**Abstract**

The MOS technology using high-k dielectrics on high carrier mobility semiconductors of InGaAs leading to a faster speed at lower power is now at the International Technology Roadmap for Semiconductors (ITRS). Great efforts have been dedicated to understand the high k/InGaAs interface. This project involves an examination of interfacial electronic structure of in-situ HfO₂ on \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As}(001)-4\times2 \) probed by synchrotron radiation photoemission. The dielectric film was prepared by atomic layer deposition (ALD) with the precursors as tetakis[ethylmethylamino] Hafnium (TEMAHf) and water. Samples were kept under ultra-high-vacuum (UHV) throughout from MBE/ALD preparation to data-acquisition photoemission chambers in the synchrotron-radiation facility. The high-resolution work with a short inelastic mean-free path allowed for the first time identification of atom-to-atom interaction at this interface. This work has elucidated the mechanism and nature of the bonding between the Hf atom in TEMAHf and In/As atoms of the reconstructed \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As}(001)-4\times2 \) surface. The TEMAHf precursor is either intact, or loses one ethylmethylamino ligand to become tri-EMAHf. The initial purge of the TEMAHf precursors effectively passivates all the edge As atoms in the top row. As a result, no oxygen bonding was found at either surface As or In atoms. The subsequent water purge reacts with tri-EMAHf to facilitate the growth of hafnium oxides there. The interface obtained by ALD in the present study found no evidence of oxygen bonding to any atom in the trough. The unpassivated surface In atoms may contribute to the frequency dispersion in the accumulation region of \( n-\text{In}_{0.53}\text{Ga}_{0.47}\text{As MOSCAP} \).
**Abstract**

The MOS technology using high-k dielectrics on high carrier mobility semiconductors of InGaAs leading to a faster speed at lower power is now at the International Technology Roadmap for Semiconductors (ITRS). Great efforts have been dedicated to understand the high k/InGaAs interface. This project involves an examination of interfacial electronic structure of in-situ HfO2 on In0.53Ga0.47As(001)-4x2 probed by synchrotron radiation photoemission. The dielectric film was prepared by atomic layer deposition (ALD) with the precursors as tetrakis[ethylmethylamino] Hafnium (TEMAHf) and water. Samples were kept under ultra-high-vacuum (UHV) throughout from MBE/ALD preparation to data-acquisition photoemission chambers in the synchrotron radiation facility. The high-resolution work with a short inelastic mean-free path allowed for the first time identification of atom-to-atom interaction at this interface. This work has elucidated the mechanism and nature of the bonding between the Hf atom in TEMAHf and In/As atoms of the reconstructed In0.53Ga0.47As(001)-4x2 surface. The TEMAHf precursor is either intact, or loses one ethylmethylamino ligand to become tri-EMAHf. The initial purge of the TEMAHf precursors effectively passivates all the edge As atoms in the top row. As a result, no oxygen bonding was found at either surface As or In atoms. The subsequent water purge reacts with tri-EMAHf to facilitate the growth of hafnium oxides there. The interface obtained by ALD in the present study found no evidence of oxygen bonding to any atom in the trough. The unpassivated surface In atoms may contribute to the frequency dispersion in the accumulation region of n-In0.53Ga0.47As MOSCAP.
15. SUBJECT TERMS
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Final Report for AOARD Grant 134033 “Probing/manipulating the interfacial atomic bonding between high $\kappa$ dielectrics and InGaAs for ultimate CMOS”

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Professor Minghwei HONG
Graduate Institute of Applied Physics and Dept. of Physics, National Taiwan University
1, Section 4, Roosevelt Rd., Taipei, Taiwan, Republic of China
Phone: 886-2-3366-5193; FAX: 886-2-2363-9984; mhong@phys.ntu.edu.tw

Professor J. Raynien KWO
Department of Physics, National Tsing Hua University
Hsinchu, Taiwan, Republic of China
Phone: 886-3-574-2800; FAX: 886-2-2365-5404; raynien@phys.nthu.edu.tw

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Abstract:
The MOS technology using high-$\kappa$ dielectrics on high carrier mobility semiconductors of InGaAs leading to a faster speed at lower power is now at the International Technology Roadmap for Semiconductors (ITRS). Great efforts have been dedicated to understand the high $\kappa$’s/InGaAs interfaces. We then present the interfacial electronic structure of in-situ HfO$_2$ on In$_{0.53}$Ga$_{0.47}$As(001)-4x2 probed by synchrotron radiation photoemission. The dielectric film was prepared by atomic layer deposition (ALD) with the precursors as tetraakis[ethylmethylamino] Hafnium (TEMAHf) and water. The samples were always kept under ultra-high-vacuum (UHV) from the MBE/ALD preparation chambers to data-acquisition photoemission chamber in the synchrotron-radiation facility. The samples have never exposed to air. The high-resolution work with a short inelastic mean-free path allowed us for the first time to identify atom-to-atom interaction at this interface. The Hf atoms maintain four-fold coordination, both after the initial TEMAHf deposition and the subsequent water purge. The Hf atoms initially bond to the As dangling bonds of the surface As atom located on the edges of the raised ridges. One EMA ligand is removed in this process. Subsequent water exposure substitutes OH ligand for one or more remaining EMA ligands. These in turn react with TEMAHf to form O-Hf-O bonds allowing the hafnium oxides to grow. The surface In atoms on the terrace of the raised ridges were partially removed, but none bonded of the precursor atoms. Correlations between the interfacial electronic structure and the electric performance (Appl. Phys. Lett. 100, 172110 (2012)) is discussed.

Introduction:
The growth of high-$\kappa$ dielectrics via atomic layer deposition (ALD) has attracted much attention because it provides a well-controlled thickness in an atomic layer-by-layer fashion, as well as unprecedented uniformity and conformity. The precursors, trimethylaluminium (tri-MA, or TMA) and tetraakis-ethylmethylaminohafnium (tetraakis-EMAHf, or TEMAHf), are most commonly employed. Subsequent exposure to water converts the precursors to the oxides Al$_2$O$_3$ and HfO$_2$, respectively. Measurements of quasi-state capacitance-voltage (QSCV) and conductance-voltage at various temperatures of the ALD-HfO$_2$ on In$_{0.20}$Ga$_{0.80}$As(001)-4x2 interface delivered an interfacial trap density ($D_{it}$) in the midgap at least four times smaller than that of the Al$_2$O$_3$ on the same substrate. Moreover, ALD HfO$_2$ on higher Indium-content InGaAs such as In$_{0.53}$Ga$_{0.47}$As(001)-4x2 showed the absence of a midgap $D_{it}$ peak and excellent

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thermal stability at high temperatures, and device performances of very high drain currents/ transconductance.\textsuperscript{2,3} Clearly, HfO\textsubscript{2}, along with a higher dielectric constant, is a better candidate for the gate dielectric than Al\textsubscript{2}O\textsubscript{3} in device applications.

After years of studies on metal-oxide-semiconductor field effect transistor (MOSFET), superior performance would always depend on having the semiconductor surface effectively passivated by the dielectric oxide. Indeed, the research that has been carried out in the absence of native substrate oxides\textsuperscript{1,2,4,5} showed that the CV curves and x-ray photoelectron spectroscopy (XPS) spectra are different from those in the presence of native oxides.\textsuperscript{5-11} Equally significant is the absence of the notorious As-As dimers. Knowledge of the interfacial electronic structure of an initial precursor, say, TEMAHf, with InGaAs is essential to understanding the cause of the superb electric performance of HfO\textsubscript{2}/InGaAs. To date, little or no relevant information is available in the established literature.\textsuperscript{4,7,8,10}

**Experiment:**

The interfacial electronic structure of ALD HfO\textsubscript{2} produced by (TEMAHf+H\textsubscript{2}O) on In\textsubscript{0.53}Ga\textsubscript{0.47}As (001)-4x2 was probed by high-resolution synchrotron radiation photoelectron spectroscopy (SRPES). SRPES is renowned for its high sensitivity to a change in the surface electronic environment. The \textit{n}-type In\textsubscript{0.53}Ga\textsubscript{0.47}As(001) layer with Si dopants was grown on N\textsuperscript{+} epi-ready InP(001) substrate, using molecular beam epitaxy (MBE). The In\textsubscript{0.53}Ga\textsubscript{0.47}As/InP samples were then moved through ultra-high-vacuum (UHV) transfer modules, under a background pressure of 10\textsuperscript{-10} Torr, to an arsenic-free UHV chamber, in which the samples were heated up to \textasciitilde460 \textdegree C to obtain (4x2)-reconstructed In/Ga stabilized In\textsubscript{0.53}Ga\textsubscript{0.47}As(001) surface. They were then transferred under UHV to a customized ALD reactor\textsuperscript{12} for the deposition of Tetrakis [ethylmethylamino]Hafnium (TEMAHf) followed by the water purge. The samples then were stored in a portable UHV module and carried to the National Synchrotron Radiation Research Center (NSRRC) for the SRPES measurements. The vacuum condition for the sample transfer was always kept in the mid 10\textsuperscript{-10} Torr range. The energy resolution was about 60 meV, and the binding energy scale (EB) was established according to the Fermi edge of Ag.

**Results and Discussion:**

**Figure 1** shows scans for the Hf 4f, In 4d, Ga 3d, and As 3d core levels on clean as well as precursor-covered surfaces. At the 120-eV photon energy used here, the cross section of the Ga 3d and In 4d core level is approximately of the same magnitude. The data in Fig. 1 show that TEMAHf pulses readily adhere to the InGaAs surface with its functional groups intact as well as with the loss of one ligand. After the first water exposure the O 2s state is already in evidence and grows with repeated cycles of TEMAHf and water exposure. The nature of system indicates the increasing growth of hafnium oxide and the presence of Hf-OH ligands. The inset shows an enlarged plot of the combined Hf 4f, In 4d, and Ga 3d region, with the spectra all normalized to the same area. New features seen in the TEMAHf-covered data are tails on both ends of the overlapping spectra. They are attributed to contributions from the Hf 4f core level, in view of the relatively large spin-orbit splitting of 1.71 eV\textsuperscript{13} and the expected large line width. The tails are separated by an energy so large that they must be due to two chemically distinct types of Hf. The difference in binding energy could represent a difference in the ligand structure or in final state screening. The feature at the low-EB tail is labeled Hf* and the other one Hf**. As can be seen in **Fig. 1**, the former component is steadily enhanced in intensity by repeated cycles of deposition, while the intensity of the Hf** component remains unchanged. These observations are essential to the formulation of the data analysis and lead the interpretation of our results.

A fit for the clean As 3d core levels is shown in **Figs. 2(a) and (b)**, and for Ga 3d/In 4d in

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Figs. 3(a). The fitting algorithm can be found elsewhere.\textsuperscript{14} Although the In content was 20% in the previous report,\textsuperscript{14} the surface core-level shifts (SCLSs) were almost identical; namely, they are \( -0.192, -0.331, \) and \(+0.263\) eV for As \( 3d \), Ga \( 3d \), and In \( 4d \) core levels, respectively. Guided by known reconstruction of the \( \beta' \textsuperscript{3}'(4x2) \) model for the reconstructed \( \text{In}_{0.53}\text{Ga}_{0.47}\text{As}(001)-4x2 \) surface (see Fig. 4(a)),\textsuperscript{15} we assigned the In(S) state to the undimerized In atoms in the top row and the As(S) state to the As atoms located on the edges of the raised ridges. Note that the signs of the As and In states are consistent with the shifts expected for monovalent anions and cations with a dangling bond. However, it is surprising to observe a negative SCLS for Ga because it is opposite to that found for the SCLS of Ga in other GaAs surfaces.\textsuperscript{16} This signal is not due to surface Ga atoms, but Ga atoms that reside in the second layer with a small percentage. They are correspondingly labeled Ga(SS) for “subsurface”. This indicates that the In surface segregation has completely displaced Ga atoms from the surface.

Panels (c), (d) and (e) of Fig. 2 show the development of the As \( 3d \) spectrum with increasing exposure to TEMAHF and water. After TEMAHF deposition a new component intrudes on the As \( 3d \) spectrum, namely the Hf \( 5p_{1/2} \) core-level. The properties of the broad Hf \( 5p_{1/2} \) line were established from data with a thick passivating layer, where the binding energy relative to the bulk As line (and lifetime width) could be determined. The reacted As labeled as As* appears with an SCLS of \(-0.135\) eV. The fact that no other As-related component is resolved suggests effective coverage of TEMAHF on every surface As atoms on the edges of the raised ridges. However, the relatively large line width of the As* state suggests that there is not a single well defined chemical environment for all the former As surface atoms.

Since all the Ga \( 3d/\text{In} \) \( 4d/\text{Hf} \) \( 4f \) lie in a 5-eV energy window, and in order to obtain convergence to physically acceptable results in the least-squares optimizations, the energy separation between the bulk components of Ga and In was fixed to known values. Furthermore, the SO splitting and SO ratio of the Hf \( 4f \) core level were fixed at 1.71 eV and 0.72, respectively.\textsuperscript{13} The resulting constrained set of six doublets was then able to provide a satisfactory representation of the experimental data. The result, shown in Fig. 3(b), indicates that adsorption of the TEMAHF precursor alone produced no new components for either the In \( 4d \), or the Ga \( 3d \) states. In the fit, the binding energies of the \( 7/2 \) state of the Hf* and Hf** components were at 17.08 and 17.85 eV, respectively. The In(S) SCLS is reduced to \( 0.142 \) eV, and the SCLS for the Ga(SS) component remains virtually unchanged, but after water exposure it is changed significantly, see below. An observed decrease of the intensity of the In(S) component suggests that some top-row In atoms were etched off. It may be relevant that in a previous investigation with a 0.8-nm thick \( \text{HfO}_2 \), diffusion of In to the surface of the dielectric \( \text{HfO}_2 \) was observed.\textsuperscript{1}

The fitted results to the core-level spectra suggest the formation of an As-Hf bond, in which the As* state in Fig. 2(c) forms a bond with the Hf* state in Fig. 3(b). One EMA ligand is removed from a TEMAHF molecule to couple it to the solid. See the schematic drawing in Fig. 4(b). Note that no As atoms are removed by the adsorption of precursor and the Hf remains in a 4-fold coordination. The loss of one EMA ligand and the location on the raised ridge makes the Hf atom of the adsorbed molecule much more accessible to water vapor than it is in the complete TEMAHF molecule. There it is surrounded by 8 aliphatic methyl and ethyl groups with a total of 32 H atoms at the surface.

The core-electron binding energy of the Hf* state in the overlayer is circa 1 eV smaller than that found in the oxidation of metallic Hf to \( \text{HfO}_2 \).\textsuperscript{17} While both are presumably referenced to the Fermi level, there are residual concerns when comparing data on metallic and insulating systems in which the final state screening is completely different in character. Neglecting that concern and taking the results at face value, one could conclude that the residual OH and EMA ligands in the oxide layer are responsible for the reduced binding energy. With above argument, we
understand that the Hf core hole in TEMAHf (the Hf** state) is screened only by the polarization of its four nitrogen ligands, while that in the growing tri-EMAHf (the Hf* state) is now part of a solid with much better screening based on charge flow from the solid. Smaller electronegativity of As versus N would give rise to a smaller binding energy of the Hf* state than the Hf** state.

In the water-purged tri-EMAHf/InGaAs surface (1 cycle of operation), the model function still represents the As 3d spectrum without the need for an additional component. However, the SCLS of the As* component is reduced to 100 meV. Additional cycles produce little or no change in the line shape, meaning that the first cycle has already determined the interfacial electronic structure. This is certainly in contrast to the deposition of TMA, which requires at least 10 pulses before the edge-row As atoms are covered. The model function with the six doublets represents the line shape of the Ga 3d/In 4d/Hf 4f core levels equally well, and no oxidized indium component is found in the spectrum. The Hf 4f core levels of the two components now appear at 18.04 eV and 17.01 eV EB. The bonding nature of the Hf* component which grows with continued deposition must be altered by the introduction of water, as OH ligands and Hf-O-Hf bonds replace the EMA ligands. We now label it HfO. The most significant observation is that there is so little change in binding energy from Hf* to HfO and take that as an indication that the Hf remains tetravalent throughout this process. The large line width can of course accommodate small differences in binding energy expected for the possible range of ligand chemistry. Throughout all this the Hf** component remains unchanged (see Figs. 3(d) and 1) suggesting that represents physisorbed unmodified TEMAHf that resists the effect of water vapor.

FIG. 1. Scans of Hf 4f, In 4d, Ga 3d, and As 3d core levels for cycling purges of TEMAHf and H2O.

FIG. 2. A fit to the As 3d core levels excited with a photon energy of 120 eV, where (a) clean and $\theta_e = 0^\circ$, (b) clean and $\theta_e = 60^\circ$, (c) TEMAHf only and $\theta_e = 0^\circ$, (d) 1 cycle of TEMAHf + H2O, (e) 2 cycles of TEMAHf + H2O, (f) 3 cycles of TEMAHf + H2O, (g) 10 cycles of TEMAHf + H2O at RT, and (h) 10 cycles of TEMAHf + H2O at elevated temperature. The symbol $\theta_e$ indicates the emission angle. Raw data are indicated using dots, and solid lines are the fit results.

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The reaction of the As-bonded Hf with a H$_2$O molecule breaks another Hf-EMA bond. The replacement of another Hf-N bond by a Hf-OH bond is energetically favorable. It leaves an OH group attached to the Hf and uses the remaining H to convert the EMA ligand into an EMA molecule that is emitted. No ionized products or dangling bonds are created. This process leaves the Hf with four ligands, one to As, one to OH, and two to EMA, and in its most stable tetra-valent state. It creates no dangling bonds. It also leaves the OH site well exposed for future reaction. In principle, all three remaining EMA ligands can be removed by this process, but only one is required to form a minimally connected string of Hf-O-Hf atoms. Schematic drawing for 1 cycle of operation is represented in Fig. 4(c).

During the next exposure to TEMAHf, the exposed Hf-OH site is available for reaction. An EMA ligand of the incoming TEMAHf molecule receives the H of the OH, becoming an EMA.
molecule that is emitted, while the remaining Hf-O forms a bond to the Hf of incoming TEMAHf molecule. The result is a Hf-O-Hf bridge, similar to that found in the bonding of HfO$_2$. This process, too, is energetically favorable since it forms a strong Hf-O bond and emits a neutral molecule. This does not imply that the overlayer will eventually consist of crystalline HfO$_2$ or even an amorphous compound with that composition. It only indicates a local chemical identity with the strong oxygen bonding of the dioxide. Indeed, it is very likely that OH and possibly even some EMA ligand will remain on the Hf atoms that grow into this overlayer.

In a fit, the subsurface Ga atoms now show a +0.32 eV SCLS labeled as Ga(SS)* in Fig. 3(c). Because those Ga atoms have no dangling bonds and are below the dangling bond As surface atoms, the positive shift is unlikely due to oxygen bonding. The objection to the proposed oxygen bond is based on the fact that it would require the breaking of an existing bond to As and creating new dangling As bond. One possibility is that the bonding of the surface As to Hf will change electronic environment at the Ga(SS) site, but a crude estimate suggests that the change would be in the wrong direction. Another possibility is that the change in surface energy produced by the overlayer counteracts the forces driving the surface segregation allowing an exchange between surface In and subsurface Ga. The Ga would then have an expected positive SCLS and could even be oxidized.

In InGaAs, the top row In atoms could become traps for electrons, if not effectively passivated. The positive SLCS of those atoms indicates that the electronic state is deficient in charge, and could therefore attract and accumulate electrons upon application of a positive gate bias to $n$-In$_{0.53}$Ga$_{0.47}$As MOSCAP. Consequently, the unpassivated surface In atoms and charge deficiency of the subsurface Ga atoms may contribute to the finite frequency dispersion in the accumulation region for $n$-In$_{0.53}$Ga$_{0.47}$As MOSCAP.$^{1,2}$

On the other hand, the CV data showed that the room temperature capacitance of the inversion region of $n$-In$_{0.53}$Ga$_{0.47}$As MOSCAP increases monotonically with increasing negative gate bias. The relatively larger frequency dispersion in the accumulation region for $p$-In$_{0.53}$Ga$_{0.47}$As MOSCAP was attributed to interface traps in the band gap near the valence band of In$_{0.53}$Ga$_{0.47}$As or to border traps located near the oxide/ In$_{0.53}$Ga$_{0.47}$As interface.$^2$ It has been suggested that the border traps at the valence band edge are mainly due to As dangling bonds or to Ga-Ga dimers.$^{18}$ In the present study, the reacted surface As atoms (As(S*)$_4$), although totally passivated, still show sizable chemical shift from the bulk, which might be responsible for the CV behavior.

In summary, we have elucidated the mechanism and nature of the bonding between the Hf atom in TEMAHf and In/As atoms of the reconstructed In$_{0.53}$Ga$_{0.47}$As(001)-4x2 surface. The TEMAHf precursor is either intact, or loses one ethylmethylamino ligand to become tri-EMAHf. The initial purge of the TEMAHf precursors effectively passivates all the edge As atoms in the top row. As a result, no oxygen bonding was found at either surface As or In atoms. The subsequent water purge reacts with tri-EMAHf to facilitate the growth of hafnium oxides there. The interface obtained by ALD in the present study found no evidence of oxygen bonding to any atom in the trough. The unpassivated surface In atoms may contribute to the frequency dispersion in the accumulation region of $n$-In$_{0.53}$Ga$_{0.47}$As MOSCAP.

References:


List of Publications and Significant Collaborations that resulted from your AOARD supported project:

List of Publications:


Encyclopedia Chapter and Conference Proceedings:


Conference presentations (Invited)


5. “High k dielectrics on high carrier mobility semiconductors for ultimate CMOS - accomplishments
and the remaining challenges”, The National Nano Device Laboratories (NDL), Hsinchu, Taiwan, June 26, 2014.


8. “High k dielectrics on high carrier mobility semiconductors for ultimate CMOS - accomplishments and the remaining challenges”, TSMC, Hsinchu, Taiwan, April 24, 2014.


11. “Pushing the ultimate CMOS and beyond”, M. Hong, NTU/TSMC/NSC Industrial-Academia Big League Center, National Taiwan University, Taipei, Taiwan, December 31, 2013.


14. “Pushing the ultimate CMOS and more – a physicist’s role”, Dept. Physics, National Chung Hsing University, Taichung, September 27, 2013.

15. “Realization of III-V MOSFETs using High k Gate Dielectrics on InGaAs Semiconductors”, J. Kwo and M. Hong, the 2013 Asia-Pacific Radio Science Conference (AP-RASC’13), Howard International House, Taipei, Taiwan, September 3-7, 2013.


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Conference presentations (Contributed)


Interactions with industry or with Air Force Research Laboratory scientists or significant collaborations that resulted from this work:

Collaboration with Dr. Gail Brown of Air Force Research Laboratory (AFRL), Dayton, Ohio 45433, USA, resulting in two SCI papers and two conference presentations.

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