



# Reflectance measurements for real-time optical control of epitaxial growth

For epitaxial growth - like for any other thin-film growth process - it is essential to have real-time information about the actual growth rate. Optical reflectance is usually the method of choice because it gives direct access to the growing layers and can be implemented through a simple normal incidence view-port. However, information gained by reflectance measurements is not limited to growth rate. State-of-the-art spectroscopic reflectance systems give access to the layer composition and the surface roughness and also allow for full fingerprinting and control of very complex multi-layer structures such as lasers or filter devices.

Epitaxial growth is the basic step in manufacturing III-V-based optoelectronic and electronic devices, which significantly determines their performance. Therefore, a careful control of the epitaxial process is indispensable. In the last few years, optical sensors have become outstanding tools for performing such in-situ online control. The common working principle is based on the interaction of light with the growing thin films.

Optical reflectance is one of the methods that can be applied. During epitaxy, it enables to derive such characteristic properties of the growing layers as growth rate, layer thickness, composition of ternary compounds and surface roughness. Optical reflectance sensors are used for calibrating, monitoring and controlling growth processes and thereby improve significantly the productivity of MOCVD and MBE systems. In addition, reflectance measurements can also be used for calibration of absolute substrate temperature and for emissivity corrections of process pyrometers.

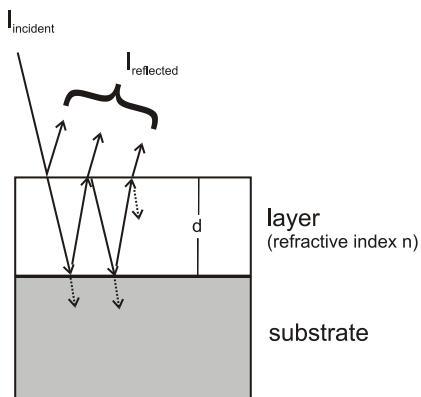
During reflectance measurements, light of a certain wavelength impinges on the layer under investigation and is partly reflected. The intensity of the reflected light is usually measured by a photo diode or a CCD array. The fraction of the intensity being reflected is called reflectance  $R$ . For a single interface between vacuum and a (bulk) material with refractive index  $n$  and extinction index  $k$  it is given by:

$$R = I_{\text{reflected}} / I_{\text{incident}} = (n-1)^2 + k^2 / (n+1)^2 + k^2$$

Both quantities  $n$  and  $k$  show dispersion, i.e. they depend on the wavelength of the incoming light. They also change with the composition of the layers and with the actual substrate temperature.

## Growth rate and layer thickness

During epitaxy, a convenient way to gain information from reflectance measurements is to take the so called reflectance-transients. In this case, the intensity of light reflected at the substrate-layer-system is measured at one single wavelength as a function of time. If the growing layer is (at least partially) transparent at the wavelength of the incoming light, these transients show an intensity-modulation, related to interference effects. The period of the modulation can be used to measure the thickness and growth rate of the layer.



**Fig. 1:** Reflection of light at a transparent layer growing on top of an absorbing substrate. The intensity of the reflected light is given by the superposition of all reflected beams.

As the incoming light is only partly reflected from the surface, another part penetrates into the layer (see Fig.1). Then it is partly reflected at the interface between layer and substrate. This reflected beam travels back to the layer surface, where the same process repeats: the beam is partly reflected and partly leaves the sample. The overall intensity of the reflected light is then given by the superposition of all reflected beams. As there is a phase difference between the single beams, constructive or destructive interference occurs, leading to an intensity-modulation of the reflected light, commonly known as Fabry-Pérot oscillations.

The phase difference and thus the intensity of the reflected light depend upon the thickness of the growing layer as well as on the optical constants of the materials and the wavelength of the light. For normal incidence, constructive interference and maximum reflectance occur if the path difference between two beams is equal to an even number of the half wavelength:

$$2nd = m\lambda$$

For destructive interference and minimum reflectance the path difference is equal to an odd number of the half wavelength:

$$2nd = (m + \frac{1}{2}) \lambda$$

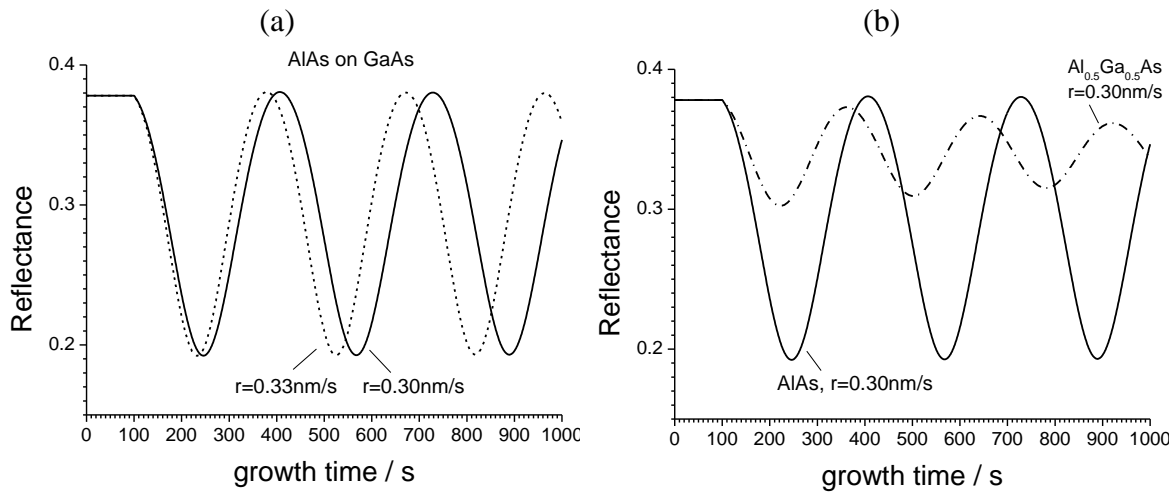
(with n: refractive index, d: thickness of the layer, m: even number)

Thus, by measuring Fabry-Pérot oscillations, the layer thickness can be derived from the intensity-transients of the reflected light. As during epitaxy the layer thickness increases continuously with time t, also the growth rate  $r = d/t$  can be estimated:

$$\text{maximum reflectance: } r \cdot t = m\lambda/2n \quad \text{minimum reflectance: } r \cdot t = (m + \frac{1}{2}) \lambda / 2n$$

Even these simple considerations, which only take reflectance minima and maxima into account, show how different process parameters influence the reflectance-transients (of course, a detailed evaluation of the complete shape of the reflectance transient leads to much more accurate quantitative results):

- high growth rates yield a short period in intensity-modulation, whereas low rates lead to long periods
- optically thicker materials (lower-energy band-gap semiconductors) lead to shorter periodicity
- optically thinner materials (wider band-gap semiconductors) lead to a slower modulation.
- changes of the layer composition (and thus n and k) lead to a change of  $R_{\max}$  and  $R_{\min}$  in addition to the thickness dependence of the signal. This is shown in figure 2 for the growth of AlGaAs on GaAs.

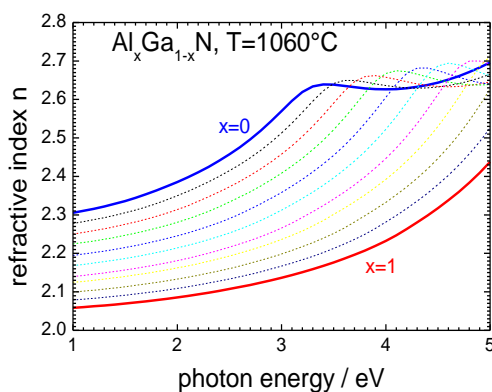


**Fig. 2:** Growth of AlAs and AlGaAs on GaAs at  $T = 582^\circ\text{C}$ : (a) For different growth rates of AlAs the amplitude of the reflectance oscillations remains the same, but the periodicity is shorter for the higher growth rate. (b) For different compositions of AlGaAs the amplitude of the reflectance oscillations changes completely (due to the different refractive index) while the periodicity is only slightly modified.

### Composition of ternary materials

For ternary materials this change in modulation amplitude, caused by changes of  $n$  and  $k$ , additionally allows to determine the composition of the growing layer. Therefore, the refractive index  $n$  of the growing layer is derived from the amplitude of the Fabry-Perot-oscillations in the measured reflectance transient.

The composition is then found by comparing this value with the refractive indices of a database that contains the values of the respective compound for the full range of compositions and all relevant growth temperature. As an example, figure 3 shows a database for  $\text{Al}_x\text{Ga}_{1-x}\text{N}$ . For optimum sensitivity to both composition and growth rate, reflectance measurements at more than one wavelength of the incoming light are necessary.

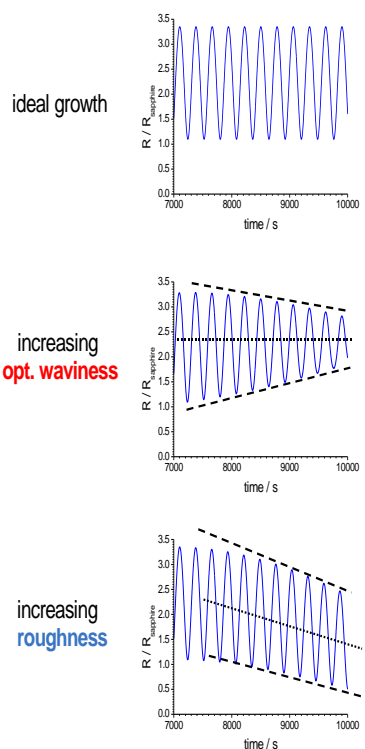


**Fig. 3:** Refractive index of AlGa<sub>1-x</sub>N at a growth temperature of  $1060^\circ\text{C}$  (valid for fully relaxed, unstrained AlGa<sub>1-x</sub>N). Data bases like this are used for on-line determination of the refractive index  $n$  of ternary compounds.

## Surface quality

Changes in the reflected intensity also arise from other effects than those discussed so far. Of course, there is a decrease in interference amplitude if the growing layer absorbs the wavelength of incoming light, i.e.  $k_{\text{layer}} \neq 0$ . In this case, the intensity is attenuated according to  $e^{-k \cdot 2\pi d / \lambda}$ . With the increasing layer thickness, the intensity-maxima and -minima approach the constant value of reflectance characteristic for the layer surface only.

Furthermore, surface roughness and optical waviness are important parameters that influence the reflectance transients. Surface roughness refers to thickness fluctuations on a nanometer scale. Optical waviness appears on a micron scale. It results from local fluctuations in the layer thickness or



**Fig. 4:** The influence of surface quality on the intensity of the reflected light for GaN growing on sapphire. Increasing optical waviness (i.e. local fluctuations in thickness and/or refractive index) and increasing surface roughness changes reflectance transients in a different way.

for the reflectance of the substrate-back-side. Semiconductor wafers usually have very rough backsides. Therefore, in reflectance measurements they behave like absorbing substrates, even at a wavelength where the substrate is transparent (just scattering away the transmitted light). However, this is not true for both-side polished wafers. In this case, appropriate optical models must be applied when analyzing in-situ reflectance measurements.

in the refractive index. The latter is caused by local changes in the composition of the material or by strain effects. Surface roughness and optical waviness influence the measured reflectance differently (see Fig. 4). Consequently, by using more comprehensive layer models than the simple approach in Fig. 1, it is also possible to gain information about surface roughness, local thickness and refractive index fluctuations from reflectance measurements.

## Avoiding artefacts during in-situ reflectance measurements

**Window depositions:** In order to rule out an influence of apparatus artefacts during the reflectance measurement, the measured intensities should be normalized to a well known reference value measured in-situ under the same conditions. LayTec sensors always normalize to the appropriate (usually high-temperature) reflectance of the substrate. As a result, artefacts (as, for example, the changing transmittance of windows) are avoided.

**Transparent substrates:** For transparent substrates, one must correct the reflectance data