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Room temperature adsorption of Au on cleaved GaAs (110) (*)

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Résumé. — On étudie les premières étapes du dépôt sous ultravide de l'or, de 10^{-3} à environ 10^2 monocouches (mc), sur une surface (110) initialement propre de GaAs clivé maintenu à température ambiante. Des renseignements d'ordre cristallographique, compositionnel et électronique sur la surface sont obtenus à partir de la diffraction des électrons lents, de la spectroscopie des électrons Auger, de la spectroscopie de perte d'énergie des électrons, et de la spectroscopie de rendement de photoémission. Après des effets dus à la reconstruction de surface à des recouvrements inférieurs à 0,1 mc, on observe la formation d'un composé bidimensionnel qui introduit une densité spécifique d'états déterminant la position du niveau de Fermi ; un modèle est proposé conduisant à une épaisseur de 8 Å environ et une composition de quatre atomes d'or par maille superficielle de GaAs. Ensuite, des îlots d'or de plus en plus larges se forment sur la surface.

Abstract. — The early stages of ultrahigh vacuum deposition of Au, from 10^{-3} to about 10^2 monolayers (ML) on an initially clean cleaved (110) surface of GaAs kept at room temperature are studied. Crystallographic, compositional and electronic surface data are obtained using low energy electron diffraction (LEED), Auger electron spectroscopy (AES), electron loss spectroscopy (ELS) and photoemission yield spectroscopy. After surface reconstruction effects at coverages below 0.1 ML, the formation of a two-dimensional compound is observed which brings a specific density of states determining the Fermi level position ; a model is developed leading to a compound about 8 Å thick with four Au atoms per GaAs surface unit cell. The formation of more and more spread islands of Au follows.

1. Introduction.

In earlier work we have studied the initial steps of the interface formation between Ag [1] or Cu [2] and the clean cleaved surface of GaAs (110) at room temperature. It was found that silver grows epitaxially on GaAs, as confirmed by Ludeke *et al.* [3] giving a film with a saw-toothed profile after deposition of several monolayers, but without disturbing significantly the bonds between Ga and As atoms up to the interface. On the contrary, it was found that Cu first formed a two-dimensional compound uniform layer with the first two monolayers of GaAs, changing thoroughly the electronic structure of the surface

region, before growing more or less spread metallic islands.

In order to find an explanation for these strikingly different behaviours of two elements which are neighbouring noble metals of the first column in the periodic table, it was interesting to study under the same conditions the behaviour of Au, the third element of the family. A few studies have been devoted already to the Au/GaAs (110) system. In particular, the group at Stanford University [4-7] using UV and X-ray photoemission spectroscopies proposed at first an interface formation dominated by intermixing, even at room temperature, up to the highest coverages (7×10^{16} atoms cm^{-2}), and accompanied by the development of a band bending which could reach 1.3 eV on an n-type GaAs substrate [6]. Recently, Pan *et al.* [8] have not excluded a non laminar Au film growth ; if it is indeed the case, the experimental data become much more difficult to interpret since the surface is then inhomogeneous. Earlier, Hiraki *et al.* [9] concluded from Auger electron spectroscopy and ion sputtering measurements to an outdiffusion of Ga atoms and the formation of an alloy at the

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interface. Brillson *et al.* [10, 11] using Al as a probe proposed also the formation of an alloy. In the present work, we take advantage of the simultaneous use of several complementary techniques which have been already successfully employed to study several similar systems [1, 2, 12], to get a more accurate picture of the first steps of the Au/GaAs (110) interface formation. In particular, the band bending changes on both n- and p-type substrates upon increasing metal deposition will be compared with the Schottky barrier value of 0.98 eV obtained under similar initial conditions by Chekir, Barret and Vapaille [13] on n-type GaAs substrates. The same authors have demonstrated the existence of acceptor states at the interface which cover a large part of the gap from the conduction band edge down to almost 0.4 eV from the valence band edge.

2. Experimental.

The ultrahigh vacuum apparatus used for the experiments has been described elsewhere by Bolmont *et al.* [12] : it allows *in situ* preparation of the surface (cleavage and/or annealing), metallization, and measurements by low energy electron diffraction (LEED), Auger electron spectroscopy (AES), electron loss spectroscopy (ELS) and photoemission yield spectroscopy (PYS).

A set of monocrystalline GaAs samples provided by RTC-La Radiotechnique (Caen, France) has been studied, the free carrier densities of which were respectively $n = 2 \times 10^{16} \text{ cm}^{-3}$ and $p_{zn} = 1.6 \times 10^{18} \text{ cm}^{-3}$. The sample $6 \times 4 \text{ mm}^2$ in cross section was cleaved and gave a sharp 1×1 LEED pattern and no trace of impurity at the sensitivity of AES.

Gold was evaporated from a Knudsen-type cell with a graphite crucible. The evaporation rate was carefully measured in absolute value, using a quartz microbalance calibrated by interferometric measurements. The stability and reproducibility of the Au flux were regularly controlled with the quartz balance which could replace the sample at its very position. Depending on the coverage range under study from less than 10^{-3} to more than 10^2 ML, four deposition rates were used, roughly 10^{-4} , 10^{-3} , 10^{-2} and $10^{-1} \text{ ML s}^{-1}$ where 1 ML (monolayer) corresponds to $8.8 \times 10^{14} \text{ atoms cm}^{-2}$, that is the number of Ga and As atoms in the (110) atomic plane. The sticking coefficient of Au is taken as one since no trace of the metal was ever found outside the area in direct view from the crucible. After cleavage, the sample remained always close to room temperature, even during metal depositions : this point is particularly important in the case of Au on GaAs (110) since a short rise in temperature to less than $100 \text{ }^\circ\text{C}$ induces irreversible changes at the interface, as we shall report elsewhere.

The photoemission yield *versus* photon energy between 4 and 6.5 eV was recorded after cleavage and after each Au deposit. LEED, AES and ELS

measurements were performed at some intermediate coverages but usually neither after cleavage nor after coverages below 10^{-1} ML. During the evaporations as well as during the measurements, the total pressure in the vessel remained always below 4×10^{-10} torr. The contamination by residual gases was carefully checked and the time schedule of a whole experiment was chosen to make the effect of contamination negligible.

3. Results.

3.1 LOW ENERGY ELECTRON DIFFRACTION (LEED). — As Au is deposited, the sharp 1×1 diagram of the clean GaAs (110) surface fades away slowly. It becomes barely visible at 6 ML coverage in a high background, then disappears. Contrary to the case of Ag [1] no evidence of an epitaxial growth of Au metal is found from LEED. The behaviour of Au resembles more that of Cu [2]; with the latter element, however, the GaAs (1×1) diagram fades away more slowly.

3.2 AUGER ELECTRON SPECTROSCOPY (AES). — The typical variations of the low energy Auger peak intensities of Ga and As relative to their respective after-cleavage values are shown in figure 1 as a function of Au coverage θ , together with the evolution of the low energy Au Auger peak intensity referred to its value at the highest Au coverage. The curves which are drawn in figure 1 are variation laws of the form $\exp(-\theta/\theta_e)$ for curve A with $\theta_e = 2.3 \text{ ML}$, and $1 - \exp(-\theta/\theta_e)$ for curve B with $\theta_e = 4.6 \text{ ML}$. Comparison with curve A shows that the shape of the Ga and As decrease is compatible with the formation of a uniform absorbing layer. On the other hand, the shape of the Au increase is compatible with such a

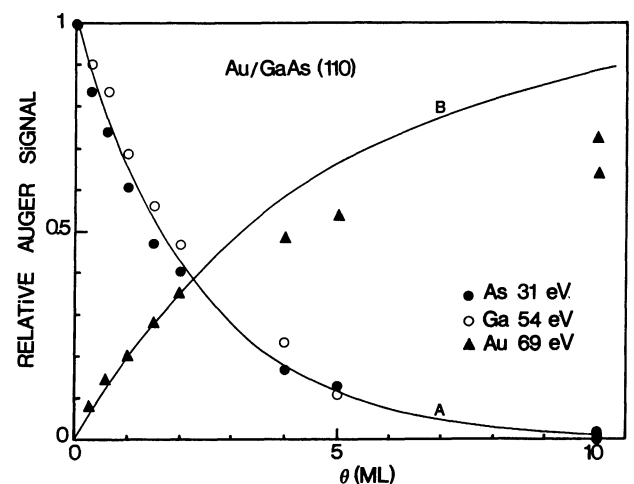


Fig. 1. — Peak to peak Auger signals of As at 31 eV and Ga at 54 eV relative to their respective after-cleavage values, and of Au at 69 eV relative to its value after a 50 ML deposit, plotted against Au coverage on a clean cleaved GaAs (110) surface. Primary beam energy : 2 keV. Curves A and B are exponential variation laws discussed in the text.

growth process up to about 2 ML as can be seen from comparison with curve B, but the rate of increase of the Au Auger peak becomes smaller at larger coverages. Moreover, one should notice that below about 3 ML coverage, the relative intensity of Ga is slightly but systematically higher than the relative intensity of As (this is also clear in figure 6 where more Auger data at low coverages are displayed).

Besides the data reported in figure 1, complementary observations have been made. Firstly, the saturation of the 69 eV Au peak occurs beyond a few tens of ML. Secondly, the behaviours of the « high energy » Auger peaks, that is 1 228 eV As and 1 082 eV Ga on the one hand, 239 eV Au and 2 025 eV Au on the other hand, which probe a deeper layer since the electron escape depth is larger than for the « low energy » peaks, are similar to those shown at low energy : roughly exponential decrease of both Ga and As signals, monotonous increase of the Au Auger signals. The change in growth process at about 2 ML which is observed with Au (69 eV) is also observable with all high energy Auger peaks. Thirdly, the Au Auger peaks at 69 and 239 eV display an energy shift beyond 10 ML coverage towards lower energies which reaches 3 eV. No such shift is observed on the 2 025 eV peak. It should be noticed that the 5d level of Au is involved in the transitions leading to the Auger peaks at 69 and 239 eV while it is not in the 2 025 eV peak.

3.3 ELECTRON LOSS SPECTROSCOPY (ELS). — Figure 2 shows the doubly differentiated electron energy loss spectra of a cleaved GaAs (110) surface covered with Au layers of different thicknesses, from 0 to 200 ML. The spectrum at zero coverage is well known and fairly well understood [14-16]; the energies of the main peaks are indicated in the figure. For Au coverages up to 10 ML the main loss peaks of GaAs remain well pronounced and the plasmon peaks (surface at 10.4 and bulk at 16.3 eV) are still observable at 20 ML. The peak at 3.6 eV is seen to decrease up to 6 ML where it starts to be masked by a growing peak at 2.8 eV. The peak at 5.9 eV keeps an apparently constant amplitude independent of Au coverage. The two peaks at 2.8 and 5.9 eV are recognized as losses of Au metal [17]. The evolutions of the electron loss spectra upon Au coverage on GaAs (110) appear qualitatively similar to the case of Cu deposition [2] but they differ from the case of Ag deposition where significant changes are already observed at 2 ML coverage [16].

3.4 PHOTOEMISSION YIELD SPECTROSCOPY (PYS). — As in our previous studies of similar systems [1, 2, 12], the results of PYS measurements are presented in terms of effective densities of filled states $N_f^*(E)$ which are deduced from the yield spectra by taking the first derivative with respect to photon energy. In figure 3 a selected set of typical densities of states obtained after increasing Au coverages are shown in the case of a low doped n-type GaAs substrate. Both the semilogarithmic and linear plots are presented

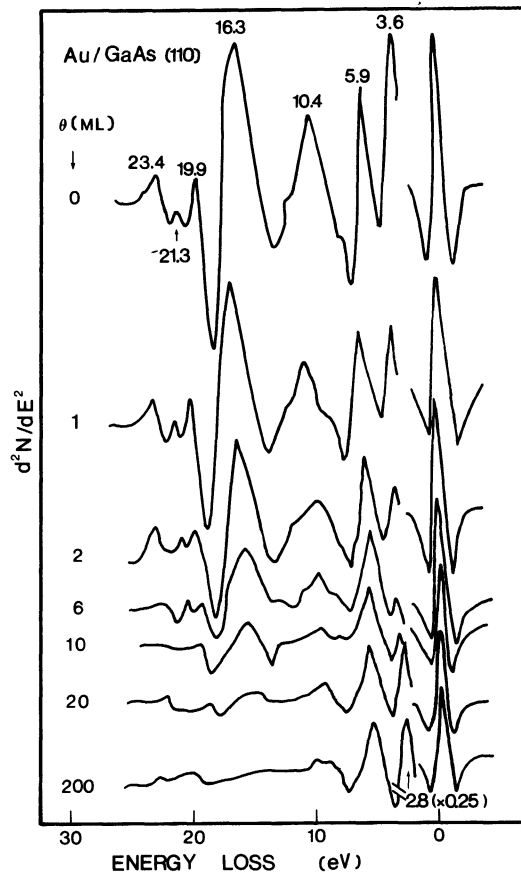


Fig. 2. — Doubly differentiated electron loss spectra of a clean cleaved GaAs (110) surface covered with Au layers of different thicknesses. Primary beam energy : 120 eV.

to show more clearly the behaviour of the curves respectively at low and high photon energies in the studied range. Complementary data are also obtained for p-type substrates.

The work function ϕ can be deduced by fitting the Fermi function at the low energy threshold of the yield curves. Comparison of the measured densities of filled states on both n- and p-type substrates leads to a good estimate (within 0.05 eV) of the ionization energy Φ of the semiconductor at least in the low coverage range, that is where the surface can be considered as statistically uniform. These determinations have been discussed in earlier publications [1, 12] and in the present case, the results are summarized in figure 4.

On n-type substrates, ϕ_n switches at Au coverages in the 10^{-3} ML range from its value $\phi_{n0} \approx 4.05$ eV for a well cleaved clean surface to a value of about 4.9 eV, that is a band bending of 0.85 eV. It increases again to reach a value close to 5.05 eV that is a band bending roughly equal to 1 eV, in the 1 ML coverage range. It agrees with the Schottky barrier value of 0.98 eV found on Au-GaAs (110) diodes [13]. On p-type samples, ϕ_p changes continuously upon increasing Au coverage, mostly below 0.1 ML. At high Au

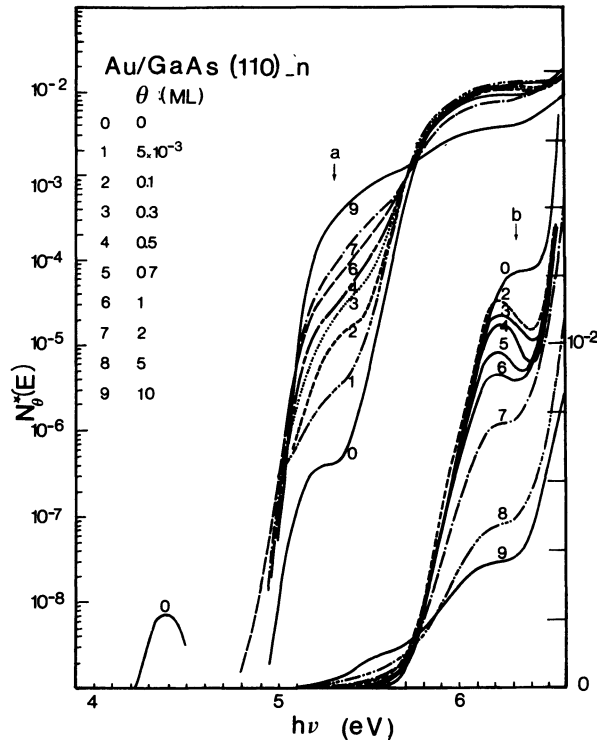


Fig. 3. — First derivative of the photoemission yield spectra with respect to photon energy, called « effective density of filled states » $N_{\theta}^*(E)$, plotted in arbitrary units against photon energy for an n-type GaAs sample at different Au coverages. Both semilogarithmic (left-hand scale and curves) and linear (right-hand scale and curves) plots of the same set of data are presented.

coverage, the work function φ_{Au} is found to be 5.1 ± 0.05 eV.

If the changes of the density of filled states are now considered, there is an obvious increase in the low energy region, labelled a in figure 3, which corresponds to the gap of bulk GaAs. Besides this increase, there is an obvious decrease, labelled b in figure 3, which corresponds to the upper part of the valence band of bulk GaAs. In order to better visualize this decrease, the variation of the density of filled states at θ coverage, with respect to zero coverage, simply obtained by taking the difference $N_{\theta}^*(E) - N_0^*(E)$, is shown in figure 5. It is limited to the low coverage range, here about 2 ML, where the ionization energy can be safely considered as a constant. It can be seen that, besides the main decrease about 1 eV below the valence band edge E_{vs} , other features are observed closer to E_{vs} . This will be discussed in the following section. The density of states decrease reported here has also been observed in the case of Cu [2] while nothing of the sort is seen in the case of Ag [1].

4. Discussion.

In order to get a comprehensive picture of the Au-GaAs (110) interaction from the set of experimental results which has been presented, the approach will be the following. Considering the LEED data and

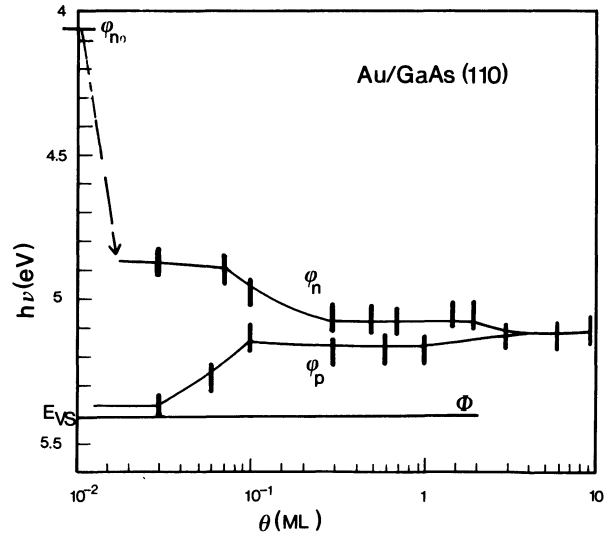


Fig. 4. — Variations of the work function φ_n (n-type substrate), φ_p (p-type substrate) and ionization energy Φ of GaAs (110) versus Au coverage as deduced from $N_{\theta}^*(E)$ curves such as in figure 3.

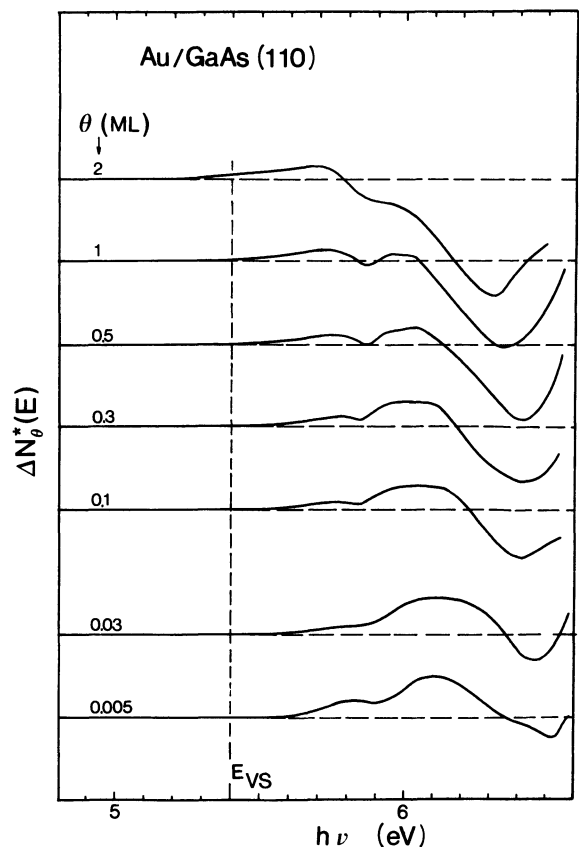


Fig. 5. — Variations of the effective density of filled states on GaAs (110), $\Delta N_{\theta}^*(E) = N_{\theta}^*(E) - N_0^*(E)$, at low Au coverages θ . E_{vs} marks the valence band edge of GaAs at the surface.

PYS results at low Au coverages, a first picture will be proposed. It will then be refined through AES data at low coverages and checked by PYS and ELS results at higher Au coverages.

However, before entering this discussion, the behaviour of the system at very low coverage will be briefly mentioned : an Au coverage below 10^{-2} ML, as any defect or impurity, induces, if not already there, an irreversible band bending on n-type GaAs (110) samples. The nature of the defect or of the impurity has only a weak influence on the pinning position of the Fermi level at the surface, usually around mid-gap or slightly closer to the valence band edge. Several ideas, which are not mutually exclusive, have been proposed to explain this general behaviour of the cleaved GaAs surface based on : i) the generation of GaAs defects in the surface region, assisted by the energy released in the adsorption reaction [18]; ii) the generation of specific bonds at the forming interface [19]; iii) the change of surface reconstruction around surface defects or impurity [1]. Since the deposition of gold does not bring new information on this question, no further comment will be developed here. It should only be noted that the changes observed in the density of states which are presented in the two lowest curves in figure 5 are related to the same effects and the discussion would be the same as in the case of Ag [1].

The observations which are specific of Au deposition, as well as of Cu deposition [2], are (i) a second increase of the work function ϕ_n beyond 0.1 ML (Fig. 4) and (ii) a growing depletion of the density of states about 1 eV below the valence band edge (Fig. 5). This behaviour can only be explained, as in the case of Cu [2], by a strong change in the bonding between As and Ga atoms in the very first layers of the system. In particular, it is not compatible with a layer by layer growth of a metal film : the attenuation of the valence states of GaAs through such a film would be much less than the one shown in figure 3 at 1 or 2 ML coverage. However, since the LEED diagram remains almost unchanged upon more than one monolayer deposition, one is led to consider the formation of a mixed two-dimensional layer which keeps the GaAs lattice periodicity, at least along the surface. Then, once this mixed layer is completed, and since the LEED diagram still persists, the formation of Au islands must be considered.

One has to check if this two-step process is compatible with the other data, in particular the AES results which, as we have seen, also point out to two growth processes. In figure 6, all the data points of the low energy Auger peaks are displayed up to 2 ML Au coverage together with curves calculated within a model which is consistent with the first step of the adsorption process described above. For simplicity, the model assumes the superposition, over pure GaAs, of an homogeneous and uniform two-dimensional alloy associating Ga, As and Au atoms with constant proportions and at a constant atomic density taken as that of GaAs. In a continuum approximation, the Auger signal for a substrate species (Ga

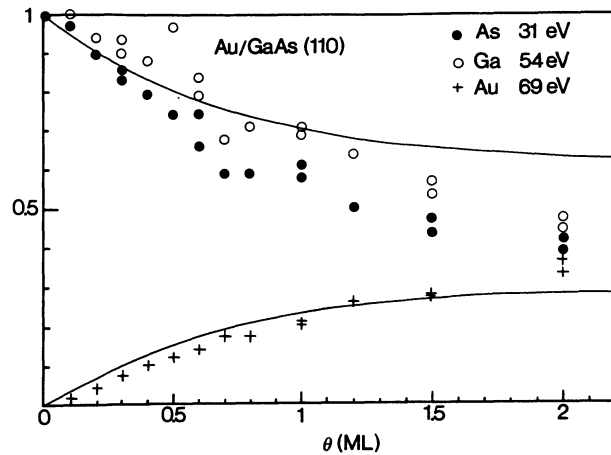


Fig. 6. — Relative peak to peak Auger data of As (31 eV), Ga (54 eV) and Au (69 eV), at low Au coverages and corresponding curves calculated following the model as explained in the text. Primary beam energy : 2 keV.

for instance) is referenced to the signal from pure GaAs, and the Au Auger signal to that from a thick layer of pure gold so that :

$$\frac{I_{\text{Ga}}(d)}{I_{\text{Ga}}(0)} = \exp(-\gamma_{\text{Ga}} d) + 2 \eta_{\text{Ga}} [1 - \exp(-\gamma_{\text{Ga}} d)]$$

and

$$\frac{I_{\text{Au}}(d)}{I_{\text{Au}}(\infty)} = \eta_{\text{Au}} \frac{N_{\text{GaAs}}}{N_{\text{Au}}} [1 - \exp(-\gamma_{\text{Au}} d)]$$

where d is the thickness of the alloy ; N_{GaAs} and N_{Au} are the atomic densities of GaAs and Au respectively ; η_i is the atomic proportion of species i in the alloy ($\sum_i \eta_i = 1$) ; $\gamma_i = \frac{1}{\lambda_p} + \frac{1}{\cos \alpha \lambda_i}$ with λ_p being the primary electron penetration depth, and λ_i the escape depth of the considered Auger electrons of species i emerging at angle $\alpha = 30^\circ$ from the normal to the surface into the CMA detector. The thickness d , expressed in \AA , is related to the coverage θ , expressed in ML, by the relation : $d = 2 \theta / \eta_{\text{Au}}$. It is further assumed that, in the alloy, $\eta_{\text{Ga}} = \eta_{\text{As}}$ so that we are left with one parameter : η_{Au} . For the low energy Auger peaks (Fig. 6), we have used $\lambda_p = 22 \text{\AA}$ and $\lambda_i = 5 \text{\AA}$ for the three peaks considered. The best fit of the Au (69 eV) Auger peak variation (Fig. 6) is found for an alloyed layer containing 40 % of Au, 30 % of Ga and 30 % of As atoms and which would be complete at about 2 ML coverage. The model, with appropriate penetration and escape depths, and with the above alloy composition accounts also for the high energy Auger peak data. The intensity variation of the As (31 eV) and Ga (54 eV) Auger peaks calculated for $\eta_{\text{Au}} = 0.4$ and drawn in figure 6 remains smaller than the actual variations of the data points. One reason can be invoked to explain this discrepancy. During the alloying process, that is

up to 2 ML, Au atoms transform the local bonds which may justify a decrease of the Auger transition probabilities of both As and Ga. The alloy which, in our model, forms a layer 8-10 Å thick, would correspond to the association of 4 Au atoms with about 3 GaAs substrate layers per GaAs surface unit cell. This is compatible with the conservation of the LEED diagram. It is clear in figure 6 that the Au signal is somewhat delayed at the beginning, showing that the Au atoms bury below the first GaAs atomic plane : it agrees with the strong perturbation of the GaAs bonds deduced from photoemission. Beyond 2 ML, the Au Auger signals increase at much lower rates than expected from a uniform metal layer; this observation is consistent with the proposed formation of Au islands during the second step of the growth process.

Some other remarks can still improve the picture developed above. Firstly, the Ga Auger signal at 54 eV remains slightly higher than the As one at 31 eV as soon as the Au coverage becomes significant (at about 0.1 ML and up); assuming the electron escape depths are the same at these two energies, this observation can be understood under the condition that the burying process of the first two monolayers of Au atoms occurs together with some displacements of the As and Ga atoms of the first substrate layers where, on the overall, the Ga atoms go outwards and the As inwards. Secondly, the chemical shift observed in the Auger transitions involving the Au 5d band can be explained as follows. The initial positions of the peaks are characteristic of Au forming an alloy with GaAs while the shifted Auger peaks, which correspond to a deeper 5d band, are associated with Au metal. The initial positions persist up to several ML : it can be understood only if Au islands form after the alloyed layer is completed, leaving then the Auger peaks of alloyed Au well observable beyond the limit which would be reached in the case of a uniform deposition. The evolution of the ELS spectra, where the decrease of the substrate signal upon Au deposition appears also delayed, is explained through the same argument. The islands which start to form beyond 2 ML do not cover a fixed portion of the surface but should be considered as

spreading more and more over the surface most of which is covered up between 6 and 10 ML Au coverage. Finally, considering the density of states at the Fermi level on the one hand (semilog plot in Fig. 3) and the work function behaviour in figure 4 on the other hand, the metallic character of the Au deposit shows up only beyond 2 ML. It further demonstrates the burial of the first two monolayers of Au : in the case of Ag, the metallic character shows up just beyond 1 ML [1] while in the case of Cu, where the initial formation of an alloyed layer has been demonstrated [2], the appearance of the metallic character is delayed, as in the case of Au.

In summary, the set of results obtained through different techniques of surface physics enables to propose a model of the Au-GaAs (110) interface formation at room temperature which is the following. First, 2 ML of Au atoms (4 atoms per GaAs surface unit mesh) intercalate between the first three atomic planes of the substrate. They form an ordered alloyed layer about 8 Å thick which keeps the surface periodicity of the substrate. Then, metallic Au islands start to form which progressively cover the whole surface. Full coverage is reached between 6 and 10 ML of Au but the layer is not yet uniform. It would not be reasonable, at the present stage, to try to be more specific about the actual positions of the As, Ga and Au atoms in the « two-dimensional » compound ; in that respect, an information giving first neighbours distances around the different atoms, by SEXAFS (surface X-ray absorption fine structure) for example, would be mostly welcome.

It remains to conclude the comparison between the three metals, i.e. Cu, Ag, Au, the interaction of which with GaAs (110) has been studied with the same methods, under the same conditions. It appears that whereas Ag forms an interface of practically perfect abruptness, both Cu and Au give an intermediate two-dimensional alloyed layer. It cannot be related to the respective atom sizes and the important parameter seems to be the position of the d-band with respect to the Fermi level : it is closer for both Cu and Au than for Ag. The study of the interface formation with Cu or Au on a cooled substrate should be of strong interest in that respect.

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