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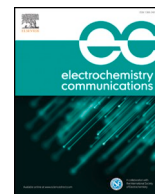
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Stoichiometry loss induced by ionic bombardment of InP surfaces: A challenge for electrochemistry combined with XPS

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ABSTRACT

Indium phosphide (InP) surfaces are greatly affected by ionic bombardment. We investigate the resulting surface perturbation through the use of the complementary analytical techniques of electrochemistry and X-ray photoelectron spectroscopy (XPS). Following bombardment, modifications to the surface were identified by a reduction in the dark open circuit potential in comparison to the pristine state. Through XPS studies, it was found that the sputtered surface was enriched with a metallic-like In contribution, which oxidized upon exposure to air. Cyclic voltammetry measurements confirmed this observation, with initial cathodic features related to an oxidized metallic In-enriched layer on the InP surface. Repeated cyclic voltammetry experiments resulted in the formation of a more In-rich overlayer due to a specific oxidation/reduction phenomenon. This behavior is very similar to that obtained by cathodic decomposition on InP surfaces.

1. Introduction

The semiconductor industry utilises multiple procedures such as plasma treatment, ionic bombardment, thermal annealing and electron beam patterning during device fabrication. However, all of these processes can induce surface and/or bulk perturbations within the semiconductor (SC) film. The SC properties can thus be modified during the manufacturing procedure; therefore, in order to determine the level of disturbance caused by the fabrication processes, additional analysis is required. One possible technique involves the use of electrochemistry. Since the bulk and/or surface properties of SCs govern the SC/electrolyte junction, electrochemistry can sensitively detect any perturbations, as well as their nature and origin [1–3]. Furthermore, as electrochemical techniques can be performed either in the dark or under illumination [4], a variety of approaches are available, with *in situ* tracking of the resulting modifications. By varying the cathodic and/or anodic mechanisms [1,3,5], partial or even complete recovery of the initial surface can be achieved. Thus, electrochemistry is particularly helpful in evaluating the perturbation caused by ionic bombardment and regenerating the surface.

Indeed, the electrochemical responses before and after the treatments are linked to electrical, chemical and structural evolutions and, consequently, to the nature and size of these changes. When electrochemistry is combined with a surface analysis technique such as X-ray photoelectron spectroscopy (XPS) [6], the exact surface chemistry can

be studied, and this combined approach therefore provides a more detailed description of the modified surface.

This letter focuses on evaluating the perturbation induced by ionic Ar⁺ bombardment of indium phosphide (InP). The consequences of such a bombardment and its associated etching process have already been investigated through other studies, which examined different bombardment sequence durations and beam intensities [6–8]. In the case of a binary alloy [6], the main question related to ionic bombardment is the possible differential abrasion effect, and more specifically, its size and surface distribution. For InP, Ar⁺ bombardment generates a surface stoichiometry loss with In enrichment, which has already been investigated by XPS [7,8]. To better understand the surface evolution and its impact on the SC properties, we coupled the XPS investigation with an electrochemical study using cyclic voltammetry. This technique is well suited to measurement of the In enrichment, due to its low In detection threshold and its ability to promote indium-specific oxidation/reduction cycles [9]. This letter discusses how the combination of XPS and electrochemistry can provide an accurate diagnostic of the perturbation induced by Ar⁺ ionic bombardment. The results of the electrochemical study of the InP bombarded surface are also compared with those obtained for an InP surface subjected to cathodic decomposition, which has been found to promote a well-defined response of an In-rich outer layer on InP [10–15].

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2. Materials and methods

N-type InP (1 0 0) wafers were purchased from InPACT Electronic Materials Ltd, with a doping level of $1\text{--}2 \times 10^{15} \text{ cm}^{-3}$. Square samples ($0.5 \text{ cm} \times 0.5 \text{ cm}$) were mounted as electrodes, with an ohmic contact at the rear side, in an electrochemical holder compatible with further XPS analyses under UHV conditions. Before the electrochemical or XPS analysis, the initial surfaces were chemo-mechanically polished in bromine methanol (< 2%), rinsed with pure methanol, ultra-pure water and subsequently dried under a nitrogen stream. In order to remove oxides, the samples were dipped for a few minutes in 2 M HCl, rinsed in ultra-pure water and dried again. Experiments combining XPS and electrochemistry were carried out by minimizing the time of transfer (< 5 min) between the electrochemical cell and the spectrometer introduction chamber to limit surface oxidation.

XPS studies were performed using a Thermo Electron K-Alpha⁺ spectrometer using a monochromatic Al-K α X-ray (1486.6 eV) and a spot size of 400 μm , with 12 kV as primary energy and 6.0 mA as emission intensity. The Thermo Electron procedure was used for calibration and the spectrometer verified using Cu and Au, following the ASTM-E-902-94 recommendation. The Constant Analyzer Energy mode with 10 eV pass energy was selected. The data were processed using the Thermo Avantage[©] data system. The XPS atomic composition was obtained using a Shirley background. Values of sensitivity factors, tabulated transmission factors, and inelastic mean-free paths were obtained from the Avantage[©] library. Bombardment was performed with an Argon MAGCIS dual ion source, using accelerated Ar⁺ monoatomic ions at an energy of 1000 eV, for specified ionic bombardment durations and with an ion beam size leading to a crater close to 4 mm². The ion gun was placed at an incidence angle of 30° with respect to the surface of the samples, in the absence of rotation.

Electrochemical studies were carried out with a 2273 PARSTAT Princeton Applied Research Potentiostat. A three-electrode cell configuration was used with a saturated mercurous sulfate electrode (MSE) as the reference and Pt foil (large area) as the counter electrode. Linear sweep voltammetry was carried out with a scan rate of 20 mV sec⁻¹. Cyclic voltammetry (CV) was performed at pH 10 (Alfa Aesar, pH 10 ± 0.01 at 25 °C) in a buffered aqueous solution, in order to maintain the chemical stability of the In-rich film on the InP surface during anodic and cathodic cycles [16]. Dissolved oxygen was removed by purging the bath with N₂ gas. All electrochemical experiments were started at the open circuit potential.

3. Results and discussion

The Ar⁺ bombardment induces etching of the InP surface at a constant rate. As previously observed, preferential abrasion occurs, which can be related to the differences in atomic number between indium and phosphorus [6–8]. A well-defined modification of the XPS signature is observed as soon as a steady-state abrasion process is reached, *i.e.*, after several tenths of seconds under our operating conditions. In comparison with the initial XPS spectra, a strong variation in the intensities associated with the phosphorus and indium contributions is observed. The XPS atomic ratio In/P (deduced from In4d and P2p levels) evolves from 1.00 to 1.35 ± 0.05 . In order to identify the chemical nature of the excess In atoms generated by the bombardment, the different core levels of In are studied, with particular attention to In4d.

Modifications of the energy distribution of the In4d spectra are observed (Fig. 1a., comparing the “reference” and “after bombardment” spectra). Similar changes are found for the other indium core levels (not shown here), with the emergence of new contributions at lower binding energies (BEs). The In MNN Auger transition is also affected. Before Ar⁺ bombardment, the In4d_{5/2} peak is positioned at $17.8 \pm 0.1 \text{ eV}$ and the initial double structure associated with the 4d spin-orbit splitting is visible. This is no longer the case after bombardment, when

spectral features suggesting the existence of possibly three contributions appear. Among the different contributions observed, the low BE one at $\text{In}4d_{5/2} = 17.2 \pm 0.2 \text{ eV}$ (labeled In_{low BE} on Fig. 1a) can be considered as a characteristic of In atoms that are more metallic than those of InP. As a reference, the In4d_{5/2} photopeak of a pure In metal sample (not shown here) has a BE of $16.7 \pm 0.1 \text{ eV}$ [17,18]. The slight difference in the low BE contribution toward a pure metal position leads to questions on the real nature of the corresponding sites and further insight is provided by the electrochemistry results given later in this work. Note that the characteristic low BE In4d deformation is reproducible as soon as a steady-state abrasion etching time is reached (10 sec). As in the case of In4d, a modification of the P2p signal is systematically observed after bombardment (Fig. 1b, comparing the “reference” and “after bombardment” spectra), with a loss of its well-defined spin-orbit splitting and a slight shifting of the peak position (reference P2p_{3/2} photopeak at $129.1 \pm 0.1 \text{ eV}$, BE and bombarded peak at $128.7 \pm 0.1 \text{ eV}$, BE). This modification of the BE distribution is explained by the appearance of a second contribution, which must be dissociated from the P sites inside the InP network. Upon initial observation, it was assumed that this can be attributed to the presence of elementary phosphorus within the modified top surface. Thus, after bombardment, the InP surface shows a complex outer layer, which is In-rich, with In mainly in a metallic-like form, along with a loss of P, leaving superficial elementary P. This perturbed outer layer is thin since the InP matrix features are still visible.

When the modified InP surfaces are transferred from UHV conditions to the electrochemical cell, the effect of air contamination has to be considered as it can alter the behavior of the film during immersion and subsequent electrochemical characterization. To study this, XPS was performed on the bombarded surface after exposure to air for 5 min (Fig. 1a and 1b, “5 min air after bombardment” spectra), which is representative of the sample before immersion. The In4d/P2p ratio remains constant (1.35 ± 0.05) but new contributions are observed at higher BE for both elements, indicating that the outer layer is effectively altered in this interval. For In, this is consistent with the high air reactivity of cleaned In metal samples already reported [19–21], leading to partial oxidation of the initial metallic In included in the outer layer and the emergence of oxide contributions to the In4d peak. In the case of P, oxidation can be excluded, since no oxide contribution is observed at 133.0 eV BE. The evolution of the P2p spectroscopic signature rather argues for a rearrangement of the chemical network of the outer layer, probably in relation to the incorporation of oxygen. Consequently, after its transfer in air, the bombarded surface is even more complex and must be considered as an ultra-thin overlayer which is still In-rich, composed of metallic-like In, which is partially oxidized and in which a few elemental phosphorus atoms are incorporated.

Following bombardment and air transfer, the InP surface is then immersed in the electrochemical cell at open circuit potential for five to ten minutes. A domain without any current (reproducible and stable in time) is observed around the dark open-circuit potential (d-OCP) value of -0.9 V/MSE (Fig. 2a), suggesting that the interface is chemically stable just after immersion. Note that the d-OCP of the bombarded surface differs significantly from the reference value (-0.7 V/MSE), confirming the modification of the InP surface.

The first electrochemical peculiarity of the I vs V curves is the presence of well-defined, specific and spontaneous double cathodic features as soon as a sufficiently negative overpotential is applied (potential lower than -1.5 V/MSE , Fig. 2a). This cathodic response characterizes the modified surface since, normally, this potential range does not display any significant cathodic current (Fig. 2c) [11,12,15]. The two novel cathodic peaks appearing during this first negative scan are situated at $-1.5 \pm 0.1 \text{ V/MSE}$ and $-1.8 \pm 0.1 \text{ V/MSE}$ (labeled as 1 and 2 in Fig. 2a). Their intensity ratio varies from one experiment to another, but interestingly their signatures (shape and position) remain stable whatever the duration of the bombardment. According to XPS results, these peaks seem to be related to the presence of the In-rich

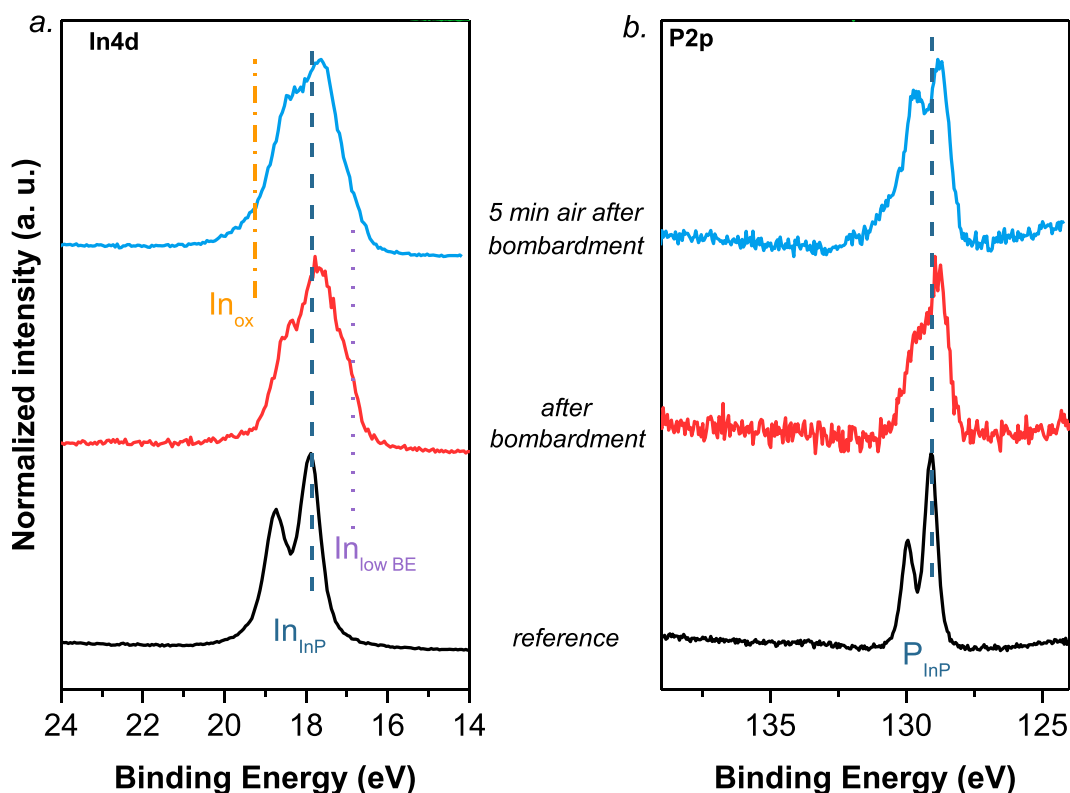


Fig. 1. Evolution of high-resolution (a) In4d and (b) P2p XPS spectra. From bottom to top: reference sample (oxide-free initial InP surface); after bombardment of the sample (InP surface bombarded with Ar^+) and 5 min air after bombardment (InP surface bombarded with Ar^+ and exposed to air for 5 min). For both In4d and P2p plots, the broken lines indicate regions of In_{InP} and P_{InP} (blue dashed line), In_{ox} (yellow dash-dot line) and the $\text{In}_{\text{low BE}}$ (purple dotted line) peak features. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

overlay. Moreover, the reduction phenomenon implies the presence of oxide phases, in agreement with surface analyses results, which are stable at pH 10 [13,16] as confirmed by the steady and specific d-OCP values. Different interpretations can be proposed for the presence of the specific double cathodic features. First, the presence of different oxide compounds (hydroxide, oxide), leading to reduction peaks at different overvoltages appears possible. Another explanation could be the

heterogeneous distribution and roughness of the oxide phase on the InP substrate, leading to different electron circulations at the outer layer/InP interface and explaining the different overvoltages observed during the cathodic transfer. When the potential range is extended toward more negative values, the characteristic hydrogen evolution current associated with water reduction is observed at -1.9 ± 0.1 V/MSE (Fig. 2a). This hydrogen evolution threshold is similar to that of an

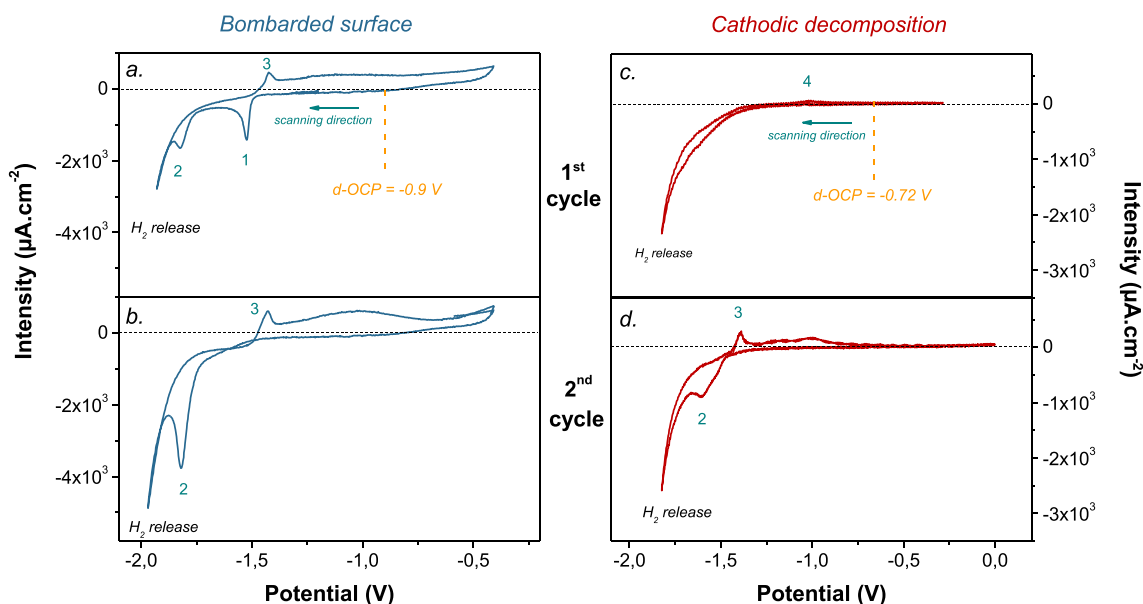


Fig. 2. Intensity versus potential curves for the first (top spectra) and the second (bottom spectra) cycles for InP surface bombarded for 120 sec (a and b) and after cathodic decomposition (c and d).

unperturbed electrode (Fig. 2c), meaning that the buffer bath does not interfere with the modified electrode [13]. The bombardment does not induce the significant shift in the overpotential that would be required to cause hydrogen evolution on *n*-type InP.

The second main characteristic observed on the *I* vs *V* curves for the bombarded surfaces is the anodic response detected at -1.4 V/MSE during the following reverse positive scan (peak 3 on Fig. 2a). This sharp anodic peak is only observed when the cathodic features at -1.5 ± 0.1 V/MSE and -1.8 ± 0.1 V/MSE are present on the initial potential scan, and is followed by a plateau, corresponding to the total oxidation of the previously reduced film. When the negative potential scan is reversed before reaching peak 2 on the first scan, the anodic response with its specific peak at -1.4 V/MSE is still observed but with a lower intensity and a less well-defined plateau. This intermediate response cannot allow us to discriminate between our above assumptions about the exact layer configuration. Nevertheless, it must be emphasized that this complex electrochemical response is only related to the post-bombardment layer behavior, excluding any contribution from cathodic side reactions.

Finally, a third peculiarity is evidenced by cyclic voltammetry, showing a difference between the first and the following negative scans. After the first specific anodic response, the second negative scan only conserves one well-defined cathodic peak at -1.8 ± 0.1 V/MSE (labeled 2 on Fig. 2b), similar to the peak 2 previously described on Fig. 2a, but with a higher current intensity. When successive scans are prolonged with identical negative and positive potential limits, a stable voltammogram shape is obtained, characterized by a lone cathodic feature, an anodic peak and a plateau. All these observations demonstrate that the outer layer induced by Ar^+ bombardment is electrochemically active. A very specific and quasi-stable response is observed as long as the domain of water reduction and its side InP decomposition is not reached. Indeed, when the potential limit includes the hydrogen evolution reaction, the shape of the voltammogram is unchanged but progressive increases in the current intensities of its cathodic and anodic features are observed, correlated with the increase in the number of extended negative scans.

It is important to highlight the reproducibility of the electrochemical behavior of the bombarded surfaces, i.e. that consistent features were observed irrespective of the duration of the bombardment (when > 10 s). This demonstrates the regularity of the InP surface state, but with fluctuations in the distribution of compounds (variability in the peak intensities).

All these observations suggest that the reduction and oxidation peaks are specific to the outer In-rich layer; in other words, that a bombarded surface is characterized by a specific electrochemical signature at pH 10. To better comprehend the nature of this outer layer, a comparison with an InP cathodically decomposed surface is essential [11,13,15]. On *n*- or *p*-InP, during strong hydrogen evolution, a side reaction causes cathodic decomposition whatever the pH of the solution. This decomposition creates an In overlayer on the InP surface which modifies the electrochemical behavior of the surface. The voltammogram obtained for a decomposed *n*-InP electrode (taken from the same wafer as the bombarded one) is shown in Fig. 2b and d and appears to match the one observed on a bombarded surface (steady state) and thus reinforces the hypothesis of an In-enriched surface after Ar^+ bombardment. This specific electrochemical behavior is related to the reversible $\text{In}(\text{OH})_3/\text{In}$ transformation of the superficial In film induced by the cathodic decomposition [11–16].

The similar electrochemical behaviors of bombarded and cathodically decomposed surfaces indicate that the partially oxidized In-rich metallic layer at the surface drives the electrochemical behavior. According to the voltammograms, the InP surface, with its ultra-thin metallic-like overlayer, is transformed into InP covered with an ultra-thin mixed In oxide/In metal layer which can exhibit cathodic and anodic features. Note that, on the bombarded surfaces, the position and the intensity of the peaks are very reproducible and remain constant

when complete voltammetric cycles are performed, in agreement with the rather constant stoichiometry loss. To generate a higher amplitude of the global voltammetric cycle, the negative limit must be overcome to increase the amount of In through the cathodic decomposition mechanism. Again, the position of the cathodic feature remains the same. Another proof of the complete transformation of the In enrichment in the oxide overlayer through electrochemistry measurements is given by HCl dipping. When the bombarded surface is dipped in HCl after a sufficient time for interaction with air, a regeneration of the initial surface is observed using XPS or electrochemistry.

4. Conclusion

After ionic Ar^+ bombardment, InP surfaces undergo preferential sputtering (P loss) associated with a surface perturbation under a few nanometers with a specific enrichment of metallic-like In, the peculiarly low P content being attributed to the presence of remaining elemental P. Brief exposure to air leads to oxidation of the metallic indium. A double structure is observed in the cathodic response on all bombarded surfaces in the first negative scan of the *I* vs *V* curve, showing that the oxidized part of the enriched In surface can be spontaneously reduced. This first scan gives rise to a specific voltammogram which demonstrates the reversible oxidation/reduction transformation of the film. After stabilization, this behavior is similar to that observed during the cathodic decomposition of a pristine InP surface. A good correlation between the XPS and electrochemical information is, thus, illustrated here. This work demonstrates that interfacial electrochemistry can be used as an excellent analytical tool to study the perturbations caused by an ion beam, providing additional electrical and analytical information not accessible through an XPS surface approach alone, or via other related characterization methods. The multiple possible variations of the electrochemical bath combined with a variety of controls lead to a plethora of (electro)chemical characterization possibilities, alongside a route for regeneration of the initial surface.

CRedit authorship contribution statement

S. Béchu: Writing - original draft, Formal analysis, Methodology, Investigation. **D. Aureau:** Investigation, Writing - review & editing. **J. Vigneron:** Investigation, Methodology. **A.-M. Gonçalves:** Investigation, Writing - review & editing. **M. Frégnaux:** Investigation, Methodology. **M. Bouttemy:** Investigation, Writing - review & editing. **A. Etcheberry:** Investigation, Methodology, Formal analysis, Writing - review & editing, Supervision.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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