Defect Creation in InGaAs/GaAs Multiple Quantum Wells – I. Structural Properties

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1. Introduction

Defect creation as an inherent process in epitaxial growth has impeded the realization and practical application of many electronic and optoelectronic devices. Physical properties and hence final device performance depend critically on the crystal quality of the epitaxial structure [1-5]. In multiple-quantum-well (MQW) solar cells, which appear to offer a superior approach compared to their homojunction counterparts [6-8], crystal quality deterioration still inhibits tapping of their full potential [9].

We report here on investigations of defect creation in InGaAs/GaAs MQWs and describe new findings about the main structural features, such as type, density, and spatial distribution of crystal defects, as dependent on growth conditions. Observations are interpreted in the framework of a model describing defect creation as a multi-stage process, starting from accommodation of initial elastic stress and point defect (PD) creation on the growth front, followed by inward diffusion and accumulation at pre-dislocation clusters, and finally transformation to extended defects in the volume of epitaxial structure [10-12]. The crystalline properties of these structures as dependent on growth conditions are related to optical properties in the companion paper [13].

2. Experimental Details

The In_xGa_{1-x}As/GaAs MQW samples were grown by molecular beam epitaxy (MBE) on undoped (001)-oriented GaAs-wafers. MQW growth was preceded by a deoxidation step at 580°C, followed by the growth of a 250-nm-thick GaAs buffer and AlGaAs cladding layers at a deposition temperature of 595°C. Completion of deoxidation and the surface morphology transformation were monitored by *in situ* reflection-high-energy electron diffraction (RHEED). Growth rates of MQW structures were chosen as ~0.1 nm/sec and 0.08 nm/sec for InGaAs and GaAs layers, respectively, adjusted ~20% In composition.

Three sample sets were grown in order to allow defect creation in the InGaAs MQWs to be studied systematically as a function of deposition temperature, total MQW thickness, and average elastic strain.

- (I) Four samples, MQW period 8+8 nm (well and barrier thickness), grown at various deposition temperatures between 475-520°C, with ×5 periodicity;
- (II) Two samples, MQW period 8+8 nm, grown at 490°C, with periodicity ×5 and ×10;

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- (III) Three structures, grown at 490°C, periodicity $\times 10$, with MQW periods 4+8 nm, 8+8 nm, and 8+16 nm

These grouped structures were then analyzed to determine the influence of the growth parameters on defect creation. Crystalline properties were investigated with a PANalytical X'Pert Pro MRD high-resolution x-ray diffractometer (HRXRD) in double- and triple-crystal (DC and TC) modes for acquisition of DC and TC ω -2 θ and TC ω rocking curves (RC). In TC geometry, an analyzer crystal in front of the x-ray detector is used to spatially separate coherent and diffuse scattered radiation. Shape, position, and full-width-at-half-maximum (FWHM) of diffraction peaks in respective RCs were examined to obtain structural information about the substrate and the epitaxial layers to suggest the type, density, and spatial location of the crystal defects resulting from epitaxial growth [10-12,14-16]. Symmetrical (004) and asymmetrical (224) ω -2 θ / ω TC reciprocal space maps (RSM) revealed the spatial distribution of diffuse scattered radiation, and hence showed structural features of the created defects. Asymmetric RSMs enable estimates to be made of the extent of relaxation of initial elastic stress, and correct specification of the preferred type of crystal defects [10-12].

Plan-view and cross-section transmission electron micrographs were compared to results from RHEED and HRXRD characterization.

3. Results and Discussion

3.1 Deterioration regimes at different deposition temperature

Changes in crystal deterioration between deposition temperatures of 475°C and 520°C are observed in DC ω -2 θ scans (Fig. 1). These curves are dominated by (004) GaAs diffraction and MQW superlattice (SL) peaks. In the curves for samples grown at temperatures $\leq 505^\circ\text{C}$, coinciding angular positions of the SL peaks indicate the target In composition of $\sim 20\%$ and MQW period of ~ 16 nm. Interference patterns with clear SL peaks and interference fringes over a wide angular range correspond to a total MQW thickness of 80 nm and low deterioration of vertical coherence. RSMs reveal low density of diffuse scattered radiation and zero relaxation of the initial elastic stress. In cubic lattice heterostructures, this indicates low density of 60° dislocation loops (DLs) as the primary extended defects created in the volume of the epitaxial structures and on the interface(s), along with a lack of closing-edge segments (Lomer dislocations) formed where the 60° DLs intersect in the volume of the MQWs [10-12].

In contrast, the DC ω -2 θ scan of structure $T_{\text{dep}}=520^\circ\text{C}$ reveals a lower In composition of $\sim 18.5\%$, as the SL interference pattern is shifted towards the substrate peak. Further, the SL peaks and the interference fringes are diffuse as a result of inferior vertical coherence. Quantitative results obtained from the FWHM of MQW peaks in TC ω -2 θ scans demonstrate diminished vertical coherence ~ 75 nm (Table 1), while samples $T_{\text{dep}} \leq 505^\circ\text{C}$ exhibit larger vertical coherence length ~ 97 nm.

TC ω RCs (Fig. 2) are used to specify type, density, and spatial distribution of preferred defects. The structures grown at $T_{\text{dep}} \leq 505^\circ\text{C}$ (Fig. 2a) reveal ω RCs with a narrow central coherent peak on wider base and a clear interference wing on the tails indicating lateral ordering of the secondary 60° DLs on the MQW/substrate interface [10-12,14]. On the contrary, weak lateral ordering of small defects occurs at elevated temperature, as indicated by the broad range of scattering contributions in the respective TC ω RC (Fig. 2b). Azimuthal asymmetry observed for the lower growth temperatures is not present in this structure. The analysis of shape and linewidths in TC ω RCs allowed estimates to be made of the densities of 60° DLs as listed in Table 1. The density of both primary and secondary 60° DLs decreased with increasing deposition temperature until a minimum ($\sim 3 \times 10^5 \text{ cm}^{-2}$ and $\sim 1.5 \times 10^7 \text{ cm}^{-2}$ for primary and

secondary DLs, respectively) is reached for a growth temperature of 505°C. Diffuse scattering on the tails of the ω RCs caused by these loops is also minimal for this sample.

Symmetric and asymmetric RSMs (not shown) confirm the differences in crystal quality deterioration. While symmetrical RSMs for structures $T_{\text{dep}} \leq 505^\circ\text{C}$ reveal narrow diffraction spots connected by a streaky truncation rod due to high vertical coherence of entire MQW structure, wide areas of diffuse scattering are observed in the RSM of the structure grown at 520°C. The latter indicates deterioration of crystallinity with a broad range of length scales that is attributed to InGaAs decomposition leading to reduced In composition ($\sim 18.5\%$) and the creation of small scattering objects like In clusters. Asymmetrical RSMs with coinciding Q_x diffraction vector for (224) GaAs and InGaAs SL spots indicate a lack of initial stress relaxation for structures grown at $\leq 505^\circ\text{C}$ because primary DLs having their bottom edge segment in the volume of the substrate do not contribute to relaxation. Likewise, the density of secondary DLs is not sufficient to initiate relaxation. Asymmetrical RSMs in which the position of substrate and InGaAs SL spots causes specific Q_x gap ($T_{\text{dep}} = 520^\circ\text{C}$), indicate an initial stage of stress relaxation ($\sim 2\text{-}3\%$).

Cross-section transmission electron microscopy (TEM) of the structure grown at 520°C revealed a structural instability in the MQW structure with regard to the growth of morphological perturbations. In contrast to the other three samples, the MQW interfaces are not smooth, but show lateral thickness undulations with a period of ~ 100 nm (Fig. 3). This type of deterioration is attributed to the structural instability caused by the elevated growth temperature. This growth interval 505-520°C resembles the critical temperature (surface diffusivity) predicted by [17] based on a theoretical stability analysis of strained-layer superlattice epitaxial growth. In this critical range, MQW growth is supposed to transition from a stable to an unstable regime, characterized by the lateral modulations in layer thickness.

Structural analysis identified the deposition temperature of 505°C as the optimum for low deposition rate ($\sim 0.1 - 0.15$ nm/sec) epitaxial growth of the $\text{In}_{0.2}\text{Ga}_{0.8}\text{As}$ MQWs. Further, two different regimes of crystal deterioration were found. At the optimal growth temperature and below, primary and secondary 60° DLs with pronounced lateral ordering served as the primary crystal defects that stabilize initially deteriorated epitaxial growth. DL densities decrease with increasing deposition temperature up to the optimum. Exceeding this temperature, a different type of deterioration, characterized by a range of lateral length scales and diminished vertical coherence is created. InGaAs decomposition as a material instability occurs at elevated growth temperature and a structural instability is manifested in lateral layer undulations.

An In composition $\sim 20\%$ was chosen for this particular study. However, there is possibly a different optimal temperature for each In composition and deposition rate with highest crystallinity and two distinct regimes of primary crystal deterioration below and above this temperature.

3.2 Effect of MQW periodicity

The increase of MQW periodicity from $\times 5$ to $\times 10$, for a fixed growth temperature of 490°C and MQW period of $8+8$ nm, is characterized by greater deterioration of crystal quality. Whereas the vertical coherence length of the $\times 5$ structure (~ 98 nm) resembles its physical thickness (~ 80 nm), the $\times 10$ structure reveals vertical coherence (~ 110 nm) in comparison to MQW total physical thickness (~ 160 nm). This reduction results from an increased density of secondary DLs, their intersection, and creation of coherency-deteriorating edge segments (Lomer dislocations) in the volume of the SL.

TC ω RCs collected around the SL peaks reveal a strong decrease in coherent scattering and a prominent increase of diffuse scattering on the tail of the RC as the total MQW thickness is doubled. The increase in diffuse scattering is related to the increase in total density of secondary 60° DLs from

$\sim 1.6 \times 10^7 \text{ cm}^{-2} (\times 5)$ to $\sim 6 \times 10^8 \text{ cm}^{-2} (\times 10)$ and augmented formation of edge segments resulting from DL intersection in the volume of MQW. The lateral coherence of the epitaxial layers is thus reduced.

Enhanced creation of secondary DLs and reduced vertical and lateral coherence are visible in the (004) RSMs (Fig. 4). While the narrow SL spots of the $\times 5$ MQW structure (Fig. 4a) are connected by a continuous narrow surface truncation rod due to high spatial coherence, Fig. 4b shows a broken truncation rod and diffuse SL spots visually elongated in the lateral direction.

The increase in secondary DL density shifts creation of next “tertiary” DLs close to the interface. This type of 60 DLs originates from single-atom pre-dislocation clusters, which gradually occupy vacant positions on the interface (minimal distance between these loops is $\approx |b| / \epsilon_{xx}$, where $|b|$ is the modulus of the Burgers vector and ϵ_{xx} the initial elastic strain). These positions became available between already created primary and secondary DL extraplanes, which, diminishing atomic migration on the growth front, enable the creation of single-atom pre-dislocation clusters. Single-atom size of these pre-dislocation clusters induces secondary elastic stress to create DLs close to the interface and finally onset relaxation of the initial elastic stress [12], but insufficient to let created dislocation core glide into the substrate. In fact, the compressively strained SL is fully stressed in the $\times 5$ structure, whereas a relaxation of $\sim 12\text{-}15\%$ is observed in the (224) RSM for the $\times 10$ structure.

Contrasting these two samples with different MQW periodicity (total thickness), while T_{dep} and MQW period were kept fixed, revealed a cumulative thickness-related deterioration as the elastic strain is accommodated during epitaxial growth by the gradual occupation of pre-dislocation positions on the interface(s).

3.3 Effect of MQW period

Growth of $\times 10$ -periodicity MQW structures was carried out at a common deposition temperature. Starting from the MQW period $8+8 \text{ nm}$ (well and barrier widths), as used in all structures discussed so far, the thickness of the strained well layers was reduced to 4 nm . In a second approach, the strained layer thickness was kept at 8 nm , while the unstrained barrier width was doubled to 16 nm . This set of samples allows an evaluation of how changes in the total incorporated elastic strain affect and possibly mitigate defect creation related to lattice-mismatched epitaxial growth.

The sample with the lowest MQW period of $4+8 \text{ nm}$ revealed best crystal quality with a sharp coherent SL peak in the TC ω RC (Fig. 5). FWHM of the central and diffuse peaks of symmetric RCs allowed estimates to be made of the density of primary and secondary DLs of $\sim 5\text{-}7.5 \times 10^4 \text{ cm}^{-2}$ and $\sim 1 \times 10^7 \text{ cm}^{-2}$, respectively. From the linewidth of the SL peaks in the TC ω -2 θ scans, the vertical coherence length of the MQW was calculated to be $\sim 120 \text{ nm}$, which coincides with the physical thickness of the structure. Symmetrical RSM (Fig. 6a) revealed SL spots elongated along the vertical diffraction vector forming a faint surface truncation rod. The narrow lateral extent of diffraction spots correlates with a weak diffuse scattering contribution in TC ω RC. Total density of the primary and secondary DLs was insufficient to onset noticeable creation of tertiary DLs and relaxation of the initial elastic stress. The growth surface, deteriorated by InGaAs deposition was “healed” by barrier GaAs layers, diminishing the depth of dislocation trenches, the main deteriorating factor on the growth surface [11]. Both effects terminated the defect creation at an early stage, individually for each SL pair (well and barrier), as less strain needed to be accommodated and fewer surface migration obstacles to overcome.

In contrast, the samples with increased MQW periods of $8+16 \text{ nm}$ and $8+8 \text{ nm}$ with thicker strained layers showed reduced crystal quality. Coherent scattering was considerably weakened and diffuse contributions were enhanced, as apparent from the TC ω RC (Fig. 5). The structure with $8+16 \text{ nm}$ demonstrates an intermediate stage of crystal perfection, between those observed in the $4+8 \text{ nm}$ and the

8+8 nm structures. For [110] azimuth direction, coherent scattering from the MQW structure is rather intense and a primary DL density of $\sim 7\text{--}8 \times 10^5 \text{ cm}^{-2}$ is estimated from the FWHM of this peak (Fig. 5). Visual azimuthal asymmetry of the ω RCs for [1-10] direction corresponds to the anisotropic spatial distribution of extended defects. This distribution results in secondary DL density estimates differing by about an order of magnitude, namely of $\sim 1.3 \times 10^7 \text{ cm}^{-2}$ and $1.7 \times 10^8 \text{ cm}^{-2}$ for [110] and [1-10] azimuths, respectively. The anisotropic spatial distribution of secondary DLs is also confirmed by the different degree of relaxation of the strained layers. Whereas the lower DL density along [110] and the respective number of tertiary DLs on the interface is insufficient to initiate the relaxation of initial elastic stress, higher DL density along [1-10] led to creation of a sufficient number of DLs close to the epitaxial interfaces for a relaxation of $\sim 5\%$ in the [1-10] direction. This corresponds to the linear density of tertiary DLs at the bottom interface of $\sim 1.8 \times 10^4 \text{ cm}^{-1}$.

Despite the increased total number of primary and secondary DLs in the volume, the calculated vertical coherence length (based on the FWHM of 0SL peak in ω -2 θ scans) is close to the physical thickness (240 nm), as for the 4+8 nm structure. The unstrained barrier layers of doubled thickness retained the crystalline properties of the MQW structures. The barrier layers, serving as a defect protector, helped to realize defect “healing” after a single deteriorated well layer is grown. During growth of further strained-well layers, these were exposed to fewer extended defects originating from the previous strained layers.

In the 8+8 nm structure, the density of secondary DLs in the volume increased to $\sim 6 \times 10^8 \text{ cm}^{-2}$, which is more than an order of magnitude higher than in the 4+8 nm structure and about half an order of magnitude higher than for the 8+16 nm structure. The spatial distribution of extended defects had no azimuthal asymmetry. The vertical coherence length was estimated as $\sim 110 \text{ nm}$, which is $\sim 30\%$ below the 160 nm physical thickness of the MQW structure. The increased thickness of the strained layers led to higher deterioration of every single quantum well as larger elastic strain had to be accommodated. In turn, more secondary and tertiary DLs were created and the density of closing-edge segments (Lomer dislocations) increased. The latter was manifested in reduced vertical coherence length as compared to the physical thickness and relaxation of the initial elastic stress.

In direct comparison, the decrease of the strained layer width (8+8 nm vs. 4+8 nm) reduced the crystalline deterioration more effectively than increasing the unstrained layer thickness (8+8 nm vs. 8+16 nm), as defect creation was terminated at an earlier stage. For a multi-stage defect creation process, this early stage is characterized by the creation of primary DLs as the main deterioration cause. Increasing the unstrained layer thickness used as “healing” element, resulted in reductions in the total amount of PDs created on the growth front and hence to the lower density of secondary DLs. The likelihood of tertiary DL and Lomer dislocation formation close to substrate/epi-structure interface and in the MQW volume was hence reduced. The latter explains why the extent of stress relaxation decreased from $\sim 15\%$ to 0 – 5%.

4. Discussion and Conclusions

The experimental results emphasize that defect creation in epitaxial growth is a complicated multi-stage process. All types of 60° DLs originate from PDs, created at the growth front. Upon inward diffusion of PDs they accumulate in pre-dislocation clusters and finally transform to DLs under secondary elastic stress, induced by these clusters. In a continuous process, primary and secondary DLs reconstruct the growth surface for the generation of tertiary DLs. Creation of these DLs stabilizes the growth procedure.

Three sets of InGaAs/GaAs MQW structures were grown by MBE and characterized by HRXRD and TEM with regard to their crystal quality. Probing the influence of the deposition temperature revealed two

distinct deterioration regimes, separated by the optimal growth temperature of $\sim 505^{\circ}\text{C}$. Deposition below this temperature resulted in the creation of primary and secondary 60° DLs as the main deterioration type, with a decreasing density trend as the temperature was increased and a minimum at the optimal temperature. Elevated temperature led to InGaAs decomposition and a structural instability of the MQW growth, forming laterally undulated MQW layers and interfaces.

Increasing the periodicity and total thickness of the MQWs revealed a cumulative deteriorating effect. The intersection of already created DLs for continued MQW growth increased the number of edge segments created at substrate/epi-structure interface and in the volume of the MQWs. The former led to the onset of relaxation of initial elastic stress, while the latter led to deterioration of the spatial coherence.

Three distinct stages of defect creation were obtained by varying the MQW period. While a diminished thickness of the QW layers (4+8 nm structure) revealed primary DLs as the main type of deterioration, the 8+16 nm structure led to enhanced creation of secondary DLs representing the succeeding deterioration stage. Closing-edge segments were formed at a density on the substrate/MQW interface not sufficient for noticeable stress relaxation. The 8+8 nm structure represented an even more advanced stage of defect creation in which the creation of tertiary DLs on the interface, and most importantly, their intersection in the volume of MQW and the location of closing-edge segments close to epitaxial interfaces were sufficient to initiate relaxation of elastic stress.

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Table 1: Structural parameters as obtained from high-resolution x-ray diffraction around (004) InGaAs/GaAs MQW peaks.

T_{dep} [°C]	475	490	505	520
FWHM TC ω-2θ (0SL peak) [arc sec]	176	174	176	228
FWHM TC ω RC (-1SL peak) [arc sec]	12.2	11.2	9.4	10.1
$L_{\text{vert.co.}}$ (MQW structure) [nm]	97	98	97	75
$N_{\text{primary DLs}}$ [cm⁻²]	5.5×10^5	4.3×10^5	3.0×10^5	3.7×10^5
$N_{\text{secondary DLs}}$ [cm⁻²]	2.7×10^7	1.6×10^7	1.0×10^7	1.2×10^7
Relaxation [%]	0	0	0	2-3

All figures single column fitting images.

Fig. 1: DC ω - 2θ scans in (004) reflection of four $\times 5$ MQW structures, period 8+8 nm (well + barrier width) grown at different temperatures. SL peaks resultant from MQW periodicity and fast interference fringes related to total MQW thickness are clear for structures $T_{\text{dep}} \leq 505^\circ\text{C}$. Scan for the structure $T_{\text{dep}} = 520^\circ\text{C}$ demonstrates decrease of In composition and reduced vertical coherence of the MQW.

Fig. 2: TC ω RC in (004) reflection on -1SL of structure grown at: (a) optimal T_{dep} and (b) elevated temperature. Pronounced interference wings on the curve tails (a) contrast broad range of diffuse scattering (b) as a result of lateral ordering of extended defects.

Fig. 3: Cross-section TEM of the $\times 5$ MQW structure grown at $T_{\text{dep}} = 520^\circ\text{C}$. Lateral layer undulations (period ~ 100 nm) result from elevated deposition temperature and partial In decay.

Fig. 4: Symmetrical (004) reciprocal space maps for two 8+8 nm structures grown at the same deposition temperature and MQW period, but varied MQW periodicity. Extended diffraction spots for $\times 10$ structure illustrate spatial coherence diminution and initial stage of relaxation caused by secondary DLs.

Fig. 5: TC ω RCs in (004) reflection of three MQW structures with varied MQW period. Three different, preceding stages of defect creation are realized. For the 8+16 nm structure RCs for two azimuthal directions [110] and [1-10] are shown, whereas the two other samples do not show azimuthal anisotropy.

Fig. 6: Symmetric (004) RSMs for three MQW structures with different MQW period. The streaky vertically running surface truncation rod suggests high vertical coherence due to low density of edge segments, but laterally elongated diffraction spots indicate high deterioration due to intersecting secondary DLs and edge segments created in the MQW volume.

a) $T_{\text{dep}}=505^{\circ}\text{C}$

b) $T_{\text{dep}}=520^{\circ}\text{C}$

A high-resolution transmission electron micrograph (TEM) showing a cross-section of a semiconductor heterostructure. The structure consists of three main layers: a GaAs substrate at the bottom, an AlAs layer in the middle, and a Multiple Quantum Well (MQW) structure at the top. The MQW structure is composed of several thin, alternating layers of different materials, creating a periodic potential. The AlAs layer is a uniform, light-colored band. The GaAs substrate is a darker, uniform band at the bottom. A scale bar in the lower right corner indicates a length of 50 nm.

MQW

AlAs

50 nm

A short, thick horizontal black line used as a scale bar to indicate the 50 nm length.

GaAs

a) $\times 5$

$Q_y \times 10000$ (rlu)

b) $\times 10$

$Q_y \times 10000$ (rlu)

counts/s

a) 4+8 nm

b) 8+8 nm

c) 8+16 nm