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# Band Offsets and Interfacial Properties of HfAlO Gate Dielectric Grown on InP by Atomic Layer Deposition

Lifeng Yang<sup>1</sup>, Tao Wang<sup>2</sup>, Ying Zou<sup>1</sup> and Hong-Liang Lu<sup>2\*</sup>

## Abstract

X-ray photoelectron spectroscopy and high-resolution transmission electron microscopy have been used to determine interfacial properties of HfO<sub>2</sub> and HfAlO gate dielectrics grown on InP by atomic layer deposition. An undesirable interfacial InP<sub>x</sub>O<sub>y</sub> layer is easily formed at the HfO<sub>2</sub>/InP interface, which can severely degrade the electrical performance. However, an abrupt interface can be achieved when the growth of the HfAlO dielectric on InP starts with an ultrathin Al<sub>2</sub>O<sub>3</sub> layer. The valence and conduction band offsets for HfAlO/InP heterojunctions have been determined to be  $1.87 \pm 0.1$  and  $2.83 \pm 0.1$  eV, respectively. These advantages make HfAlO a potential dielectric for InP MOSFETs.

**Keywords:** Band alignments, HfAlO dielectric, InP, Atomic layer deposition

## Background

As silicon-based complementary metal-oxide-semiconductor (CMOS) devices approach their fundamental limits when scaled down, the adoption of new technologies is urgently required to meet the demands for higher performance and less power dissipation integrated circuits. The integration of III–V compound semiconductors with high-*k* gate dielectrics is the leading candidate to address many of these issues [1–6]. III–V compound semiconductors including GaAs, InGaAs, and InP have drawn quite a lot of attention as alternative channel materials due to their high electron mobility and low effective mass over Si. Of these candidates, InP could be promising because it is a material with weak Fermi-level pinning effect and has a high electron saturation velocity ( $2.5 \times 10^7$  cm/s) [7]. However, there are still some bottlenecks impeding the actual implementation of InP channel material. One of the major challenges is finding thermodynamically stable dielectric on InP surface with good interfacial properties like SiO<sub>2</sub>/Si counterpart [8]. Various chemical treatments have been extensively explored to achieve effective passivation on

the InP surface. Such methods include NH<sub>4</sub>OH, (NH<sub>4</sub>)<sub>2</sub>S, H<sub>2</sub>S, and F treatment [7, 9–11].

Band alignment of high-*k*/InP interface is of great importance for InP-based MOSFET researches. Recently, Chou et al. measured the band offset between InP (100) and ALD Al<sub>2</sub>O<sub>3</sub> with various passivation methods using internal photoemission (IPE) [12]. The barrier heights from the top of InP valence band (VB) to the bottom of Al<sub>2</sub>O<sub>3</sub> conduction band (CB) and conduction band offset were determined to be 4.05 and  $2.7 \pm 0.10$  eV, respectively. With a high *k* of ~20–25, HfO<sub>2</sub> has been extensively studied as an alternative high-*k* gate dielectric both in Si-based and III–V technology. The barrier height at HfO<sub>2</sub>/InP interface is also measured using IPE by Xu et al. to be 3.89 eV for the sample without passivation treatment [13]. To improve the interfacial and thermal properties, gate dielectric engineering has been successfully adopted in high-*k*/III–V compound semiconductor technology by using a combination of Al<sub>2</sub>O<sub>3</sub> and HfO<sub>2</sub> films recently. Kim et al. reported that the In out-diffusion and the subsequent In-related phase generation can be effectively suppressed by introducing Al<sub>2</sub>O<sub>3</sub> to the HfO<sub>2</sub> film grown on InP. Moreover, intermixing of Al<sub>2</sub>O<sub>3</sub> with HfO<sub>2</sub> to form HfAlO on InP is expected to adjust the band offset. In this study, HfAlO gate dielectrics were grown on InP substrates using alternative cycles atomic layer deposition

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(ALD) of  $\text{Al}_2\text{O}_3$  and  $\text{HfO}_2$  starting with an ultrathin  $\text{Al}_2\text{O}_3$  layer. ALD has manifested itself as a technique suitable for the semiconductor industry due to its capability of growing uniform and conformal thin films [14]. Accurate controls over chemical stoichiometry and thickness at atomic scale can also be achieved by ALD. The ultrathin  $\text{Al}_2\text{O}_3$  layer was introduced to the interface between  $\text{HfAlO}$  dielectrics and InP substrate to diminish the undesirable interfacial  $\text{InP}_x\text{O}_y$  layer. The band offset and interfacial properties of the formed  $\text{HfAlO}$  gate dielectric grown on InP has been investigated using x-ray photoelectron spectroscopy (XPS). The energy band diagrams of the  $\text{HfAlO}/\text{InP}$  heterojunction is then constructed, which can provide vital information on fabricating InP MOSFET.

## Methods

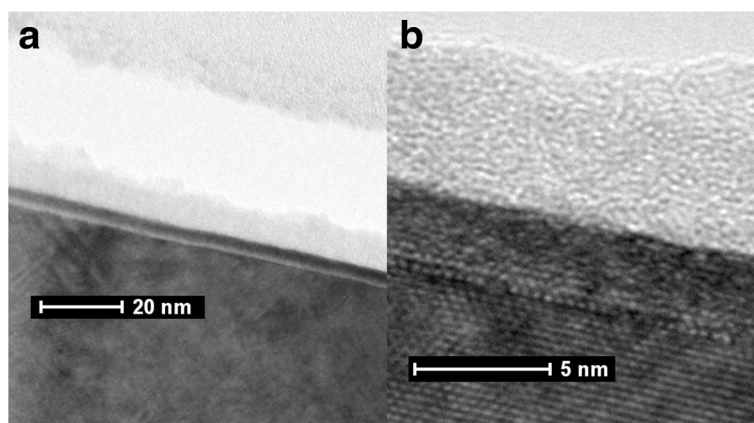
The heterostructures were prepared on *n*-type (100) InP wafers with a doping concentration of  $\sim 1 \times 10^{17} \text{ cm}^{-3}$ . Prior to the ALD process, the InP substrates were degreased using acetone and methanol for 5 min each, followed by a diluted 2% hydrofluoric acid (HF) solution etching for 1 min to remove native oxide. After cleaning, the substrate was immediately transferred to a BENEQ TFS-200 ALD reactor where  $\text{HfO}_2$  and  $\text{HfAlO}$  thin films were prepared at 300 °C. Thin  $\text{HfAlO}$  film was grown using alternative cycles ALD of  $\text{Al}_2\text{O}_3$  and  $\text{HfO}_2$  starting with  $\text{Al}_2\text{O}_3$ . The precursors used were trimethylaluminum (TMA) and  $\text{H}_2\text{O}$  for  $\text{Al}_2\text{O}_3$  and tetrakis(ethylmethylamido)hafnium (TEMAH) and  $\text{H}_2\text{O}$  for  $\text{HfO}_2$ . Four sets of samples were prepared for XPS measurements: (1) a 4 nm thick  $\text{HfO}_2$  film grown on InP substrate to detect the interface property of the  $\text{HfO}_2/\text{InP}$  heterojunction; (2) a 30 nm thick  $\text{HfAlO}$  film grown on InP substrate to measure the valence band maximum (VBM) and  $\text{Al } 2p_{3/2}$  of bulk  $\text{HfAlO}$ ; (3) a 4 nm thick  $\text{HfAlO}$  film grown on InP substrate to determine the energy difference between  $\text{Al } 2p_{3/2}$  and  $\text{In } 3d_{5/2}$  at the

$\text{HfAlO}/\text{InP}$  heterojunction's interface; and (4) a clean InP substrate (HF-dipped) to measure the VBM and  $\text{In } 3d_{5/2}$  of bulk InP.

High-resolution transmission electron microscopy (HR-TEM) was used to obtain the images of high-*k*/InP interface at atomic scale. Both TEM with low and high magnification were performed to investigate the interfacial profile and fringe atom arrangement. Accurate film thicknesses can also be measured directly. The XPS spectra were recorded using a Thermo Scientific ESCA-LAB 250 XPS system equipped with a monochromatic  $\text{Al } K\alpha$  source ( $h\nu = 1486.6 \text{ eV}$ ). The source power is 150 W (15 kV  $\times$  10 mA) at a takeoff angle of 90°. Scans with a step of 0.05 eV and pass energy of 20 eV were performed for 20 times for binding energy of specific elements. The chemical compositions for the prepared  $\text{HfAlO}$  thin film were detected to be 14.23% for Hf, 22.36% for Al, and 62.55% for O, respectively. Valence band scans with a step of 0.01 eV and pass energy of 20 eV were performed for 30 times for valence band spectra. Charge correction was performed using the known position of C 1s spectra at 284.8 eV. The XPS spectrometer energy scale was also calibrated using Cu  $2p_{3/2}$ , Ag  $3d_{5/2}$ , and Au  $4f_{7/2}$  photoelectron lines located at 932.67, 368.26, and 83.98 eV, respectively.

## Results and Discussion

Figure 1 gives the TEM images of the as-deposited  $\text{HfO}_2$  film grown on InP. As shown in Fig. 1(a), the dielectric film in amorphous structure is observed in a large area with low magnification. Moreover, it can be seen clearly from the Fig. 1b, by high magnification, that an interlayer ( $\sim 1 \text{ nm}$ ) exists at the interface between the  $\text{HfO}_2$  film and the InP substrate. The visible interlayer is inferred to consist of In-O and P-O compounds which can be assigned to the diffusion of In and P atoms into the  $\text{HfO}_2$  film.



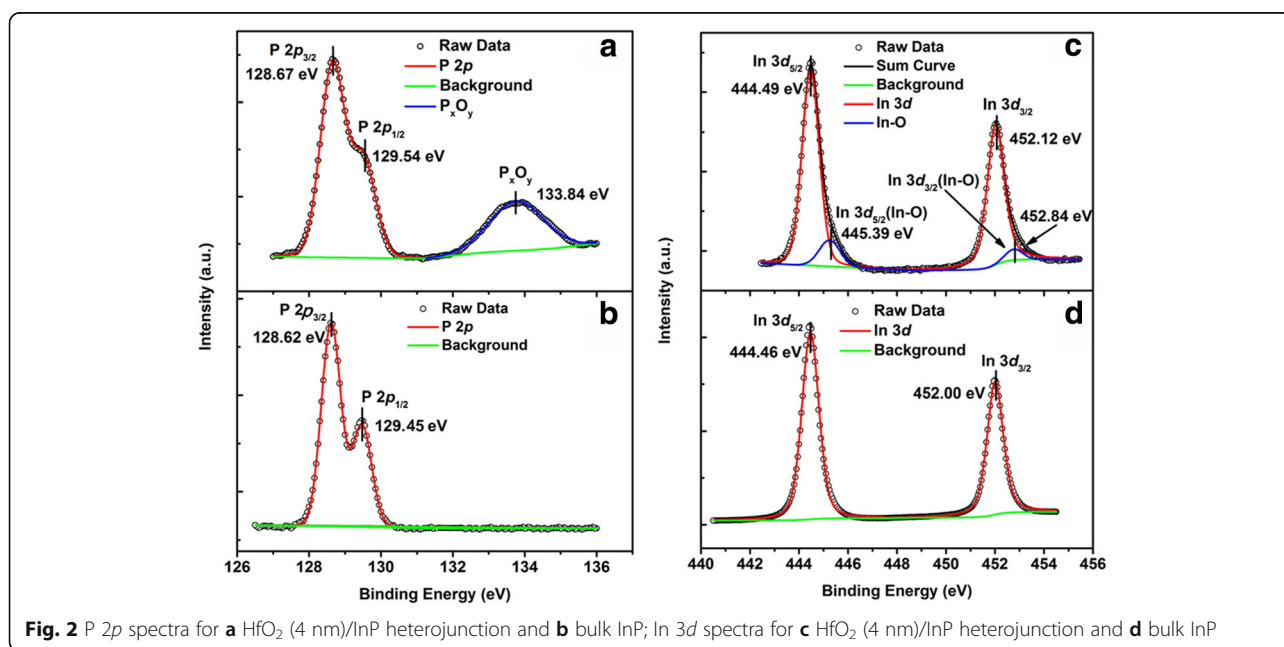
**Fig. 1** HR-TEM images of as-grown  $\text{HfO}_2$  thin films on InP substrate, with **a** low magnification and **b** high resolution

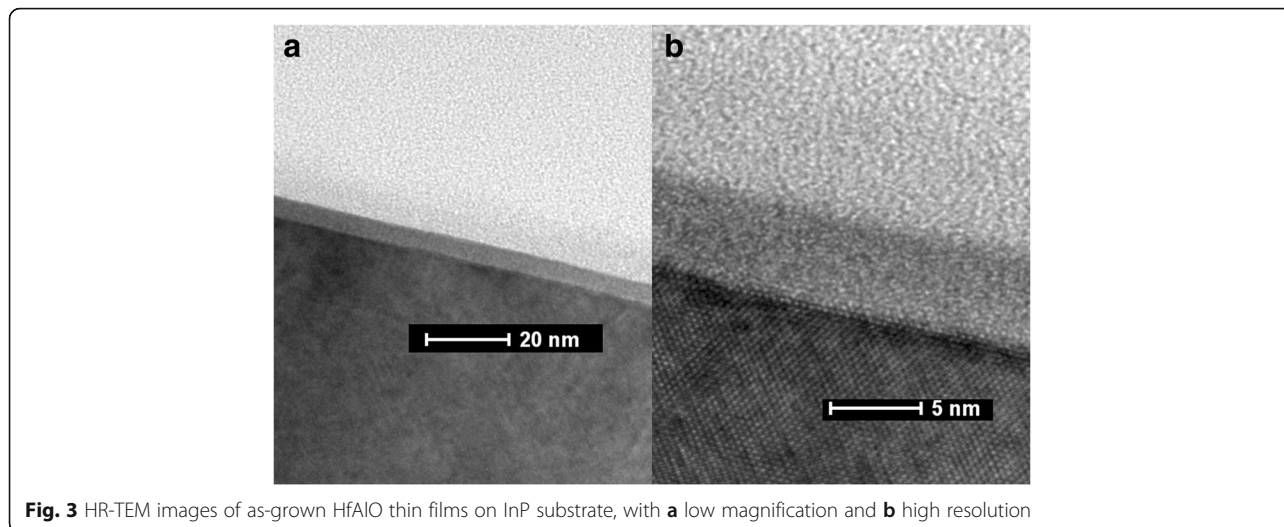
To further investigate the effect of  $\text{HfO}_2$  film deposition on the interfacial properties and the components of the interlayer, In  $3d$  and P  $2p$  XPS spectra for sample (1) (4 nm thick  $\text{HfO}_2$  sample on InP substrate) and sample (4) (bulk InP sample) are investigated and presented in Fig. 2. According to Fig. 2a, the P  $2p$  core level spectrum attributed to the In-P bond from the substrate is composed of two peaks, P  $2p_{3/2}$  (128.67 eV) and P  $2p_{1/2}$  (129.54 eV), with a spin orbit split (SOS) of 0.87 eV. Compared with the P  $2p$  XPS spectra of bulk InP sample, shown in Fig. 2b, an increase of the full width at half maximum (FWHM) of the P  $2p$  for  $\text{HfO}_2/\text{InP}$  sample was observed in the spectra range of 127.2–130.6 eV. This result is in agreement with the findings obtained by Dong et al. [15, 16]. They suggested that there is an increase in the distribution of the chemical bonding environments at the InP/ $\text{HfO}_2$  interface during the ALD process. It is ascribed to the increased surface disorder introduced by the reaction between TEMA and the InP substrate. Moreover, a new peak for 4-nm-thick  $\text{HfO}_2/\text{InP}$  is clearly observed at a binding energy of 133.84 eV. Investigation by Lu et al. shows the peak is assigned to the P-O bonding [11]. Besides, according to Fig. 2(d), the In  $3d$  core level spectrum attributed to the In-P bond from the InP substrate is composed of two spin-orbit split peaks, In  $3d_{5/2}$  (444.46 eV) and In  $3d_{3/2}$  (452.00 eV), with a SOS of 7.54 eV [17]. However, as shown in Fig. 2(c), new peaks located at 445.39 and 452.84 eV are also found in the In  $3d$  spectrum for  $\text{HfO}_2$  (4 nm)/InP sample, which are attributed to In-O bonding and cannot be detected in bulk InP. This spectrum further confirms the formation of  $\text{InP}_x\text{O}_y$  layer at the interface of  $\text{HfO}_2/\text{InP}$  heterojunction. According to

investigations by Chen et al. and Driad et al., respectively, the high interface state density will result in a large frequency dispersion, a low breakdown voltage and a large gate leakage current for InP metal-oxide-semiconductor capacitors with  $\text{HfO}_2$  deposited directly on InP substrates [18, 19]. What is more, when the  $\text{HfO}_2/\text{InP}$  heterojunction is fabricated into MOSFETs, the interlayer will cause a low transconductance and a large subthreshold swing.

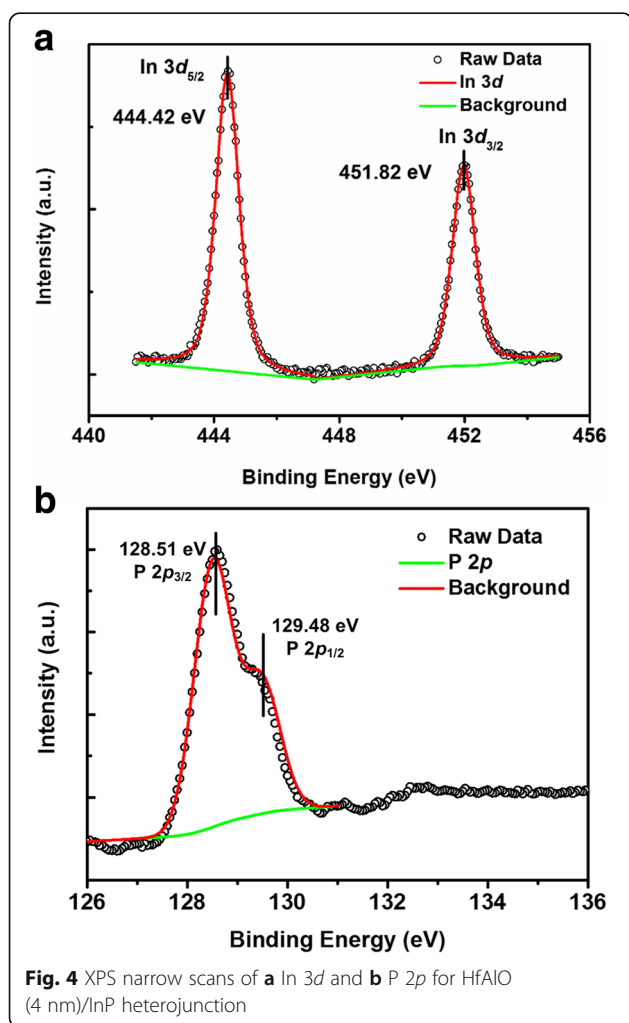
An interfacial layer is easily formed as  $\text{HfO}_2$  deposited directly on InP substrate. The  $\text{HfAlO}$  layer is deposited on InP started with an ultrathin  $\text{Al}_2\text{O}_3$  layer. Figure 3 shows the HR-TEM images with low and high magnification for the  $\text{HfAlO}$  (4 nm)/InP sample, which reveal an abrupt interface between  $\text{HfAlO}$  and InP. Chemical composition analysis for the  $\text{HfAlO}$  (4 nm)/InP sample was revealed by XPS as well, as shown in Fig. 4. Compared with the In  $3d$  and P  $2p$  spectra from the 4-nm-thick  $\text{HfO}_2$  grown on InP substrate, it is clear that In-O or P-O component completely vanish. The formation of a clean interface strongly indicates the effect of “self-cleaning,” and similar effect of thin  $\text{Al}_2\text{O}_3$  film grown on GaAs by ALD has been studied by Hinkle et al. [20]. The results imply that a thin  $\text{Al}_2\text{O}_3$  layer can passivate the InP surface by acting as a barrier layer to prevent the diffusion of In and P from substrate into the dielectric film. Similarly, the shape of P  $2p$  spectrum from the InP bulk peak is different with the one from the pure InP substrate. It is indicated that the FWHM of the sample also increase due to the reaction between TMA and InP substrate during the ALD process.

The band alignment for  $\text{HfAlO}/\text{InP}$  heterostructure at the interface is then investigated, which is a crucial





**Fig. 3** HR-TEM images of as-grown HfAlO thin films on InP substrate, with **a** low magnification and **b** high resolution



**Fig. 4** XPS narrow scans of **a** In 3d and **b** P 2p for HfAlO (4 nm)/InP heterojunction

standard to measure the ability to suppress leakage current for devices. To determine the valence band offset ( $\Delta E_v$ ) of HfAlO/InP, Kraut’s method is employed, which is based on the assumption that the energy separation between the valence band edge and core level (CL) of the substrate remains the same before/after dielectric deposition [21–23]. In this work, In  $3d_{5/2}$ , Al  $2p_{3/2}$ , and Hf  $4f_{7/2}$  are selected as reference CLs for the substrate and high- $k$  dielectrics, respectively. The CL spectra were fitted by Shirley background and a nonlinear Gaussian-Lorentzian line shape with a fixed spin orbit splitting to determine the respective CL positions. The  $\Delta E_v$  value could be extracted from the following equations,

$$\Delta E_v^{\text{HfAlO/InP}} = \left( E_{\text{In } 3d_{5/2}} - E_{\text{VBM}} \right)_{\text{bulk InP}} - \left( E_{\text{CL peak}} - E_{\text{VBM}} \right)_{\text{bulk HfAlO}} - \Delta E_{\text{CL}} \tag{1}$$

where  $\Delta E_{\text{CL}}$  is the energy difference between In  $3d_{5/2}$  CL spectrum and Al  $2p_{3/2}$  CL spectrum at the interface of HfAlO (4 nm)/InP heterojunction. All of the spectra for thin high- $k$  oxide/InP samples are referenced to the In  $3d_{5/2}$  peak from clean InP sample to compensate for charging effects. The parameters extracted via XPS, for the samples studied, are listed in Table 1 for clarity.

Figure 5 shows VB spectra of InP substrate and 30-nm-thick HfAlO sample as well as and the Al  $2p_{3/2}$  spectra of HfAlO films. The VBM positions were determined by linear extrapolation of the leading edges of the VB spectra recorded from the InP substrate and the bulk HfAlO films to the base lines in order to account for the tail induced by instrument resolution. The uncertainty of the VBM positions should be lower than 0.05 eV because considerable accordance of the fitted lines to the measured data has been obtained. Through

**Table 1** Binding energies (in eV) of core level spectra for all the samples and the valence band maximum (VBM) values (in eV) for the bulk InP and HfAlO (30 nm)/InP samples

	InP (bulk)	HfAlO (30 nm)/InP	HfAlO (4 nm)/InP
In 3d <sub>5/2</sub>	444.46	–	444.42
Hf 4f <sub>7/2</sub>	–	17.61	17.32
Al 2p <sub>3/2</sub>	–	74.46	74.21
VBM	0.79 ± 0.05	2.89 ± 0.05	–

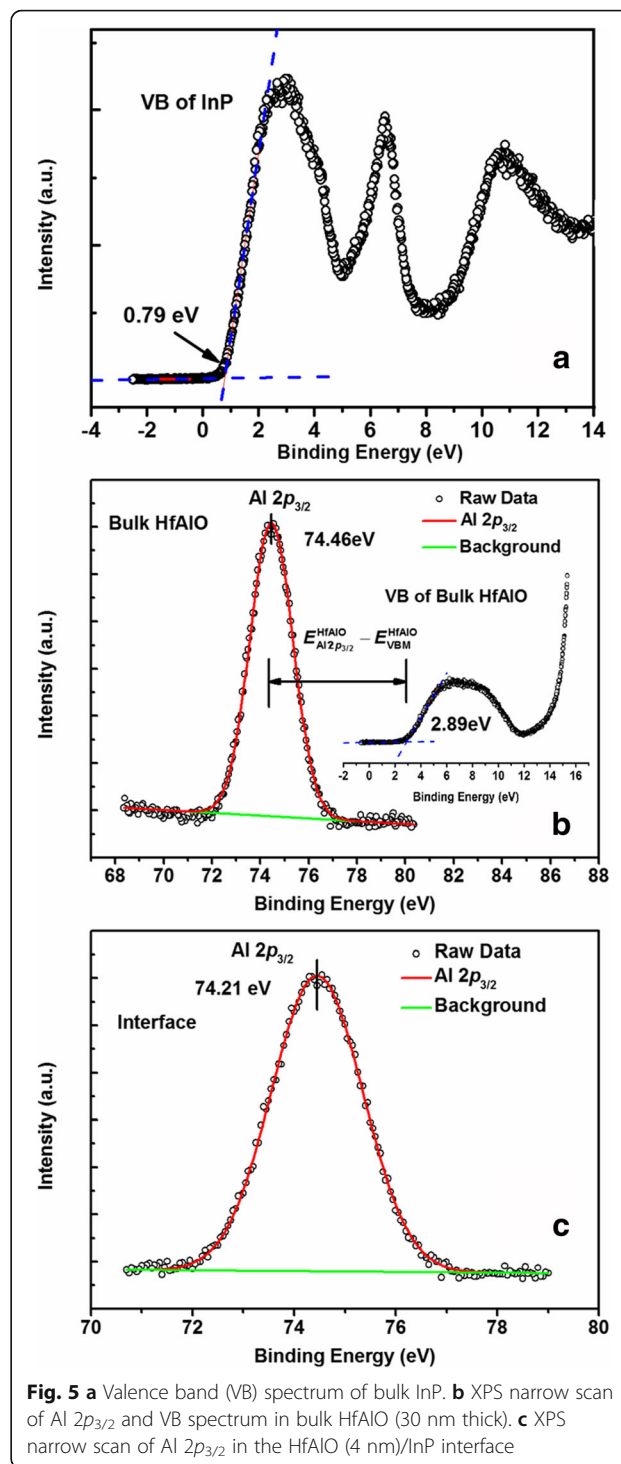
The errors in the peak positions and the VBM values are ±0.05 eV

extrapolating a linear fitting of the leading edges of VB spectra to the base lines, the VBM position is determined to be 0.79 ± 0.05 eV and 2.89 ± 0.05 eV for InP substrate and 30-nm-thick HfAlO films, respectively. The VBM value of bulk HfAlO sample is in good agreement with the previously reported one by Yu et al. [24]. Figure 5b depicts a single and symmetrical peak located at 74.46 eV in the CL spectrum of Al 2p<sub>3/2</sub>, indicating the uniform Al-O bonding state in the 30-nm HfAlO film. Furthermore, the CL spectrum of Al 2p<sub>3/2</sub> for HfAlO (4 nm)/InP heterojunction is shown in Fig. 5c. Compared with thick HfAlO (30 nm)/InP sample, Al 2p<sub>3/2</sub> peak shifts to 74.21 eV at the interface. With all the obtained values above,  $(E_{\text{In } 3d_{5/2}} - E_{\text{VBM}})_{\text{InP}}$ ,  $(E_{\text{Al } 2p_{3/2}} - E_{\text{VBM}})_{\text{HfAlO}}$ , and  $\Delta E_{\text{CL}}$  are calculated to be 443.67 ± 0.05, 71.57 ± 0.05, and 370.21 eV, respectively. As a result, the  $\Delta E_v$  value of HfO<sub>2</sub>/InP heterojunction is determined to be 1.89 ± 0.1 eV using Eq. (1). To improve the accuracy of the VBO value determined by XPS, the combination of Hf 4f and In 3d is also used to obtain the VBO of HfAlO/InP interface. Relative data is summarized in Table 1, and the VBO is determined to be 1.85 ± 0.1 eV. To reduce the measurement error, the VBO value of HfAlO/InP interface is then calibrated to be 1.87 ± 0.1 eV by averaging the two VBO values.

The conduction band offset ( $\Delta E_c$ ) at the high-*k*/InP interface can be determined by the following equation:

$$\Delta E_c = E_g^{\text{high-}k} - E_g^{\text{InP}} - \Delta E_v \quad (2)$$

The energy band gaps ( $E_g$ ) measured by spectroscopic ellipsometry are 5.70 and 6.04 eV for as-deposited HfO<sub>2</sub> and HfAlO films, respectively. Using 1.34 eV energy gap for InP [25], the  $\Delta E_c$  at the HfAlO /InP interface is thus calculated to be 2.83 ± 0.1 eV. According to the investigation above, both of the  $\Delta E_v$  and  $\Delta E_c$  values are larger than 1 eV, which means the HfAlO film supplies enough barrier heights for both electrons and holes. Compared with the  $\Delta E_c$  extracted to be 2.55 eV at the interface of HfO<sub>2</sub>/InP by Xu et al. [13], the  $\Delta E_c$  for HfAlO/InP is a little larger. Furthermore, the  $\Delta E_v$  at the interface of HfO<sub>2</sub>/InP can also be calculated to be 1.81 eV using equation (2), which is a



**Fig. 5** a Valence band (VB) spectrum of bulk InP. b XPS narrow scan of Al 2p<sub>3/2</sub> and VB spectrum in bulk HfAlO (30 nm thick). c XPS narrow scan of Al 2p<sub>3/2</sub> in the HfAlO (4 nm)/InP interface

little smaller than that of HfAlO/InP heterojunction. As a result, the dielectric HfAlO film can suppress both the electrons induced and holes induced leakage current more effectively than the HfO<sub>2</sub> film when InP is chosen as channel material. Considering the better interface condition and higher potential barrier heights, HfAlO can be relatively preferable than HfO<sub>2</sub> as the dielectric film for InP.

## Conclusions

In summary, interfacial properties for HfO<sub>2</sub>/InP and HfAlO/InP heterojunctions have been studied by XPS and HR-TEM. When HfO<sub>2</sub> is deposited on InP substrate by ALD, an undesirable interfacial InP<sub>x</sub>O<sub>y</sub> layer is easily formed at the HfO<sub>2</sub>/InP interface, which will degrade the electrical properties. Fortunately, a thin Al<sub>2</sub>O<sub>3</sub> layer can act as a barrier layer to form an abrupt interface between HfAlO and InP channel layer. The band alignment at the interface of HfAlO/InP heterojunction was studied, and the VBM for HfAlO/InP is 1.87 ± 0.1 eV at interface, leading to a ΔE<sub>c</sub> of 2.83 ± 0.1 eV for HfAlO/InP based on the analysis. This result indicates that HfAlO dielectric film can supply sufficient barrier heights for holes and electrons when InP is chosen as channel material. More researches are needed to make further efforts to optimize the heterojunction's interface condition.

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## Authors' Contributions

LFY and TW carried out the deposition along with its characterization (XPS, TEM). YZ participated in the characterization. LFY, TW, YZ, and HLL participated in the analysis and discussion of the results obtained from the experiments. HLL supervised this study. All authors read and approved the final manuscript.

## Competing Interests

The authors declare that they have no competing interests.

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