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Photoemission studies of the Cu-InP(110) interface

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The atomic and electronic structure of Cu—(n-type) InP(110) interface has been studied by soft-x-ray photoemission spectroscopy and Auger spectroscopy for copper coverages ranging from 0.7 to 80 Å. By monitoring the evolution of the P $2p$ and In $4d$ core-level spectra and the change of the Cu $3d$ level, which is degenerate with the substrate valence band, we provide evidence of strong intermixing taking place at the interface. A strong outward diffusion of phosphorous into the copper overlayer and indium segregation at the surface have been observed. A splitting of the phosphorous $L_{2,3}VV$ Auger line has been observed for Cu coverages higher than 8 Å. This splitting, similar to that observed for silicides, provides evidence of bond formation between Cu and P. On the basis of the P $2p_{3/2}$ line shift, a Schottky-barrier height of 0.6 eV has been established.

I. INTRODUCTION

Recently there has been an increased effort to understand the microscopic nature of metal-semiconductor contacts. This issue is challenging both from an academic and a practical point of view. A growing amount of experimental data and theoretical work improved the knowledge of processes which dominate Schottky-barrier formation at metal-semiconductor interfaces only in some cases. No general consensus has yet been reached. This may be partially due to the fact that in most theoretical models only phenomena intrinsic to abrupt metal-semiconductor interfaces were taken into account. Contrarily, the latest experiments show that deposition of metals onto atomically clean cleaved surfaces leads to nonabrupt and nonordered interfaces.[1] Moreover, it has been recently suggested by Brillson and co-workers that interfacial reaction may significantly influence the height of the Schottky barriers on InP(110).[2] This behavior is at variance with less reactive but also nonabrupt interfaces on GaAs where a wide range of different adatoms were shown to give very similar Fermi-level—pinning positions.[3] The independence of barrier height on chemical nature of the metal was one of the basic observations that led to the unified defect model for Schottky-barrier formation recently proposed by Spicer and co-workers.[3,4] In this model, the barrier height is due to the formation of extrinsic semiconductor states at or near metal-semiconductor interfaces. These states are produced by the perturbation of the surface by adatoms at the very beginning of the interface formation.

Since this model has been proposed it has proven to be very successful in the analysis of experimental data for various systems.[5] Also, in the case of InP, recently published data by Williams *et al.*[5] received the most satisfactory understanding for a range of metals via unified defect models. In this work, the existence of two pinning levels has been established and related to two defect levels, one a donor relatively close to the conduction band and the other a donor or an acceptor level at $\sim$0.5 eV below E_c. High Schottky barriers resulted for so-called "nonreactive" metals like Au, Ag, and Cu. More strongly reactive metals such as Al, Fe, or Ni yielded low barrier contacts. The Schottky-barrier height was established mainly on the basis of C-V and I-V methods.[6] Contrary to this report, widespread pinning positions at significantly different energies have been reported for a range of metals (Al, Ti, Ni, Au, Pd, Ag, and Cu) in the aforementioned work of Brillson.[2] In this case, analysis of the shift of the core lines for growing metal coverage has been used. The existing body of data on Schottky barriers (SB) on InP raises two questions: (1) Does experiment show that the unified defect model needs readjustment to include chemical reactivity at the interface and (2) how conclusive are results based on the core-line—shift analysis for systems where strong chemical reactivity may lead to chemical shifts that are difficult to deconvolve from the band bending? To answer these questions further, both experimental and theoretical studies of the influence of chemical reactivity on barrier height and detailed characterization of the interfacial reacted layer are necessary. The recent data[2,5] suggest that for this kind of study InP is of special interest among III-V compounds. However, until recently this material has received much less attention from experimentalists and practically none from theoreticians.

In this paper, we report results of a soft x-ray photoemission study of the Cu—(n-type) InP junction. Using the synchrotron at the Stanford Synchrotron Radiation Laboratory (SSRL), we probe the evolution of the In 4d and P 2p core levels. We also study the change of the electronic structure in the region of the valence band of the host material. The main goals are the following:

(1) To give information on composition of the interfacial compound through the analyses of core-level intensities.

(2) To obtain information on local bonding monitoring the valence-band spectra and chemical shifts of the core levels and change of the line shape of Auger transitions.

(3) To establish the height of the SB and its change with the width of the metal layer.

A similar study of P 2p and In 4d core-level spectra on the Cu-InP interface has been recently published by Brillson *et al.*[2,7] However, due to the higher photon flux offered by the storage ring at Stanford, we were able to get both a better resolution and signal-to-noise ratio and disprove certain findings of this earlier work. Moreover, tuning the photon energy and thus varying the escape depth of photoelectrons with different final-state energies, we could clarify certain issues concerning compositional distribution of the overlayer. It is, however, worthwhile to note that because of the short escape depth of photoelectrons with kinetic energies up to 200 eV, photoemission offers a limited possibility of monitoring the products of interfacial reaction near the junction itself for thicker metallic overlayers. Because of this and also in order to get better insight into the structural and electronic proprieties of the Cu-InP junction, we continue this study using other experimental techniques. Some results obtained using Auger electron spectroscopy will be presented here. A full report of these studies will be published later. To our knowledge, we present here the first study of the evolution of the valence-band spectra for the Cu-InP interface.

II. EXPERIMENTAL

This experiment was performed on the 4° line at SSRL. The radiation was incident on the InP surface at an angle of about 14° from the plane of the surface. A double-pass cylindrical-mirror analyzer (CMA) was used for angle-integrated electron-energy analysis. The overall energy resolution was, in all the cases, better than 0.35 eV. The data were collected with the PDP-11/34—based acquisition system. Clean n-type InP(110) ($n=6\times10^{17}$ cm^{-3}) surfaces were prepared by cleaving in an ultrahigh-vacuum chamber with base pressure $\sim5\times10^{-11}$ Torr. Copper was evaporated by thermal deposition from a bead source carefully outgassed before cleaving of the crystal. The rate was calibrated with the use of a Sloan quartz crystal-thickness monitor. The pressure during evaporation was maintained below 2×10^{-10} Torr. Reference spectra from Au and In were taken from thick metal layers evaporated *in situ* on a stainless-steel substrate in electrical contact with the sample holder and grounded to the analyzer. Details of the method used to determine the surface Fermi energy have been discussed earlier.[8]

During this experiment we determine relative shifts of the core level with accuracy of about ±0.05 eV, the values of core-level energies measured relative to the Au Fermi level are accurate within ±0.1 eV.

III. RESULTS AND DISCUSSION

A. In 4d and P 2p core-line shifts and shape evolution

Figure 1 illustrates modifications of P 2p and In 4d core-level spectra with increasing thickness of Cu deposited onto the cleaved n-type InP(110) surface. The presented energy-distribution curves (EDC's) were taken at photon energies of 185 eV (P 2p) and 80 eV (In 4d), and for both lines they correspond to photoelectrons of extremely short escape depth (~5 Å). This condition assures surface sensitivity in the experiment. As can be seen in Fig. 1, the P 2p level shows a decrease in intensity and rigid shift of the EDC in the initial stage of SB formation. This shift is related to band bending. Contrary to P 2p, the In 4d level undergoes a more complicated change and in addition to band bending we observe the appearance of a new chemically shifted doublet on the high-kinetic-energy side of the In 4d peak. The 0.25-eV shift of the better-resolved $p_{3/2}$ component of the P 2p doublet is completed for the smallest Cu coverage of $\sim\frac{3}{4}$ monolayer (ML) (0.7 Å) studied in this experiment. No further shift of this level is observed for higher Cu coverages. The comparison of the Fermi-level position E_F with the position of the valence-band maximum for clean cleaved InP showed that the cleave used in this experiment was partially pinned with E_F of the clean surface located 0.35 eV below the conduction-band minimum (CBM). The position of the Fermi level—0.6 eV

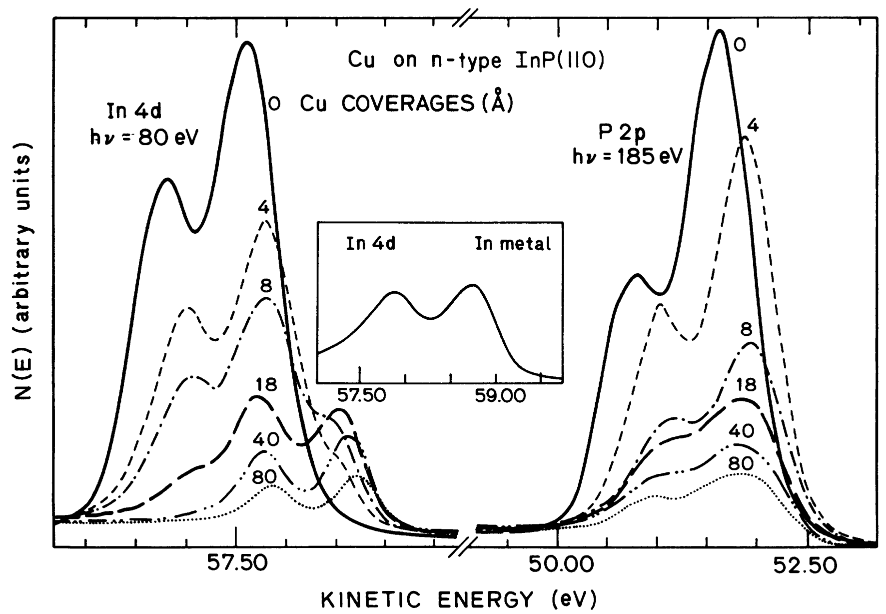

FIG. 1. Photoemission of In 4*d* and P 2*p* core levels taken at photon energies of 80 and 185 eV, respectively, and normalized to the incident photon flux for various Cu coverages.

below the CBM—derived by us for the Cu-InP interface from the shift of P $2p_{3/2}$ binding energy is in close agreement with the value of 0.65 eV established by Brillson and co-workers on the basis of the In 4*d* shift analysis.[2] In our case, the analysis of In 4*d* shift also provides the same result in the early stage of the barrier formation (coverage ≤ 8 Å) although caution must be taken to properly deconvolute additional structure from the core line of the substrate. For higher metal coverages the photoemission from the In 4*d* core level is dominated by segregated indium. This point will be discussed below.

Contrary to the results of Brillson *et al.* no evidence of additional structure in the P 2*p* core line has been detected in this experiment. This additional structure was attributed by those authors to new Cu—P bonding.[2,7] To clarify this point, the EDC's of P 2*p* core lines were also measured for different photon energies, and it was found that in all the cases no additional structure could be resolved. We note, however, changes in the proportion and substantial broadening of the constituents of the spin-orbit (SO) split doublet. Comparing the data for different photon energies, we conclude that SO splitting of the P 2*p* level is within experimental error conserved. Owing to higher photon flux offered by the synchrotron at Stanford we have reason to believe that our data of deeper core levels have both better resolution (for P 2*p* at 185 eV, our estimated overall resolution is 0.32 eV) and better signal-to-noise ratio in comparison with the previous data for InP.[2,7] The fact that we do not observe additional structure in P 2*p* of the type presented by Brillson does not rule out the possibility of chemical interaction between P and Cu. Contrarily, as will be discussed later, on the basis of the line-shape change of the P $L_{2,3}VV$ Auger transition, we find such reaction probable. We conclude here that, if such a reaction is taking place, the respective chemical shift related to this reaction is smaller than the resolution of our experiment and may be responsible for broadening of the P 2*p* spectra for higher coverages.

Contrary to P 2*p* emission, which seems to be only slightly affected by chemical reaction, the In 4*d* core level shows dramatic change with increasing Cu deposition (compare Fig. 1). For this line our data are in better agreement with the results of Brillson. In both cases an additional doublet chemically shifted toward lower binding energy has been found. The chemical shift grows with increasing Cu coverage toward the binding energy of the bulk indium core level. A similar change of In 4*d* core-level emission has been found by us[9] and others[2,5] for InP interfaces with reactive metals. Angle-resolved-photoemission studies for Ni-InP systems show that an appreciable fraction of In resides on the surface of the Ni film.[5] Our data also confirm this assignment for the Cu-InP system. We observe the following:

(1) For the highest coverage of 80 Å, the binding energy of the chemically shifted peak and its SO splitting ($\Delta_{SO} = 0.85$ eV) are identical to those ob-

tained for In metal (see inset, Fig. 1). This shows that a metallic In—In bond is present for species showing chemically shifted emission.

(2) Increase of photon energy above $h\nu = 80$ eV causes relative decrease of the ratio of shifted to nonshifted peaks. This fact is illustrated in Fig. 2 where the spectra of InP + 18 Å Cu for $h\nu = 80$ and 120 eV have been compared (spectrum taken at 120 eV has been shifted by 40 eV and rescaled to equal intensity in the lower-kinetic-energy shoulder arising from In 4*d* photoemission from the "deep" interface region). Although the change of the escape depth is less than 20%,[8] the change is quite visible. Since in all cases we monitor the change of the same In 4*d* line, it is obvious that the observed effect is due to the increase of sensitivity to the surface region for 80 eV and not a cross-section effect.

Additional arguments showing that In is segregated to the surface come from the change of intensities of In 4*d* emission for different photon energies and will be discussed later.

B. Valence-band EDC's

Photoemission spectra of the valence-band (VB) for different Cu coverages are shown in Fig. 3. These data were taken at an excitation energy of 80 eV. In the top panel of the figure, the VB of clean Cu(110) is given for comparison. Similar data have also been taken at $h\nu = 120$ eV. For both energies the peak of the Cu 3*d* electrons gives a dominant contribution at high Cu coverages. This is due to the relatively high cross section for photoionization from this level. In consequence, the evolution of the features related to the *sp* VB is, for higher coverages, difficult to analyze. However, the data for the submonolayer coverages are of special interest as they may provide information on the interface states and their relation to the initial metal-chemisorption geometry.[10]

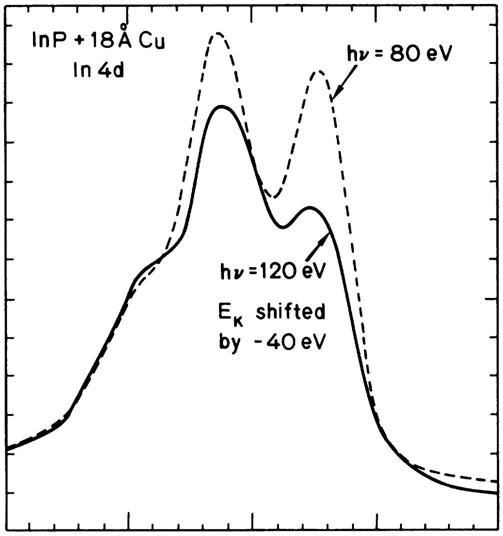

FIG. 2. Photoemission spectra of In 4*d* core levels taken at energies of 80 and 120 eV. 120-eV spectrum has been shifted by 40 eV and rescaled to match low-kinetic-energy shoulders.

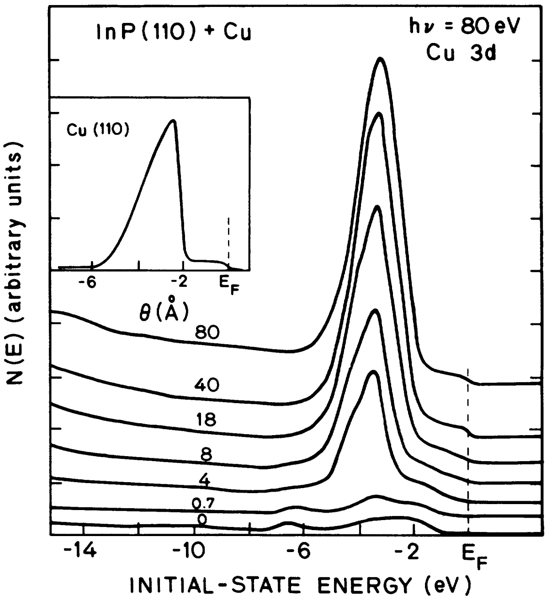

FIG. 3. EDC's for Cu(110), InP(110), and InP(110) + Cu as obtained with $h\nu = 80$ eV.

The spectra of cleaved InP(110) (clean and with $\frac{3}{4}$ ML of Cu) taken at 80 and 120 eV are presented separately in Fig. 4. Over a wide range of energies, we could match all features attributed to the InP surface (including In 4*d* and P 2*p* core levels) simply by multiplying the InP + $\frac{3}{4}$ ML of Cu spectrum by a constant representing attenuation of bulk features of the spectrum by the metallic overlayers. This procedure enables us to subtract contributions related to the Cu 3*d* emission. As can be seen, all features of the clean surface are preserved after this initial evaporation, and apart from the Cu 3*d* peak arising 2.9 eV below the VB edge no additional peak or shoulder is detected. This means that either no adatom states are present in the VB for this interface or the respective feature is energetically degenerate with the Cu 3*d* band and not resolved in this experiment.

The difference-curve 3*d* peak is much narrower than the 3*d* peak for the highest coverage of 80 Å studied in this experiment or Cu metal [top panel of

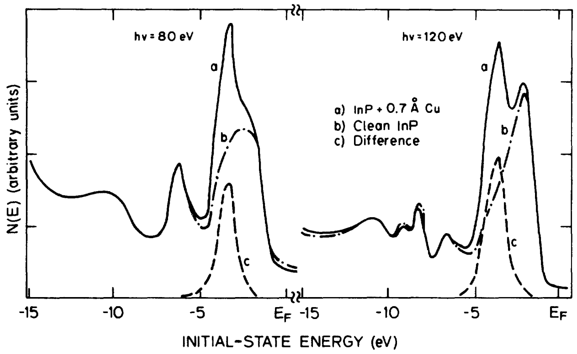

FIG. 4. EDC's for cleaved InP(110) and for InP(110) + $\frac{3}{4}$ ML of Cu taken at $h\nu = 80$ and 120 eV. Curves have been rescaled to match structures related to clean InP photoemission (including In 4*d* levels—not shown—and P 2*p* levels that can be seen in the second order for the 120-eV spectrum near -9 eV) and subtracted to sort out Cu 3*d* contributions.

Fig. 3 (Ref. 11)]. Also, the binding energy of Cu 3*d* at the initial stage of interference formation is significantly higher than the value in bulk metal. The changes in the full width at half maximum and the binding energy of the 3*d* peak as a function of the Cu coverage are shown in Fig. 5. Similar narrowing and shifting to higher binding energy of the 3*d* band at the initial stage of SB formation has been observed by Abbati *et al.* and Rossi *et al.* for Cu-Si interfaces.[12,13] On the basis of the temperature dependence of the Cu 3*d* contribution, they attribute the evolution of the peak position and width to atomic intermixing. It is possible that a similar mechanism is responsible for changes at Cu-InP interfaces, for which analysis of core-line intensities strongly suggest that intermixing is taking place. If we adopt this interpretation, a smooth change of binding energy would give an argument that Cu is evenly dispersed in the adlayer rather than sharp three-dimensional islands. Unfortunately, the interpretation of the 3*d*-band change is not unique and, as pointed out by Abbati *et al.*, similar behavior is expected and observed for a thin (~ 1 ML) Cu layer evaporated on nonreactive substrates and for small Cu particles.[14] The reduction of width and change in binding energy is in this case attributed to the decrease of the *d*-*d* overlap and possible reduction of *sp*-*d* Coulomb interaction.

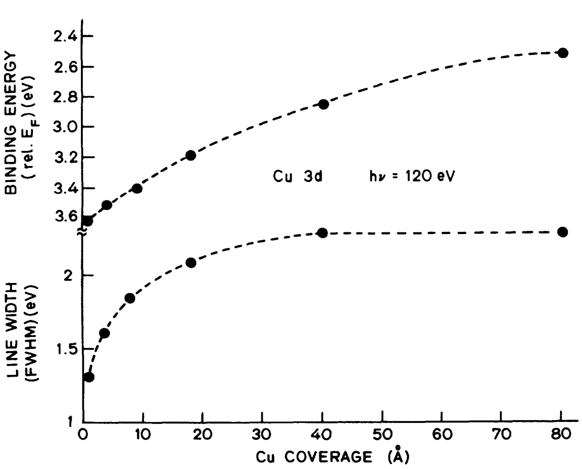

FIG. 5. Coverage dependence of 3*d* linewidth and binding energy.

Unambiguous separation of the aforementioned "size effect" from the change related to intermixing induced rehybridization is, in the case of the Cu-InP junction, difficult. However, the following experimental facts suggest that most of the Cu 3*d*—level change is due to intermixing:

(1) The 3*d* peak shape is for all coverages more symmetrical than in pure Cu (compare spectra of Fig. 3).

(2) The change of the width and binding energy is relatively slow in comparison to junctions with a sharp interface and thus small intermixing (for example, when comparing evolution of the 4*d* level of silver on the Ag-Si interface[15]).

We conclude that evolution of the 3*d* level gives evidence that Cu-InP intermixing leads to a broad interface.

Additional information related to the interfacial reaction comes from the analysis of the line shape of the VB related P $L_{2,3}VV$ Auger transitions. These electron stimulated Auger data were taken in a separate chamber (base pressure of 8×10^{-11} Torr) on sputter-cleaned and annealed InP (100) wafers. Figure 6 shows the change of P $L_{2,3}VV$ Auger line shapes for different Cu coverages. The lowest deposition of 0.75 Å shows that the Auger line is very similar (slightly broader) to that of the clean InP crystal (not shown), and further Cu evaporations cause an increase of the width of the line. At 8-Å coverage additional Auger transitions can be resolved. Further evaporation produces a well-resolved doublet structure with ~4.3-eV splitting. Similar study of the $L_{2,3}VV$ Auger peak of Si was successfully used by many groups to identify the products of chemical reaction at Si interfaces.[13,16–18] It was shown that, in the case where chemical reaction (leading to formation of silicides or intermixed alloys) is taking place, the Si $L_{2,3}VV$ transition exhibits splitting resulting from the formation of new bonds, and thus a different distribution of Si valence electrons. The theoretical basis

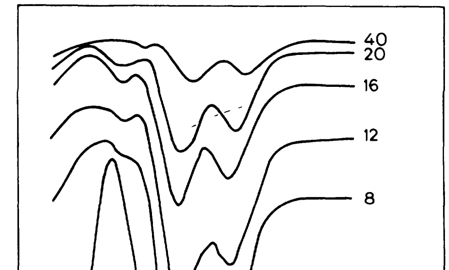

FIG. 6. Auger P $L_{2,3}VV$ line shapes for InP(100) + Cu vs coverage at room temperature.

for the interpretation of the Si $L_{2,3}VV$ transition has been developed by Sawatzki,[19] Feibelman,[20] Jennison,[21] and others.[22] It was shown that for the Si $L_{2,3}VV$ transition the Coulomb interaction between the two holes created is small compared to the bandwidth, and thus this transition carries information about the local density of states of Si. Moreover, the Si LVV transition was successfully interpreted as a self-convolution of partial $3p$ density of states, with the sensitivity to p symmetry being an Auger matrix effect. This interpretation of Si LVV transitions has been extended to the study of silicides. The change of the shape and splitting was attributed to the redistribution of the local density of $3p$ valence electrons of Si dissolved in the metal. This redistribution stems from hybridization of the $3p$ states with metal d band and gives the direct evidence of interfacial alloy (or silicide) formation.[23] On the basis of our data it is highly probable that the same interpretation can be extended to the P $L_{2,3}VV$ Auger line. We interpret the splitting of the line as indication of Cu—P bonds and formation of a new interfacial compound. It is worthwhile to note that the observed splitting has a value similar to that observed for silicides. The problem of interfacial reaction will be discussed in the next section.

C. Core-level intensities

Core-level intensities give information on the composition change of the interface with increasing Cu coverage. As pointed out before, due to the extreme surface sensitivity this information is, however, limited to the first few upper layers of the interfacial compound and only for relatively small coverages provides direct insight related to the junction itself. But, even sampling relatively narrow regions, we are able to conclude (on the basis of our data) that the Cu-InP interface, in agreement with data presented in previous paragraphs, is broad and that strong intermixing is taking place.

In order to compare intensities of the photoelectrons emitted from the particular core levels, the areas under the different peaks have been computed and normalized to the corresponding areas observed on the clean surface (for P $2p$ and In $4d$) or for the maximum coverage studied (Cu $3d$). Figure 7 shows the intensity variation as a function of Cu coverage for two photon energies. The first set of curves [Fig. 7(b)] corresponds to kinetic energies of photoelectrons of about 50 eV, i.e., close to the minimum of the escape depth. In the second set [Fig. 7(a)] photoelectrons from particular core levels leave the crystal with E_k close to 100 eV. Although the escape depth of photoelectrons versus kinetic-energy dependence in the case of InP is not exactly known, we expect it to be similar to that of GaAs.[8,24] Thus, we expect that the relative change of escape depth between these sets of data is small (about 20%). We observe that even a small change in escape depth causes a noticeable change in the intensity profile. This shows that In segregation, as discussed before, is indeed restricted to the near-surface layer. Contrary to indium, no noticeable difference in the change of intensity (data taken at 185 and 235 eV) has been observed for the P $2p$ peak. From the figure it is evident that the intensity of this level does not fall exponentially as anticipated for so sharp an interface and continuous overgrowth of metal. Contrarily, we observe a high amount of phosphorus in the upper layer of the copper. It is also worthwhile to notice that even for the highest coverage (80 Å) of Cu, the intensity of the P $2p$ core level exceeds 20% of its value for the clean InP substrate; and, even more surprisingly, a very small change of $2p$ intensity is observed be-

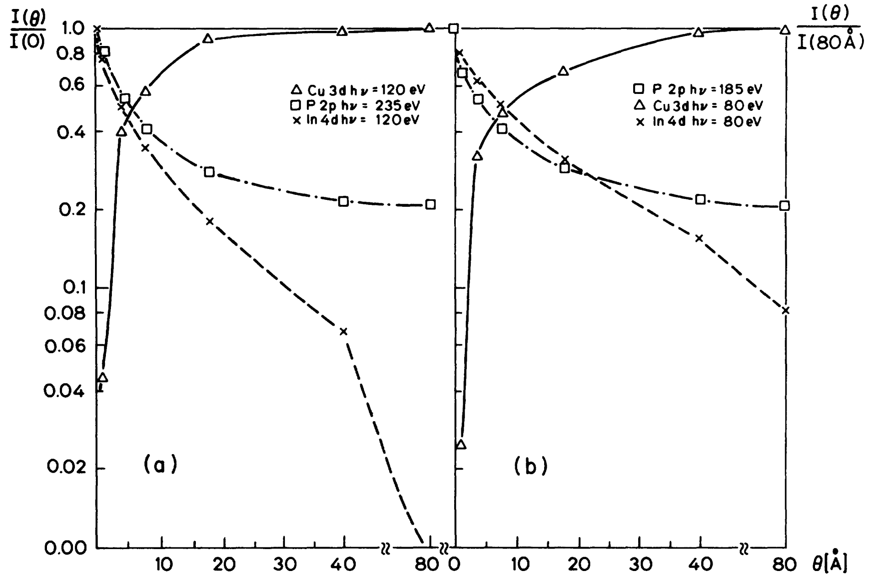

FIG. 7. Relative intensity profiles for Cu 3*d*, In 4*d*, and P 2*p* levels for InP(110) + Cu for different photon energies. P 2*p* and In 4*d* core-level intensities have been normalized to their respective values for clean cleaved InP(110), and that of Cu 3*d* has been normalized to the maximum coverage spectra.

tween 40 and 80 Å of Cu deposited. It is known from the bulk thermodynamics that Cu and P form a relatively low-temperature eutectic—Cu_3P.[25] It is interesting to check whether this kind of compound can be formed during the Cu-InP formation. This problem cannot be solved on the basis of the data presented above and awaits further investigation. The strong subeutectic reaction is, however, well documented for the Cu-Si interface. In particular, on the basis of Auger electron spectroscopy (AES) and photoemission studies for the Cu-Si interface, it was shown that the interfacial compound consists of a Cu_3Si eutectic.[13] Keeping in mind that segregation of about 1 ML of Si to the surface is observed for the Cu-Si system and is typical of all silicon-silicide interfaces, we have a strikingly similar picture of the interface profile based on our data for a Cu-InP junction. In our case, the surface energy is lowered by the segregation of indium. The segregation of In to the surface also seems to be the rule for InP interfaces and was observed for overlayers with strong interfacial intermixing.[2,5,9]

IV. CONCLUSIONS

On the basis of photoemission and AES studies we provide evidence of strong intermixing on the Cu-InP interface. We observe significant outward diffusion of P into the interface together with Cu-P band formation. An indium overlayer has been shown to segregate to the surface.

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