

Photoemission spectroscopy of the evolution of In-terminated InP(100)-(2×4) as a function of temperature: Surface- and cluster-related In 4*d* lines

Y. Chao,¹ K. Svensson,² D. Radosavkić,³ V. R. Dhanak,^{4,5} M. R. C. Hunt,⁶ and L. Šiller¹

¹*Department of Physics, University of Newcastle upon Tyne, Newcastle, NE1 7RU, United Kingdom*

²*Applied Physics Department, Chalmers University of Technology, Göteborg SE-41296, Sweden*

³*School of Physics and Astronomy, The University of Birmingham, Birmingham B15 2TT, United Kingdom*

⁴*CCLRC, Daresbury Laboratory, Warrington, Cheshire, WA4 4AD, United Kingdom*

⁵*Surface Science Centre, Liverpool University, Liverpool L69 3BX, United Kingdom*

⁶*School of Physics and Astronomy, University of Nottingham, Nottingham, NG7 2RD, United Kingdom*

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The evolution of clean, In-terminated InP(100)-(2×4) surfaces is investigated by synchrotron-radiation-excited photoemission spectroscopy as a function of annealing temperature. As-prepared InP(100)-(2×4) surfaces are found to be free of metallic indium, and the 4*d* core level shows two clear surface components. A third, indium-cluster-related component appears after annealing above 360±10 °C, due to phosphorous desorption, and is accompanied by a corresponding reduction in intensity in the In-P surface component. Further annealing leads to a decrease in binding energy of the indium-cluster-related peak due to increased metallicity and hence core-hole screening in the clusters. The increasingly metallic nature of the indium clusters is also revealed by the appearance and growth of a Fermi edge in valence-band spectra.

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Despite the large body of literature regarding the composition and structure of the In-rich InP(100)-(2×4) (Refs. 1–7), surface there is still disagreement in several areas. For example, in photoemission studies it has been difficult to assign the number of low-binding-energy components of the In 4*d* core line which are associated with “free” indium in an unambiguous way. The In 4*d* core level has been fitted with three surface and one bulk component by some groups,^{1,2} while others have fitted with two surface components and one bulk component.^{4,5,7} Perhaps the main problem is that researchers are unable to distinguish the low-binding-energy component(s) related directly to the surface reconstruction from that due to “free” indium (by “free” indium we mean indium in excess of that required for a perfect 2×4 reconstruction) found in the form of clusters on the surface. An additional difficulty in the unambiguous assignment of a particular In 4*d* component to indium clusters is that, as we shall demonstrate in this paper, the binding energy of the cluster-related component changes according to the size, and hence screening efficiency, of the indium clusters.

The above debate about possible models for the In-rich (2×4) InP(100) surface reconstruction arises from the discovery that this surface does not behave analogously to the GaAs(100) surface.⁸ It has recently been suggested on the basis of scanning tunneling microscope (STM) studies,^{6,9} total energy calculations,^{10,11} and angle-resolved photoemission spectroscopy^{8,11} that simple In-In dimers or trimers are not solely present on this surface, as originally expected, but that there is evidence that there is also a considerable fraction of mixed In-P dimers^{6,8–11} or even P-P dimers^{6,12} present. Our recent studies of the chemisorption of C₆₀ on InP(100)-(2×4) (Ref. 13) are in agreement with these findings—chemisorption of a fullerene leads to changes in the spectral features of both the fullerene and phosphorus, but not of the indium. This result requires the presence of

phosphorous atoms at the outermost layer of the InP(100)-(2×4) surface, which are likely to desorb from the surface in the initial stages of annealing.

In addition to providing further insight into the way in which the InP(100)-(2×4) surface changes under annealing, information gleaned about the nature of the indium clusters is important in its own right. The properties of clusters have been the focus of much research interest over the years^{14–17} partly due to the desire to understand the transition from the atomic to bulk solid state¹⁸ and partly due to the potential applications of clusters in fields as diverse as heterogeneous catalysis,¹⁹ nonlinear optics,^{20,21} and nanoscale electronics.^{22,23} There are a number of ways of growing clusters on substrates, such as *in situ* size and energy-selected cluster deposition²⁴ and thermal evaporation^{25,26} and *ex situ* chemical preparation.²⁷ In the case of III-V semiconductors, clusters of the group-III elements are formed simply by heating, sputtering, or reacting with atomic hydrogen.^{15,26,28–30} Once prepared and then oxidized, indium clusters, for example, might become important in the formation of InO_x thin films, widely known as sensor elements.^{31,32}

In air the InP(100) surface naturally oxidizes so that an ion bombardment and annealing treatment are necessary in order to obtain an In-rich (2×4) reconstruction and has been studied in the past.^{33–35} The periodicity of the surface has been confirmed by low-energy electron diffraction (LEED) and STM.⁶ Indium clusters produced by annealing oxidized InP(100) over a wide temperature range (250–550 °C), have been observed by photoemission spectroscopy (PES).³³ No detailed studies of the In clusters were made since the emphasis of that work was mainly to investigate the influence of Sb deposition on the surface thermal stability. Reflection electron microscopy³⁴ (REM) has also been used to study the growth of indium clusters on InP(110) at 650 °C (above the thermal decomposition temperature 370 °C of the InP

surface³⁰). PES has also been used to investigate argon-ion-bombardment-treated InP(110) surfaces over a wide temperature range.³⁵ The starting surface in that work was already cluster covered, due to preferential phosphorous sputtering above ion energies of 60 eV.³⁶ To our knowledge, no work has been done on the growth of clusters of indium starting with cluster-free In-terminated InP(100)-(2×4) surfaces. In this work we study the effects of heating a perfect (to within experimental sensitivity) InP(100)-(2×4) surface in a temperature range between 360 and 500 °C, and present a detailed analysis of the changes induced in In 4*d* and valence-band spectra. Clean InP(100) as a high-carrier-mobility material has rising technological potential, and its surface core-level shifts (SCLS's) and morphology has been studied widely by x-ray photoemission spectroscopy (XPS), short-range PES (SRPES), and STM.^{1-6,35,37-42} In studies of the InP(100)-(2×4) surface the proposed that number of In 4*d* core-level components for the clean surface varies, as mentioned above, with the presence of three surface and one bulk component suggested by some groups,^{1,2} while others use two surface components and one bulk component.^{4,5,7} The aim of this work is to try directly to identify and assign the low-binding-energy component of the In 4*d* core line by following its development as a function of temperature. In addition, STM work suggests that the most probable model for a InP(100)-(2×4) surface is a two-phosphorus dimer—two missing dimer model, with the prediction of a highly mobile second layer of In-In dimers which may decompose to form the In clusters.⁶

The samples used in these experiments were cut from polished InP(100) wafers (S-doped, *n*-type with $n = 2 \times 10^{17} \text{ cm}^{-3}$, wafer thickness 400 μm , reported resistivity $1.5 \times 10^{-2} \Omega \text{ cm}$, purchased from Wafer Technology Ltd.). The samples were cleaned by chemical etching using a sulphuric acid and hydrogen peroxide solution [$\text{H}_2\text{SO}_4:\text{H}_2\text{O}_2:\text{H}_2\text{O}(3:1:1)$] for 10 min at 60 °C.^{8,43} They were then rinsed with de-ionized (DI) water and dipped in an $(\text{NH}_4)_2\text{S}_x$ solution for 20 min at 65 °C to avoid oxide formation. Subsequently, they were again rinsed in running DI water and then rapidly introduced into an ultrahigh-vacuum (UHV) chamber at a pressure below 5×10^{-10} mbar. Samples were mounted on a Ta plate and, in general, evenly heated by electron bombardment. However, one of the samples was unevenly heated in order to get a full temperature gradient across the surface. This was achieved by fixing one end of the sample tightly to the sample holder while the other end was loosely held (henceforth we will call this the “temperature-gradient-heated sample”). All the data presented in this paper correspond were obtained from the evenly heated samples unless explicitly stated otherwise. Sample temperatures were measured by a *K*-type thermocouple in close proximity to the sample holder, using slow heating and cooling to ensure proper temperature equilibration. An In-terminated InP(100)-(2×4) reconstruction^{2,7,8,43} was prepared by performing several cycles of sputtering (Ar^+ ions, 0.5 keV beam energy, 1 μA sample current) and annealing according to a well-established recipe, until no sulphur, carbon, and oxygen could be detected by XPS, and sharp LEED patterns characteristic of a clean surface were

observed.^{8,43} Photoemission spectra were acquired with a SCIENTA SES-200 analyzer using beamline 4.1 of the synchrotron radiation source (SRS), Daresbury. All In 4*d* spectra shown in this paper were obtained with 50 eV photon energy. All spectra were acquired in normal emission with the synchrotron light incident at 60° to the normal. The overall resolution of spectra acquired using synchrotron radiation is estimated at ~ 0.3 eV, using the Fermi edge of the metallic clusters. In addition, we acquired spectra with He II radiation from a discharge lamp where the overall resolution was improved to ~ 0.09 eV.¹³ Spectra acquired at this improved resolution were used to determine initial fitting parameters such as the number of surface components, their energy position, and the position and width of the bulk In 4*d* component. Binding energies were referenced to the Fermi level of a Ta plate in good electrical contact with the sample, and care was taken to ensure that no observable surface photovoltages were generated over the range of flux and energies used. The temperatures of the temperature-gradient-heated sample were calibrated by comparing area ratios of all components with those of other spectra that were taken from evenly heated samples (in all cases the samples were heated for an equal time of 2 min). We estimate that the temperature variation across the spot exposed to synchrotron light (~ 1 mm) on the temperature-gradient-heated sample was not more than 10 °C. Area ratios for a given component are defined as the sum of the In 4*d*_{3/2} and 4*d*_{5/2} peak areas for that component divided by the total area of the core line (sum of all components).

Figure 1(a) shows In 4*d* core-level photoemission spectra obtained with 50 eV photon energy from clean and heated samples. Each curve indicates a new sample evenly heated to the labeled temperature. One can see a rise in the shoulder on the low-binding-energy side of the In 4*d* core line, associated with formation of In clusters due to the loss of phosphorus.³⁵ As the temperature of the surface becomes higher, the number and/or size of the clusters increases. Exactly the same behavior was observed on the temperature-gradient-heated sample: see Fig. 1(b). Initial atomic force microscopy (AFM) measurements, to be published elsewhere,⁴⁴ of a gradient-heated sample, across the whole length of the surface indicated the presence of a large number of clusters with strong increase in cluster coverage with temperature. In addition to the obvious growth of the cluster-related component, as we scan from the end annealed at the lower temperature to the higher, one can see an additional effect: an asymmetric narrowing of the In 4*d* line on the higher-binding-energy side (indicated with an arrow). In order to understand this behavior and follow the cluster growth mechanism further we studied the evolution of the shape of the In 4*d* core levels for both evenly heated samples and the temperature-gradient-heated sample in detail. Figures 2(a) and 2(b) show the In 4*d* photoemission spectra of the clean surface and after annealing at 470 °C, respectively. The experimental spectrum was fitted to three (for the clean surface) or four (for the annealed surface) mixed doublets with a Shirley background. During the fitting procedure, spin-orbit splitting was kept fixed at 0.86 eV; the branching ratio was 0.65. The position of the bulk component was kept fixed

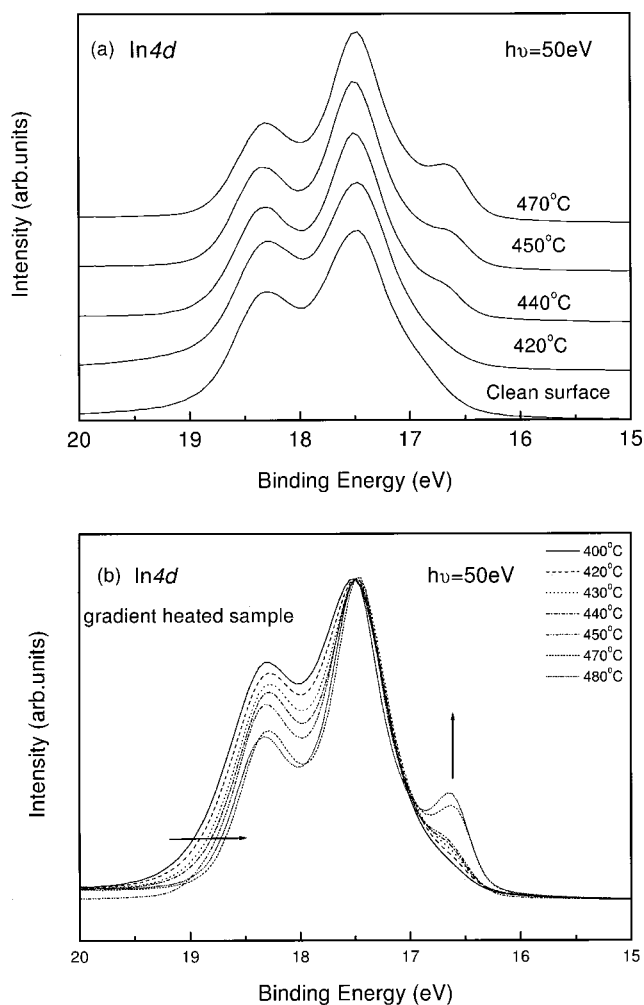


FIG. 1. (a) In 4d photoemission spectra ($h\nu=50$ eV) of the cluster-free InP(100)-(2 \times 4) surface and of four samples heated to the temperatures denoted in the figure. A clear rise of the shoulder on the low-binding-energy side is observed. (b) In 4d photoemission spectra ($h\nu=50$ eV) of the temperature-gradient-heated sample.

within ± 0.02 eV, and the width was allowed to vary by ± 0.05 eV. The initial parameters for fitting the In 4d core lines were obtained from fits to spectra of the clean InP(100)-(2 \times 4) surface¹³ obtained with He II radiation—these spectra were acquired at an improved resolution of 0.09 eV, but at the expense of enormously increased acquisition time. The clean surface was fitted to three rather than four components since the addition of a fourth component led to no significant increase in the quality of the fit, as would be expected if a fourth doublet was genuinely present. It should be noted that the full width at half maximum (FWHM) of the In 4d lines is slightly larger for the clean (2 \times 4) surface than for the annealed surface (0.60 and 0.51 eV, respectively). We attribute this difference to the presence of defects on the as prepared (2 \times 4) surface, which are subsequently reduced in density by the annealing procedure.

The In 4d core-level spectrum of the clean surface can be decomposed into a bulk-related doublet (In-1) and two surface contributions (In-2 and In-3), which exhibit core-level

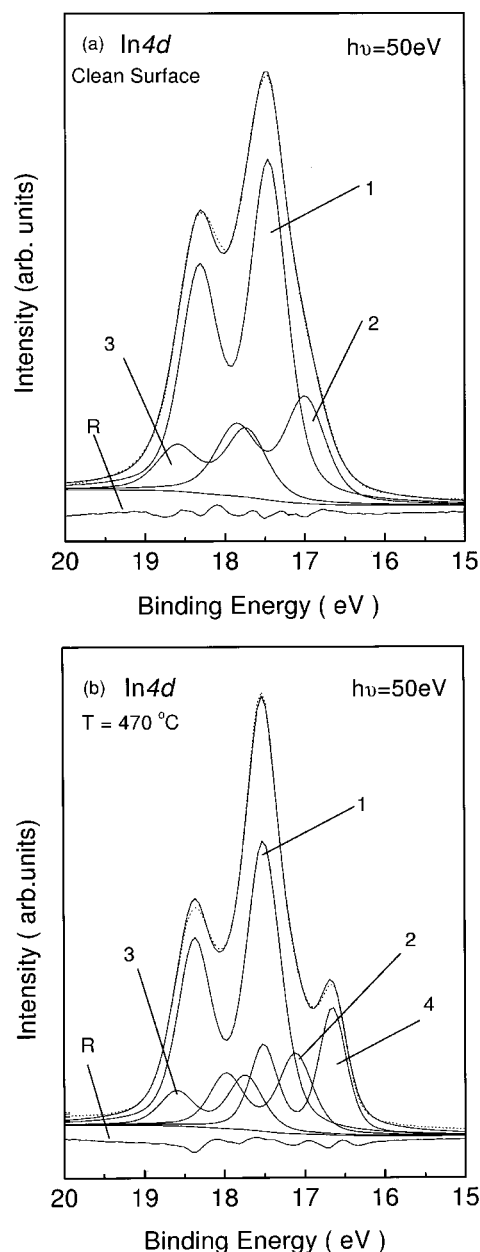


FIG. 2. (a) In 4d photoemission spectrum ($h\nu=50$ eV) of clean InP(100)-(2 \times 4). The experimental spectrum (dotted line) was fitted to a Shirley background and three mixed doublet components: bulk (1) and surface (2) and (3) components. The line marked R shows the residuals of the fit. (b) In 4d photoemission spectrum ($h\nu=50$ eV) obtained after annealing a clean surface at 470°C. The experimental spectrum (dotted line) was fitted with a Shirley background and four mixed doublet components: bulk (1), surface (2) and (3), and indium cluster (4) components. The line marked R shows the residuals of the fit.

shifts compared to the dominant bulk component of -0.40 ± 0.03 eV and $+0.30 \pm 0.03$ eV, respectively, which is consistent with previous studies.^{4,5,38} The In-3 component has been attributed to three-fold coordinated indium surface atoms, which exhibit charge transfer, to three-fold coordinated P surface atoms.^{7,38} The component labeled In-2 might be associated with four-fold coordinated surface indium atoms,

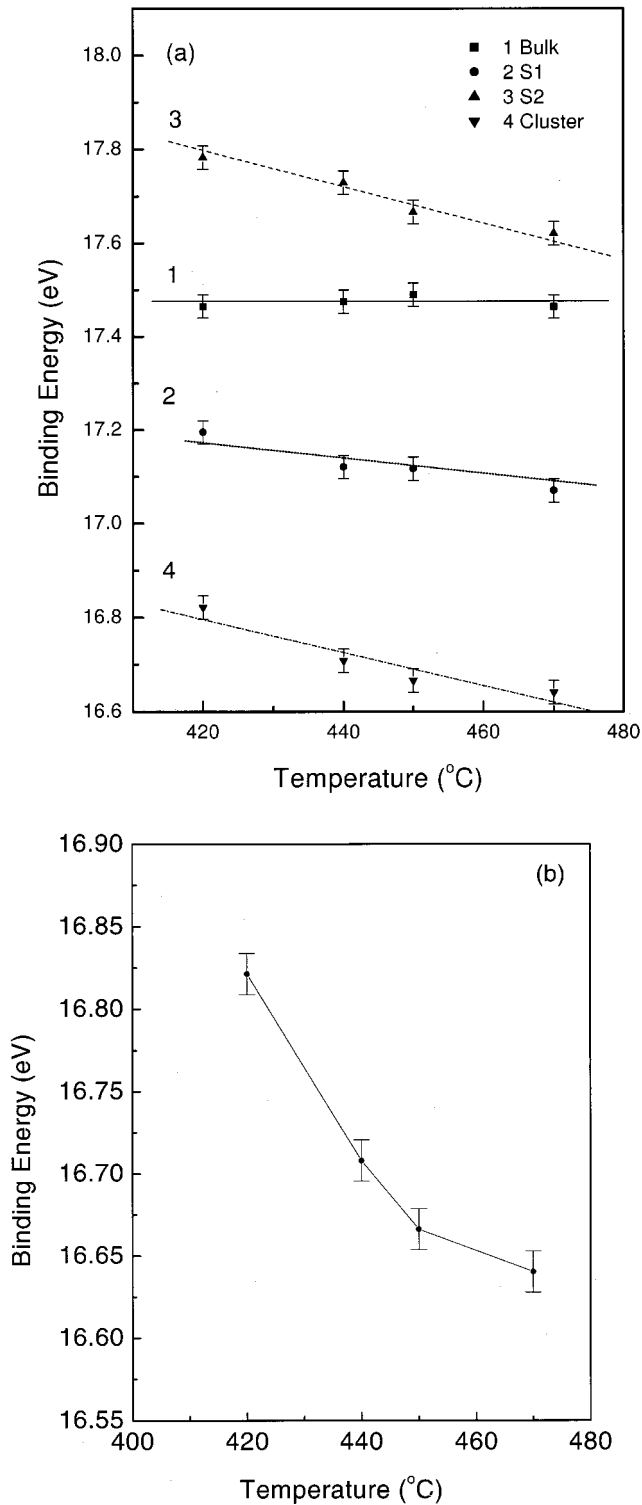


FIG. 3. (a) Binding energy positions of all components for evenly heated samples and (b) binding energy position of the indium-cluster-related component against temperature for evenly heated samples.

which are characterized by charge accumulation.³⁸ After the sample was heated above 360 °C, the surface showed thermal decomposition, as a result of which the In 4*d* spectra had to be fitted with four doublets and a Shirley background.^{1,2} The

fourth doublet (In-4) we relate to contributions from metallic indium clusters, formed from surface indium liberated during the annealing process.

It is worth mentioning the differences in In 4*d* fitting procedures between our work and that of others. Some groups fit the In 4*d* photoemission spectrum of the *cluster-free* InP(100) surface with four doublets and a Shirley background^{1,2} and others with three doublets and a Shirley background.^{4,5,38,40} After experimentation with fitting parameters we found that three doublets (one bulk and two surface components) were enough; after annealing to temperatures beyond the P thermal desorption point, metallic indium appeared on the surface, requiring a fourth doublet. During the fitting procedure, the branching ratio and spin-orbit splitting should be kept fixed for all components, though in Ref. 2 the branching ratio was allowed to vary considerably. In Fig. 3(a) we show the general trend of all four components for evenly heated samples. It is obvious that a shift in the both surface components and the cluster-related component is observed. Since the In-3 (second surface component) is related to charge exchange between the indium and phosphorus,³⁸ it is to be expected that, as one begins to lose phosphorus atoms, this component will become weaker and probably some redistribution of charge will occur. The reduction in intensity of this component is the main reason for the apparent narrowing of the In 4*d* with temperature in Fig. 1(b).

Figure 3(b) shows in greater detail how the position of the cluster-related spectral component of the evenly heated samples shifts with annealing temperature. When the annealing temperature is increased gradually from 420 to 470 °C, the cluster-related component position shifts to lower bind-

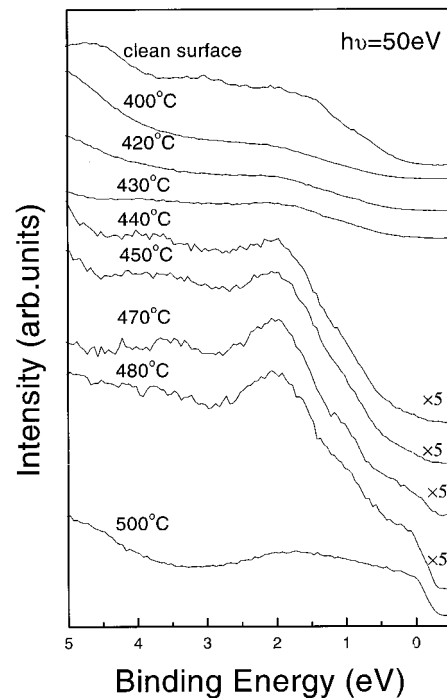


FIG. 4. Valence-band spectra ($h\nu=50$ eV) collected at normal emission from InP(100)-(2×4) surfaces after various annealing steps.

ing energy by 0.2 eV. Examination of the coverage dependence of core electron photoemission intensity and binding energy for simple, noble-metal, and transition-metal clusters supported on amorphous carbon¹⁶ has shown that very small metal-atom clusters, containing fewer than 30 atoms, are not fully metallic and final-state core holes are screened by polarization of neighboring atoms. Consequently, the binding energy depends only weakly on the cluster size.¹⁶ In larger metallic clusters, conduction electrons screen the hole, with the missing charge appearing at the surface of the cluster. In this regime, the core electron binding energy shifts to lower values with increasing cluster size.^{16,23} The variation in binding energy of the cluster-related component of the In 4*d* line indicates that, for temperatures above 420 °C, the indium clusters fall into the latter regime.

To further investigate the evolution of the InP surface during annealing, we recorded valence-band spectra after annealing at each temperature. Figure 4 shows the normalized valence-band spectra. After annealing to 440 ± 10 °C and above, a Fermi edge begins to become apparent in the spectra, indicating that the indium clusters, or a proportion thereof, have become fully metallic. By carefully inspecting the features of the valence band, one can also find that the valence-band maximum (VBM) shifts from 1.70 to 2.05 eV when the annealing temperature is increased from 400 to 500 °C. This might be interpreted as the appearance of new donor states in the gap during the annealing procedure which donate electrons and shift E_F closer to vacuum level. This

appears as a shift of the valence band to a higher binding energy.

In conclusion, the annealing of clean InP(100)-(2 × 4) surfaces, free from excess indium, was investigated by PES. Above the thermal desorption temperature of phosphorous (360 °C),^{31,34} a metallic indium component gradually appears in the In 4*d* line. As-prepared In(100)-(2 × 4) surfaces are free of metallic indium and the 4*d* core level shows only two clear surface components. A third component, indium cluster related, due to phosphorous desorption appears after annealing and is accompanied by a corresponding reduction in intensity in the In-P surface component. By careful inspection of the features of the In 4*d* and valence-band spectra, it is found that a visible Fermi edge appears when the temperature approaches 440 °C. The cluster component of the In 4*d* line shifts by 0.2 eV in binding energy when the annealing temperature is raised from 420 to 470 °C. This shift in the binding energy of the cluster-related core-level component is interpreted in terms of improved core hole screening with increasing cluster size.

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