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# Reflectance-difference spectroscopy as a probe for semiconductor epitaxial growth monitoring

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#### ABSTRACT

We report on real-time reflectance-difference (RD) spectroscopic measurements carried out during the homoepitaxial grow of GaAs under As overpressures in the range from  $P_{As} = 6 \times 10^{-7} - 5 \times 10^{-6}$  Torr. We found that the time-dependent RD spectrum is described in terms of two basic line shapes. One of these components is associated to the orthorhombic surface strain due to surface reconstruction while the second one has been assigned to surface composition. Results reported in this paper render RD spectroscopy as a powerful tool for the real-time monitoring of surface strains and its interplay with surface composition during growth.

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#### 1. Introduction

Reflectance-difference spectroscopy (RDS) is a noninvasive, contrasting technique that suppresses the bulk isotropic component of the optical reflectance spectrum of cubic semiconductors enhancing its surface-associated component. Both its noninvasive character and surface specificity make RDS a very attractive tool for monitoring the epitaxial growth of zincblende semiconductors, as it was first reported by Aspnes and collaborators [1]. The interpretation of reflectance-difference (RD) spectra, nevertheless, poses some challenges as the surface may become anisotropic for a number of physical mechanisms [2], including surface electric fields [3],  $\alpha$  and  $\beta$  dislocations [4], surface reconstruction strains [5,6] and surface dimers [7,8]. At the same time, time-resolved RD spectra measured during epitaxial growth would lead to a great deal of information on the kinetics of epitaxial growth, provided we can resolve them into their different components and determine the time-evolution of such components during growth.

Previously we reported on real-time RDS of homoepitaxial GaAs (001) grown by molecular beam epitaxy (MBE) [9]. For the growth we employed a As overpressure ( $P_{As}$ ) of  $1 \times 10^{-6}$  which led to a rich surface reconstruction evolution as growth progressed as well as to

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http://dx.doi.org/10.1016/j.jcrysgro.2015.02.061 0022-0248/© 2015 Elsevier B.V. All rights reserved. considerable changes in RD spectrum line shape. We showed that the time-evolution of RD spectra during growth is well described in terms of two independent components, each with a specific physical origin. In this paper we report on the results of a study carried out within a range of As overpressures and demonstrate that our previous results can be extended to more general growth conditions.

#### 2. Experimental details and results

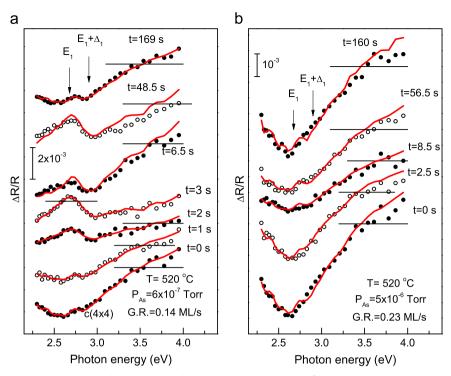
Epitaxial growth was carried out on (001) GaAs substrates in a solid-source MBE chamber (Riber 32P). Epitaxial growth was carried out under four As overpressures ( $P_{As}$ ) ranging from  $6 \times 10^{-7}$  to  $5 \times 10^{-6}$  Torr at a growth rates from 0.14 to 0.23 ML/s as determined from RHEED oscillations. Growth substrate temperature was 520 °C in all cases. Epitaxial growth was initiated/interrupted by opening/ closing the Ga shutter and lasted for about 45 s. Time-resolved spectroscopic RD measurements were performed with a rapid RD, 32-channel spectrometer attached to the epitaxial growth chamber. More details on the RD spectrometer are given elsewhere [10]. In order to correlate RD changes with surface reconstruction changes, RHEED patterns were acquired concurrently with RD spectra.

To prevent components associated to dislocations in the measured RD spectra, a 0.3  $\mu$ m thick GaAs buffer layer was grown prior to carrying out the experiments. Further, to avoid electro-optical contributions that may hinder the analysis of the RD line shapes, we did not intentionally dope the GaAs films.

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**Fig. 1.** Filled and open circles: representative time-resolved RD spectra during MBE GaAs homoepitaxy for two As overpressures. Panels (a) and (b) correspond to  $6 \times 10^{-7}$  Torr and  $5 \times 10^{-6}$  Torr, respectively. Growth temperature and growth rate are as indicated. Spectra have been displaced vertically for the sake of clarity with zeros as indicated with horizontal lines. Growth started at t=0 and was interrupted at t=44.5 s. Time shown next to each spectrum corresponds to time elapsed after starting growth. Lowermost spectra in both panels were measured just before starting growth, while uppermost spectra were measured long after closing the Ga shutter. Continuous lines correspond to line shapes synthesized in terms of two the basic line shape components  $S_1(E)$  and  $S_2(E)$  as discussed in the text.

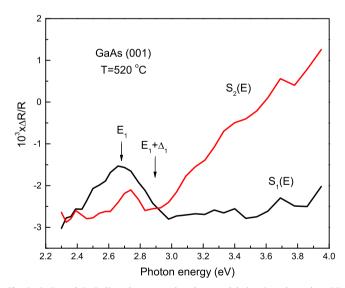
In Fig. 1 we show time-resolved RD spectra of GaAs homoepitaxy for (a)  $P_{As} = 6 \times 10^{-7}$  Torr and (b)  $P_{As} = 5 \times 10^{-6}$  Torr. Lowermost spectra in both cases correspond to GaAs surfaces under As flux, just before starting growth. For  $P_{As} = 6 \times 10^{-7}$  the evolution of the RD spectrum is rather involved. This is in correspondence with the evolution of the GaAs surface reconstruction which changes from  $c(4 \times 4)$  before starting growth, to  $(2 \times 4)$  after the deposition of about 0.5 ML GaAs, and to a Ga-rich  $(4 \times 2)$  phase after steady state growth is reached. In comparison, the RD spectrum evolution for  $P_{As} = 5 \times 10^{-6}$  Torr is simpler according to the less varied surface reconstruction changes. We note, nevertheless, that irrespective of  $P_{As}$  values, there is a change in RD spectrum line shape as growth progresses.

#### 3. RD line shape analysis

The rather complex evolution of RD spectra during epitaxial growth is indicative that they comprise at least two components, each one with its own time-evolution. We may further expect these two components to result from different physical processes. As reported previously [9], a Singular Value Decomposition (SVD) [11] analysis of the experimental, time-dependent RD spectra measured during MBE GaAs homoepitaxial growth shows that for  $P_{As} = 1 \times 10^{-6}$  the RD spectrum is written in terms of two independent components, allowing thus write

$$\frac{\Delta R}{R} = c_1(t)S_1(E) + c_2(t)S_2(E),$$
(1)

where  $\Delta R/R$  is the experimental RD spectrum,  $S_1(E)$  and  $S_2(E)$  are independent line shape components, each one associated to a specific physical mechanism, and  $c_1(t)$  and  $c_2(t)$  are coefficients that account for the time-evolution during growth of components  $S_1(E)$  and  $S_2(E)$ , respectively.



**Fig. 2.**  $S_1(E)$  and  $S_2(E)$  line shapes employed to model the time-dependent RD experimental spectra. These line shapes were obtained from a SVD analysis of time-dependent RD spectra measured during the MBE homoepitaxial growth of GaAs under a  $1 \times 10^{-6}$  Torr As overpressure.

Provided we have access to  $S_1(E)$  and  $S_2(E)$  line shapes, as well as to their physical interpretation, a great deal of information on epitaxial growth dynamics may be obtained from the fitting of Eq. (1) to realtime RD spectra. In this regard, we note that while the SVD analysis yields by construction orthogonal independent line shapes, in our case these line shapes may correspond to linear combinations of  $S_1(E)$  and  $S_2(E)$  and may have therefore a mixed physical origin.

Previously we took advantage of the substantial changes in RD line shape that occur during the homoepitaxial growth of GaAs Download English Version:

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