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Ge growth on GaAs(001) surfaces studied by reflectance anisotropy spectroscopy

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Surface modifications induced by germanium deposition onto clean GaAs(001) substrates have been monitored by reflectance anisotropy spectroscopy (RAS) and low energy electron diffraction (LEED). The clean GaAs(001) surfaces onto which Ge has been evaporated were $c(4\times4)$, (2×4), and (4×1) reconstructed. Regardless of the initial surface reconstruction, after deposition of 0.5 monolayers of Ge and further annealing at 850 K, we have *always* observed a (1×2) LEED pattern and the same characteristic RAS spectrum. On the contrary, overlayer structures obtained at intermediate stages between the clean surface and this (1×2) phase depend upon the initial surface reconstruction. Modifying the (1×2) reconstructed surface by deposition of additional monolayers of Ge or exposure to atmosphere, we have separated the surface, interface, and bulk contributions to the RAS spectra. Finally, monitoring the characteristic linear-electro-optical feature appearing at E_1 and $E_1+\Delta_1$ bulk critical points, we discuss how its change in amplitude and sign could be connected to a variation of the substrate doping induced by annealing.

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I. INTRODUCTION

Among the very first heterojunctions studied,[1–9] Ge/GaAs interface has deserved attention for the good lattice-match existing between the substrate and the growing overlayer ($<=0.1\%$). Thus, Ge/GaAs has become a prototypical system to investigate the heterojunction evolution and the band-offset. Several papers were dedicated to the comprehension of the factors determining the interface evolution and its properties, investigating the role of either local microscopic features of the interface (morphology of the overlayer, surface orientation, electronic properties of the starting surface) or different preparation methodologies. For this reason, a few of the initial studies were mainly focused onto Molecular Beam Epitaxy (MBE) grown samples, providing different surface reconstructions of the starting GaAs(001) surface, useful to understand the role of the substrate stoichiometry. As a result, some well defined surface reconstructions were identified by Low-Energy-Electron-Diffraction (LEED) at the early stages of Ge/GaAs interface formation, but detailed models for the surface structure were not known.

More recently, the Ge/GaAs interface has been examined using spectroscopies highly sensitive to local morphology, as Scanning Tunnelling Microscopy (STM)[10,11] and Medium-Energy-Ion-Scattering (MEIS).[12] The results have confirmed that the early stages of the interface formation are characterized by different phases with definite LEED patterns (1×2 and 2×1), interpreted as built by Ge-As and Ge-Ga dimers, respectively. The existence of such reconstructions was related to the temperature of the substrate during and after Ge deposition.

A first attempt to explain the experimental results on the base of first principles pseudopotential calculations has been reported by Srivastava and Jenkins,[13] who have obtained atomic geometry, electronic states and bonding structures for both (1×2) and (2×1) superstructures, respectively built up with Ge-As dimers and Ge-Ga dimers.

Also optical techniques, in particular Reflectance Anisotropy Spectroscopy (RAS), have been applied to Ge/GaAs(001).[14,15] The possibility of having different dimers on the same GaAs substrate has suggested that this system could be an ideal case to investigate the origin of the anisotropic signal measured with RAS. Although the potential of RAS to study fundamental aspects of surfaces and interfaces has been clearly demonstrated,[16–20] important questions, mainly regarding its theoretical interpretations, are still under debate. In fact, while—at the beginning of its development—explanation of spectral features was normally limited to symmetry-based arguments, it has been more recently shown that experimental spectra can be well interpreted on the base of accurate calculations.[21–24] Thus precise correlations between theoretical results and optical features are obtained if the assumed atomic structure is realistic.

Following this approach, in our previous paper we have both experimentally and theoretically investigated the structure of the Ge/GaAs interface resulting after submonolayer deposition of Germanium onto the (2×4) reconstructed GaAs(001) surface and subsequent annealing.[15] We have obtained a well ordered and stable (1×2) phase, passing through a disordered (2×4) phase, and ruling out the previously proposed intermediate (2×1) structure.

In this paper, we have extended our RAS and LEED investigation to deposition of Germanium onto $c(4\times4)$ and (4×1) reconstructed GaAs(001) surfaces, that are respectively the more As-rich phase and a case of Ga-rich reconstruction. We present also the evolution of the optical properties of the (1×2) phase at higher Ge coverage. Finally, in order to clarify the origin of different contributions to RAS spectra for the (1×2) surface phase, we have modified the sample surface either depositing additional Ge layers or exposing it to air. The different behavior of the various spectral features identified with this procedure are consequently discussed and interpreted.

The paper is organized as follows. In Sec. II we describe the samples and the experimental set-up. Experimental results are presented in Sec. III and discussed in Sec. IV. In Sec. V conclusions are drawn.

II. EXPERIMENTAL

The samples were epitaxial, *n*-doped GaAs layers, grown by MBE on GaAs(001) substrates and capped with an amorphous As protective layer. Two different doping concentrations were used: $n=1\times10^{16}$ cm^{-3} and $n=1\times10^{18}$ cm^{-3}, hereafter indicated as LD (Low Doping) and HD (High Doping), respectively.

After transfer from the growth chamber through air, the samples were investigated in an Ultra-High-Vacuum (UHV) chamber equipped with a four-grid reverse view LEED optic. The RAS set-up (described elsewhere[25]) was placed in front of a strain-free quartz window of the UHV chamber. The samples were decapped at 620 K, yielding $c(4\times4)$. Further annealing to 670 K and 820 K yielded (2×4) and (4×1) surfaces, respectively. Ge evaporation was performed using a calibrated MBE cell. During deposition, the pressure in the chamber (normally in 10^{-11} mbar range) was better than 2×10^{-8} mbar. The substrate was kept at 600 K and 700 K during submonolayer and higher coverage Ge deposition, respectively.

RAS spectra are commonly reported in terms of $\Delta r/r=\Delta\rho/\rho+i\Delta\theta$, where $r=\rho\exp(i\theta)$ is the complex reflectance, and Δr is defined as $\Delta r=r_{\bar{1}10}-r_{110}$. The subscript denotes the polarization of light at normal incidence with respect to directions on the sample surface. In the following we will show only spectra of Re($\Delta r/r$), that is the real part of RAS signal, always recorded after samples were cooled at room temperature (RT). On clean GaAs(001) surfaces (2×4) [(4×1)] reconstructed, As-As (Ga-Ga) dimer bonds are aligned along [110] ([110]).

III. RESULTS

A. Ge on (2×4)

As-rich (2×4) surfaces were prepared from HD (Fig. 1, curve a) and LD (Fig. 2, curve a) samples. In both RAS spectra, superimposed on the anisotropies at 2.9 eV and 4.4 eV typical of (2×4) reconstruction,[17,24,26] there are bulk related structures at E_1 (2.9 eV), $E_1+\Delta_1$ (3.1 eV), and E_0' (4.3 eV) critical points, still evident after exposure to air (Fig. 1, curve d). The different amplitudes of oscillations in curves 1a and 2a (due to the Linear Electro Optical effect, LEO[27]) are consequence of the different doping concentrations.

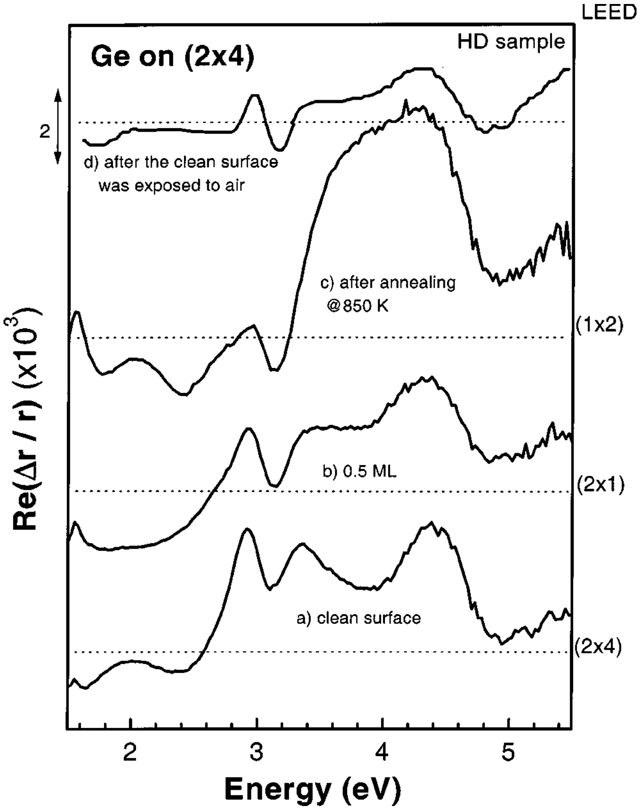

FIG. 1. Re($\Delta r/r$) spectra of a GaAs(001)-(2×4) surface covered with Ge overlayers. The doping concentration of the GaAs sample (indicated as sample HD) was $n=1\times10^{18}$ cm^{-3}. RAS spectra were measured at: (a) clean (2×4); (b) 0.5 ML of Germanium; (c) after annealing @850 K; (d) after the clean surface was exposed to air. Each curve has its own zero line. The LEED pattern corresponding to each surface phase is also indicated.

0.5 ML of Ge were deposited on HD and LD substrates kept at 600 K. RAS spectra are shown in Fig. 1 (curve b) and Fig. 2 (curve b), respectively. After deposition, a strong reduction of the peak intensity at 2.9 eV is apparent as well the development of a negative contribution below 2.6 eV. The broad positive structure centered at about 4 eV seems less affected by coverage. LEED exhibits a weak (2×1) for sample HD, and a weak (1×1) for sample LD.

After annealing (850 K, 5 min) the LEED pattern becomes (1×2). RAS spectra undergo a variation in the low energy side (Fig. 1, curve c and Fig. 2, curve c) with two negative structures that are now visible at about 1.8 and 2.4 eV. However, the 4 eV peak is evidently predominant.

Passing from clean (2×4) to the Ge-covered (1×2) surface, the LEO oscillation decreases. For sample LD, it almost vanishes after 0.5 ML, being again visible—*with opposite sign*—after annealing at 850 K.

Figure 3 displays RAS spectra after deposition of 2 ML (HD sample, $T_{\text{sub}}=700$ K). The LEED pattern is (2×2),

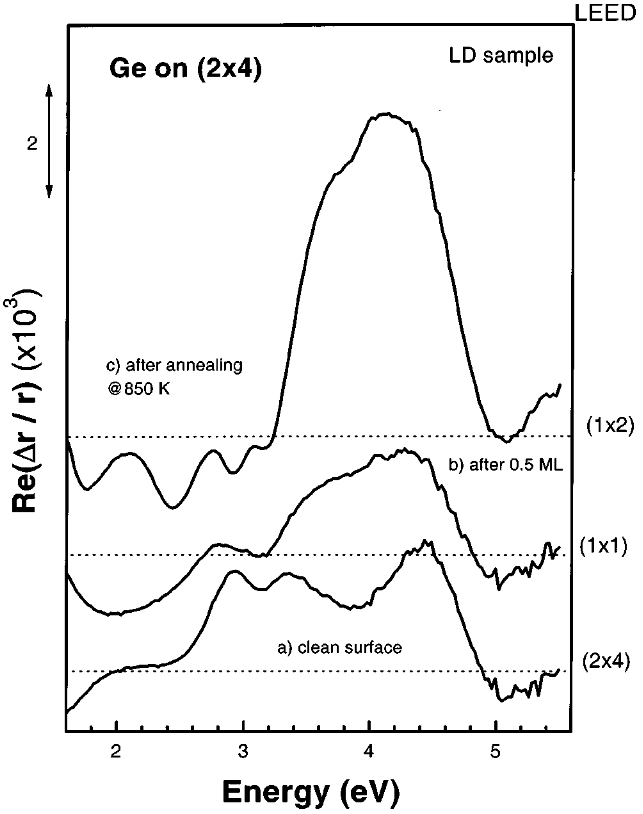

FIG. 2. Re($\Delta r/r$) spectra of a GaAs(001)-(2×4) surface covered with Ge overlayers. The doping concentration of the GaAs sample (indicated as sample LD) was $n = 1\times10^{16}\ \mathrm{cm}^{-3}$. RAS spectra were measured at: (a) clean (2×4); (b) 0.5 ML of germanium; (c) after annealing @850 K. Each curve has its own zero line. The LEED pattern corresponding to each surface phase is also indicated.

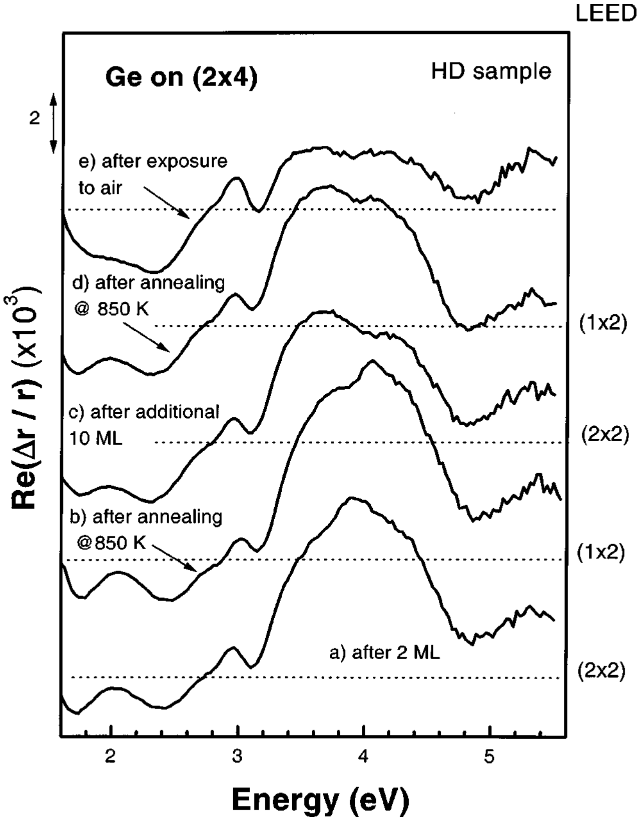

FIG. 3. Re($\Delta r/r$) spectra of a GaAs(001)-(2×4) surface covered with Ge overlayers. The spectra were measured at: (a) 2 ML of Ge; (b) after annealing @850 K; (c) after additional 10 ML of Ge (total coverage: 12 ML); (d) after annealing @850 K; (e) after exposure to air. Each curve has its own zero line. The LEED pattern corresponding to each surface phase is also indicated.

with sharp spots, then again (1×2) after annealing at 850 K. This reconstruction does not change after ten additional ML of Ge ($T_{\mathrm{sub}} = 700$ K). Further annealing at 850 K has no effect. After exposure to air, the negative signal below 2.6 eV is still present (Fig. 3, curve e). Other structures are visible at 3.6 eV and 4.2 eV.

To monitor the interface evolution at higher Ge coverage regime, 21 ML were deposited on the (1×2) phase (LD, $T_{\mathrm{sub}} = 700$ K) (Fig. 4, curve a). The sample was then annealed at 850 K (Fig. 4, curve b). Negative minima at 1.8 and 2.4 eV are detected. A residual LEO oscillation is present at 2.9 eV. The LEED pattern was (2×2), weak after Ge deposition and clear after annealing. The RAS spectrum measured for the same samples stored in air for few days is almost identical (Fig. 4, curve c).

B. Ge on $c(4\times4)$

The RAS spectrum of clean As-rich $c(4\times4)$ surface is characterized by a minimum at about 2.8 eV plus LEO oscillation at 2.9–3.15 eV, as already reported [28] (Fig. 5, curve a). An additional weak structure is detectable at about 2.3 eV, as in Ref. 28.

After deposition of 0.5 ML of Ge ($T_{\mathrm{sub}} = 600$ K), we observed a faint (1×2) LEED pattern. The corresponding RAS spectrum is reported in Fig. 5, curve b. The minimum at 2.8 eV is reduced, and the weak feature at 2.3 eV disappeared.

After the sample was annealed at 700 K for 5 min, the LEED pattern became a weak (2×3). The corresponding RAS spectrum (Fig. 5, curve c) mimics the 2×4 case: a dominant peak at 2.9 eV, plus ancillary features at 3.4 and 4.3 eV. After a new annealing at 850 K the LEED pattern changes, becoming (1×2). The RAS spectrum has now the same line shape as obtained when identical treatment has been applied to (2×4) surface, with two minima around 1.8 and 2.4 eV, plus a broad maximum at about 4 eV.

C. Ge on (4×1)

Finally, we studied the effect of Ge deposition onto a (4×1) Ga-rich surface. RAS line shape for the clean surface (Fig. 6, curve a) is characterized by the large, negative peak at 2.2 eV typical of Ga-termination, and the LEO oscillation at 2.9–3.15 eV.[17,24]

When 0.5 ML of Ge are deposited on this surface ($T_{\mathrm{sub}} = 600$ K), LEED detects a rather good (1×2) order. In RAS spectrum (Fig. 6, curve b) the two negative features at 1.8 eV and 2.4 eV appear. (1×2) order is still present after annealing at 850 K (Fig. 6, curve c). RAS spectra do not show any particular difference with respect to other GaAs(001) surfaces similarly treated.

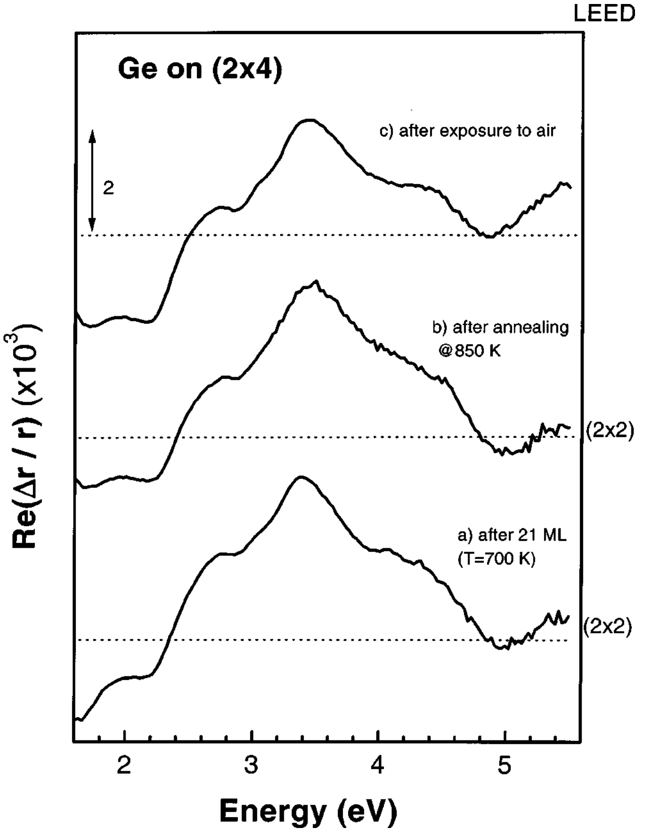

FIG. 4. Re($\Delta r/r$) spectra of a GaAs(001)-(2×4) surface covered with Ge overlayers. The spectra were measured at: (a) 21 ML of Ge; (b) after annealing @850 K; (c) after exposure to air. Each curve has its own zero line. The LEED pattern corresponding to each surface phase is also indicated.

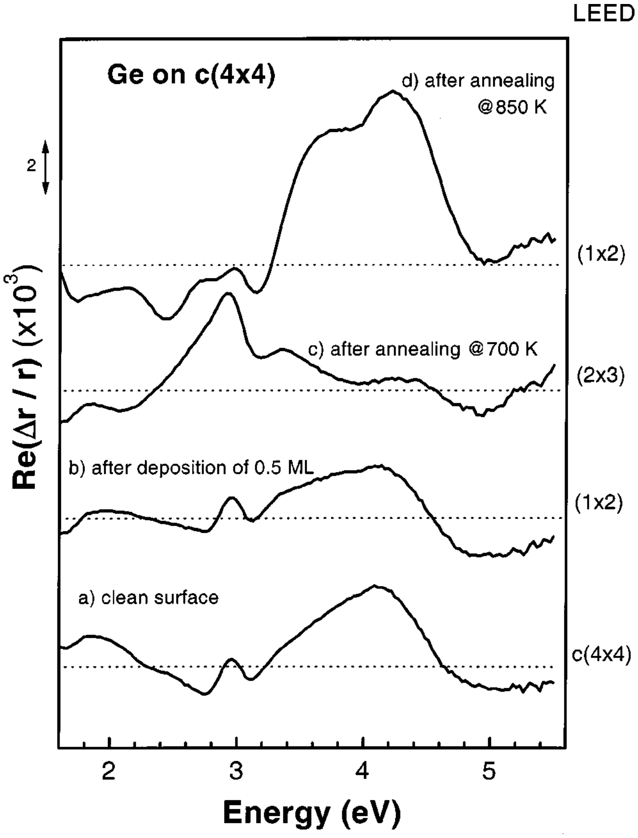

FIG. 5. Re($\Delta r/r$) spectra of a GaAs(001)$c(4\times4)$ surface covered with Ge overlayers. The spectra were measured at: (a) clean surface; (b) after deposition of 0.5 ML of Ge; (c) after annealing @700 K; (d) after annealing @850 K. Each curve has its own zero line. The LEED pattern corresponding to each surface phase is also indicated.

After additional deposition of 2 ML ($T_{\mathrm{sub}}=700$ K) and subsequent annealing ($T_{\mathrm{sub}}=850$ K, 5 mins), the surface showed a (1×2) order.

IV. DISCUSSION

A. Surface and interface structure

Considering the Ge/GaAs interfaces that we have prepared on various (001) surface phases, the present experimental data allow to extract some general conclusion.

(1) For all GaAs(001) surfaces, the (1×2) *phase always occurs after 0.5 ML of germanium are deposited and the sample is annealed at 850 K.* The corresponding RAS spectra have a well defined and characteristic line shape: two negative peaks at about 1.8 eV and 2.4 eV, plus a broad positive feature centered around 4 eV. This RAS spectrum is characteristic of the Ge-Ga dimer structure.[10,11,15]

(2) On the contrary, the intermediate state between the clean surface and the ordered (1×2) *phase depends on the starting surface.*

We have already shown that, in sample HD, after deposition and annealing of 0.5 ML of Ge, the (2×4) surface changes to (1×2), passing through a disordered (2×4) phase.[15] For sample LD (this work) the intermediate phase has a (1×1) LEED pattern. We never observed the previously proposed intermediate (2×1) phase.[10]

When a clean $c(4\times4)$ surface is covered with 0.5 ML of Ge ($T_{\mathrm{sub}}=600$ K), we have measured a weak (1×2) LEED pattern and RAS spectra without the typical features of Ge-Ga dimers [on clean GaAs(001) the transition temperature for $c(4\times4)\rightarrow(2\times4)$ is in the range 400–450 °C]. In this case, (1×2) order originates from a disordered $c(4\times4)$ produced by deposition, and must be distinguished from the (1×2) phase due to Ge-Ga dimers formation. This new phase could consist of a Ge-As mixture (1.5 ML of As and 0.5 ML of Ge), producing mixed Ge-As dimers at the surface, directed along the [110] direction. When this surface is annealed (Fig. 5, curve c), the (2×3) LEED pattern indicates the formation of a structure similar to the one already reported as intermediate between $c(4\times4)$ and (2×4) on a clean (001) surface.[29] The (1×2) originated from Ge-Ga dimers would be not affected by a 700 K annealing, since this temperature is too low to produce an effective variation of stoichiometry for this structure. Only at the higher annealing temperature of 850 K, Ge-Ga dimers are formed, and the RAS spectra exhibit the typical line shape of the (1×2) Ge-Ga terminated surface.

A (1×2) LEED pattern is also observed after deposition

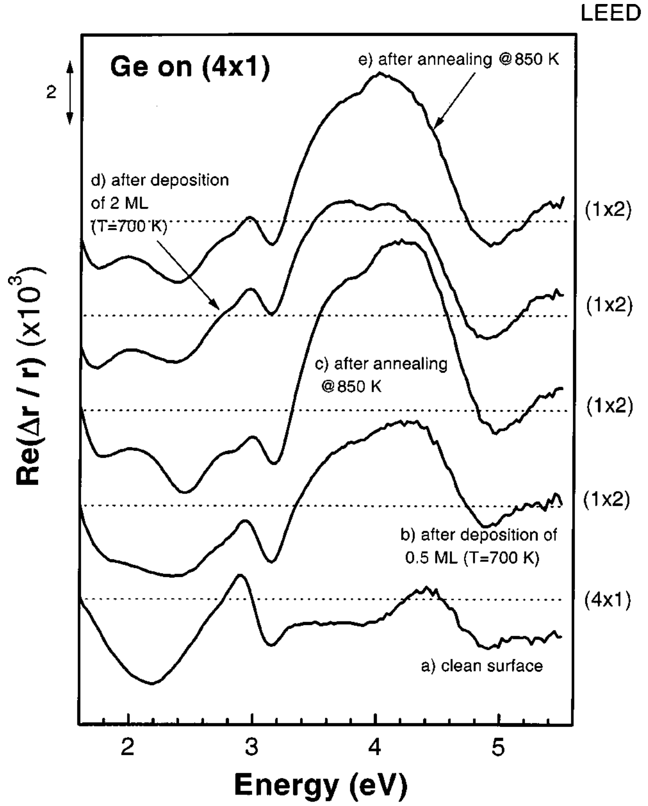

FIG. 6. Re($\Delta r/r$) spectra of a GaAs(001)-(4×1) surface covered with Ge overlayers. The spectra were measured at: (a) clean surface; (b) after deposition of 0.5 ML of Ge; (c) after annealing @850 K; (d) after additional 2 ML of Ge (total coverage: 2.5 ML); (e) after annealing @850 K. Each curve has its own zero line. The LEED pattern corresponding to each surface phase is also indicated.

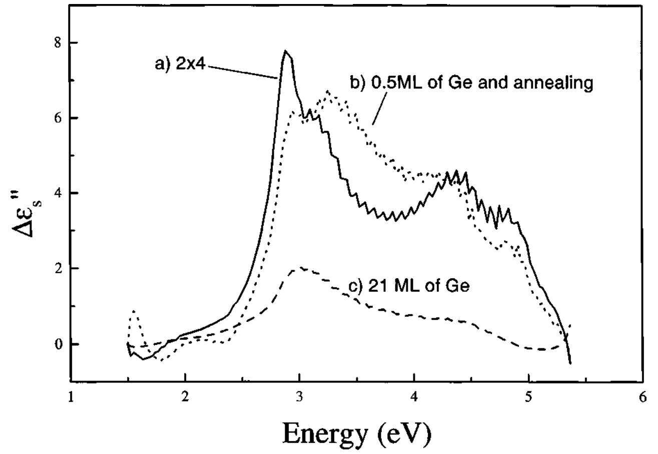

FIG. 7. $\Delta\varepsilon_s''$ computed by KK analysis from Re($\Delta r/r$) spectra of GaAs(001)-(2×4) surfaces covered with increasing Ge overlayers: (a) (full line) clean surface [corresponding RAS spectrum: curve a, Fig. 1]; (b) (dotted line) after 0.5 ML of Ge and annealing @850 K [RAS spectrum: curve c, Fig. 1]; (c) (dashed line) after 21 ML [RAS spectrum: curve d, Fig. 4].

and annealing of 0.5 ML of Ge onto the clean (4×1) Ga-rich surface: in this case it originates from Ge-Ga dimers formed at the surface as the overlayer is grown, consistently with the Ga-richness of the (4×1) surface. No As desorption and consequent intermediate phase must occur before Ge-Ga bonds are formed.

We conclude that *only* the *intermediate phase* between the clean surface structure and the (1×2) Ge-Ga dimer structure (characteristic of the Ge/GaAs interface) depends upon the *starting surface reconstruction*. For As-rich clean surfaces, during desorption of excess As, an intermediate phase occurs before the (1×2) surface is formed. On the contrary, for Ga-rich surfaces the (1×2) phase is formed directly upon Ge deposition at 600 K.

The anisotropy spectrum measured at the (1×2) Ge/GaAs(001) interface is well accounted by calculations:[15] on this basis, we attributed two negative features (at 1.8 and 2.4 eV) to back-bonds of the surface Ge-Ga dimers. This attribution agrees with the observation that: (i) such structures are visible *just after deposition* when Ge/GaAs interface develops onto a Ga terminated layer; (ii) such structures survive after the sample is exposed to air, as they come from buried interface states of the Ge overlayer.

The broad peak at about 4.1 eV—significantly reduced after surface contamination—deserves a closer analysis. Within the three-layer model,[30] the complex RAS signal is expressed by:

$$\frac{\Delta r}{r}=\frac{4\pi i d}{\lambda}\,\frac{\Delta\varepsilon_s}{\varepsilon_b-1}, \tag{1}$$

where $\Delta\varepsilon_s=\Delta\varepsilon_s'-i\Delta\varepsilon_s''=\varepsilon_s^{[\bar{1}10]}-\varepsilon_s^{[110]}$ is the anisotropy of the surface (or overlayer) dielectric function between [110] and [110] directions, ε_b the complex bulk dielectric function, d the thickness of the layer, and λ the photon wavelength. In general, in order to obtain $\Delta\varepsilon_s'$ and $\Delta\varepsilon_s''$ from experimental Re($\Delta r/r$) we need an additional Kramers–Kroenig (KK) relation.[31,32]

The resulting $\Delta\varepsilon_s''$ curve computed from the RAS spectrum of Fig. 1, curve c, is displayed in Fig. 7, curve b. For a comparison, we have also reported $\Delta\varepsilon_s''$ for the clean (2×4) phase, extracted from spectrum a of Fig. 1. The peak at 2.9 eV, characteristic of the (2×4) reconstruction, has been cut down by Ge deposition and annealing, while the structure at E_0' bulk critical point is only slightly affected. Besides the already discussed negative peaks at 1.8 and 2.4 eV, a new feature has now appeared at about 3.3 eV. We attribute this structure to the Ge overlayer, as also tested by its evident sensitivity to contamination.

For higher Ge coverage, our experiments were limited to GaAs(001)(2×4) surfaces (Fig. 3, curve c, and Fig. 4). The spectral line shape changes in the high-energy range, where a broad structure evolves at about 3.4 eV. Below 3 eV, the spectrum is essentially unaffected, as one expects for interface-related anisotropies (see discussion above). The occurrence of a (2×2) LEED pattern comes probably from superposition of (1×2) and (2×1) domains on the surface of the Ge epilayer: the surface most likely has the same structure as Ge(001), locally dimerized with (1×2) or (2×1) superstructure.[33] On a single domain surface, the RAS spectra should be similar to the ones measured for Ge(001) surfaces;[34] but any eventual anisotropy contribution of

Ge-Ge dimers cancels out for the presence of differently oriented domains. Also the behavior versus gas contamination is different: no modification is evident after exposure to air.

Computing by KK analysis the overlayer dielectric function from RAS data, we have obtained the curve c of Fig. 7. The difference between experimental Re($\Delta r/r$) and computed $\Delta\varepsilon_s''$ is large: the peak at 3.4 eV, prominent in the RAS spectra of Fig. 4, does not exist in the $\Delta\varepsilon_s''$ curve. It is an artifact of the dependence of Re($\Delta r/r$) upon the substrate and overlayer dielectric functions. The resulting line shape—with two structures at E_1 and E_0' critical points—is evidently a relic of the GaAs substrate, related to modifications of the effect that surface termination has on bulk wave functions,[35,36] without any particular surface or interface contribution. Furthermore, we cannot exclude that strain effects in GaAs produced by the Ge overlayer provide an important contribution to the measured anisotropy.[37]

In conclusion, in the high coverage regime RAS spectra are *interface, overlayer, and substrate-related*, while true surface terms have vanished.

Finally, we want to comment briefly about the zero lines that have been reported in figures for each experimental Re($\Delta r/r$) curve. In fact, from Eq. (1) it is possible to show that shifts of the baseline *within the experimental energy range* could be the consequence of anisotropic absorption *out of the experimental energy range*, that introduces detectable effects in Re($\Delta r/r$) through the real part $\Delta\varepsilon_s'$ of the overlayer dielectric function anisotropy, weighted by a coefficient related to bulk absorption (see, for example, discussion in Refs. 31 and 32). In our data, however, such baseline shifts seem to be negligible.

B. Linear-electro-optical effect and doping

It is well known that in RAS spectra characteristic features appear in coincidence with bulk critical points, due to the linear-electro-optical effect (LEO). LEO is a modification of the bulk optical properties induced by the electric field consequence of band-bending at the surface.[27,38] In RAS spectra it induces oscillations at bulk critical points, detected at well-defined photon energies. The amplitude of LEO oscillation depends upon the static dielectric function ε of the crystal, the carrier concentration N and the surface band bending V_{bb}. Since LEO is linear with respect to the space-charge electric field, LEO structures invert upon going from *n*- to *p*-type material.[38]

In Sec. III we commented that LEO oscillation—clearly visible for the clean surface at E_1 and $E_1+\Delta_1$ bulk critical points—reduces strongly in amplitude and even changes its sign after Ge have been deposited onto the clean surface and then annealed. This variation could be the consequence of a change either in doping concentration (N) or in band bending (V_{bb}). In our experiment, the former effect could originate from diffusion, during annealing, of Ge atoms from the outer layers into GaAs substrate, with consequent modification of local doping. The latter could be due to a change in the electronic states distribution at the interface induced by formation of Ge-Ga dimers, moving the Fermi level.

The possibility to distinguish between alternative interpretations comes from the mentioned experimental finding that LEO sign inverts in sample LD after 0.5 Ge ML have been annealed at 850 K (Fig. 2, curve c). On the contrary, no inversion of sign occurs in similar conditions for HD samples (Fig. 1, curve c).

This effect is likely produced by Ge in-diffusion leading to *p*-doping of the substrate: Ge atoms, in a GaAs matrix, can behave as acceptor atoms.[39] The highly *n*-doped sample (HD) is only partially compensated, while the lightly *n*-doped sample (LD) is overcompensated, thus becoming *p*-doped. Given the doping level of our samples, we estimate that a low number of Ge atoms (less than 1 for 1000 atoms deposited onto GaAs) is necessary to produce the local doping variation, diffusing into the substrate for a depth comparable to the penetration length of light at the E_1 and $E_1+\Delta_1$ critical points. These values are below the detection limit of spectroscopies used to monitor interdiffusion at the Ge/GaAs interface.[40,41]

Alternatively, the diminishing/reversal of LEO could be explained in terms of the final Fermi-level position (E_F) when the interface has evolved to (1×2) structure. This position should be intermediate between the ones typical of the clean (2×4) surfaces of HD and LD samples. In the former case band bending would decrease, in the latter an accumulation layer would form, reversing the surface field and LEO sign. From photoemission data we estimate that after deposition of 0.5 ML E_F is only slightly moved from its initial value.[8] Since we know E_F due to the doping level in our samples, this little variation is not enough to explain the observed behavior of LEO. However, the temperature at which the Ge/GaAs interface was formed in Ref. 8 was lower than the one used in our experiment. This suggests that, although movement of Fermi level seems to be less likely than doping modification, it cannot be completely refused.

V. CONCLUSIONS

We have presented the optical anisotropy measured at Ge/GaAs interfaces formed on different phases of GaAs(001) surface. We have determined that, although the stable structure of the interface is *always* represented by the (1×2) Ge-Ga dimer structure, regardless of the starting condition, the intermediate phases depend upon the stoichiometry of the surface onto which Ge is deposited. The role of annealing has been investigated to define either the structure of the overlayer and the doping of the substrate, more likely modified by in-diffusion of Ge atoms caused by temperature.

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