The importance to reveal buried interfaces in the semiconductor heterostructure devices

Yoshikazu Takeda\(^1\) and Masao Tabuchi\(^2\)

\(^1\)Department of Crystalline Materials Science, Graduate School of Engineering, Nagoya University, Nagoya 464-8603, Japan

\(^2\)Venture Business Laboratory, Nagoya University, Nagoya 464-8603, Japan

takeda@numse.nagoya-u.ac.jp

Abstract. Even though several \textit{in-situ} monitoring techniques exist and are quite useful to understand the growth processes in MBE or MOVPE, we also need a technique to reveal the buried interfaces along which carriers are transported and recombine to emit light. The interface is modified during the capping (overgrowth) and also during the device fabrication processes after growth. We need to correlate the interface structures in the devices and the device performances. The only technique we have at present is the X-ray CTR scattering measurements. We discuss the limits of the \textit{in-situ} monitoring and the necessity to reveal the buried interfaces non-destructively, either \textit{in-situ} or \textit{ex-situ}.

1. Introduction

We are interested in a nondestructive and \textit{ex-situ} measurements of heterostructures (a basic part of device structures) where carriers run and recombine in the device operations.

In the MBE (molecular beam epitaxy) we use an electron beam (RHEED) as a tool to observe surface structures in the atomistic level. However, the active layer is capped to form heterostructures and we do not know what happens during and after the capping. In the MOVPE (metalorganic vapor phase epitaxy), optical measurements (reflectivity and ellipsometry) are often used. These techniques are also sensitive to the top surface of the growing crystal.

TEM (transmission electron microscopy) is a powerful technique to investigate the atomistic structure of materials. However, the inevitable process of thinning often causes unpredictable damages and modification of the structures. In the strained layers, the strain is released due to the thinning and then the real buried structure disappears.

Thus, the nondestructive and atomistic measurements and analyses are very important to understand what the real structure is, what the relationship between structures and device performances is, and then to control the heterostructures.

The measurement of X-ray reflectivity around the Bragg point gives X-ray CTR (Crystal Truncation Rod) [1-3]. It is so called because the measured X-ray intensity distribution looks like a “rod” due to the termination of the periodicity (\textit{i.e.}, the surface) of a crystal. CTR may be used hereafter since it is short to express. We have used this CTR extensively to analyze buried heterostructures since the rod shape (reflectivity) is largely modified not only due to the surface structures but also due to the layer structures underneath the surface.
We describe in this paper the limits of the in-situ measurements and necessity of the measurements on the buried interfaces, either in-situ or ex-situ, during or after the growth process of semiconductor heterostructures.

2. Importance of hetero-interfaces

Figure 1 shows a schematic drawing of a double heterostructure (or a single quantum well, depending on the thickness of the GaInAs layer) that is used in such optical devices as double heterostructure lasers, quantum well lasers and high efficiency LEDs, and in such transport devices as HEMTs and HBTs.

In the optical devices, the well thickness and the barrier height directly change the emission wavelength and a fluctuation of them changes the half width of the spectra.

In the HEMTs, carriers are transported along the hetero-interface. The roughness of the interface is one of the major scattering mechanisms that limit the carrier mobility and drift velocity. In the HBTs, electrons transported from emitter to base are strongly influenced by the notch and/or dip at the interface.

Even in the basic research on the band structure at the interface, it is quite often assumed that the hetero-structures are fabricated as designed since there are no techniques to reveal the atomistic structure of the interfaces.

TEM has been used to investigate the atomistic structure of materials. However, the inevitable process of thinning often causes unpredictable damages and modification of the structures. In the strained layers, the strain is released due to the thinning and the real buried structure disappears. From the TEM images it is difficult to evaluate quantitatively the composition grading around the interface.

Therefore, we need to develop a technique to observe and reconstruct the real structure of the hetero-interfaces quantitatively in the atomistic scale.

3. in-situ measurements

3.1. RHEED in MBE.

In the MBE growth, where the reactor pressure is better than $10^{-3}$ Pa, RHEED is a common equipment installed in most MBE reactors to observe the surface structures before and during the growth process. The RHEED intensities oscillate due to the formation of two-dimensional islands (Fig. 2a).

Fig. 2 (a) Change of surface roughness due to coverage by 2D nucleation. (b) RHEED intensity oscillation due to the change of surface roughness and recovery with coverage. The intensity decreases due to roughening of the surface caused by 2D nucleation on 2D islands.
during growth, and to monitor the growth speed to control the layer thickness [4-5]. The oscillation in intensity of the specular beam is understood to come from the change of the surface morphology, i.e., from flat to rough due to 2D nucleation and then back to flat as shown in Fig. 2 (a). This simple understanding is widely accepted and used to control the thickness of the growing layer by counting the number (or time) of the oscillation cycle.

However, as shown in Fig. 2 (b), the oscillation intensity decreases with the thickness since the surface usually does not go back to a perfect flat. Therefore, this technique is not effective at an arbitrary thickness.

3.2. Optical probe in MOVPE. In the MOVPE, the electron beam cannot be used since the reactor pressure is atmospheric or not lower than 1/10 of atmospheric pressure. The optical beam is commonly used to probe the growing surface. Reflectivity, absorption, and ellipsometry are used.

In Figure 3 a schematic drawing of the SPA (Surface Photo-absorption) technique where the angle $\phi$ of the p-polarized incident light is adjusted to the Brewster angle of the substrate materials [6]. If some material that has a different refractive index covers the substrate surface, a signal should be detected.

Clear oscillation is observed in the growth of InAs on InP and InP on InAs as shown in Fig. 4. In this case, not only the oscillation but also a drastic change of the intensity is observed. The change is considered to reflect the different materials, i.e., InP and InAs. From this change it may be possible to analyze the composition at the interfaces. However, a possible change of the InAs well layer and the interface between InAs and InP after, for example 60 s, is not traceable from this figure and a further analysis was not conducted. To reveal the interface structures after a further growth or after other processes of the device fabrication, other techniques are required.

Similar oscillations are observed in the reflectivity measurements and ellipsometry measurements.

4. Change of interface structures after overgrowth
4.1. InP/GaInAs/InP. We successfully revealed the hetero-interface structures of an InP/GaInAs/InP single quantum well with a thin GaInAs well layer, grown by MOVPE, using X-ray
CTR measurements ex-situ and analysis [7-10].

Figure 5 illustrates the CTR scattering intensity profile from InP wafer (a) and that from InP/Ga$_{0.47}$In$_{0.53}$As(10 ML)/InP single quantum well with well-thickness of 10 ML (b). In (b) the profile is largely modulated due to the heterostructure. The modulated structure contains information such as the InP cap layer thickness, the surface roughness of InP top surface, the composition of GaInAs, the thickness of GaInAs, and the composition grading at the interfaces. The change of the profile is schematically illustrated in Fig. 6 caused by several physical properties of heterostructures.

The CTR scattering measurements have extensively been applied to many kinds of heterostructures.

Fig. 5 (a) shows a model calculation of CTR intensity distribution profile for InP wafer and (b) shows that for InP/Ga$_{0.47}$In$_{0.53}$As/InP. The profile is largely modulated due to the heterostructure buried underneath InP.

Fig. 6 Change of the CTR shape for a perfectly matched InP/GaInAs(2 ML)/InP (center), due to strain (top left), surface roughness (top right), GaInAs thickness (bottom left), and interface roughness (bottom right).
One of the obtained examples of composition profiles is shown in Fig. 7. The bottom interface (GaInAs on InP) is abrupt, but both Ga and As composition gradings near the upper interface (InP on GaInAs) are considerable. This indicates a square well potential is not applicable to this single quantum well.

4.2. Change of dots before capping. Figure 8 (a) shows an AFM image of InAsP quantum dots grown by MOVPE at 430°C. When they are annealed in the TBP+TBAs ambient during the temperature change from 430°C to 600°C, smaller dots coalesce to yield larger dots as shown in Fig. 8 (b). The dot size in Fig. 8 (b) is too large and usually misfit dislocations are introduced into the dots, which work as nonradiative recombination centers. The temperature raise was conducted to cap the dots with a better quality GaInP layer. However, Fig. 8 indicates that a capping layer to freeze the dots at the smaller size is necessary before raising the temperature to grow a thicker layer for a barrier layer.

4.3. Photoluminescence with different caps. The InAsP dots were capped by different materials at 430°C and after raising the temperature to 600°C the GaInP layer was deposited. As sown in Fig. 9 no clear peaks from dots were observed from any samples of the low-temperature capping with GaInP, InP, or GaAs. The only peak was observed from the sample without capping.

Though the measurements of the interface structures are not conducted for these samples, it is clear that the disappearance of the PL is due the interface and the capping layer. Quite a delicate control of the interface formation is required in this case.

5. Discussion

In-situ monitoring of growth is certainly necessary to understand how the initial growth starts and how high the growth speed is. However, in-situ monitoring is not useful enough to observe the interface structures after overgrowth, i.e. the buried interface structure, as described in Section 4. The active regions of semiconductor devices are always buried. We do need a technique that reveals the real structure of the hetero-interfaces and quantum wells that are typical examples of buried interfaces in semiconductors.

The only technique we know is the X-ray CTR scattering measurements and have demonstrated its usefulness. One of the difficulties of the CTR is a complicated analysis because quite a few numbers of parameters are deduced from a curve-fitting. Another problem is that the sample structure is almost limited to a single quantum well, though the technique can be applied to a further complicated layer...
structures. The parameters are too many and it is practically impossible to conduct the curve-fitting for more complicated layer structures.

6. Conclusion
We described in this paper the limits of the in-situ measurements and necessity of the measurements on the buried interfaces, either in-situ or ex-situ, during or after the growth process of semiconductor heterostructures.

We have successfully used the X-ray CTR scattering technique extensively to analyze buried heterostructures since the rod shape (reflectivity) is largely modified not only due to the surface structures but also due to the layer structures underneath the surface.

We described also the limits of the X-ray CTR scattering technique.

Acknowledgements
The authors thank S. Kawamura for his sample preparation of dots. This work was supported in part by the Grant-in-Aid for Scientific Research (S) #18106001 and (B) #19360006 from the Japan Society for the Promotion of Science.

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