

Detection of surface states anisotropies at $GaAs(001)(2 \times 4)$ decapped surfaces

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The *surface or bulk* origin of the optical anisotropies detected by reflectance anisotropy spectroscopy (RAS) at $GaAs(001)(2 \times 4)$ surfaces has been extensively investigated in the last years and a quite general agreement has been reached that the dominating character would be bulk-like. Nevertheless, a very recent paper [F. Arciprete et al., Phys. Rev. B **69**, 081308(R) (2004)] has again issued the presence of *surface states* contributions in optical anisotropies, revealing a structure at 2.5 eV due to surface states, in addition to the well known features around 2.9 eV and 4.5 eV related to the bulk critical points E_1 and E_0' . We have carried out a new experiment to prove this conclusion by following the changes in the optical anisotropy of a $GaAs(001)(2 \times 4)$ surface in the range 2.0-5.0 eV induced by GaAs(001) is known to be not reactive. Due to its surfactant effect, codeposition. The interface GaAs(001) is known to the Ag overlayer. We show that at the early stages of deposition (nominally at 0.25 monolayer) an evident modification of the RAS spectrum is detected at 2.5 eV, well below the photon energy (2.9 eV) where bulk-like anisotropies appear. We relate this modification to the disappearance of surface states characteristic of the (2 × 4) reconstruction, in excellent agreement with previous conclusions and experiments.

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1 Introduction

The detection of surface states by optical techniques has always dealt with the low specific sensitivity of photons to the surface layer of solids, and particular experimental methods have been developed to extract the surface contribution from the overwhelming bulk signal [1, 2]. For example, in surface differential reflectance (SDR) the change of reflected-light intensity induced by contamination or chemisorption of foreign atoms (usually oxygen or hydrogen) allows to isolate the signal due to transitions involving surface states. Oxygen molecules saturate dangling bonds removing the related states [2, 3] localized in the surface layer: in this way, surface states of $Ge(111)(2 \times 1)$ [4] and $Si(111)(2 \times 1)$ [5] have been clearly identified inside the bulk energy gap of the solid.

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Also the different symmetry properties of surfaces with respect to bulk are useful to enhance the surface contribution: on a (001) surface of centrosymmetric materials, measuring the reflectance signal at nearly normal incidence for two independent, perpendicular polarizations (namely along $[\bar{1}10]$ and [110] directions), since the bulk is expected to be isotropic it is straightforward to connect the anisotropy signal to the surface, having different symmetry properties due to new bonds formed upon reconstruction. On this assumption reflectance anisotropy spectroscopy (RAS) has been developed [6, 7], since its early applications to study the optical properties of semiconductor surfaces in Ultra-High-Vacuum (UHV) [8].

If a surface contribution is detected at photon energies where the bulk is not absorbing (such as for $Si(111)(2 \times 1)$ at 0.45 eV and $Ge(111)(2 \times 1)$ at 0.5 eV), its interpretation as due to surface electronic states is evident, while more care is necessary where the absorption of the bulk material is not negligible. This analysis of optical spectra has regarded results obtained by SDR for the cleavage surfaces of GaAs and GaP in the range 1-4 eV [9, 10], and Ge and Si surfaces above 1.5 eV [11-13]. Bulk transitions were found to contribute to the features detected by SDR above the bulk energy gap, and independent arguments were developed to identify the surface contribution [11, 12]. In the same years, analogous analysis of experimental data obtained by RAS has not been performed, and the measured anisotropies, although often detected above the energy gap and in particular in coincidence with bulk critical points, were originally explained as mainly originated from surface states transitions just on the basis of an a priori statement: the optical anisotropy results from the symmetry lowering at the surface of cubic crystals like group IV, II–VI and III–V-semiconductors. A heuristic interpretation in terms of dimers present at the clean surface, originally used for GaAs, was then exported to other semiconductor surfaces

In contrast with this assumption, further experimental and theoretical research concluded that the optical anisotropy of (001) surfaces is not dominated by surface dimers, and that only by a detailed knowledge of the structure of the uppermost surface layers realistic calculations of the spectra could be obtained [14].

Two important contributions have motivated a deeper comprehension of RAS spectra: (i) the experience in studying the *bulk* anisotropies of oxidized samples (GaAs, GaP, Si) [6, 15], which has clearly evidenced that for centrosymmetric materials well defined structures could appear at bulk critical points because of the particular condition of the surface (due to roughness, oxidation, strain, etc.); (ii) from calculations, the understanding of the effect that the surface has on bulk states [16, 17]: bulk wave functions can slightly modify their symmetry characteristic in the neighborhood of the surface layer, so contributing to optical anisotropies.

It is now well established that in RAS spectra the *bulk-modified-by-the-surface* contribution has a fundamental part in the total anisotropy, and that the surface acts as a perturbation of the otherwise isotropic bulk wave functions. In the important example of clean $GaAs(001)(2 \times 4)$ surfaces, about ten years of long-lasting discussions and experiments have then conducted to interpret the measured anisotropic features in terms of bulk-like states, on the base of new and realistic calculations as well as of accurate, specific experimental findings.

However, true surface states should contribute to the optical properties of the surface, and the oscillator strength of the surface electrons should be exhausted. Very recently theoretical and experimental research has again reconsidered this issue [18–22]. Density functional theory within the local density approximation (DFT-LDA) calculations of the RAS signal have represented the spectrum by summing contributions involving surface states (SS) or bulk/surface and surface/bulk states (BS and SB) transitions [19, 20]. A peak at about 2.5 eV, preferentially polarized perpendicularly to the As-dimer axis, characterizes the SS term. At energies higher than 2.7 eV, the presence of bulk states is predominant. In a further experiment, RAS and HREELS (High Resolution Electron Energy Loss spectroscopy) have been applied in the same UHV chamber to Molecular Beam Epitaxy (MBE) grown GaAs(001) surfaces [21]. The different surface sensitivity of the two techniques has demonstrated that surface states of the clean As-rich phase contribute to the spectrum with a structure at 2.5 eV, mainly polarized along the [001] direction. Also sum rule criteria have confirmed the surface character of this structure [12].



Dealing with the problem of surface preparation, it could be reasonable that the weak surface term has been not evidenced in the past due to a poorer quality of GaAs(001) surfaces prepared by decapping [23]. Consequently, we have set up a new experiment to check if this surface contribution could be evidenced in RAS spectra of the (2×4) reconstructed phase also for decapped surfaces.

In our case, in order to quench the surface reconstruction we have evaporated silver onto the clean $GaAs(001)(2 \times 4)$ surface: (i) Ag is not reactive with GaAs(001), and the resulting interface is atomically sharp, without diffusion into the substrate [24–26]; (ii) Ag can be easily evaporated and is compatible with Ultra High Vacuum (UHV); (iii) the addition of Sb changes dramatically the Ag layer morphology from clustering to nearly epitaxial, as confirmed by Auger electron spectroscopy (AES) and atomic force microscopy (AFM) (not shown here). Sb acts as a surfactant, perhaps reducing the barriers experienced by metal atoms and limiting their diffusion [27].

We have simultaneously evaporated Sb and Ag on $GaAs(001)(2 \times 4)$ surfaces, following the evolution of the system in the coverage range 0-10 ML by RAS, Low Energy Electron Diffraction (LEED) and Auger. As expected, the growth results nearly epitaxial. The zero-line of the RAS is not observed to vary at the different coverage values, at variance from oxidation [28, 29], allowing a careful analysis of the modification of the lineshape induced by silver deposition. We observe that at 0.25 ML an evident modification of the spectrum happens around 2.5 eV. This modification is quenched at higher coverage. On the contrary, the peak at 2.9 eV is progressively modified, until it disappears at 2 ML. We ascribe this different behaviour to the character of the anisotropies: surface at 2.5 eV, bulk at 2.9 eV and at 4.5 eV. The energy position and sign of the 2.5 eV anisotropy fits the surface term fore-seen in LDA-DFT calculations [19, 20].

2 Experimental

The samples were epitaxial, n-doped ($n = 1 \times 10^{16} \, \text{cm}^{-3}$) GaAs layers, grown by MBE on GaAs(001) substrates and capped with an amorphous As protective layer. All the experiments were performed in a UHV chamber equipped with a four-grid reverse view LEED optic and Cylindrical Mirror Analyzer (CMA) for AES analysis. The RAS set-up (described elsewhere [30]) was placed in front of a low-strain quartz window of the UHV chamber, with light impinging at nearly normal incidence on the surface. The samples were decapped at 620 K and then annealed at about 670 K, yielding the (2 × 4) reconstruction.

The controlled contamination of the surface has been often used to check the surface character of electronic properties. In our case, however, the exposure to oxygen is not useful to detect slight modifications of the optical anisotropy: in fact, a shift of the zero-line observed during oxidation complicates any conclusion [28, 29]. Also hydrogen is not a good choice, because of its characteristic, aggressive behaviour with GaAs surfaces: it strongly alters the surface structure by removing As atoms from the surface layer [31].

Instead we have evaporated Ag, well known to produce a sharp interface on GaAs(001) surfaces [24]. Ag and Sb co-evaporation was performed using a calibrated source (Ag rate: 0.1 ML/s; Sb rate: 0.025 ML/s), monitored by a quartz-crystal microbalance. The quantities of Ag and Sb deposited at each stage were in the ratio 4:1, at variance with Ref. [27], where Ag and Sb were deposited on the surface in the ratio 10:1. Our complementary atomic force microscope (AFM) and AES measurements (not shown here) demonstrate that by this method deposition of a uniform layer is successfully achieved. During evaporation, the pressure in the chamber (normally in the 10^{-11} mbar range) was always better than 6×10^{-10} mbar. The substrate was held at room temperature during all measurements.

RAS spectra are commonly reported in terms of the complex quantity $\Delta r/r = \Delta \rho/\rho + i\Delta\theta$, where $r = \rho \exp{(i\theta)}$ is the complex reflectance. In our case, Δr is defined as $\Delta r = r_{1\overline{10}} - r_{110}$, where the subscript denotes the polarization of light with respect to directions on the sample surface. On clean GaAs(001)(2 × 4) surfaces, As-dimer bonds are aligned along [110]. In the following we will show only the real part of RAS signal [Re($\Delta r/r$)], although also the imaginary term has been always measured and recorded. All the spectra have been measured in the range 2–5 eV.

3 Results and discussion

The evolution of RAS spectra as a function of Ag coverage is shown in Fig. 1. The spectrum of the clean (2×4) phase exhibits the typical features already discussed elsewhere [14, 19–21]: a pronounced maximum at 2.9 eV (in the following cited as P1), a shoulder at about 3.3 eV (P2), plus another structure at about 4.5 eV (P3). The corresponding LEED pattern was a clear (2×4) with low background (not shown here). The Auger analysis showed no contaminants present on the surface layer after decapping.

A little deposit of Ag (0.2 ML of Ag plus 0.05 ML of Sb: total 0.25 ML) causes an attenuation of the spectrum, particularly evident between 2.9 and 4.5 eV. The LEED is still (2×4), but with a detectable background. The low energy part of the spectrum appears to be modified by deposition: this appears in Fig. 1, where the onset of the spectrum is pinned at 2.2 eV just after the first metal deposition.

At higher coverage (up to 1 ML) the anisotropy is reduced in the full spectral range. At 2 ML, the RAS spectrum has definitely changed, and traces of the previous (2×4) are not visible anymore at LEED inspection: in the RAS spectrum, only a strong, large feature is apparent at about 4 eV, plus a very faint one at 3 eV. No other variations are perceptible up to 10 ML, with the exception of the photon energy region around E'_0 .

In Fig. 2 we report the variation induced by coverage in the RAS spectrum of the clean surface: to highlight the anisotropy modification, the spectra measured at different deposition stages have been subtracted from that of the clean surface. At 2.5 eV, a negative dip (P0) is clearly visible, whose amplitude has already saturated at 0.25 ML coverage. At higher energies, i.e. above 2.5 eV, the difference mimicks the signal typically measured at clean $GaAs(001)(2 \times 4)$. This part of the spectrum shows a distinct dependence on coverage: changes are weak in the submonolayer range, but pronounced after several monolayers. Up to 1 ML, the spectrum changes mainly around 3 eV, the E_1 bulk critical point. At 2 ML the change is extensive, but restricted at the bulk critical point energies. At further coverage, anisotropy changes mainly at 4.5 eV, in correspondence with the E_0' critical point. These experimental results present an interesting agreement above 2.5 eV with previous data obtained during oxidation of decapped As-rich GaAs(001) surfaces [28]. However, there are also important differences (principally at 2.5 eV) depending upon the lower quality of the surface in Ref. [28] and the shift of the zero-line observed during oxygen chemisorption.

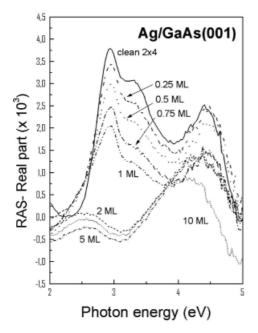


Fig. 1 Real part of RAS signal in the photon energy range 2.0–5.0 eV, measured at increasing coverage values onto a clean GaAs(001)2 × 4 surface: clean, 0.25 ML, 0.5 ML, 0.75 ML, 1 ML, 2 ML, 5 ML, 10 ML. The reported coverage corresponds to the total amount of material (silver and antimony) deposited onto the surface.



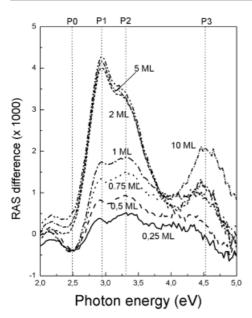


Fig. 2 Evolution with coverage of the real part of RAS signal reported in Fig. 1. All the spectra have been obtained substracting the spectrum for a certain coverage from the clean surface spectrum. Dotted lines marks the energy position of structures P0 (at 2.5 eV), P1 (at 2.9 eV), P2 (at 3.3 eV) and P3 (at 4.5 eV) discussed in the text.

It has been already noted in literature that: (i) P1 and P3 coincide with bulk critical points E_1 and E_0' , respectively [15, 17–19, 21]; (ii) the sign of P1 (positive, according to the assumed expression for RAS signal) is consistent with a higher reflectivity for light polarized along the direction of As-dimers at the surface; (iii) P1 is sensitive to contamination (oxygen [28], hydrogen [31], indium [32], germanium [33]), while P3 is weakly dependent upon surface conditions; (iv) the amplitude of P1 results proportional to the number of As-dimers present at the surface layer [34].

Co-deposition of Ag and Sb at room temperature results in the growth of a continuous 2D-overlayer in the case of Ag homoepitaxy [27]. The ratio Ag/Sb used in this experiment has been chosen to avoid clustering also for the heteroepitaxy of Ag on GaAs(001): the disappearance of the $GaAs(001)(2 \times 4)$ related anisotropy at a coverage between 1 and 2 ML confirms that the growth is layer-by-layer (see Fig. 1), in agreement with AES data.

The different behaviour of the spectral features P0–P3 is well accounted for if different origins are inferred for the states involved in transitions: surface states at 2.5 eV, bulk states modified-by-the-surface between 2.7 and 5 eV. The polarization of the 2.5 eV anisotropy (negative, that is along [110] direction) is a significant argument to support its attribution to the surface, being consistent with theoretical calculations of RAS spectra [19, 20], experimental RAS data on MBE grown surfaces [21] and HREELS data [35]. In the latter case, the anisotropy at 2.5 eV is huge (about 20% of the total signal) and by far dominating the whole spectrum, at variance with RAS results where the effect is extremely reduced (less than 1% of the total reflection at those energies). However, this finding is easily accounted for by the different sensitivity to bulk and surface of two techniques. Moreover, we have obtained our samples by decapping, while in Ref. [21] MBE freshly grown surfaces were studied, having a better ordering of the reconstruction. A strong argument in favour of the surface state nature of the 2.5 eV feature is its sensitivity to submonolayer deposition: this result evidences uniquely the different origin of the low energy and high energy features. Also mixed transitions (bulk/surface or surface/bulk), that theoretical calculations have shown to give significant contributions to the anisotropy signal, would be consistent with the experimental behaviour at 2.5 eV, without modifying our conclusions.

The spectral variation measured above 2.7 eV and observed to scale with coverage up to the saturation of the surface is connected to the bulk-modified-by-the-surface states, perturbed by the metal adsorption. This term has been attributed to bulk states perturbed at critical points $(E_1, E_1 + \Delta_1)$ by the anisotropic strain field produced by surface dimers [36]. When adsorption of metal atoms eliminates dimers, the intensity of the strain field weakens, and the related anisotropy reduces. A similar explanation can be

applied also for oxygen adsorbed at the surface. As expected this effect begins with the first stages of growth, when the true surface term has already reached saturation, and lasts up to the complete coverage. This result is consistent with conclusions of a noteworthy recent paper, where, by studying the optical anisotropy of (001)-GaAs surface quantum wells, the authors have shown that electronic states perturbed by the anisotropic surface strain field produce a RAS signal at 2.9 eV [22].

After the complete coverage of the substrate, besides a weak feature at 2.5 eV also visible on as-grown Sb-terminated GaAs(001) surfaces [37], the anisotropy mainly comes from the photon energy region close to the E'_0 bulk critical point. Near 4 eV (exactly at 3.9 eV) there is the characteristic plasmon loss of bulk silver [38]. Since HREELS measurements demonstrate the existence of a clear anisotropy of this plasmon on silver surfaces [39], we cannot exclude that the collective oscillation of the layer electrons is to some extent responsible of the wide structure centered at 4 eV, slightly dependent upon coverage. However, as in the difference spectra of Fig. 2 the main variation always appears at 4.3 eV (E'_0), we consider most probable that the origin again lies in the effect that the surface layer has on GaAs bulk wavefunctions.

In conclusion, by monitoring the modification that the RAS spectrum of a clean decapped $GaAs(001)(2\times4)$ surface undergoes during the growth of a nearly epitaxial layer of silver, we have evidenced a true surface state contribution to the optical anisotropy, in excellent agreement with previous experimental data taken on MBE grown surfaces. The quenching of surface states in the early stages of deposition produces a structure at 2.5 eV absorbing for light polarized along [110] direction. This finding definitely demonstrates that also on decapped surfaces the contribution of true surface states can be successfully measured.

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