did no diffuse to a step edge. The dark features (red box) are hypothesized to be incomplete terraces.

Initially, the decapped surface, Fig. 2(a) was exposed to 600 Langmuir (L) of atomic hydrogen at 100 °C, Fig. 2(b), to determine the damage induced by the atomic H. Table 1 shows quantification

of the density of surface islands and dark sites after the initial decapping, subsequent H clean, and subsequent anneals. Atomic H dosing increases the island defect density by about 300x. The well-defined terraces on the decapped surface are no longer present after H exposure and the surface appears amorphous. The surface was annealed at 200 °C for 30 min, Fig. 2(c). With the additional thermal energy, surface atoms diffuse more readily and step edges become visible (red arrow) and the surface begins to regain order. The 200 °C anneal decreased the island density by 1.5 x and the dark site density by about 2x. After an additional series of anneals with a max temperature of 386 °C for 30 min, Fig. 2(d), step edges become very distinct and a high degree of order is obtained. Each surface terrace has an increased density of islands compared to the decapped surface, but the terrace sizes are similar. Annealing up to 437 °C reduces the bright site density to within 10x of the density on the decapped samples and an anneal up to 475 °C results in an island density only 3x larger than the decapped sample. It is well known that atomic H induces etching by formation of volatile AsH₃ consistent with the probable source of the initial surface roughening after atomic H exposure.[9] Annealing allows the surface atoms to have sufficient mobility to coalesce into terraces, although there is formation of islands within each terrace.[10, 11] It is

Table 1. Density of islands anddark sites as a function of surfacepreparation method. H cleaninginduces large number of islandsand dark sites but these can bedramatically reduced using hightemperature anneals.

Surface	Density of Islands (/µm2)	Density of dark sites (/µm2)
Decapped	200	11,613
600 L H dose at 100°C	68,800	6,900
After 200°C Anneal	50,200	3,900
After 386°C Anneal	9,403	2,388
After 437°C Anneal	2.400	800
After 487°C Anneal	600	300
Air Exposed+ H clean @ 100°C + 475C		
anneal Air Exposed+ H clean @	1600	2000
275°C	5000	N/A

expected this process will be more challenging on InGaAs (110) than InGaAs (001) since MBE growth on InGaAs(110) is more challenging than on InGaAs(001) consistent with lower diffusion rates at a given temperature. The atomic H exposure and annealing reduces the dark site density below the dark site density on the decapped sample; this would be consistent with hydrogenation increasing the mobility of some of the substrate atoms.





Adecapped sample was exposed to air for 30 min and formation of an amorphous oxide layer was observed, Fig. 3(a). The dominant oxide species present are most likely As₂O₃, Ga₂O₃, and In₂O₃.[12] After exposure to 600 L of atomic H at 100 °C, the surface remains amorphous and the formation of a terrace is observed, Fig. 3(b). The sample was annealed to 386 °C for 30 min and subsequently to 472 °C for 30 min, Fig. 3(c). There are dark features (red box) as well as bright features (blue circle) which are hypothesized to be incomplete terraces and atoms which have not been able to diffuse to a step edge, respectively. Table 1 indicates that the surface has 8x the island density of the decapped sample and 6x less dark defects. The average height between the topmost terrace and the bottommost terrace in a 100 x 100 nm² image is 8.8 Å which corresponds to 3 atomic steps. There is a large variation in the size of surface islands and that is reflected in the average terrace area of $194 \pm 122 \text{ nm}^2$. This suggests that atomic H alone at 100 °C is insufficient to both clean the surface and restore the ordered and atomically smooth surface. An additional anneal is required to ensure the surface atoms have a high enough mobility to diffuse across the surface and form terraces.

In an attempt to eliminate the post clean anneal, an air exposed sample was cleaned with 600 L of atomic H at 275 °C, Fig. 4(a). The amorphous oxide layer has been completely removed without an anneal and atomic terraces and islands are clearly visible. This is likely due to the surface atoms having enough mobility to coalesce into terraces and fill any excess etching due to H cleaning. The average height between the highest and lowest terrace is 10.9 Å which corresponds to 4 atomic terraces. Additionally the average terrace area is 184.7 ± 233 nm² which highlights the difficulty in obtaining atomically flat surfaces after atomic H exposure. Table 1 quantifies the density of islands on this surface. A subsequent anneal to 350 °C for 30 min was performed, Fig. 4(b), but there was not a significant difference in density of terraces or etch pits. This experiment demonstrates it is possible to remove the native oxide from InGaAs (110) but it also demonstrates the difficulty in removing the etching induced roughness.

Conclusion:

To ensure an unpinned Fermi level on InGaAs (110) surfaces it is crucial to remove any contamination and oxides prior to atomic layer deposition of the high-k dielectric layer. An effective way to clean (110) surfaces has been demonstrated by utilizing an in-situ atomic hydrogen source. The cleaning was performed at both 100 °C and 275 °C, and it was found that the higher cleaning temperature eliminates the need for a post clean anneal leaving the surface with over 10x lower defect density than the low temperature clean surfaces; however, the residual defect density after high temperature cleaning is higher than on than the decapped surface consistent with the lower diffusion rates for substrate atoms on InGaAs (110) than InGaAs (001). This method has previously been shown to be effective on the (001) surface as well, allowing for an entirely in-situ cleaning method for three dimensional MOSFET architecture.

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10.4028/www.scientific.net/SSP.219

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10.4028/www.scientific.net/SSP.219.47