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Interface positions measured at the lattice constant scale by Auger analysis on chemical bevels

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Résumé. — Les résultats expérimentaux relatifs à des profils Auger obtenus sur des biseaux chimiques d'hétérostructures épitaxiées ($\text{InP}/\text{Ga}_x\text{In}_{1-x}\text{As}/\text{InP}$) sont reportés. L'analyse théorique de ces résultats montre que la position des interfaces et la largeur des puits quantiques peut être déterminée avec une précision de $\pm$ une maille cristalline (limite ultime). L'évolution du gradient de concentration est aussi suggérée. Des puits quantiques de $\sim 3,5$ nm d'épaisseur (6 mailles cristallines) sont bien résolus.

Abstract. — Experimental results of Auger profiles obtained on chemical bevels of hetero-epitaxial structures ($\text{InP}/\text{Ga}_x\text{In}_{1-x}\text{As}/\text{InP}$) are reported. The theoretical analysis of these results shows that the positions of the interfaces and widths of the quantum wells can be determined with an accuracy of $\pm$ one lattice spacing (ultimate limit). The evolution of the concentration gradient is also suggested. Quantum wells of ~ 3.5 nm thick (6 lattice spacings) are well resolved.

1. Introduction.

In the past few years, many intense efforts have been focused on the elemental characterization of solid-solid interfaces [1-5]. These efforts have been stimulated by the needs of the semiconductor industry where a precise knowledge of the interfaces positions and their concentration gradient are of tremendous interest. The most popular techniques for depth profiling use ion bombardment associated with secondary ion mass spectroscopy (SIMS) or Auger electron spectroscopy [1-7]. Unfortunately, due to statistics of sputtering, cascade mixing and instrumental effects, the interface resolution Δz reaches the range 1.5 to 15 nm at a profile depth z of 100 nm. Δz increases with z [1], thus, deeply buried quantum wells such as those obtained by the epitaxial growth of III-V compound semiconductor heterojunctions (for semiconductor superlattices and semiconducting lasers [8]) cannot be well resolved.

An interesting alternative to conventional depth profiling (combining sputter-etch removal and AES) is the chemical bevelling, where a thin film analysis by in-depth profiling is obtained as a result of a surface analysis of a bevel resulting from a chemical etching [4, 5, 9, 10].

The first purpose of this paper is to explain the Auger profiles obtained on such a bevel. For this reason, in the first part, the Gallon model [11] is used to simulate mathematically these profiles. In the second part the experimental results obtained by chemical bevelling on epitaxial heterojunctions are analysed with the help of the theory.

2. Models.

2.1 MODEL FOR THE CHEMICAL BEVEL. — In figure 1, a schematic diagram of a bevel is given at the atomic scale for the interface between an element A and an element B.

In such an ideal situation, the lattice spacing « a » between two equidistant atomic layers gives rise to terraces, each step being « d » wide ($d = Ma$) and « a » in height, where M is the linear magnification of the bevelling. Linear magnification of several 10^3 can be easily obtained [9-10]; this means that « d » is in the micron range for a lattice constant of 0.5 nm. With a smooth grinding, we can expect that the surface looks like the surface obtained by crystal growth on crystalline terraces (see Fig. 1 : dotted lines). When the incident electron beam is just at the middle of a step, the Auger signal comes from a very large area (large with respect to the atomic scale) and one can suppose that deviations from the idealized situation are negligible.

2.2 MODEL FOR THE AUGER EMISSION. — We refer to the Auger intensities $I_0, I_1, I_2, \dots, I_j$ measured when the incident electron beam is just at the middle $M_0, M_1, M_2, \dots, M_j$ of the steps $n^\circ : 0, 1, 2, \dots, j$ (Fig. 1). We assume that the Auger signal comes from an area which doesn't overlap with the adjacent terraces ($j - 1$ and $j + 1$) for the j th terrace (point M_j). This means that the incident electron beam width d_0 and the backscattered electron beam width d_R are : $d_0, d_R < d$.

When the concentration of element A in the substrate is a constant ($C_A = C_0$ for $j \leq 0$) and presents a gradient $j > 0$: $C_A = C_1$ for $j = 1$, $C_A = C_2$ for $j = 2$; etc., the Auger signal obtained on the j th terrace is :

$$I_j = K \left[\sum_{n=1}^j C_n k^{j-n} + C_0 \sum_{n=0}^{-\infty} k^{j-n} \right], \quad (1)$$

where

$$k = e^{-\alpha} = \exp - a/\lambda \cos \theta. \quad (2)$$

Fig. 1. — Schematic diagram of the bevel. Full line : ideal situation ; dotted line : more realistic situation where the steps have widths d which can fluctuate around their mean value d_0 , where $d_0 = Ma$, « a » is the step height (0.5 nm), and M is of the order of several 10^3 . Concentration of the element A, constant in the substrate ($j \leq 0$), varies ($j > 0$) for non abrupt interfaces. $C_j = 0$ ($j > 0$) for an abrupt interface. The atomic concentration of the element B is a complement to the unity of the atomic concentration of the element A.

Expression (1) corresponds to an application of the Gallon model [11] where the first sum represents the contribution of the layers above the substrate, the second sum the contribution of the layers composing the substrate. $\lambda \cos \theta$ is the escape depth of Auger electrons of interest and K an instrumental factor which disappears when Auger intensities are normalized ($y_j = I_j/I_0$) to the intensity of a bulk sample of uniform and known concentration, i.e. $I_0 = KC_0/(1 - k)$. When an abrupt interface ($C_{n>0} = 0$) is analysed by scanning the incident electron beam along the Ox axis (Fig. 1), the reduced Auger intensity is expected to follow the law :

$$y(x) = \exp\left(-\frac{x}{M\lambda \cos \theta}\right) = \exp\left(-\frac{z}{\lambda \cos \theta}\right). \quad (3)$$

In fact, the above relation holds only for the discrete points M_0 ($x_0 = 0$); M_1 ($x_1 = d = Ma$); M_j ($x_j = jd = jMa$). For a very localized Auger response function ($d_0, d_R \sim 0$), the curve is expected to be step-like with equal step height when relative intensities are reported in a log scale

$$\text{Log} \frac{y(x_{j+1})}{y(x_j)} = -\frac{a}{\lambda \cos \theta}.$$

Nevertheless, for increasing values of the Auger response function (d_0, d_R close to d), the step like curve is smoothed out and approaches the variation given by expression (3). When elements A and B are interchanged ($C_0 = 0$; $C_{n>0} = 1$), the reduced Auger intensity is expected to follow :

$$y(x) = 1 - \exp\left(-\frac{x}{M\lambda \cos \theta}\right). \quad (4)$$

When a quantum well (of thickness t in depth) consists of two abrupt interfaces of element A, the Auger profile obeys the sum of expressions (3) and (4), taking into account the translation $OO' = Mt$ of the origin of the second curve. When the quantum well is composed by an element A in between two abrupt interfaces of B, the expected profile is a complement to unity to the above described situation.

Conclusions for these points are :

— Expressions (3) and (4) shows that a log-scale is a natural scale for intensities : $\text{Log } I/I_0$ when the element of interest composes the substrate (case α), $\text{Log } (1 - I/I_0)$ when the element of interest composes the uppermost layers (case β).

— For an abrupt interface the slope of the straight line (log scale) allows the escape depth of Auger electrons to be deduced.

— The exact interface position does not correspond to relative intensity of about 50 %. It corresponds either to the beginning of the decrease from unity (case α) or the beginning of the increase from zero (case β).

— The quantum well profiles are asymmetric.

— There is no change in the evaluation of the widths if the height of the terraces corresponds to the atomic spacing ($a/2$ in cubic zinc sulfide structure) instead of the lattice spacing « a ».

3. Experimental results.

The experiment was developed on an $\text{InP}/\text{Ga}_{0.47}\text{In}_{0.53}\text{As}$ double heterostructure obtained by epitaxy (low pressure MOCVD). On the same substrate (InP) four successive quantum wells ($\text{Ga}_{0.47}\text{In}_{0.53}\text{As}$) were built with a thickness ratio expected to decrease like 4:3:2:1. Details on chemical bevelling are given in previous works [10, 14]. Measured with a Talysurf, the mean amplification coefficient was found to be $M = 1430$. The setting conditions of the Auger electron microscope (PHI model 590) were the following : $I_0 \sim 40$ nA, $V_0 = 5$ kV, d_0 (spot size) < 0.5 μm .

The profiles shown on the top of figures 2 and 3 have been obtained with phosphorus LMM Auger transition ($E_K \sim 121$ eV). Figure 2 shows a detailed profile of the second quantum well. It is asymmetric. Experimental points reported on the bottom by using a log scale are on straight lines. The extrapolation of these lines allows the positions of the interfaces to be determined.

Fig. 2. — Top : Auger profile obtained on the second quantum well (from the substrate, see Fig. 3) by using phosphorus LMM Auger line ; InP/ $Ga_{0.47}In_{0.53}As$ /InP double heterostructure. Bottom : the measured intensities reported on a log scale. ($\bullet$) are related to a 100 % intensity which corresponds to the maximum intensity of the signal (right hand side on the top). ($+$) are related to a 100 % intensity which corresponds to the maximum intensity of the signal in the left hand side (see top curve). The extrapolation procedure allows determination of the positions of the interfaces on the log scale and on the experimental curve (top). Note that the fluctuations of the experimental points around the straight line are very weak even near the surface. In the middle the geometry of the bevel is suggested.

The width of the quantum well is estimated to be 5.3 nm, a thickness which nearly corresponds to 9 lattice spacings ($a = 0.58687$ nm). The interface on the substrate side (right hand side in Fig. 2) is very abrupt. The escape depth of Auger electrons ($\lambda \cos \theta = 0.84$ nm) is reasonably in agreement with the tabulated values $0.6 < \lambda < 0.9$ nm [12, 13]. On this interface, the concentration gradient concerns only one lattice spacing (at maximum). It is an ultimate limit. For the interface on the other side, it is not necessary to subtract the remaining contribution (to the Auger signal) of phosphorus atoms composing the substrate because of the large thickness of the quantum well (the minimum Auger signal inside the well being close to zero). The apparent escape depth measured on the other interface is larger than the previous one : $\lambda \cos \theta \sim 1.9$ nm.

Fig. 3. — Top : wide scan spectrum of four quantum wells (of different thicknesses) obtained on the same InP substrate. Profile obtained by using phosphorus LMM Auger line. Bottom : the measured intensities are reported on a log scale in order to determine the positions of the interfaces and widths of quantum wells. The experimental values are : 9.44 nm ; 5.3 nm ; 4.13 nm ; 3.55 nm. Middle : the geometry of the full bevel is suggested (in this figure, the broken lines correspond to the extrapolation procedure used to take into account the remaining contribution of atoms being on the substrate side).

This can be due to the following 3 experimental points :

- i) local modification of the amplification coefficient M ,
- ii) superficial layer mixing created before analysis by the ion cleaning bombardment (Ar^+ , 5 keV, 2 min),
- iii) the presence of residual oxide having different thickness and chemical composition on InP and InGaAs.

With the help of only a single profile, it is difficult to determine the main cause. However, the profiles obtained for additional elements (Ga, In, As, O) will allow us to elucidate this effect. If these three points can be neglected, the escape depth value can also be explained by a concentration gradient at this interface. With the help of expression (1) the best fit between theory and experiment has been obtained by choosing the following atomic concentration gradient : $C_1 = 0.25 \pm 0.1$; $C_2 = 0.5 \pm 0.1$; $C_3 = 0.75 \pm 0.1$. It concerns only 3 or 4 lattice spacings. This has to be verified by other experiments, which are now in progress.

On the top of figure 3 a wide scan of all the bevel is shown. The procedure described above has been used. For the interface on the surface side of the thinnest wells (where the minimum Auger signal is not close to zero), the remaining contribution of atoms on the other side of the well is deduced by an extrapolation of the log curves related with the interfaces on the substrate side (broken line in Fig. 3). The intensities of interest are measured between the corresponding extrapolation line (broken lines) and the registered signal line. The following widths of the quantum wells have been estimated : 9.44 nm ; 5.3 nm ; 4.13 nm ; 3.55 nm. They correspond to 16, 9, 7 and 6 lattice spacings, respectively, the accuracy being estimated to ± 1 lattice spacing. One can observe that a quantum well of about 3.5 nm thick is well resolved.

4. Discussion.

The observed asymmetrical profile can be explained by our theoretical model, elaborated from an ideal surface shown on figure 1. From this fact we can deduce that the chemical etching induces damages restricted to the first top layers. This chemical etching can also induce local modification of the magnification coefficient M at the edges of the well. At the interfaces, we can notice for the logarithmic curves (Fig. 2), a departure from a linear behaviour corresponding to one or two monolayers.

Due to the limited number of experimental points in figure 3, which is a general view of the 4 wells, we cannot perform data analysis as for figure 2. The lack of experimental points in figure 3 explains the large scattering of the slope of the different straight lines.

5. Conclusion.

Solid-solid interfaces can be characterized by the accurate knowledge of the interface position, their chemical composition and their concentration gradient. For the interface position, the ultimate limit corresponds to one lattice spacing. The experimental results reported here show that this limit can be reached for heteroepitaxial structures by chemical bevelling and Auger profiling. The same methods hold also for an estimate of the concentration gradient determination. The first results have been reported here, and they need to be confirmed by additional experiments which are now in progress.

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