

Chemical and Thermal Stability of Alkanethiol and Sulfur Passivated InP(100)

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InP(100) surfaces treated with $\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$ and $\text{C}_n\text{H}_{2n+1}\text{SH}$ are examined by contact angle measurement, X-ray photoelectron spectroscopy, and atomic force microscopy to determine the chemical and thermal behavior of these passivated surfaces. The surfaces coated by octadecanethiol ($n = 18$) self-assembled monolayers (SAMs) are found to be more stable toward oxidation than the S-passivated surface. The chemical stability of octadecanethiol SAMs in various environments is examined. The thiol monolayer is found to be stable in 0.1 M HCl but degrades in 0.1 M NaOH, boiling chloroform, and water. The behavior of these surfaces at elevated temperatures under a vacuum is also investigated. The octadecanethiol-coated InP(100) is stable up to 473 K, above which the films begin to degrade. Unlike other substrates on which the entire molecule including the sulfur headgroup desorbs together, on InP, the sulfur headgroup remains on the surface even after annealing to 673 K. These observations suggest that the desorption occurs by S–C bond cleavage as well as In–S bond cleavage. The sulfur of S-passivated InP is found to be more thermally stable than that of the octadecanethiol monolayer, perhaps due to their different bonding geometries and hence energies.

I. Introduction

The surface chemistry of semiconductors is of great significance to device manufacturing and fundamental properties. InP is a III–V compound semiconductor with a direct band gap ($E_g = 1.34$ eV), which has found applications in many areas¹ including active optoelectronics, photovoltaic devices, and high-speed electron devices such as photodetectors, light-emitting diodes, lasers, high-speed heterojunction bipolar transistors, and more recently microelectromechanical systems (MEMS).² Despite these potential applications, InP devices suffer from surface instabilities, particularly oxidation upon exposure to air. In contrast to Si,³ Ge,⁴ and GaAs,⁵ surface modification of InP has not been studied as thoroughly.

The chemical modification of InP surfaces with S-containing compounds such as hydrogen sulfide (H_2S),^{6,7} cadmium sulfide (CdS),⁸ ammonium sulfide ($(\text{NH}_4)_2\text{S}$),^{9,10} and sodium sulfide ($\text{Na}_2\text{S}\cdot 9\text{H}_2\text{O}$)^{11,12} has been reported.

The sulfidation of InP surfaces has been shown to effectively reduce the surface-state density that causes rapid recombination velocity and Fermi-level pinning and is shown to be thermally stable up to ~ 730 K.⁹ There is a general agreement that the InP surface, treated by an aqueous sulfide solution such as $(\text{NH}_4)_2\text{S}$, has an indium sulfide layer on the surface without P–S bonding.¹⁰ However, the state of sulfur on InP has not yet been fully characterized. The formation of alkanethiol $\text{C}_n\text{H}_{2n+1}\text{SH}$ self-assembled monolayers (SAMs) on InP has been demonstrated; the bonding of alkanethiols to InP surfaces and the effect on photocurrent have been studied.^{13–15} The deposition procedure was carried out at temperatures in the range of 52–55 °C, with a deposition time of 18 h, and in an inert environment. When compared to the formation of alkanethiol monolayers on Au, some dissimilarities may be expected, reflecting the differences in the reactivity of the surfaces, because the InP is made up of two components and InP surfaces oxidize rapidly in air. A study using angle-resolved X-ray photoelectron spectroscopy (XPS) on the dodecanethiol SAMs also suggested that the sulfur binds to In atoms on the surface.¹³

In this study, we report on the chemical and the thermal evolution of InP(100) surfaces passivated with sulfur and alkanethiol monolayers performed at room temperature and in air. The water contact angles and the angle-resolved XP spectra of the octadecanethiol-coated and the S-passivated InP are reported immediately after formation, after exposure to ambient air for 1 day, and upon exposure to elevated temperature in a vacuum to characterize the stability of these modified surfaces and to provide information on the bonding at the surface.

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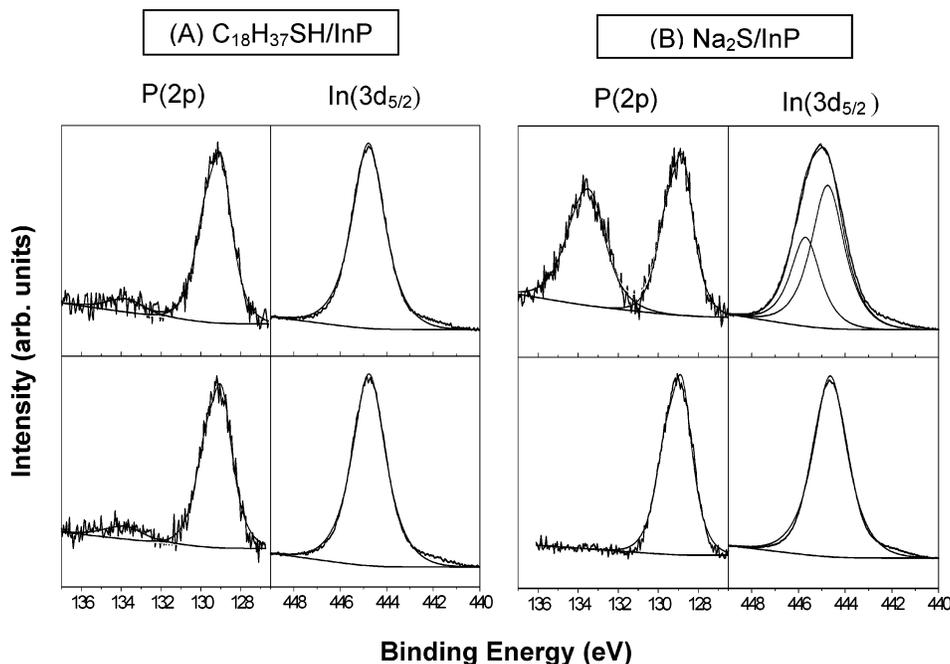


Figure 1. P(2p) and In(3d_{5/2}) regions of the XP spectra for (A) C₁₈H₃₇SH/InP and (B) Na₂S/InP immediately (bottom) and stored in ambient air for 1 day after treatment (top). All the peaks are fitted by a mixture of Gaussian and Lorentzian of ratio 4:1. The P(2p) peaks are fit using one doublet with a 2:1 area ratio and a splitting of 0.84 eV.

II. Experimental Section

S-Doped InP(100) wafers (Crystacomm, with a dopant density of $3.3 \times 10^{16} \text{ cm}^{-3}$ and resistivity of $0.0012 \text{ } \Omega \text{ cm}$) are used in these studies. The as-received samples are first sonicated in acetone for 10 min to dissolve organic contaminants and blow-dried with N₂. The samples are then cleaned using a UV ozone treatment. After cleaning, the InP samples are immersed in a 1:1 mixture of HF and 18 M Ω deionized water for 10 min to remove the native oxide layer. After etching, the water contact angle is 26° and XPS shows a small chemically shifted P(2p) peak at 133.8 eV, corresponding to phosphorus oxide. Oxide may have grown during rinsing and transfer of the etched sample to the XPS apparatus. For the SAM formation, the InP samples are subsequently cleaned in deionized water and 2-propanol. After blow-drying with N₂, the samples are immediately immersed in 0.002 M alkanethiol solutions in 2-propanol with several chain lengths: $n = 3$, propanethiol (Aldrich, 99%); $n = 8$, octanethiol (Aldrich, 97%); and $n = 18$, octadecanethiol (Aldrich, 98%). The samples are left in the solution for various durations. We have determined that 1-day immersion is adequate for obtaining high-quality films. To prevent substantial evaporation of 2-propanol, the container holding InP samples is sealed with Parafilm. After monolayer formation, the InP samples are sonicated in neat 2-propanol for 10 min and blow-dried again with N₂. For the S-terminated InP, HF-etched samples are cleaned with deionized water, blow-dried with N₂, and then dipped in 1 M Na₂S solution (Na₂S·9H₂O, Alfa Aesar, 98%) for 10 min. After the S-passivation, the InP samples are sonicated in deionized water for 10 min and blow-dried again with N₂. These steps are all carried out in ambient air (20 °C and 50% relative humidity).

Contact Angle Analysis. A Rame-Hart model 100 contact angle goniometer is used to measure static contact angles on sessile drops. The volume of the drops is about 3 μL . Water and hexadecane contact angles are used for the qualitative evaluation of SAM packing and for the calculation of surface energies by combining a geometrical mean approach and the Young equation.¹⁶

X-ray Photoelectron Spectroscopy. The XPS experiments are performed in an ultrahigh vacuum chamber with a base pressure of 10^{-9} Torr, equipped with an Omicron EA 125 electron energy analyzer and an Omicron DAR400 X-ray source. Mg K α excitation is used for the spectra reported here, with an energy of 1253.6 eV and a power of 300 W. The takeoff angle is 20° for

grazing angle analysis. The spectra are analyzed using curve fitting with a mixture of Gaussian and Lorentzian of ratio 4:1 to determine peak positions. The deconvolution is performed after subtraction of the background signal with the Tougaard method.¹⁷ The component peak parameters are constrained to experimental values deduced from measurements on reference samples. To study the thermal evolution of the modified surfaces, the substrate temperature is ramped in the XPS chamber at 2 K/s to a specified annealing temperature, held for 10 min, and then cooled to room temperature for the XPS measurements. The steps are successively repeated for increasing annealing temperature to probe the thermal stability of the layers.

Atomic Force Microscopy. A Digital Instruments Nanoscope III is used to image the sample surfaces before and after various modifications. The samples are scanned in ambient air at room temperature, using the tapping mode to minimize possible damage to the sample surfaces.

III. Results and Discussion

III.a. Formation and Chemical Stability. Figure 1 shows the P(2p) and In(3d_{5/2}) regions of XP spectra obtained on octadecanethiol-coated and S-passivated InP(100) surfaces. All XP spectra are obtained at the takeoff angle of 20° to increase the surface sensitivity. The curve fitting results are listed in Table 1. There are four main peaks: In(3d_{5/2}) at 444.7 eV for InP; chemically shifted In at 445.7 eV corresponding to indium oxide; a P(2p) peak corresponding to InP at 129 eV; chemically shifted P at 133.8 eV, corresponding to phosphorus oxide.^{12,13} In the case of octadecanethiol SAMs immediately after formation (Figure 1A, top), there is a small peak at 133.8 eV corresponding to phosphorus oxide. Since this peak does not appear to change after 1 day of storage in air (Figure 1A, bottom), we postulate that this peak is due to the long reaction time of 1 day, with the solution exposed to air. However, we observe significant oxide formation for the S-passivated samples after 1 day of storage in air (Figure 1B). The oxides of InP could be amorphous nonstoichiometric phases based on a mixture of indium oxide and phosphorus oxide compounds.^{18,19} It is not easy

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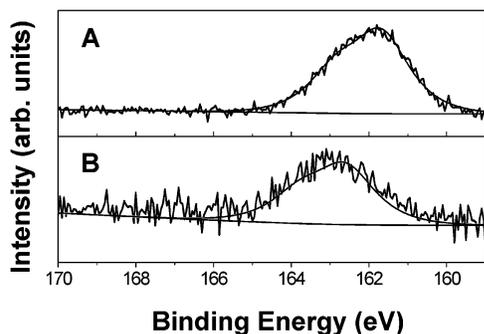


Figure 2. S(2p) region of the XP spectra for (A) Na₂S/InP and (B) C₁₈H₃₇SH/InP. The peaks are fit using one S(2p) doublet with a 2:1 area ratio and a splitting of 1.18 eV.

Table 1. The Water Contact Angles (deg) and the P(2p) and In(3d_{5/2}) Peak Shape Analysis (% of Occupied Area) of C₁₈H₃₇SH/InP and Na₂S/InP Immediately after Treatment and Stored in Ambient Air for 1 Day

		$\theta_{\text{H}_2\text{O}}$ (deg)	P(2p)		In(3d _{5/2})	
			P–In, P–S (%)	P–O (%)	In–P, In–S (%)	In–O (%)
C ₁₈ H ₃₇ SH	immediately	106	90.6	9.4	100	0
	after 1 day	104	91.4	8.6	100	0
Na ₂ S	immediately	16	100	0	100	0
	after 1 day	64	51.9	48.1	64.3	35.7

to define their structure, but the water contact angle on native oxide is about 62°. It is very useful to obtain information on degree of oxidation of indium and phosphorus as a function of exposure time to ambient. As shown in Table 1, the extent of phosphorus oxidation is much more than that of indium. The data support that the S headgroup preferentially bonds to In.

Figure 2 shows the S(2p) region of XP spectra obtained on the S-passivated and octadecanethiol-coated InP(100) surfaces, immediately after treatment. Since the spectra consist of (2p_{3/2}) and (2p_{1/2}) peaks with an intensity ratio of 2:1, which is theoretically determined by the spin-orbit splitting effect, the two shoulders should be assigned to one species. The peak for the S(2p_{3/2}) of S-passivated InP is observed around 161.6 eV, whereas the peak for the S(2p_{3/2}) of the octadecanethiol monolayer is observed around 162.4 eV. There is no peak at 168 eV corresponding to sulfur oxide in either case. The sulfur has been confirmed to lie in the range of 161–162 eV for bonding to indium (electronegativity, 1.7), in the range of 162 eV for thioalkanes on Au (2.4), in the 163–164 eV range with bonding to phosphorus (2.1), and at binding energies of 163–168 eV when bonded to oxygen (3.5).^{12,13,20} This trend in the sulfur binding energies indicates that the more electro-positive the sulfur atom, the higher its binding energy in XPS. These comparisons suggest that the sulfurs in both cases are binding to the indium but the sulfur states are different between S-passivation and thiol monolayers. For the S-terminated InP, sulfur binds with two indium atoms, while for the alkanethiol monolayer, the sulfur binds to the substrate through one indium and carbon. X-ray degradation effects that cause the S(2p) peak to shift to higher binding energies, due to the formation of polysulfidic or disulfidic species, are not observed in this study, in contrast to alkanethiols on Au.^{21,22} In addition, the S(2p) XP spectra, not shown here, revealed that the peak at 161.6 eV in S-passivated InP is not removed completely

Table 2. The Contact Angles (deg) and the Calculated Surface Free Energy (mJ/m²) Values of C₁₈H₃₇SH/InP as a Function of Storage Time in Ambient Air

		$\theta_{\text{H}_2\text{O}}$	θ_{HD}	γ	γ^p	γ^d
C ₁₈ H ₃₇ SH	immediately	106	39	22.8	0.4	22.4
	after 1 day	104	35	23.1	0.6	22.5
	after 7 days	90	25	28.3	3.3	25.0

* $\theta_{\text{H}_2\text{O}}$, water contact angle; θ_{HD} , hexadecane contact angle; γ , surface free energy; γ^p , polar force of surface free energy; γ^d , dispersive force of surface free energy.

after a UV ozone cleaning for 10 min, while octadecanethiol-coated InP shows the clean surface with no S(2p) peak after the UV ozone exposure.

The water contact angle data for the samples immediately after preparation and after 1 day of storage in laboratory air are reported in Table 1. The water contact angle on the Na₂S-treated InP(100) is 16° immediately after treatment, and it increases to 64° after 1 day. This latter value is similar to that obtained on oxidized InP(100), suggesting that the surface after 1 day is strongly oxidized. This is also consistent with the P(2p) and the In(3d_{5/2}) spectra shown in Figure 1.

The water contact angle on the octadecanethiol-coated InP(100) is 106° for the as-prepared sample. This value is somewhat smaller than that reported for this monolayer on Au(111) (ca. 110°)²³ but compares well to the thiol-coated Ge(111) (ca. 101°).²⁴ The water contact angles on the short chain alkanethiol monolayers indicate the different reactivity of InP from that of Au. The water contact angle for propanethiol-treated InP is 39° and for octanethiol is 80°, whereas for Au, the water contact angles are ca. 92° and ca. 110°, respectively.²⁵ The data indicate that it is very hard for the short chain length monolayers to form on InP(100). Table 2 shows the water contact angle ($\theta_{\text{H}_2\text{O}}$) and the hexadecane contact angle (θ_{HD}) obtained on the octadecanethiol-coated InP as a function of storage time in ambient air. In contrast to the sulfur-passivated surface, the water contact angle on the octadecanethiol-coated InP shows only a slight decrease to 104° after 1 day of aging. After 7 days, $\theta_{\text{H}_2\text{O}}$ decreases to 90°, while θ_{HD} decreases to 25°. This result, consistent with their lower initial contact angles, indicates that the films on InP(100) are not as well packed and ordered as they are on Au(111). However, the surface energy calculated¹⁶ using the measured contact angles for the as-prepared octadecanethiol-coated InP shows that the octadecanethiol-coated InP is similar to a hydrophobic HDPE (high-density polyethylene) surface.^{25,26}

Atomic force microscopy is used to image the surfaces before and after the sulfur and thiol passivation treatment. The AFM image of the HF-etched sample shows essentially no change in the topography and roughness as seen in Figure 3A. The surface topography also remains unchanged following the sulfur passivation and the octadecanethiol coating (Figure 3B,C).

The chemical stability of octadecanethiol-coated InP is also investigated in various solutions. The results are shown in Figure 4. After immersion in boiling chloroform or water, $\theta_{\text{H}_2\text{O}}$ starts to drop. After 30 min of immersion

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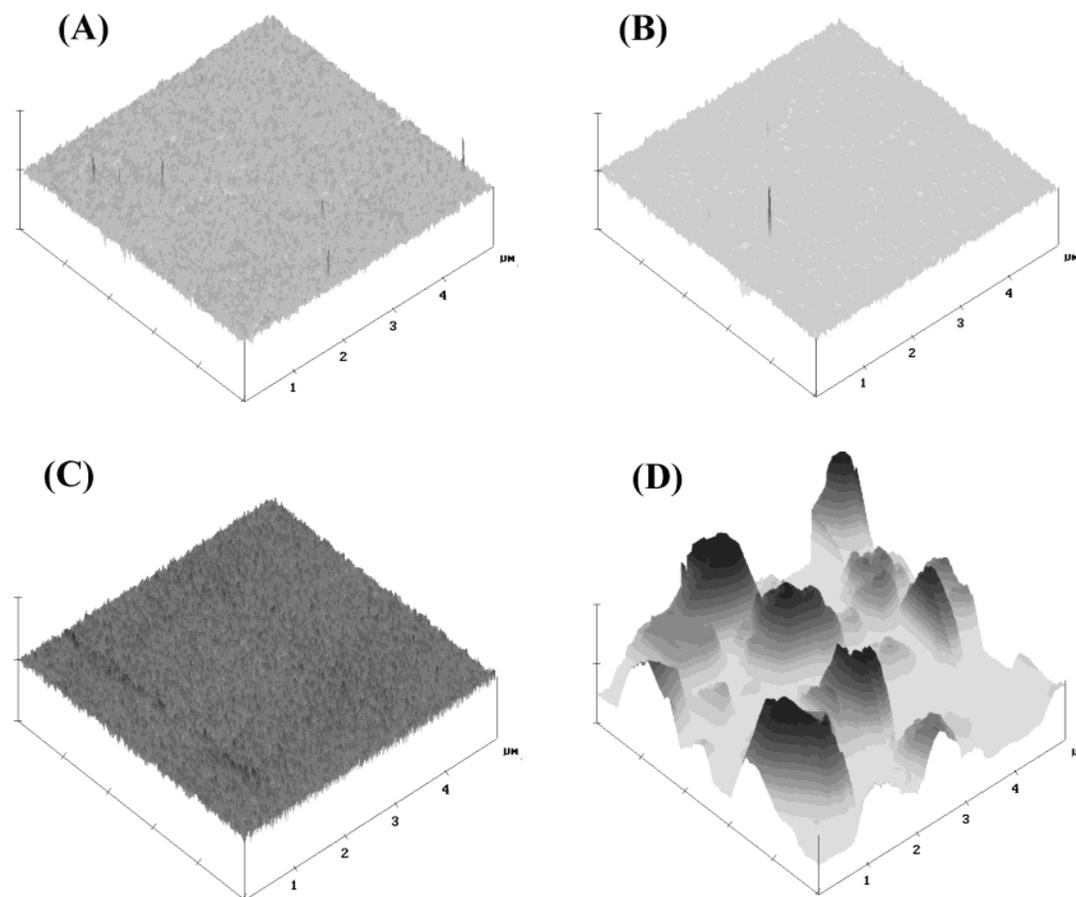


Figure 3. AFM images of (A) 1:1 HF treated InP, (B) $C_{18}H_{37}SH/InP$, (C) Na_2S/InP , and (D) $C_{18}H_{37}SH/InP$ stored in 0.1 M NaOH for 5 days. The rms roughness is (A) 0.4 nm, (B) 0.4 nm, (C) 0.2 nm, and (D) 220 nm.

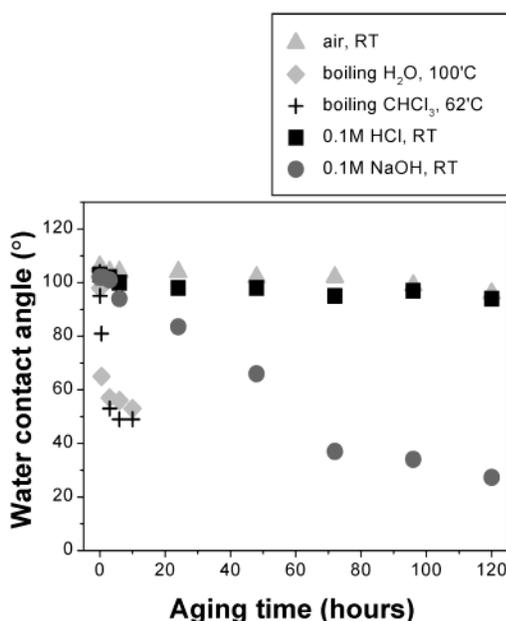


Figure 4. The water contact angles of $C_{18}H_{37}SH/InP$ under different environments and temperatures as a function of time.

in boiling chloroform or water, θ_{H_2O} decreases to about 65° and 81° , respectively. After 10 h of immersion in boiling chloroform and water, θ_{H_2O} reaches about 49° and 53° , respectively. This behavior may be attributed to chloroform and water attacking the sulfur bonds at the monolayer/InP interface with the help of thermal energy, leading to desorption of the thiol molecules and oxidation of the InP surface. θ_{H_2O} of thiol monolayers in 0.1 M HCl shows

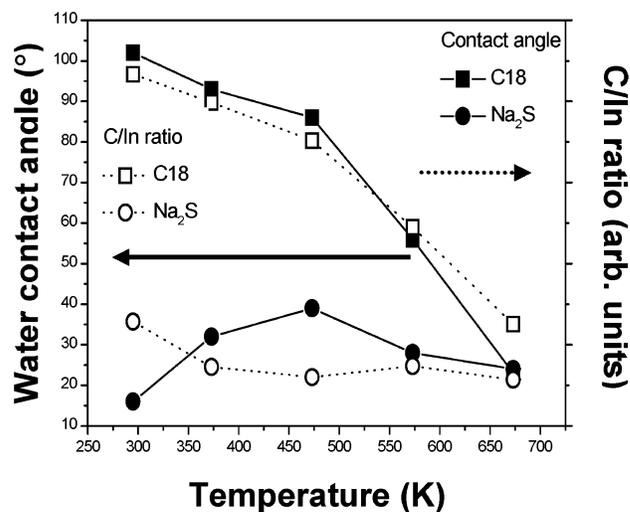


Figure 5. Water contact angles and the C/In ratio of the XP peak area as a function of annealing temperature.

similar behavior to storage in air, whereas in 0.1 M NaOH, it decreases to about 27° after 5 days of immersion. This treatment also increases the surface roughness. Figure 3D is the AFM image of the surface after a 5-day immersion with root-mean-square (rms) roughness of 220 nm. The surface roughness is attributed to the anisotropic etching of InP in NaOH solution. These results suggest that 0.1 M basic solution is enough to etch the InP substrate, while 0.1 M acidic solution affects neither the thiol monolayer nor the InP substrate.

III.b. Thermal Behavior. The water contact angles and the XP peak intensities for the sulfur-passivated and

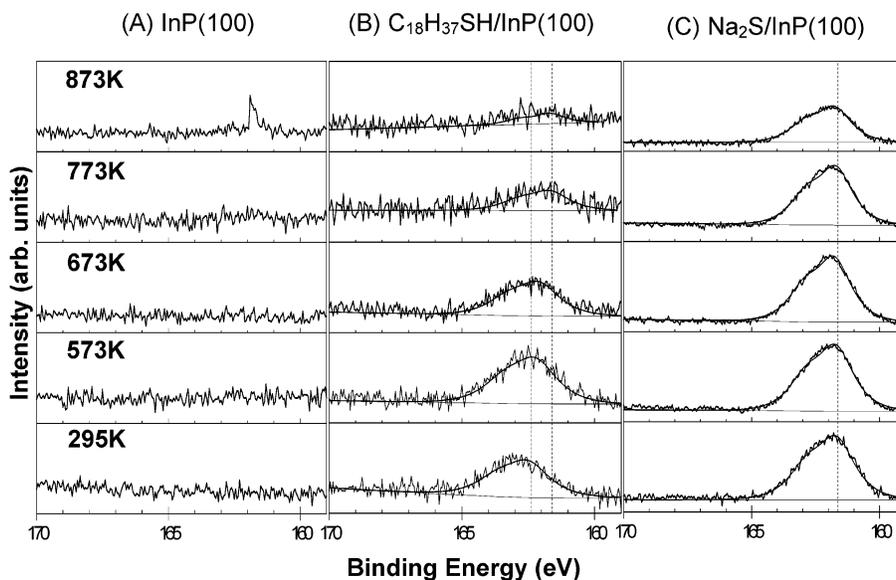


Figure 6. S(2p) region of the XP spectra for (A) InP, (B) $C_{18}H_{37}SH/InP$, and (C) Na_2S/InP as a function of annealing temperature. The peaks are fit using one S(2p) doublet with a 2:1 area ratio and a splitting of 1.18 eV.

the octadecanethiol-coated InP(100) are measured as a function of annealing temperature in a vacuum. The contact angle data and the C(1s)/In(3d) intensity ratios are plotted in Figure 5 as a function of annealing temperature. The data show that the water contact angle on the thiol-coated InP decreases slightly until 473 K and then decreases more rapidly above 473 K. The C(1s)/In(3d) intensity ratios in the angle-resolved XPS follow a similar behavior. For the S-passivated InP, the water contact angle spans the range of 16° – 39° and the C(1s)/In(3d) ratios show a steady decline with temperature.

Additional insight to the thermal behavior of the S-passivated and the thiol-coated InP(100) is obtained by examining the S(2p) peak in XPS (Figure 6). First, the S(2p) region of XP spectra of the HF-treated InP is obtained as a function of annealing temperature to examine the possible effect of the sulfur present in the InP substrate as a dopant. No S(2p) peak is observed in this case (Figure 6A), confirming that the concentration of sulfur as a dopant is not high enough to be detected in XPS. For the octadecanethiol-coated InP(100), the intensity of the S(2p) peak at 573 K is similar to that at 295 K. This is to be contrasted to the C(1s)/In(3d) intensity ratio that shows a significant drop at 573 K, indicating a substantial loss of the hydrocarbon chain. The loss in the alkane chain is expected to reduce the attenuating effect of the remaining hydrocarbon chain on the photoelectrons. Therefore, if all the original sulfur atoms have remained on the surface at 573 K, a sizable increase in the S(2p) signal would be expected, which is not observed. These results suggest that a significant fraction of the S–In bonds have been broken and left the surface at 573 K. However, the sulfur is still detected after annealing to 873 K. Furthermore, the sulfur binding energy is observed to shift to lower energy with an increase in temperature. In the case of S-passivated InP, the sulfur signal is unchanged until 673 K, above which it begins to decrease, with no shift in the binding energy, as reported by others.⁹ The sulfur on the S-passivated InP is suggested to be located on the bridge site and displaced by phosphorus in the InP structure.²⁷

In previous studies on the octadecanethiol-coated Au(111) and Ge(111), the desorption of the entire chains

through the cleavages of the Au–S and Ge–S bonds are observed at about 450 and 550 K, respectively.^{23,24} In the case of Cu (polycrystalline), the hexadecanethiol begins to desorb at about 433 K and almost completely desorbs at 453 K.²⁸ Based upon the results reported here, we find that the octadecanethiol-coated InP(100) is quite stable until 473 K. The sulfur is detected even after annealing the samples to 873 K, suggesting that the desorption involves C–S bond cleavage, unlike the preferred cleavage of the bond between substrate and sulfur. Also, annealing at high temperature changes the sulfur of the octadecanethiol SAM into a bonding geometry similar to that of sulfur in S-passivated InP. The sulfur in the S-passivated InP is found to be more thermally stable than that of octadecanethiol-coated InP, most likely due to different bonding arrangements.

IV. Conclusions

The chemical and thermal stability of the S-passivated and the alkanethiol-coated InP(100) have been evaluated by contact angle measurement, XPS, and AFM. All modification processes are performed at room temperature in air. The octadecanethiol monolayer showed the inhibition of the InP surface from oxide formation compared to the S-passivation. For S-terminated InP, sulfur most probably binds with two indiums, while for alkane-terminated InP, sulfur binds with an indium and a carbon from the chain. The octadecanethiol SAMs showed unstable behaviors in 0.1 M NaOH solution and boiling chloroform and water. The thermal behavior showed stability until 473 K in a vacuum. The desorption of the octadecanethiol monolayer seems to be caused by the cleavage of both the S–C and In–S bonds. Work is underway to evaluate the benefits of these treatments to enhance device performance, in particular for MEMS applications.

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