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Surface evolution of InP substrates at the frontier between deoxidation and dissolution in HCl solutions

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To:
Editors of Surface and
Interface Analysis

Versailles, 12th, July, 2022

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13 Dear Dr. Abel,
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18 We are pleased to submit our research article entitled “Surface evolution of InP substrates at
19 the frontier between deoxidation and dissolution in HCl solutions” by Solène Béchu, Damien
20 Aureau and Arnaud Etcheberry for publication in the special ECASIA issue of the Surface and
21 Interface Analysis journal.
22

23 We confirm that this work is original and has not been published elsewhere nor is currently
24 under consideration for publication elsewhere. In this paper, we explore the surface reactivity
25 of InP substrate when facing different concentrations of HCl. For this purpose, we study by
26 XPS, SEM and AFM the reactivity of (100), (111) and polycrystalline InP substrates when
27 immersed into HCl solutions. We proved that after exceeding a concentration of 6 M, the
28 morphological properties of the InP substrates evolve drastically, with a deconstruction of the
29 InP network. However, this deconstruction is only morphological since all surfaces conserve a
30 perfect stoichiometry, except the (111) face B, which is initially phosphorus-rich and presents
31 an important release of PH₃ when immersed into HCl 12 M. After the HCl immersion, a small
32 enrichment in indium can be noticed, but with a really low value. This paper highlights the
33 importance of controlling the concentration of HCl during the deoxidation stage, to avoid the
34 morphological deconstruction even if the chemical properties are conserved.
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38 We believe that this work is an important tool for the surface and the semiconductor
39 communities regarding the importance of deoxidation for processing semiconductors.
40 Consequently, we think that is of interest to readers in the areas your journal’s publications.
41

42 In case you accept our manuscript, please note that we are not interested to use the colour
43 versions of our illustrations in the print version.
44

45 Please address all correspondence concerning this manuscript to me at solene.bechu@uvsq.fr.
46

47 Thank you for your consideration of this manuscript.
48

49 Sincerely,
50

Solène Béchu



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3 **1 Surface evolution of InP substrates at the frontier between deoxidation and dissolution**
4 **2 in HCl solutions**

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15 7 Keywords: X-ray photoemission spectroscopy, InP, surface evolution

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18 8
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20 9 Abstract

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23 10 Surfaces of InP substrates with different crystallographic orientations are investigated when
24 11 facing HCl solutions. The immersion of (100) InP in a wide range of HCl solutions with
25 12 different concentrations showed that the frontier between deoxidation and dissolution is around
26 13 6 M, with a morphological destructuration. However, no chemical evolutions are noticed even
27 14 at high concentrations. Similar observations were performed with other crystallographic
28 15 orientations (polycrystalline and (111) InP substrates), with topographic surface modifications
29 16 but no chemical ones.

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35 17 1. Introduction

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38 18 III-V semiconductors hold a well-established and specific place in the electronic devices
39 19 industry. Because of the exceptional adaptability of their optical and electrical properties, III-V
40 20 semiconductors offer the possibility of designing reliable components, targeted as to their use
41 21 and performing in application fields as diverse as light-emitting diodes, lasers, large wavelength
42 22 range photodetectors, solar cells, optical switches, quantum well high electron mobility
43 23 transistors...¹⁻⁴

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48 24 A strength of III-V materials chemistry is to propose well-defined quaternary, ternary and
49 25 binary alloys and to combine them by epitaxy in multi-structures, with micrometric or
50 26 nanometric layers to design energy band diagrams of hetero-structures, with tuneable optical
51 27 and/or electronical responses for the whole component.⁵ The control of these particular objects
52 28 is achieved by mastering chemical engineering of the interfaces between the layers, the
53 29 substrate surfaces supporting epitaxy, the final encapsulations, and the etchings. In such a

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3 30 context, the position of the XPS is obviously a quantitative tool for chemical or even electronic
4 31 (band-off set) characterizations.

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7 32 To understand the fundamentals of the III-V chemistry, the researchers have at their disposal
8 33 binary alloys (InP, InAs, InSb, GaN, GaP, GaAs, GaSb) perfectly mastered. Their comparative
9 34 study in the face of the same source of interaction is extremely valuable. Here again, XPS plays
10 35 a central role. Among all III-V binary alloys, InP has a prominent place since it is present in
11 36 many devices as active device materials, capping layers and even substrates for epitaxial
12 37 growths, for which surface control is essential.

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18 38 Indeed, achieving surface chemical controls of InP substrates is a necessity for the growth of
19 39 high-quality epitaxial films or the device's performance. The key to obtaining such surfaces is
20 40 the control of intentional oxidation and deoxidation stages. Regarding the deoxidation process,
21 41 each laboratory possesses its own expertise: thermal desorption of the oxide,⁶⁻⁸ ion sputtering
22 42 followed by annealing,⁹ H₂ plasma,¹⁰ wet chemical treatments...^{11,12} Within the literature, the
23 43 wet chemical deoxidation of InP is widely described with the importance of the acid choice and
24 44 how it affects oxide removal.^{11,12} HCl is an acid of choice to perform the deoxidation, however,
25 45 the interactions existing between HCl and InP are very specific, with potential dissolution.
26 46 Indeed, so lone or in association with other chemicals, HCl concentrated solutions can generate
27 47 more or less efficient dissolution, governed by an initial interaction between the undissociated
28 48 HCl molecules and the InP surface lattice. The detailed dissolution mechanisms and associated
29 49 kinetic rates are available in the literature.¹³⁻¹⁷ However if the InP thinning using HCl
30 50 concentrated solution is established, the chemical surface evolution of InP substrates is barely
31 51 investigated at high HCl concentrations not saying if, during the dissolution or even the
32 52 deoxidation processes in HCl, the surface stoichiometry of InP substrates evolves, with a
33 53 preferential etching of either indium or phosphorus.

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39 54 This work aims at unveiling the evolution of InP surfaces, either when facing a deoxidation or
40 55 a dissolution. Indeed, very few works deal with the modifications of InP substrates according
41 56 to the concentration of HCl solutions or the crystallographic orientations, especially at high
42 57 concentrations.¹⁸ In order to, XPS, Atomic Force Microscope (AFM) and Secondary Electrons
43 58 Microscope (SEM) analysis are performed to unveil those evolutions and to understand the
44 59 mechanisms lying behind.

60 2. Materials and Methods

61 2.1. Materials

(100), (111) and polycrystalline n-type InP substrates were purchased from InPact Electronic Materials Ltd. Surfaces were mirror-like finished and denoted as epi-ready quality by the supplier for the (100) and (111) substrates. Samples were directly immersed for 5 minutes (min) in HCl solutions (37 %, ACS quality, from SUPELCO) at different concentrations (respectively, 3 mol L⁻¹ (M), 6 M, 9 M and 12 M). Then, the samples were rinsed for 1 minute in deionised water, dried under N₂ flow and directly transferred within the spectrometer. Air interactions last less than 5 to 10 min.

2.2. XPS

XPS surface chemical analyses were conducted on a Thermo Electron Nexsa spectrometer equipped with a monochromatic Al-K α X-Ray source (1486.6 eV). The Thermo Electron procedure was used to calibrate the Nexsa spectrometer by using metallic Cu and Au samples intern references (Cu 2p_{3/2} at 932.6 eV and Au 4f_{7/2} at 84.0 eV). High energy resolution spectra were acquired with an X-ray spot size of 400 μ m and using a constant analyzer energy (CAE) mode of 20 eV and 0.1 eV as energy step size. Regarding the nature of the sample, charge compensation wasn't applied. XPS spectra were treated using a Shirley background subtraction, and XPS compositions were deduced using the sensitivity factors and the inelastic mean free paths from Avantage library associated with the spectrometer and the corresponding transmission function.

2.3. SEM

Secondary electron micrographs (SEM) analyses were performed using a JEOL JSM 7001F microscope with a patented "in-lens" Schottky field emission gun (FEG). Imaging analyses were realized at 15 kV accelerating voltage and 10 mm working distance.

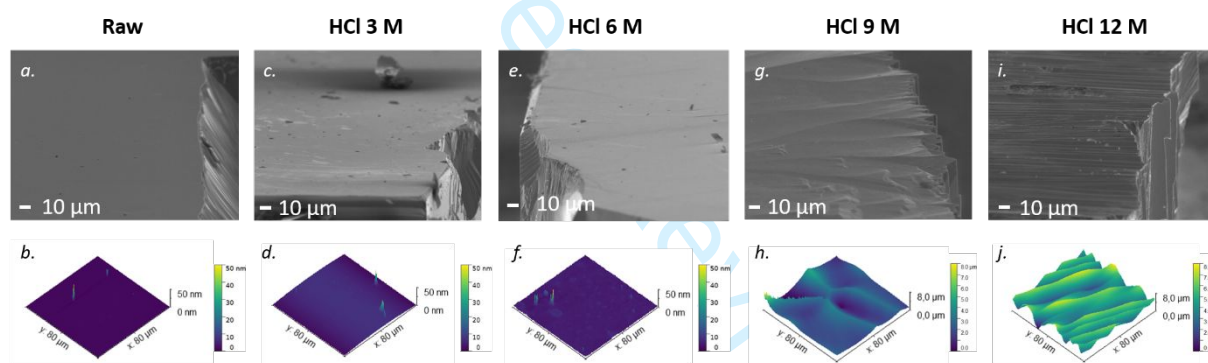
2.4. AFM

A Dimension ICON AFM (Bruker) was used to observe the morphology of the InP substrates. The AFM topography measurements were performed in air with the Peak Force tapping mode. For this purpose, a silicon tip on a nitride lever (ScanAsyst Air model, Bruker), with a 0.4 N/m spring constant and a nominal tip radius of 2 nm was used with a resolution of 256 pixels \times 256 pixels.

3. Results and Discussion

3.1. Influence of the HCl concentration for (100) InP substrates

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3 92 The immersion of (100) InP substrates within HCl solutions with different concentrations led
4 93 to a large range of morphological evolutions as observed in Figure 1. Two different regimes
5 94 can be distinguished: the low concentration range (from 0 to 6 M), where no gas emission was
6 95 observed and the high concentration range (above 6 M), with a spontaneous and strong emission
7 96 from all the InP surface in contact with the solution of an ultra-dense and characteristic fog of
8 97 micro bubbles, due to the continuous release of PH_3 .¹⁹ Within the first regime, the integrity of
9 98 the morphology is preserved (Figure 1 a. to f.) while surface degradation is noticeable in the
10 99 second regime (Figure 1 g. to j.). According to the AFM images and the increase of the surface
11 100 roughness, the degradation is evolving progressively with the HCl concentration, with the InP
12 101 substrate immersed in the 9 M HCl solution representing the beginning of the degradation
13 102 observed on the InP substrate immersed in the 12 M HCl. InP (100) substrates immersed in HCl
14 103 12 M showing a drastic topographic modification was already observed by Tuck and Baker,²⁰
15 104 but the progressivity of this morphological degradation at HCl concentrations higher than 6 M
16 105 is shown here for the first time.



106
107 Figure 1: Cross-section SEM and AFM images for raw InP (100) substrate (a. and b.), InP (100)
108 substrate immersed for 5 min in HCl 3M (c. and d.), InP (100) substrate immersed for 5 min in
109 HCl 6M (e. and f.), InP (100) substrate immersed for 5 min in HCl 9 M (g. and h.) and InP
110 (100) substrate immersed for 5 min in HCl 12 M (i. and j.).

111 As the morphological properties of the (100) InP substrate evolve with the HCl concentration,
112 it is necessary to investigate also the potential modifications of the chemical properties, with
113 XPS measurements. The In $3d_{5/2}$ and P 2p spectra are displayed in Figure 2 and the fit
114 parameters are in Table 1. Regarding the raw (100) InP substrate (Figure 2 a. and b.)
115 corresponding to the epiready configuration, two contributions are observed for the In $3d_{5/2}$ and
116 the P $2p_{3/2}$ photopeaks: ones related to the InP substrate at 445.0 eV and 129.2 eV respectively,
117 and the other ones related to the oxide phases, In-O contribution being at 445.9 eV and the P-
118 O contribution being at 133.5 eV.²¹ Quantitatively speaking, no specific enrichment in In or P
119 is noticed on the surface of the raw (100) InP substrate, as expected for an epiready substrate.

As soon as the (100) InP substrate is immersed in HCl solution, the In-O contribution disappears while a small reminiscence (inferior to 0.6 at. %) of P-O is observed. No modification of the initial position of the photopeak is noticed and the modified Auger parameters remain in the associated range of the InP (852.6 eV).²¹

One could expect that the modification of the morphology would induce changes within the photopeak answer, with an enlargement of the full-width half at maximum (FWHM) for example.²² However, as observed in Table 1, the FWHM values of the In 3d_{5/2} and P 2p_{3/2} photopeaks don't evolve, reaching 0.79 ± 0.01 eV and 0.60 ± 0.02 eV, respectively. The only noticeable difference is related to the phosphate contribution, with a larger range of value, which is attributed to the really low amount of oxide detected, inducing variabilities during the fitting procedure.

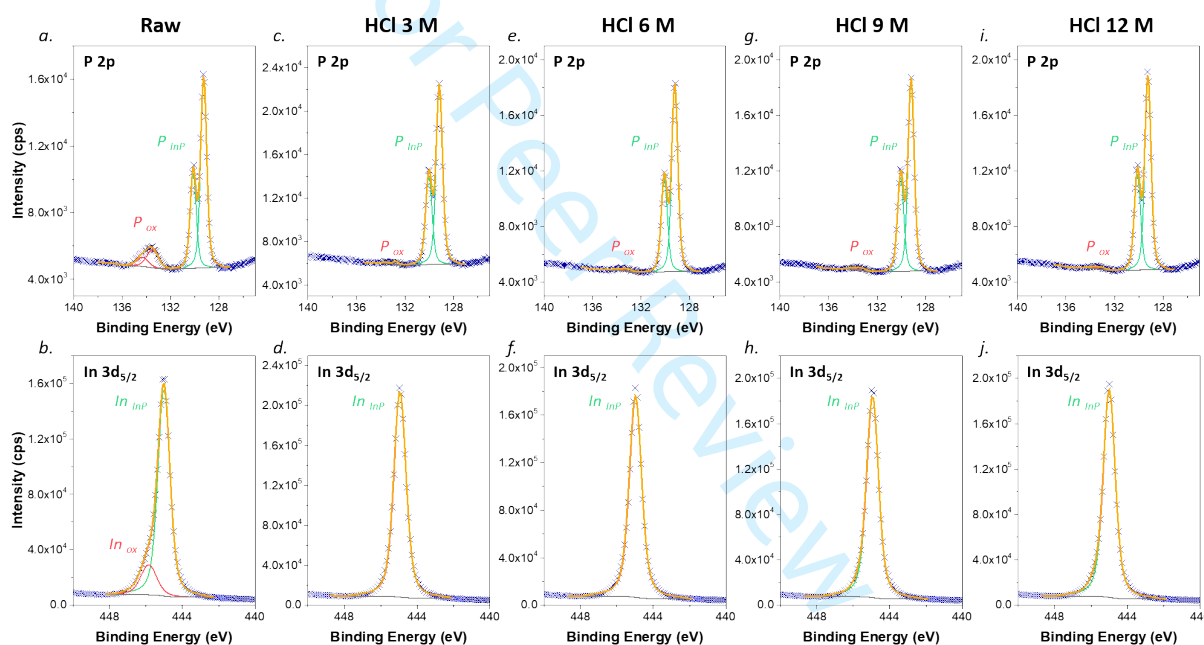


Figure 2: P 2p and In 3d_{5/2} XPS spectra for raw InP (100) substrate (a. and b.), InP (100) substrate immersed for 5 min in HCl 3 M (c. and d.), InP (100) substrate immersed for 5 min in HCl 6 M (e. and f.), InP (100) substrate immersed for 5 min in HCl 9 M (g. and h.) and InP (100) substrate immersed for 5 min in HCl 12 M (i. and j.).

Another expectation related to morphology evolution observed in Figure 1 could be an enrichment in In or P during the strong dissolution regimes (9 M and 12 M). The In/P ratios calculated in Table 1 refute this hypothesis since the In_{InP}/P_{InP} ratios remain within a range of 1.03 ± 0.05 , the raw value being at 1.04. Thus, the surface chemistry of (100) InP substrate is not modified when exposed to a large range of concentrations of HCl, even if some morphological modifications appear.

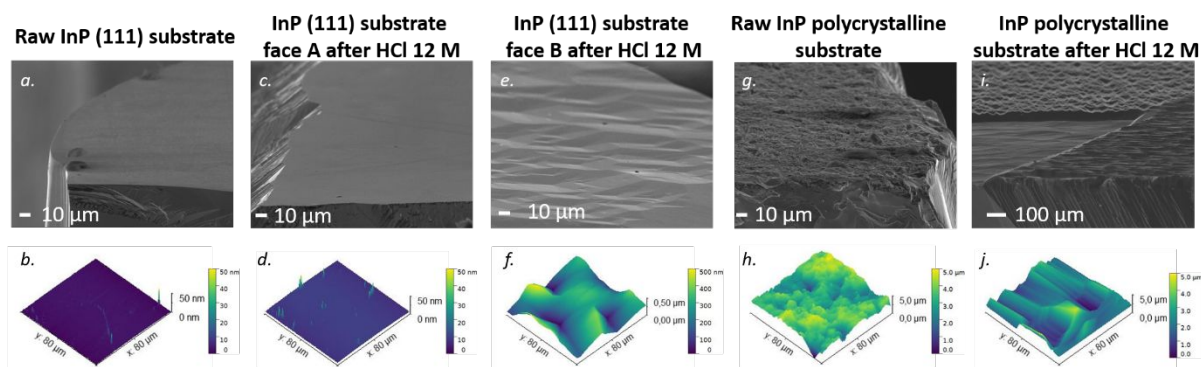
142 Table 1: Parameters fits for the XPS spectra displayed in Figure 2. The ratios are calculated
 143 from the In 3d_{5/2} and P 2p photopeaks.

	Attribution	Position (eV)		FWHM (eV)		Ratios		Modified Auger parameter (eV)
		In 3d _{5/2}	P 2p _{3/2}	In 3d _{5/2}	P 2p _{3/2}	In _{InP} / P _{InP}	In _{tot} / P _{tot}	
Raw substrate	InP	445.0	129.2	0.79	0.59	1.04	1.05	852.7
	oxide	445.9	133.5	1.03	1.12			
HCl 3 M	InP	445.0	129.2	0.80	0.62	0.99	0.97	852.8
	oxide	/	132.9	/	0.90			
HCl 6 M	InP	445.0	129.2	0.78	0.60	1.04	1.02	852.7
	oxide	/	133.2	/	1.09			
HCl 9 M	InP	444.9	129.2	0.78	0.60	1.02	1.04	852.7
	oxide	/	133.3	/	0.94			
HCl 12 M	InP	445.0	129.2	0.79	0.60	1.04	1.06	852.7
	oxide	/	133.1	/	1.07			

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145 3.2. Influence of the crystallographic orientation of the InP substrate

146 If the HCl concentration doesn't influence the chemical behaviour of (100) InP substrate,
 147 interrogations can remain about the influence of HCl when facing different crystallographic
 148 orientations of InP substrate. To elucidate it, (111) InP substrates, on face A (In-rich) and on
 149 face B (P-rich), as well as polycrystalline InP substrates, were immersed in HCl 12 M for 5
 150 minutes.



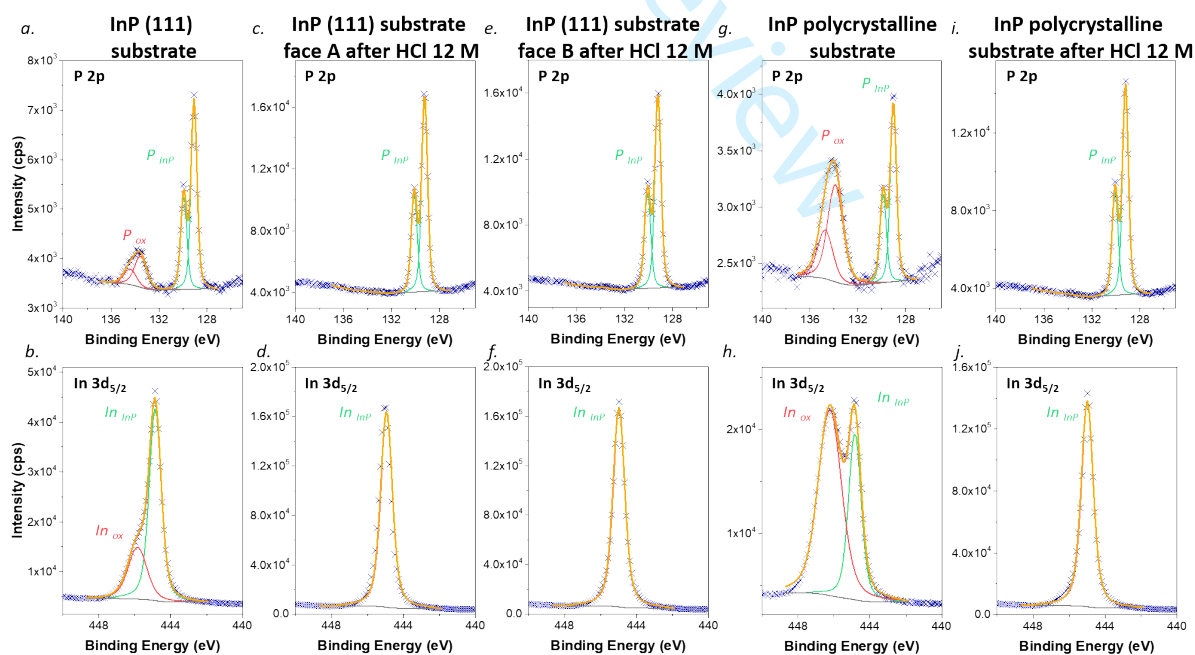
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152 Figure 3: Cross-section SEM and AFM images for raw InP (111) substrate (a. and b.), InP (111)
 153 substrate face A (In-terminated) immersed for 5 min in HCl 12 M (c. and d.), InP (111) substrate

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3 154 face B (P-terminated) immersed for 5 min in HCl 12 M (e. and f.), raw InP polycrystalline
4 155 substrate (g. and h.) and InP polycrystalline substrate immersed for 5 min in HCl 12 M (i. and
5 156 j.). Since faces A and B of the raw InP (111) substrate present similar surface aspect and
6 157 rugosity, only one was represented here.

8
9 158 Regarding the (111) InP substrates, according to the face observed, two different surface
10 159 morphologies are obtained after the HCl treatment (Figure 3 a. to f.). One first face remains as
11 160 smooth as the raw (111) substrate (whatever the face observed) when the other present a high
12 161 rugosity. The difference between the two faces can be explained with the gas release. For the
13 162 first face (Figure 3 c. and d.), only a small amount of PH_3 was released during the HCl
14 163 immersion, while for the second face (Figure 3 e. and f.), an intense PH_3 release as important
15 164 as the one observed for the (100) InP substrate is noticed, resulting in important modifications
16 165 of the topography of the surface sample. Based on Tuck and Baker previous observations,²⁰
17 166 face 1 is thus the (111) A InP, terminated with an indium layer which tempers the release of
18 167 PH_3 and face 2 is the (111) B InP, terminated with phosphorus atoms which immediately react
19 168 with the acidic environment to release an important amount of PH_3 .

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28 169 Immersing the polycrystalline InP substrate into HCl doesn't result in a smoother surface
29 170 (Figure 3 j.) but specific textures are observed, especially according to the initial grain
30 171 orientations (Figure 3 i.). This difference of grain orientation induces also a difference of
31 172 kinetics etching, as noticed with the specific step observed on Figure 3 i.



173
174 Figure 4: P 2p and In 3d_{5/2} XPS spectra for raw InP (111) substrate (a. and b.), InP (111)
175 substrate face A (In-terminated) immersed for 5 min in HCl 12 M (c. and d.), InP (111) substrate
176 substrate face B (P-terminated) immersed for 5 min in HCl 12 M (e. and f.), raw InP polycrystalline

177 substrate (g. and h.) and InP polycrystalline substrate immersed for 5 min in HCl 12 M (i. and
178 j.). Since faces A and B of the raw InP (111) substrate present similar fitting procedures, only
179 one was represented here. Fit details are available in Table 2.

180 The chemical evolution of InP substrates is also studied according to the crystallinity evolution
181 when the samples are immersed in HCl 12 M for 5 minutes (Figure 4 and Table 2). Whatever
182 the crystallographic orientation, both raw substrates present important quantities of oxides, and,
183 as soon as samples are immersed into HCl 12 M solution, the oxide contributions disappear,
184 even the P-O one. This difference with the substrate (100) is explained with a shorter transfer
185 time to the XPS analyses. Quantitatively speaking, the $\text{In}_{\text{InP}}/\text{P}_{\text{InP}}$ ratios of the polycrystalline
186 and the (111) substrates are lower than the (100) raw substrate reference. This apparent
187 modification is related to the important amount of oxide detected for the raw polycrystalline
188 and the (111) InP substrates. In $3d_{5/2}$ and P $2p_{3/2}$ photopeaks are within an extended energy
189 range, In $3d_{5/2}$ being at 445.0 ± 0.1 eV and P $2p_{3/2}$ at 129.2 ± 0.1 eV. Thus, the difference
190 between the electron escape depths of these two photopeaks becomes too important, especially
191 with the large oxide layers present at the substrates' surfaces. This difference can be overcome
192 by choosing two photopeaks in a narrower energy range, such as In $4d_{5/2}$ (17.9 ± 0.1 eV) and P
193 $2p_{3/2}$ (129.2 ± 0.1 eV). In this case, the $\text{In}_{\text{InP}}/\text{P}_{\text{InP}}$ ratios become 1.06 for the polycrystalline
194 InP substrate and 0.92 for both (111) A and (111) B faces of InP (111) substrate.

195 Regarding the (111) substrates, after HCl immersion, similar fitting parameters and quantitative
196 aspects are observed between the (100) InP substrate and the (111) face A. For the InP substrate
197 (111) face B, a small enlargement of the P $2p_{3/2}$ contribution is noticed (Table 2), which was
198 attributed to an additional contribution of the (111) face B surface of InP by Sturzenegger and
199 Lewis (elementary phosphorus, responsible for the oxidation begin).²³ In the present case, it is
200 not possible to confirm this additional contribution since the enlargement is quite small, but it
201 is worth noticing it. A slight enrichment in In is also noticed for the (111) face B surface,
202 confirming thus the morphological degradation observed, with a higher quantity of PH_3
203 released.

204 As expected,²² the raw polycrystalline substrate presents slightly higher FWHM parameters,
205 but the immersion in the concentrated HCl solution restore them within the range observed for
206 (100) and (111) InP substrates. No specific enrichment in In or P is noticed, confirming thus
207 what was observed with the (100) substrates: the dissolution in HCl solutions doesn't modify
208 quantitatively the chemistry.

209 Table 2: Parameters fits for the XPS spectra displayed in Figure 4. The ratios are calculated
 210 from the In 3d_{5/2} and P 2p photopeaks.

	Attribution	Position (eV)		FWHM (eV)		Ratios		Modified Auger parameter (eV)
		In 3d _{5/2}	P 2p _{3/2}	In 3d _{5/2}	P 2p _{3/2}	In _{InP} / P _{InP}	In _{tot} / P _{tot}	
(111) face A raw substrate	InP	445.0	129.1	0.80	0.58	0.87	0.92	852.6
	Oxide	445.9	133.7	1.19	1.35			
(111) face B raw substrate	InP	444.9	129.1	0.79	0.59	0.80	0.86	852.7
	Oxide	445.8	133.6	1.23	1.20			
(111) face A HCl 12 M	InP	445.0	129.2	0.78	0.58	1.02	1.02	852.6
	Oxide	/	/	/	/			
(111) face B HCl 12 M	InP	445.0	129.2	0.78	0.61	1.08	1.08	852.7
	Oxide	/	/	/	/			
Polycrystalline raw substrate	InP	444.8	129.0	0.84	0.62	0.81	1.05	852.6
	Oxide	446.2	133.9	1.54	1.51			
Polycrystalline HCl 12 M	InP	445.0	129.2	0.80	0.59	1.04	1.04	852.6
	Oxide	/	/	/	/			

211

212 4. Conclusion

213 To understand the interactions between HCl solutions and InP surfaces during deoxidation and
 214 dissolution, the roles of the HCl concentration and the crystallographic orientation of the InP
 215 substrates were investigating. If a concentration frontier at 6 M is noticeable for the
 216 morphological evolution of the (100) InP substrates, with the deconstruction of the InP network
 217 due to dissolution, no specific modification of the surface chemistry information is impacted
 218 by the concentration of HCl. Indeed, no enrichment in indium or phosphorus is observed, and
 219 this, within the first 10 nm of the substrate.

220 Regarding the crystallographic orientation, again, at high HCl concentrations, morphological
 221 modifications are noticed, with an important differentiation between the face A and the face B
 222 of the (111) InP substrate. However, those topographical evolutions don't influence the

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3 223 chemical surface of the InP substrate, since the $\text{In}_{\text{InP}}/\text{P}_{\text{InP}}$ ratios don't present any evolution even
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5 224 when facing HCl 12 M. Only a really small enrichment in In might be noticed in face B, with a
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7 225 more important quantity of P released during the dissolution of InP. Thus, it is possible to
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9 226 conclude that, whatever the crystallographic orientation of the InP substrates studied in this
10
11 227 paper, the HCl concentration doesn't influence the chemical quantification of the InP substrate
12
13 228 surface, even during strong dissolution of the substrates.

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