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Optical transitions and sum rules at clean semiconductor surfaces

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Abstract

The experimental results of surface differential reflectivity and reflectance anisotropy spectroscopy show that in Si(111)- 2×1 , Ge(111)- 2×1 and GaAs(001)- 2×4 a sum rule for the imaginary part of the (surface) dielectric function is verified both for the isotropic and anisotropic parts of ϵ_s'' . It is shown that the sum rule together with the dependence of the spectra upon oxygen contamination are useful in the interpretation of the optical transitions of the above surfaces. In particular, for the case of GaAs(001)- 2×4 the above analysis has allowed the distinction between optical transitions associated to true surface states and bulk states modified by the surface near the 3 eV critical point.

1. Introduction

The study of the optical properties of clean semiconductor surfaces has allowed over the years the observation of electronic surface states mainly associated to reconstruction [1, 2]. The first observations were obtained by the so-called surface differential reflectivity (SDR) technique which consists of the comparison of the intensity of light reflected by clean and oxidized surfaces [1]:

$$\left(\frac{\Delta R}{R}\right)_{\text{SDR}} = \frac{R_{\text{clean}} - R_{\text{ox}}}{R_{\text{ox}}}. \quad (1)$$

This quantity is related to the surface dielectric function, representing transitions between filled and empty surface bands. In particular, Si(111)- 2×1 and Ge(111)- 2×1 have shown large anisotropies associated to the chain structure of the surface reconstruction [1]. The existence of such anisotropies enables one to use a different technique, named reflectance anisotropy spectroscopy (RAS), that compares the normal-incidence reflectivities for two orthogonal polarizations x and y as a function of energy [2–5]:

$$\left(\frac{\Delta R}{R}\right)_{\text{RAS}} = 2 \frac{R_x - R_y}{R_x + R_y}. \quad (2)$$

The method of modulation spectroscopy applied to RAS results in a great sensitivity, allowing the observation of small anisotropies, of the order of 1% or lower, for example in the GaAs(001) reconstructed surfaces [2, 5].

The small signals observed by RAS are related to the anisotropy of both the surface and the bulk, especially near the critical points of the bulk structure. The observation by SDR and RAS of the surface structure of Si and Ge(111)-2 × 1 both below and above the indirect bulk gap has shown zero-sum behaviour [6, 7]:

$$\int_0^{\infty} (\varepsilon_s''(x) - \varepsilon_s''(y)) \omega \, d\omega = 0. \quad (3)$$

Here x and y refer to the polarizations parallel and perpendicular to the chains of the Pandey model of reconstruction, i.e. $[1\bar{1}0]$ and $[11\bar{2}]$, $\varepsilon_s''(x)$ and $\varepsilon_s''(y)$ are the principal values of the surface dielectric tensor (imaginary parts). However, we shall demonstrate that equation (3) has a general character and descends from fundamental properties of the dielectric function.

This rule may be useful in many ways, for example to distinguish between surface-state transitions and transitions involving bulk states modified by the surface, as shall be discussed below in detail.

2. Application of sum rules to SDR and RAS

It is well known [8] that real and imaginary part of the dielectric function $\hat{\varepsilon}(\omega) = \varepsilon'(\omega) - i\varepsilon''(\omega)$ are connected by the K–K (Kramers–Kronig) dispersion relations, namely for $\varepsilon'(\omega)$:

$$\varepsilon'(\omega) = 1 + \frac{2}{\pi} \wp \int_0^{\infty} \frac{\omega' \varepsilon''(\omega')}{\omega'^2 - \omega^2} \, d\omega', \quad (4)$$

where \wp means the Cauchy principal part of the integral.

Let us call ω_{\max} a cutoff frequency beyond which the absorption can be neglected ($\varepsilon'' = 0$).

For frequencies $\omega \gg \omega_{\max}$ equation (4) can be rewritten as

$$\varepsilon'(\omega) = 1 - \frac{2}{\pi \omega^2} \int_0^{\omega_{\max}} \omega' \varepsilon''(\omega') \, d\omega' \quad (\omega \gg \omega_{\max}). \quad (5)$$

For anisotropic surfaces ε' and ε'' are tensors of rank 2. However, for $\omega \gg \omega_{\max}$, $\hat{\varepsilon}(\omega)$ is a scalar, since at sufficiently high energies bound electrons behave essentially as free ones, and do not experience the crystal field. The fact that for $\omega \gg \omega_{\max}$ $\varepsilon'(\omega)$ is a scalar brings about automatically the validity of equation (3).

For $\omega \gg \omega_{\max}$, $\varepsilon'(\omega)$ can be approximated [8] by

$$\varepsilon'(\omega) = 1 - \frac{\omega_p^2}{\omega^2}, \quad (6)$$

where ω_p is the plasma frequency:

$$\omega_p = \sqrt{\frac{4\pi e^2 N}{m}}, \quad (7)$$

where N is the density of electrons and m is the free-electron mass. Therefore equation (5) becomes

$$\int_0^{\omega_{\max}} \omega' \varepsilon''(j)(\omega') \, d\omega' = \frac{\pi}{2} \omega_p^2, \quad (8)$$

where j stands for x or y .

If a group of electrons behave in a similar way (for example the electrons associated to dangling bonds in semiconductors) it may be convenient to divide the interval of integration in equations (3), (5) and (8) so that in each interval the given group of electrons can be considered separately. In this case N of equation (7) can be written as $N = N_0 n_{\text{eff}}$, where N_0 is the density of atoms and n_{eff} the number of electrons per atom in a given group [6].

For a surface layer of thickness d , equation (8) becomes

$$n_{\text{eff}}(\omega) = \frac{m}{2\pi^2 e^2 N_s} \int_0^\omega \omega' [\varepsilon_s''(\omega') d] d\omega', \quad (9)$$

where N_s is the density of surface atoms. Then $n_{\text{eff}}(\omega)$ shows a plateau when the oscillator strength of a given group of electrons is exhausted. It should be emphasized that a direct knowledge of d (which is ill defined and in some ways arbitrary) is not required for the determination of n_{eff} . In fact, the experimental values of $\Delta R/R$ yield directly $\varepsilon_s''(\omega)d$, both in SDR and RAS. Moreover, it can be noted that the knowledge of d is not necessary for the validity of equation (3).

A convenient formulation of the reflection of an electromagnetic wave by a surface was given by McIntyre and Aspnes [9] in terms of a three-phase model: vacuum, surface of effective thickness d and bulk, each phase being characterized by a complex dielectric function $\hat{\varepsilon}_j = \varepsilon_j' + i\varepsilon_j''$, where j labels the three media. For normal incidence the McIntyre and Aspnes theory gives (when the first medium is vacuum) [9]

$$\frac{\Delta R}{R} = \frac{R(d) - R(0)}{R(0)} = \frac{8\pi d}{\lambda} \text{Im} \left(\frac{1 - \hat{\varepsilon}_2}{1 - \hat{\varepsilon}_3} \right). \quad (10)$$

In the range where the oxide is transparent $R(0) \approx R_{\text{ox}}$, so that definition (1) and equation (10) are equivalent [1]. If one further assumes $d = d_{\text{ox}}$, expression (10) can be easily transformed into [10]

$$\frac{\Delta R}{R} = -\frac{8\pi d}{\lambda} \left[\frac{(1 - \varepsilon_b')(\varepsilon_s'' - \varepsilon_{\text{ox}}'')}{(1 - \varepsilon_b')^2 + (\varepsilon_b'')^2} + \frac{\varepsilon_b''(\varepsilon_s' - \varepsilon_{\text{ox}}')}{(1 - \varepsilon_b')^2 + (\varepsilon_b'')^2} \right], \quad (11)$$

where now s (surface) (or ox (oxide)) and b (bulk) refer to the second and third medium.

The general case $d_{\text{ox}} \neq d$ could be taken into account by considering in equation (11) an effective dielectric function [10]

$$(\hat{\varepsilon}_{\text{ox}})_{\text{eff}} = [\hat{\varepsilon}_{\text{ox}} + 1] \frac{d_{\text{ox}}}{d} - 1 \quad (12)$$

for the oxide.

The dependence on the bulk dielectric properties is included in the quantities

$$A = \frac{8\pi}{\lambda} \frac{(\varepsilon_b' - 1)}{(\varepsilon_b' - 1)^2 + (\varepsilon_b'')^2} \quad B = \frac{8\pi}{\lambda} \frac{(\varepsilon_b'')}{(\varepsilon_b' - 1)^2 + (\varepsilon_b'')^2}, \quad (13)$$

so that equation (11) can be rewritten in the simpler form [10]

$$\left(\frac{\Delta R}{R} \right)_{\text{SDR}} = Ad(\varepsilon_s'' - \varepsilon_{\text{ox}}'') - Bd(\varepsilon_s' - \varepsilon_{\text{ox}}'). \quad (14)$$

A and B are known functions of the bulk parameters and are shown in figure 1 for Si, Ge and GaAs. It is seen from figure 1 that B is nearly zero for energies lower than 3.5 eV in Si, 2 eV in Ge and 3 eV in GaAs. Moreover, in such an energy range, the oxide is transparent ($\varepsilon_{\text{ox}}'' = 0$) and $\Delta R/R$ is then simply proportional to $\varepsilon_s''(\omega)$, which is the quantity that enters equation (3).

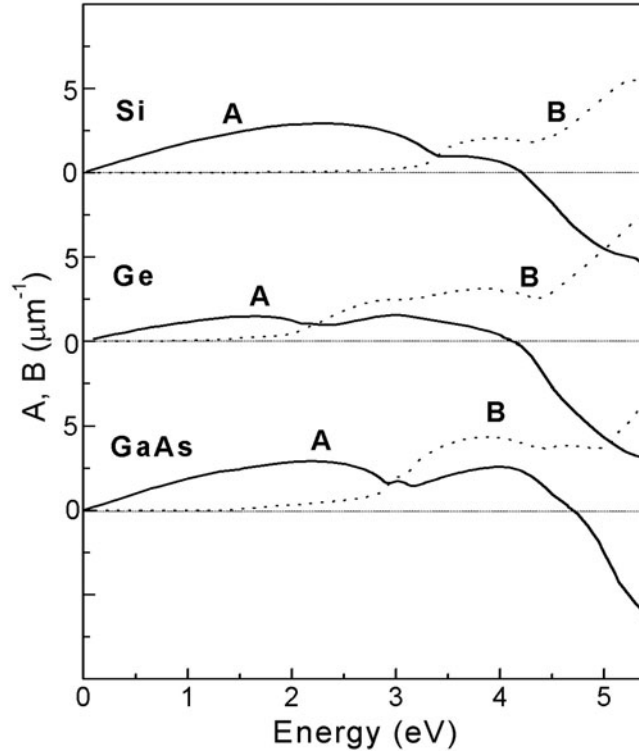


Figure 1. Energy dependence of the coefficients A (solid curve) and B (dashed curve), defined in equation (13), for Si, Ge and GaAs, in the range 0–5.5 eV. The bulk optical values are taken from [19].

In such a range rule (3) is valid also for $\Delta R/R$ and can be checked directly by experiment:

$$\int_0^{\omega_{\max}} \left[\left(\frac{\Delta R}{R} \right)_x - \left(\frac{\Delta R}{R} \right)_y \right] \omega d\omega = 0. \quad (15)$$

On the other hand, when $B \neq 0$ the change in reflectance depends also on ε'_s (even in the absence of effects associated to $\hat{\varepsilon}_{\text{ox}}$) so that the use of K–K relations is necessary to extract $\hat{\varepsilon}_s$ from the experimental data [10].

In the same way, the RAS signal given by (2) can be written, in Aspnes and McIntyre formalism, as

$$S(\omega) = 2 \frac{R_x(d) - R_y(d)}{R_x(d) + R_y(d)} \approx 2 \frac{R_x(d) - R_y(d)}{R_x(0) + R_y(0)}. \quad (16)$$

Moreover, for an isotropic substrate (or for small anisotropy) we can take in the denominator $R_x(0) = R_y(0)$ and write [7]

$$S(\omega) \approx \left(\frac{\Delta R}{R} \right)_x - \left(\frac{\Delta R}{R} \right)_y = Ad(\varepsilon''_{s,x} - \varepsilon''_{s,y}) - Bd(\varepsilon'_{s,x} - \varepsilon'_{s,y}) = Ad\Delta\varepsilon''_s - Bd\Delta\varepsilon'_s. \quad (17)$$

Since RAS experiments are often performed in a spectral region where the assumption $B \approx 0$ is not valid, it may be useful to consider briefly such a case.

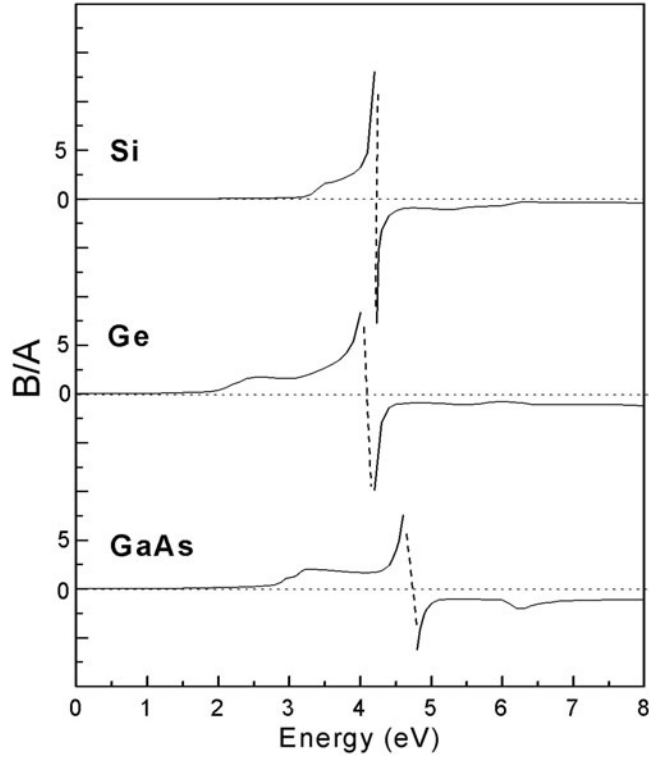


Figure 2. Energy dependence of the function B/A , for Si, Ge and GaAs, in the range 0–8 eV.

By defining the integral

$$I = \int_0^\infty \frac{\omega}{A} S(\omega) d\omega = d \left[\int_0^\infty \omega d\Delta \varepsilon_s'' \omega - \int_0^\infty \omega \frac{B}{A} d\Delta \varepsilon_s' \omega \right], \quad (18)$$

we see that it reduces to

$$I = -d \int_0^\infty \omega \frac{B}{A} d\Delta \varepsilon_s' \omega, \quad (19)$$

because of equation (3) [7, 11].

The integral I can be thought of as a reduced area of the RAS signal. It is in general different from zero.

B/A is a known function of the bulk parameters and is plotted in figure 2, omitting the short range where A changes sign and the function diverges. In fact, this short interval does not contribute appreciably to the integral since the function B/A is symmetric around the singularity and the rest of the integrand is a slowly varying function.

Figure 2 shows that B/A is positive below the frequency for which $A = 0$ and negative above it. This suggests that the influence of the term (19) on the integral I is small. Therefore $(\Delta R/R)_{\text{RAS}}$ obeys approximately an equation similar to equation (15) even when $B \neq 0$, provided $(\Delta R/R)_{\text{RAS}}$ is divided by A . The problem is discussed in detail in [7] and [11], where the effect of various oscillators at different energies is simulated.

In principle the same considerations could be done also for SDR when $B \neq 0$. In such a region, however, the oxide is absorbing, so the analysis becomes uncertain.

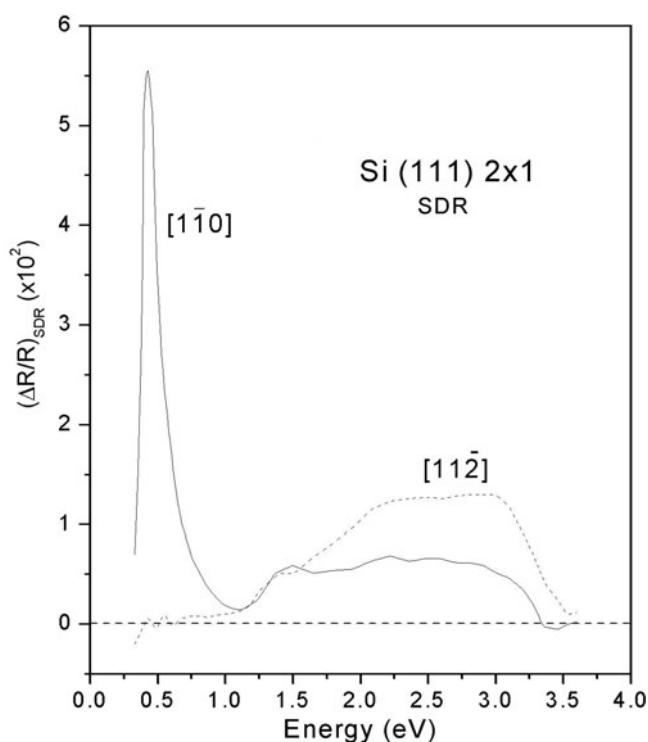


Figure 3. Surface differential reflectivity spectra of a single-domain Si(111)- 2×1 surface, for light polarized along the $[1\bar{1}0]$ and $[1\bar{1}\bar{2}]$ directions, in the range 0.3–3.5 eV.

3. Experimental results and discussion

3.1. Silicon and germanium (111)- 2×1 by SDR and RAS

$(\Delta R/R)_{\text{SDR}}$ for Si(111)- 2×1 is plotted in figure 3, in the energy range from 0.3 to 3.5 eV [12]. By cleaving the sample along the $[1\bar{1}\bar{2}]$ direction a single-domain surface has been obtained, as checked by LEED (low energy electron diffraction). Light was polarized along $[1\bar{1}0]$ and $[1\bar{1}\bar{2}]$, i.e. parallel and perpendicular to the directions of the chains of the Pandey model of the 2×1 reconstruction [13].

Since $B \approx 0$ and the oxide is practically transparent over the entire range of figure 3, ϵ_s'' and $\Delta R/R$ display the same behaviour, as expected from equation (14) and figure 1. This appears clearly by comparing figures 3 and 4 (upper part), where $\epsilon_s''d$ is plotted as a function of energy for the two polarizations. It must be noticed, however, that figure 4 has been obtained from equation (14) without neglecting the last term and determining ϵ_s' from K–K analysis [10, 14]. This brings only a minor correction in the range 3.0–3.5 eV.

From the values of $\epsilon_s''d$ given in figure 4(a) and using equation (9), the effective number of electrons per atom n_{eff} can easily be calculated, and this is plotted in figure 4(b) as a function of energy for each polarization. It is seen that both curves tend to plateaux corresponding to about 1 electron /atom, within experimental error. This shows that the dangling-bond electron exhausts its oscillator strength below 3.5 eV and that equation (3) is accurately satisfied.

Due to its strong anisotropy (see figure 3), the single-domain Si(111)- 2×1 surface is a good candidate for RAS investigation. The measurements have been recently performed,

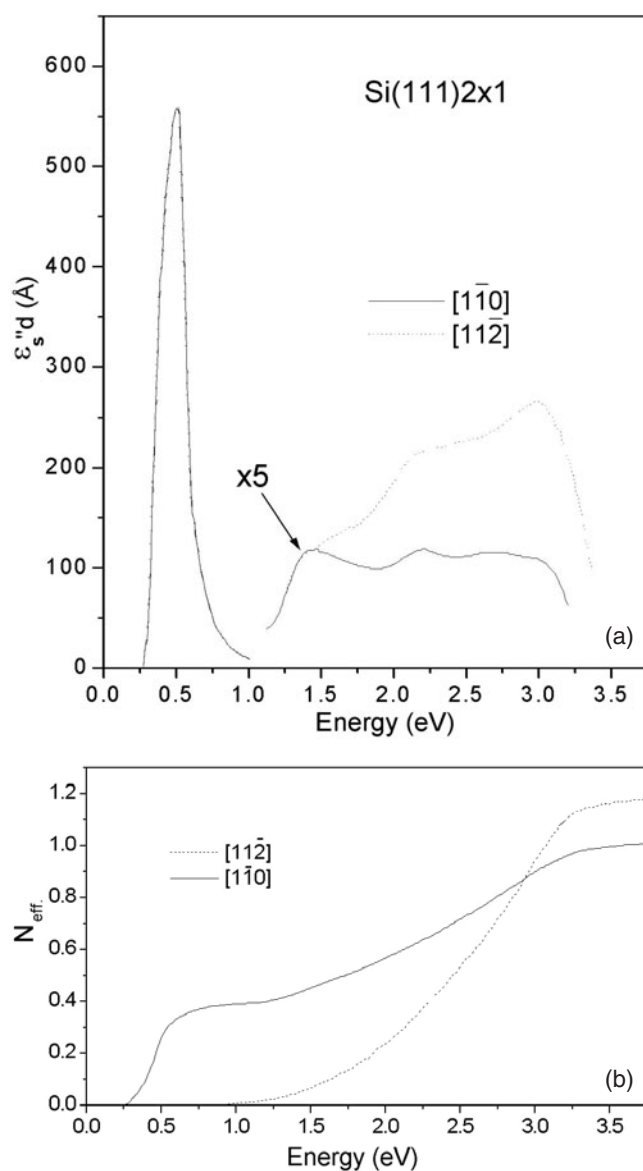


Figure 4. Top panel (a): imaginary part of the surface dielectric function $\epsilon''_s(\omega)$ times the surface thickness d for Si(111)- 2×1 , calculated from figure 3, for both light polarizations. Bottom panel (b): effective number of electrons/atom participating in optical transitions up to the energy in the abscissa, for the two polarizations of figure 3.

in the near IR–visible–UV range. The results are shown in figure 5 as a function of energy, between 0.3 and 5.5 eV. The IR part of the spectrum has been obtained separately with a novel RAS apparatus [15]. For the visible and UV range the decrease of the RAS signal upon oxygen exposure¹ is also reported.

¹ Due to a water vapour contamination in the gas line, of the order of 1% of the oxygen pressure, the absolute values of exposure in figure 5 cannot be compared with those of [12] (indeed they are smaller by a factor 2–3, as expected).

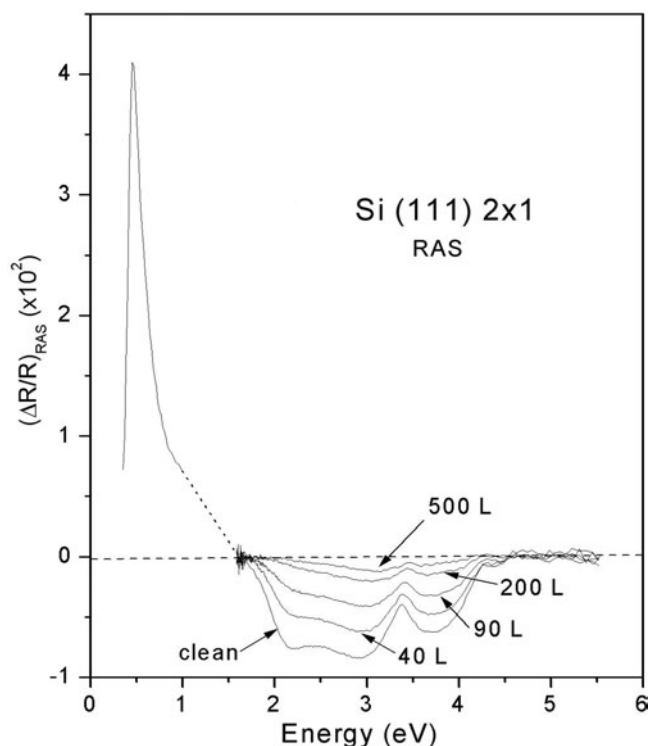


Figure 5. Reflectance anisotropy spectrum of a single-domain Si(111)- 2×1 surface, in the range 0.3–5.5 eV. The measurements in the IR range (0.3–1 eV) and visible–UV range (1.5–5.5 eV) were taken with different apparatuses and different samples. The dashed line connects the two parts of the spectrum. In the latter case the decay of the RAS signal upon exposure to (humid) oxygen was also recorded. The last spectrum (500 L) indicates the saturation value.

A comparison of figures 3 and 5 shows that SDR and RAS are in very good agreement in the common range (0.3–3.5 eV), thereby proving that the oxide does not contribute to the spectrum, at least for its anisotropic part. Moreover, the RAS curve shows a new peak at about 3.8 eV, which is outside the range of SDR data.

In order to apply the sum rule expressed by equation (3) to the experimental data of figure 5, the anisotropy of the imaginary part of the surface dielectric function $\Delta\varepsilon_s''$ has been computed, on the basis of equation (17) and using K–K analysis for $\Delta\varepsilon_s'$. The results confirm that equation (3) is satisfied for optical transitions up to 3.5 eV, while new transitions appear for light polarized along the $[11\bar{2}]$ direction, as already pointed out. The oxidation dependence of this part of the spectrum proves that also in this case surface electrons are involved. However, since dangling-bond electrons have exhausted their oscillator strength below 3.5 eV, the new contribution to $\Delta\varepsilon_s''$ must be due to another group of surface electrons, possibly of back-bond type. The application of rule (3) to this second group of electrons reveals, on the other hand, that additional optical transitions polarized in the orthogonal direction $[1\bar{1}0]$ are necessary. They are expected at energies higher than the range of figure 5. However, in this region the experiments are particularly difficult.

It should also be mentioned that ellipsometric measurements have also been performed in the energy range of figure 5 [16], and the data are in qualitative agreement with the present results. In particular the peak at 3.8 eV is observed as a clear shoulder in the $\Delta R/R$ spectrum

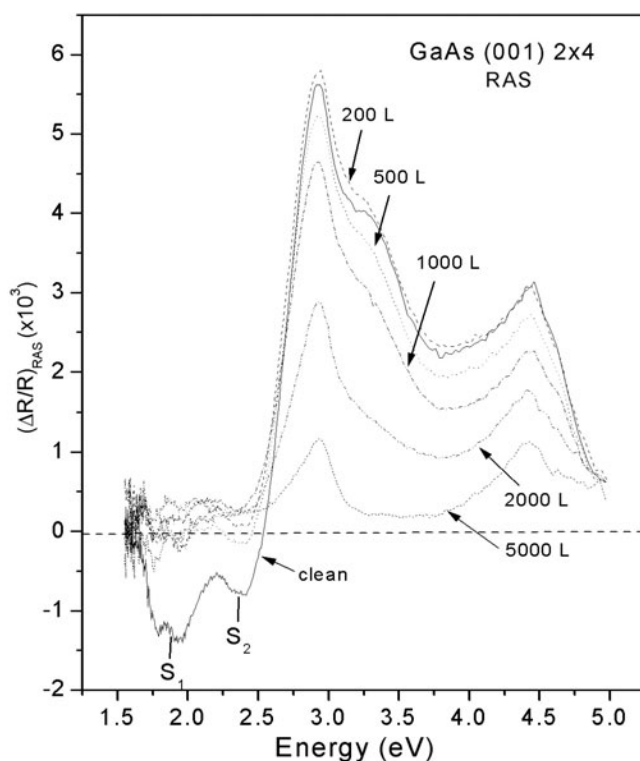


Figure 6. Reflectance anisotropy spectrum, in the range 1.5–5.0 eV, of a GaAs(001)-2 × 4 surface, clean and upon increasing exposure to oxygen (excited by the ion gauge) as indicated.

computed from the ellipsometric data. As a final remark, close inspection of figure 5 shows a residual signal present at saturation value (500 L (see footnote 1)), with a minimum near the 3.3 eV critical point, so that it can be interpreted as due to bulk transitions modified by the surface.

$\Delta R/R$ of Ge(111)-2 × 1 observed with SDR shows essentially the same behaviour as Si(111)-2 × 1 [6]. Specifically, rule (3) is verified in the range of observation (0.3–2.5 eV), though the plateau in n_{eff} occurs for a number of electrons slightly above 1 (1.4 and 1.5 for the two polarizations). This extra contribution is presumably associated to back bonds and has been discussed in detail in [6].

3.2. GaAs(001)-2 × 4 by RAS

The surface electronic properties of GaAs(001)-2 × 4 have been revisited recently by using the reflectance anisotropy technique in combination with high resolution electron energy loss spectroscopy [17]. Due to an improved quality of the samples, grown by molecular beam epitaxy *in situ*, optical transitions related to surface states have been revealed for the first time in the energy region 1.8–2.5 eV. The extreme sensitivity to oxygen contamination points to the surface character of these transitions, as confirmed by accurate first-principle density-functional calculations [17].

In figure 6 the evolution of the reflectance anisotropy spectra of clean GaAs(001)-2 × 4 upon oxygen exposure is reported. The negative part of the clean-surface spectrum below the

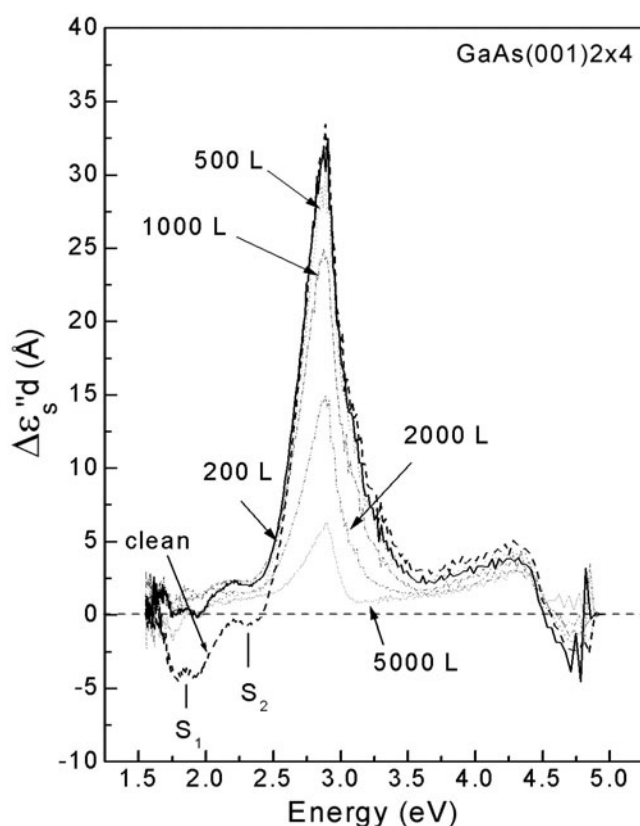


Figure 7. Anisotropy of the imaginary part of the surface dielectric function $\Delta\epsilon_s''(\omega)$ times the surface thickness d for GaAs(001)- 2×4 , calculated from figure 6.

E_1 critical point at 3 eV disappears completely after an exposure of only 200 L, thus proving that it is associated to surface states. At first sight this is the only relevant modification of the spectrum occurring at such a small oxygen exposure. On the other hand, at larger exposure values, between 500 and 5000 L, a progressive decrease of the main peaks at 3 and 4.5 eV, associated to bulk transitions modified by the surface, is observed.

In order to verify the validity of the sum rule expressed by equation (3), the imaginary part of the surface dielectric function has to be derived from the experimental data of figure 6. This is done by Kramers–Kronig analysis in the framework of the three-layer model and assuming that the oxide is an isotropic layer of thickness d [10].

The result is shown in figure 7, where $\Delta\epsilon_s''$ is reported for each oxidation stage. By comparing figures 6 and 7 it is clear that roughly speaking $\Delta\epsilon_s''$ retains the general line shape of $\Delta R/R$. However, a closer inspection at the two figures reveals some differences. For example, the pronounced shoulder in the $\Delta R/R$ spectra at about 3.4 eV (clean surface and initial oxidation stages) is no longer present in the $\Delta\epsilon_s''$ spectra.

In figures 6 and 7 two groups of optical transitions, labelled S_1 and S_2 , are visible at about 1.8 and 2.4 eV. Both of them have surface character and contribute to the negative part of the RA spectrum, being excited by light polarized along the [110] direction (perpendicular to the As dimers). In figure 7 the apparent shift of the zero line around 2.5 eV is probably associated to the presence of oscillators (transitions) outside the range of measurements (see below).

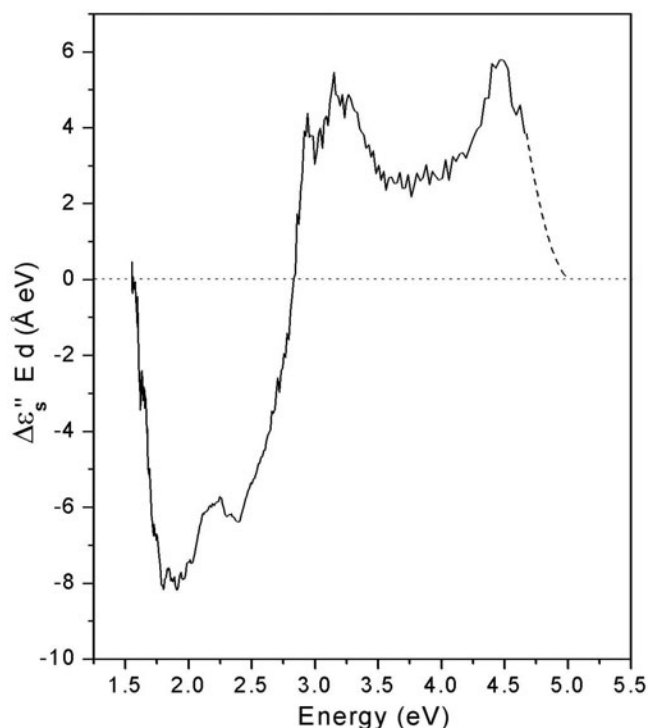


Figure 8. Difference of the two curves of figure 7 corresponding to the clean GaAs(001)- 2×4 surface and to 200 L exposure, multiplied by the photon energy E . The last part of the spectrum has been extrapolated to zero.

In order to overcome this problem and to be able to apply equation (3), we have subtracted the 200 L curve (of figure 7) from the clean one. The result is plotted in figure 8, where the difference curve has been multiplied by the photon energy $\hbar\omega$, as required by equation (3). In fact the numerical integration of the difference curve in the energy range of figure 8 (1.5–4.5 eV) yields zero within a few per cent of the total area. This shows that equation (3) is satisfied for the group of transitions S_1 and S_2 , clearly related to surface states, as also stressed by the high sensitivity to surface contamination. One may further notice that the line shape of the positive part of the difference spectrum is somehow reminiscent of the bulk dielectric function. This behaviour suggests that bulk states are also involved in the transitions. The fact that rule (3) is satisfied implies that electron(s) in the same initial state(s) is (are) involved in the transitions, exhausting its (their) oscillator strength in this range. A possible explanation is that peaks S_1 and S_2 (plus transitions above 3 eV) are related to the two types of dimers present on the surface [18]. This hypothesis is substantially in agreement with theoretical results [17].

As far as the main positive peaks at about 3 and 4.5 eV in figure 7 are concerned, which show a much lower sensitivity to oxygen contamination, one may notice that they mimic to a large extent the bulk dielectric function of GaAs. This suggests that this part of the spectrum is mainly due to bulk transitions modified by the surface and therefore it involves another group of electrons.

In principle, this group of electrons should also satisfy the sum rule given by equation (3). For this to be true the positive anisotropy shown in figure 7 should be compensated by a negative one, presumably at the energy of the last critical point near 7 eV.

4. Conclusions

The experimental results for Si(111)- 2×1 , Ge(111)- 2×1 and GaAs(001)- 2×4 show that the so-called sum rule of equation (3) is satisfied both for SDR and RAS. In addition, when (as in SDR) both the isotropic and anisotropic parts of the spectrum are detected, the sum rule allows the evaluation of the number n_{eff} of electrons per atom involved in the optical transitions. The presence of saturation (plateau) in $n_{\text{eff}}(\omega)$ indicates that a group of electrons have exhausted their oscillator strength. This kind of analysis has been applied to the Si and Ge 2×1 reconstructed surfaces, showing that the SDR spectrum up to 3.5 eV in Si and 2.5 eV in Ge is due essentially to dangling-bond transitions.

When rule (3) is apparently not satisfied, because of the limited range of measurements, it is nevertheless useful for a prediction of the lacking part of the spectrum.

Moreover, rule (3), together with the dependence of reflectivity upon oxidation, helps in elucidating the distinction between transitions associated to surface states and bulk states modified by the surface.

This analysis applied to GaAs(001)- 2×4 in the range around 3 eV has allowed us to attribute part of the spectrum to surface transitions associated to As dimers.

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