InGaAs surface preparation for atomic layer deposition by hydrogen cleaning and improvement with high temperature anneal

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Using *in situ* atomic scale imaging with scanning tunneling microscopy/spectroscopy, a combination of atomic hydrogen dosing, annealing, and trimethyl aluminum dosing is observed to produce an ordered unpinned passivation layer on an air exposed InGaAs(001)-(4×2) surface with only monatomic steps. This shows that conventional gate-last semiconductor processing can be employed to fabricate a variety of electronic devices, even on air exposed compound semiconductors. © 2011 American Institute of Physics. [doi:10.1063/1.3597791]

INTRODUCTION

Commercial III-V semiconductor devices such as high electron mobility transistors and heterostructure bipolar transistor all employ interfaces, which are grown in situ in vacuum systems to avoid air contamination of the III-V surfaces. However, these devices are not suitable for practical logic applications such as microprocessors because they cannot be scaled in size and have large off-state leakage currents. At present, the only device design which offers the compact size and low off-state currents required for microprocessors is the metal oxide semiconductor field effect transistor (MOSFET). Present MOSFETs are fabricated on silicon, but higher levels of integration (more devices per unit area) are limited by power dissipation; therefore, several hundred research papers are published each year upon the development of MOSFETs using III-V semiconductors, especially InGaAs.¹ To enable low power MOSFET operation, a low supply voltage is required, the semiconductor must have high mobility and high saturation velocity, the oxide-semiconductor interface must have a low density of interface trap states (D_{it}), and the oxide-semiconductor interface must be nearly atomically flat to insure high mobility at high field strength.

Surface channel III-V MOS devices can be fabricated with atomic layer deposition (ALD) high-K gate-first processes,^{2–5} which are similar to silicon processes for SiO₂ growth on silicon or ALD of high-K on silicon.^{6–10} The key for a gate-first process is that subsequent processing steps cannot degrade the semiconductor, the dielectric, or the oxide-semiconductor interfaces. For silicon, the only commercial ALD high-K fabrication process is a replacement gate process (a type of gate-last process) to avoid processinginduced damage.¹¹ While preparing silicon for gate-last processing is straightforward, for III-V semiconductors the key to a gate-last process is the order and cleanliness of the III-V channel prior to dielectric deposition. It has been shown that ALD of trimethyl aluminum (TMA)^{12,13} or tetrakis(ethylmethylamino)hafnium (TEMAH)¹⁴ on III-V semiconductors has self-cleaning properties by reducing the presence of As-O and Ga-O bonds. However, for high quality dielectric semiconductor interfaces, further reduction or cleaning may be required and the interface must be atomically flat. Furthermore, aggressive oxide thickness reduction [equivalent oxide thickness (EOT) scaling] is needed to fabricate small gate length devices with small subthreshold swings, and aggressive EOT scaling requires a very high uniform ALD nucleation density with no pinholes due to surface contaminants.¹⁵ The key barrier to solving a very practical problem is a surface chemistry challenge: development of a chemical process, which removes nearly all air induced defects and contaminants and leaves the III-V surface flat and electrically active for high nucleation density ALD gate oxide deposition which unpins the Fermi level.

Atomic hydrogen cleaning of III-V semiconductors has been investigated.^{16–22} Traditional atomic hydrogen cleaning is performed at elevated temperatures as a surface preparation for molecular beam epitaxial (MBE) growth. This study investigates the hydrogen cleaning at 24 and 380 °C with postcleaning annealing to determine the influences of cleaning temperature and annealing temperature on the electronic structure and surface defects, roughness, and step density. Atomic hydrogen has been shown to unpin GaAs,²² however, it induces surface etching.^{21,23,24} It has been shown that the atomic hydrogen etch rate for silicon is lower at 700 °C compared to room temperature (RT).²⁵ However, for GaAs, the atomic hydrogen etch rate increases with temperature along with the pressure of hydrogen.²⁶ Etching must be minimized since it can induce surface roughness, which is incompatible with the thin channel structures required in low-power highly-scaled devices.^{10,27,28} By employing scanning tunneling microscopy (STM) to probe in situ hydrogen cleaned surfaces, the surface features can be studied at the atomic level.

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In the present study, the samples are 0.2 μ m thick In_{0.53}Ga_{0.47}As layers grown by MBE on commercially available InP wafers. The MBE-grown InGaAs layers are doped *n*-type and *p*-type with a doping concentration of 2 × 10¹⁸ cm⁻³ of the Si and Be dopants. Following the MBE growth, all samples are capped with a 50 nm As₂ layer and shipped/ stored under vacuum before being loaded into the ultrahigh vacuum (UHV) chamber. The As₂-capped samples allow for the comparison of pristine samples to air-exposed/H cleaned samples. The samples are loaded into an Omicron UHV chamber with a base pressure below 1×10^{-10} Torr. The samples are decapped in UHV and annealed at 450–470 °C to form the InGaAs(001)-(4 × 2) surface reconstruction in the preparation chamber. Further details concerning the samples and preparation methods are published in Ref. 29.

The order and defect density of the decapped surface is quantified with scanning tunneling microscopy (STM) in the analysis chamber with a base pressure of 2×10^{-11} Torr. Afterwards, the decapped samples are transferred to the load lock and exposed to air for 0.5-30 min. Using an Oxford Applied Research TC-50 thermal gas cracker in the preparation chamber, the sample is cleaned with atomic hydrogen at various sample temperatures and dose times with a H₂ pressure of $1-2 \times 10^{-6}$ Torr. The percent of atomic hydrogen from the thermal cracker at 65 W is \sim 50%; the thermal cracker was operated at 60 W. The percentage of atomic hydrogen at the sample surface is a mixture of the recombined background H₂ and the direct stream from the thermal cracker. The background H₂ should be inert; therefore, the reduction in surface oxides is assumed to be from the atomic hydrogen produced with the thermal cracker, however, the exact dose of atomic hydrogen is unknown since the measured pressure includes both the direct and background H and H₂ sources. Following the hydrogen cleaning, the sample is annealed to 460–480 °C to regain the InGaAs(001)-(4 \times 2) surface reconstruction, and the STM is employed to determine the reconstruction, defect density, and surface roughness. The TC-50 thermal cracker employs a hot iridium tube to crack the H₂ molecules, thereby avoiding contamination that can occur with hot tungsten filaments and or the formation of ions that can occur with a plasma source; both metal contamination and ion bombardment probably would negatively influence surface cleaning, ordering, and electronic passivation. 30,31

The STM provides atomic resolution of the surface morphology, however, scanning tunneling spectroscopy (STS)^{32–35} is performed to determine the electrical quality of the surface. The decapped InGaAs(001)-(4 × 2) surface reconstruction is pinned, consistent with the presence of strained In/Ga dimers,^{29,36,37} therefore, for the surface to be unpinned upon suitable oxide deposition, surface electrical passivation is required. The TMA is synergistic because it both electrically passivates the surface and provides the monolayer nucleation density required for very thin gate oxide formation.³⁸ The deposition of TMA is performed in the load lock. The load lock is first baked overnight until it reaches a base pressure below 1×10^{-7} Torr to avoid water contamination (commercial ALD tools employ hot walls, which is a similar, but faster technique). The sample is exposed to 1×10^{-3}

 -1×10^{-2} Torr of TMA vapor for 5 s at room temperature (RT) followed by a 250 °C anneal in the preparation chamber.

RESULTS AND DISCUSSION

Figure 1(a) shows an STM image of the decapped InGaAs(001)-(4 \times 2) surface. The decapped surface includes two distinct defects: dark horizontal features (black rectangle) and bright vertical features (white rectangle). Similar defects have been studied on InAs(001)-(4×2), which effectively has the same surface structure as InGaAs(001)- (4×2) . The dark horizontal features are described as missing-rowdimers,³⁹ and the bright vertical features are described as the same In/Ga row dimers in the 4×2 , however with 8 Å spacing instead of 17 Å.⁴⁰ There are some variations in the bright vertical features, which are probably caused by excess atoms bridge bonding between the As row edge atoms. After the InGaAs(001)-(4 \times 2) surface is exposed to air for 0.5 min, an amorphous film is observed by STM, shown in Fig. 1(b). Note that the surface was annealed to 200 °C in order to achieve stable STM images. The oxide film of the As-rich 2×4 surface primarily consists of As₂O₃, Ga₂O₃, and In_2O_3 ,¹⁹ and the In-rich 4 × 2 should have similar oxides with different ratios. Figure 1(c) shows a surface exposed to air for 30 min followed immediately by atomic hydrogen

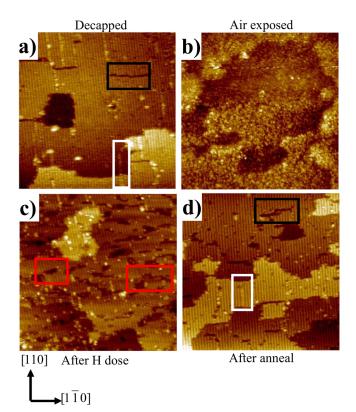
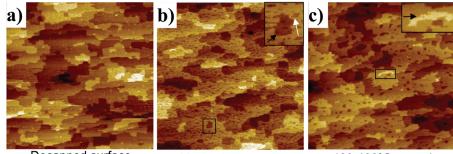


FIG. 1. (Color online) $100 \times 100 \text{ nm}^2$ filled state STM image of (a) decapped surface, (b) air exposed and annealed to $200 \,^{\circ}\text{C}$ surface, (c) surface after 30 min air exposure followed by 30 min dose of hydrogen at 380 $^{\circ}\text{C}$, and (d) after a high temperature anneal to 460–480 $^{\circ}\text{C}$. The black rectangles show dark horizontal defect features, white rectangles indicate vertical bright defect features, and red rectangles show dark surface features seen after hydrogen dosing which might be from surface etching. Reproduced with permission from ECS Trans. **35**(4), 175–189 (2011). Copyright © 2011, The Electrochemical Society.



Decapped surface

30min air exposure 30min H dose @380°C 460-480°C anneal

FIG. 2. (Color online) $500 \times 500 \text{ nm}^2$ filled state STM image of (a) decapped surface, (b) 30 min air exposure followed by 30 min dose of hydrogen at 380 °C, and (c) after a high temperature anneal to 460–480 °C. The terrace sizes improve with high temperature annealing. The hydrogen dosed surface shown in (b) has a large amount of dark surface features which are no longer present in (c). The inset in (b) indicates large (black arrow) and small (white arrow) etching features caused by hydrogen exposure. The inset in (c) indicates a small terrace (black arrow) which is consistent with an incomplete terrace. The images are corrected for global tilt. Reproduced with permission from ECS Trans. **35**(4), 175–189 (2011). Copyright © 2011, The Electrochemical Society.

cleaning at 380 °C for 30 min. The hydrogen cleaned surface shows dark features (red rectangle) consistent with monolayer etch pits; however, there is a reduction in the other defects observed on the decapped surface. Figure 1(d) shows the hydrogen cleaned surface after a 460-480 °C anneal for 10 min showing an increase in terrace size and uniformity. If the sample was not cleaned with hydrogen and only annealed to 460-480 °C anneal for 10 min, the STM image resembles that of Fig. 1(b). The densities of horizontal dark defects (black rectangle) appear to be similar to the decapped surface while the vertical defects are drastically reduced. There are some bright vertical defect features, however, these structures appear to be different from the features on the initial decapped surfaces. The difference in structure is probably caused by the removal of excess atoms by atomic hydrogen from the 8 Å spacing row dimers.

To further investigate the defect densities and etch features, a set of images was recorded over a larger area. Figure 2(a) is a $500 \times 500 \text{ nm}^2$ STM image of the decapped surface showing ~242 (manually counted) of the bright vertical defect features with large terraces. After exposure to air for 30 min (no anneal) and a 30 min dose of hydrogen at 380 °C, the number of bright vertical defects is reduced to ~58 in Fig. 2(b); however, the STM image shows a reduction in terrace size because of the dark monolayer deep etch features. Finally, if the sample is annealed to 460–480 °C, the bright defect features are further reduced to ~ 28 in Fig. 2(c), and the terrace size is restored to almost the same size as the decapped surface.

The atomic hydrogen induced etching features reduce the terrace sizes. There are two types of etching features; dark pits, shown in the inset of Fig. 2(b), which shows the removal of surface atoms in the plane of the terrace and incomplete terraces, shown in the inset of Fig. 2(c), which illustrates a terrace that has been almost completely etched with only a residual amount remaining. Comparing the decapped sample to the 30 min high temperature (HT) atomic hydrogen dosed without an annealed sample, it is clear that there is a sharp increase in the etch pits after HT atomic hydrogen cleaning. After a HT anneal, the surface shows a distinct improvement as seen in Fig. 2(c).

To quantify the effect of annealing on the etching feature density, the number of etch pits per unit area, the number of incomplete terraces per unit area, and the average fraction of the surface covered by etch pits and incomplete terraces was quantified. As shown in Table I, for the 30 min 380 °C hydrogen clean surface, HT annealing reduces the density of dark etch pits by almost a factor of 50 and the percent coverage of the surface with etching features after annealing is reduced by almost a factor of 3. The hydrogen cleaned and annealed surface has etch feature densities similar to that of the decapped surface. The quantitative analysis

TABLE I. Comparison of hydrogen dosing temperature and time with densities of etch pits, incomplete terraces, and percent surface coverage of defects (either pits or incomplete terraces). The HT anneal is $460-480^{\circ}$ C. The densities are calculated by counting the number of sites on a $500 \times 500 \text{ nm}^2$. The total percent coverage was calculated by taking the area of a large terrace and determining the percent of that area which is occupied by either the dark etch pits or the incomplete terraces. Every sample but the decapped sample was exposed to air prior to hydrogen cleaning. The values are for one sample but illustrate the trend between processing conditions. For the decapped sample there are features that resemble the etching features, however they are not etching features, and they are counted to give a comparison of hydrogen cleaned surfaces to that of the decapped surface.

Sample	Density of etch pits (/µm ²)	Density of incomplete terraces $(/\mu m^2)$	Percent surface coverage of defects (%)	Average size of etching feature (nm ²)
Decapped	20	20	6–7	1500
30 min 380 °C dose, no HT anneal	50,000	60	28	5.6
30 min 380 °C dose with HT anneal	1,000	60	10	100
5 min 380 °C dose with HT anneal	100	200	4	130
30 min 24 °C dose with HT anneal	100	1000	14	130
5 min 24 $^{\circ}\text{C}$ dose with HT anneal	800	40	16	190

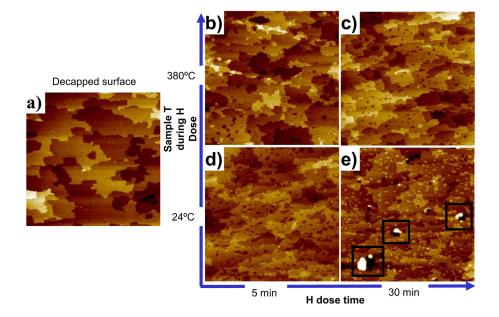


FIG. 3. (Color online) $500 \times 500 \text{ nm}^2$ filled state STM images of (a) a typical decapped InGaAs(001) surface, with a surface step coverage (SSC) of 5.66%. For STM images (b)–(e), all samples after decapping are then exposed to air for 30 min, followed by hydrogen cleaning and annealing to 460–480 °C. (b) Shows a STM image of a sample dosed with hydrogen at a sample temperature of 380 °C for 5 min with SSC = 7.65%; (c) is for 30 min at 380 °C with SSC = 13.9%. (d) Sample is dosed with hydrogen at 24 °C for 5 min with SSC = 13.4%; (e) is for 30 min at 24 °C with SSC = 15.4%. The images are corrected for global tilt.

shows that an air-exposed sample after hydrogen cleaning and HT anneal is a very similar surface to that of the decapped surface except the etch features are smaller on the hydrogen cleaned HT annealed surface while similar features are larger on the decapped sample.

Figure 3 compares the temperature and time of airexposed samples cleaned with hydrogen; all samples are annealed to 460–480 °C after hydrogen dosing. The temperature and the time influence the densities and surface coverage of the etching features. The densities of etch pits, the densities of incomplete terraces, and the percent surface coverage for the STM images in Fig. 3 are presented in Table I. The HT hydrogen dosing shows an improvement over the room temperature dosing (RT hydrogen) in the etch feature percent coverage of the surface. The 30 min RT hydrogen dosed surface shows a lower density of etch pits compared to the 5 min RT hydrogen dosed surface; however, the 30 min RT hydrogen dosed surface is dominated by the incomplete terrace features, most likely due to the hydrogen dose which has etched more than 70% of the original terraces. At elevated temperatures, it appears that the surface atoms have enough mobility during hydrogen dosing to either reduce multilayer etching or repair the effects of multilayer etching. A similar process in which surface mobility reduces roughness is well-known on the GaAs surface in migration enhanced epitaxy.^{41,42} The 30 min RT hydrogen dosed surface has bright and dark features, indicated by black squares. In Fig. 3(e) the bright features range from 2-6 nm where the dark features range from 1.5-4.2 nm. Figure 3(c) of the HT hydrogen surface does not show any of these bright or dark features indicating that the etching is uniformly occurring over the surface. This is consistent with an annealing process simultaneously occurring with etching for 380 °C hydrogen dosing preventing multilayer etching while at 25 °C only etching occurs.

Besides etch pits, step edges are a major defect that can reduce carrier mobility in the channel, because steps usually contain dangling bonds due to the undercoordinated bonding configuration. Using the scanning probe image processor's grain analysis tool, the fraction of the surface covered in edge features was quantified (step edges, horizontal, and vertical defects). Typical decapped surfaces [Fig. 3(a)] have a 5.6% surface coverage of the step edges. For samples with a 380 °C sample temperature during hydrogen dosing [Figs. 3(b) and 3(c)], show an improvement in comparison to the room temperature hydrogen dosing, [Figs. 3(d) and 3(e)]. Typical surfaces after 30 min air exposure and 5 min atomic hydrogen cleaning at 380 °C followed by 460–480 °C have 7.6% step coverage [Fig. 3(b)], which is close to that of the decapped surface.

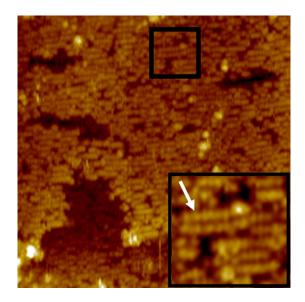
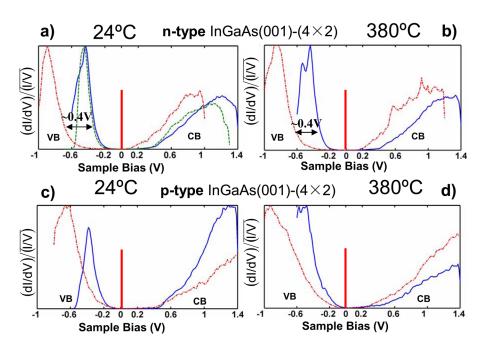


FIG. 4. (Color online) $40 \times 40 \text{ nm}^2$ filled state STM images of surface after the TMA dose at room temperature and annealed to 250 °C. The inset shows an expanded view of $7 \times 7 \text{ nm}^2$ indicated by a black square. The surface shows high ordered horizontal row features (indicated by the white arrow), consistent with a surface reconstruction. Reproduced with permission from ECS Trans. **35**(4), 175-189 (2011). Copyright © 2011, The Electrochemical Society.



Typical surfaces after a 30 min air exposure and a 30 min atomic hydrogen cleaning at 380 °C followed by 460–480 °C have 13.9% surface coverage of step edges [Fig. 4(c)], which is close to that of the decapped surface consistent with the annealing removing most etching-induced step formation. The RT hydrogen dosed samples, even with a high temperature anneal, were not as flat as the decapped samples with a percent coverage of the edges of 13.4% and 15.4% for 5 and 30 min doses followed by a 460–480 °C anneal. The role of the sample temperature of the surface morphology indicates that at 380 °C surface mobility is enhanced, resulting in smoother surfaces; however, at higher temperatures the etch rate might be increased because the GaAs (Ref. 26) atomic hydrogen etch rate increases with temperature.

The atomic hydrogen induced removal of the oxide layer involves multiple reactions. Atomic hydrogen can reduce Ga₂O₃ to Ga₂O and H₂O,^{19,20,24} and desorption of Ga₂O occurs at ~400 °C on GaAs and InGaAs.^{20,43} The influence of atomic hydrogen on In oxides has been assumed to be similar to that of Ga oxides.¹⁹ Atomic hydrogen has been shown to convert atomic As and As₂O₃ into AsH₃ and/or H₂O.⁴⁴ Atomic hydrogen on 400 °C samples of GaAs and InGaAs is known to remove As.^{19,20} Furthermore, the desorption of As oxides and As₂/As₄ occurs around 400 °C.^{19,20} This hydrogen induced volatilization of As is consistent with the reduction of any excess As defects on the hydrogen clean surfaces in the present study. The absence of any oxides in the present study is likely due to the > 300 L exposure which reduces any Ga₂O₃ or In₂O₃ to suboxides which are volatilized during annealing along with any As oxides.

To determine the electrical quality of the InGaAs(001)- (4×2) surface, electrical passivation is required because the decapped surface is pinned.²⁹ Following atomic hydrogen cleaning, the surface was exposed to TMA at room temperature and annealed to 250 °C; an STM image of the TMA-dosed surface can be seen in Fig. 4. The TMA induces a surface reconstruction, creating a bulklike bonding configu-

FIG. 5. (Color online) Normalized (dI/dV) $\overline{(I/V)}$ spectra using variable-z STS of decapped InGaAs(001)- (4×2) (solid blue line), 30 min air exposed and hydrogen cleaned for 30 min (dashed green line), and the room temperature dose of TMA followed by a 250 °C anneal (dot-dashed red line). Spectra for n-type InGaAs with hydrogen cleaning at (a) 24 °C, and (b) 380 °C. Spectra for p-type InGaAs with hydrogen cleaning at (c) 24 °C, and (d) 380 °C. Δz for spectra ranges from 0.1–0.3 nm/V. I/V is smoothed before (dI/dV)/(I/V) is calculated (see Ref. 35). The 0.4 V shift in the VB side of the dot-dashed red curve for (a) the n-type and (b) and the absence of a shift in the p-type. (c) and (d) Indicates an unpinned surface Fermi level.

ration between the Al atoms and the surface As atoms.³⁸ The TMA passivation layer has horizontal rows of dimethyl aluminum. The surface is a highly-ordered self-limiting layer that has a high nucleation density. The self-limiting and high nucleation density is necessary for EOT scaling. The STM image of the TMA-dosed surface shows that this surfaces satisfies the key processing conditions for gate oxide deposition: an atomically flat, high nucleation density, and electrically unpinned as shown next by STS.

The STS spectra for n-type and p-type decapped InGaAs can be seen in Fig. 5. The Normalized (dI/dV)/(I/V) spectra is proportional to the local density of states of the surface.^{45,46} The STS was performed with a modulation frequency of 1 kHz, a lock-in time constant of 20 ms, and a T-raster of 40 ms; therefore, it is sensitive to all trap states with a lifetime less than 1 ms. In the spectra, the zero sample bias is the Fermi level position of the surface relative to the bands. For the decapped surface (solid blue curves), the Fermi level on both the n-type and p-type samples is positioned near the valence band (VB), indicating that the decapped surface is pinned. After air exposure and hydrogen cleaning for 5 min at room temperature followed by a 460-480 °C anneal, the spectra look almost identical to the decapped surface, shown by the blue and green curves in Fig. 5(a) for n-type InGaAs. This is also the case for the p-type at room temperature (spectra not shown). The STS spectra for the dark horizontal defects, bright vertical defects, and step edges were taken (not shown). The STS spectra for the defects showed a slight change in the relative signal intensity of the filled and empty states, however, the differences were not enough to draw any significant conclusions. For InGaAs(001)-(4 \times 2), the clean defect-free reconstructed surface is pinned which would make single defect site STS very difficult because the background signal from the reconstructed surface would dominate the local signal.

To demonstrate that atomic hydrogen cleaning not only restores the surface morphology but also the electrical characteristics, TMA is deposited to document the surface Fermi level unpinning. The spectra for the TMA-dosed surface (dot-dashed red curves) shows the surface Fermi level near the conduction band for n-type samples, see Fig. 5(a), and near the VB for p-type samples. Therefore, Fig. 5(c) is consistent with the surface Fermi level being unpinned. For further details on the energy and density of the remaining trap states, capacitance voltage measurements on a metal-oxide-silicon capacitor are needed, but these require extensive processing.

The STS results were also performed on surfaces cleaned with hydrogen for 5 min with a sample temperature of 380 °C followed by a 460–480 °C anneal. The spectra for n-type and p-type InGaAs can be seen in Figs. 5(b) and 5(d), respectively. Immediately after the hydrogen cleaning and after annealing, spectra were also taken, showing almost identical features to that of the decapped surface (spectra not shown). Within the limitations of STS, the surface electrical quality appears to be the same for the air-exposed surface cleaned with hydrogen at RT and 380 °C with a 460–480 °C anneal. However, the STM indicates that there might be a difference in D_{it} based on the densities of etching features between RT and 380 °C with a 460–480 °C anneal.

In summary, atomic hydrogen cleaning is able to restore the InGaAs(001)-(4×2) reconstruction after air exposure. With STM, the removal of the oxide layer and the restoration of the clean InGaAs surface reconstruction is observed, allowing for a gate-last or replacement-gate process. The STS of InGaAs shows that the electrical quality of the hydrogen cleaned surface is similar to that of an As₂ decapped surface; more importantly, the STS is consistent with unpinning after air exposure, with the hydrogen cleaned and annealed surface followed by an organic metal molecule, which also acts as an electrical passivation and nucleation layer for oxide ALD. The difference between room temperature and high temperature hydrogen cleaning is minimal after high temperature annealing, allowing for a wide process window that must be optimized for etch rates compatible with the device structure. The key process, which is common to both room temperature and high temperature hydrogen cleaning, is the high-temperature anneal which reduces the step edge density and increases the terrace size. The process can easily be implemented with other hydrogen sources as long as the atomic hydrogen is free from high-energy ions and chemical contaminants.

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