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14. ABSTRACT
The awarded $58,000 in DURIP equipment funds per Contract No. W911NF-14-1-0426 was used to purchase and install a LayTec EpiTT 3W triple-wavelength in-situ emissivity corrected pyrometer on our existing Thomas Swan 6x2 Close-Coupled Showerhead metalorganic chemical vapor deposition (MOCVD) growth system which is used in the growth of III-As, P, Sb materials for a variety of DoD applications including the growth of InAs-GaSb and InAs-InAsSb type II superlattice (T2SL) structures and InP-InGaAs-InAlAs quantum-cascade lasers (QCLs). The

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Report Title

Final Report: Purchase of LayTec EpiTT Real-Time Optical Monitoring Equipment for In-Situ Control of Type II Superlattice Growth in an MOCVD System

ABSTRACT

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Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

57th Electronic Materials Conference, Columbus OH, Study of InAs/InAsSb type-II superlattices with in-situ optical monitoring system"

Number of Presentations: 1.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper
03/02/2017	2 James D. Justice, Theeradetch Detchprohm, Russell D. Dupuis, Honggyu Kim and Jian-Min Zuo, Zhiyuan Lin and Yong-Hang Zhang. Study of InAs/InAsSb type-II superlattices with in-situ optical monitoring system, 57th Electronic Materials Conference. 24-JUN-15, The Ohio State University, Columbus OH. : ,

TOTAL: 1

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received	Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received	Paper

TOTAL:

Number of Manuscripts:

Books

Received	Book

TOTAL:

Received	Book Chapter

TOTAL:

Patents Submitted

Patents Awarded

Awards

none

Graduate Students

NAME	PERCENT_SUPPORTED	Discipline
James Justice	0.00	
Hongmo Li	0.00	
FTE Equivalent:	**0.00**	
Total Number:	**2**	

Names of Post Doctorates

NAME	PERCENT_SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Names of Under Graduate students supported

NAME PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields: 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

Final report on program

Technology Transfer

Date: 2015/10/31
Contract No: W911NF-14-1-0426, ; GT Acct. No. 2106CZU
Project Title: Purchase of LayTec EpiTT Real-Time Optical Monitoring Equipment for In-Situ Control of Type II Superlattice Growth in an MOCVD System
Manufacturer: LayTec AG, Sesener Str. 10-13, 10709 Berlin, Germany
Institution: Georgia Tech Research Foundation, Georgia Institute of Technology, School of ECE, Atlanta GA 30332-0250
Pricipal Investigator: Prof. Russell D. Dupuis
ARO Grants Officer's Representative: Dr. William W. Clark, (919) 549-4314, e-mail: William.W.Clark@US.Army.mil
Period of Performance: 2014/08/01-2015/07/31
Funded Amount: $58,000.-

Abstract:

The awarded $58,000 in DURIP equipment funds per Contract No. W911NF-14-1-0426 was used to purchase and install a LayTec EpiTT 3W triple-wavelength in-situ emissivity corrected pyrometer on our existing Thomas Swan 6x2 Close-Coupled Showerhead metalorganic chemical vapor deposition (MOCVD) growth system which is used in the growth of III-As, P, Sb materials for a variety of DoD applications including the growth of InAs-GaSb and InAs-InAsSb type II superlattice (T2SL) structures and InP-InGaAs-InAlAs quantum-cascade lasers (QCLs). The system is used by graduate students and postdocs who are working on this MOCVD reactor to grow a variety of device-related structures. The epitaxial growth of these multiple-period, ultra-thin layer device structures was optimized via this LayTec EpiTT system to maintain growth conditions during relatively long growth runs and precise growth rates, interface switching, and growth temperatures in order to obtain high-quality device structures. This additional in-situ, real-time growth monitoring tool, when added to our existing MOCVD system has provided accurate and reliable measurement of these critical growth parameters during the epitaxial growth as well as to provide information on the interface roughness and a complete archival record of the actual growth details during the run. With this LayTec system, we have reestablished growth process to achieve 60 pairs of InAs-InAsSb T2SL on GaSb susbtrate with highly uniform composition across the structure.

1 *III-V Superlattice Materials Growth Issues and Solutions*

The Thomas Swan 6x2 Close-Coupled Showerhead (CCS) metalorganic chemical vapor deposition (MOCVD) epitaxial growth system is currently used primarily for InAs-GaSb and InAs-InAsSb type II superlattices in Dupuis' Laboratory at Georgia Tech under ARO MURI titled "FUNDAMENTAL STUDY OF DEFECT REDUCTION IN TYPE-II SUPERLATTICE MATERIALS" under contract No. W911NF-10-1-0524. This MOCVD system was purchased in 2003 by Georgia Tech as part of Dupuis' "start-up" package at a cost of ~$1.5M and was installed in Dupuis' Class 100 MOCVD Cleanroom using Georgia Tech and State of Georgia funds. As such, it represents a significant investment by Georgia Tech in the infrastructure to support this research contract.

Our research activities in this, and other, DoD-related III-V As-, P-, and Sb-based materials and device research programs, would greatly be accelerated and improved due to the increased control and in-situ monitoring of the growth which are critical in the development of advanced III-V materials and device structures, especially for complex structures like Type II superlattices, multiple-quantum-well lasers, and quantum-cascade laser diodes. In many cases, the real-time monitoring of the wafer bowing, epitaxial temperature, and growth rates would be beneficial in the growth of the highly-strained III-V quantum-well active regions, highly mismatched and strain-compensated superlattices, and thick *p*-type and *n*-type optical cladding and waveguide regions in laser diodes, the separate absorption and multiplication regions in advanced heterojunction avalanche photodiodes, and the heterojunction-based electron channel regions in HFETs. We proposed to accomplish this improvement in research program performance and in materials and device yields by the addition of an in-situ optical temperature and growth-rate monitor added to the existing reactor chamber components for the III-V As+P+Sb MOCVD system which is currently is dedicated to use for the growth of T2SLs in the ARO-sponsored program.

The energy gap vs. lattice constant for the III-V alloys with Column V As, P, and Sb atoms are shown below in Figure 1. The growth of many III-V heterostructure devices requires the control of materials three (ternary alloys) or more elements (e.g., quaternary or pentenary alloys) or the switching of the Column III and/or Column V elements across the heterostructure interfaces. This process of interface switching control is a critical one for all heterostructure devices, especially for device structures employing many such interfaces and the growth of thick epitaxial structures, e.g., T2SLs and QCLs. Shown below in red markers is the particular case for the InAs-GaSb T2SL and "Ga-free" InSb-InAsSb T2SL structures of interest for the on-going ARO sponsored contract in Dupuis' lab. Before the installation, the optimization of the growth of these complex heterostructures was carried out by extensive time-consuming and expensive growth condition optimizations. Furthermore, it is expected that slight shifts in the growth conditions are necessary due to subtle changes in the condition of the growth system, e.g., the coating on the reactor walls and the "showerhead" inside the MOCVD system. Consequently, shifts from the optimized growth conditions can occur during a run which can cause device structures to be adversely affected, which will not be obvious until expensive device processing and characterization testing are performed. Real-time in-situ monitoring can make this process of optimization and control much more efficient. The LayTec EpiTT 3W in-situ "real-time" monitoring system which is the subject of this funding will be extremely useful in optimizing the MOVD growth of heterostructures composed of all of many of these important "lattice-mismatched" III-V heterostructures, e.g., InAs-GaSb, InP-InGaAs-InAlAs-InAlGaAs, InP-InGaAs-InGaAsP-InAlAsP, etc.

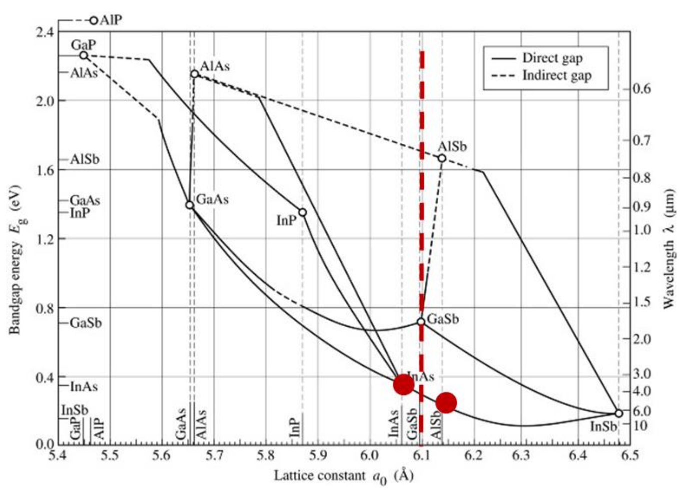

Figure 1: Energy bandgap vs. lattice constant for III-V As-P-Sb alloys of interest. Note that for all technologically

important III-V heterostructures except AlAs-GaAs ternary alloys, control of the lattice constant for heterostructures is a critical issue. The red dots show the InAs and InAsSb lattice constants useful for strain-balanced T2SL structures on GaSb substrates. The dashed vertical red line shows the GaSb lattice constant.

2 *Insatllation of LayTec EpiTT 3W on III-V As+P+Sb MOCVD Growth System as an Upgrade In-Situ Monitoring Equipment*

The LayTec Epi TT 3W triple-wavelength in-situ emissivity corrected pyrometer for real-time growth temperature monitoring system was installed on our III-V As+P+Sb MOCVD growth system on November 19-20, 2014 by an LayTec engineer. The functionality and performance of the system was then verified by growing a step-graded-composition heterosturcture of AlGaAs layers on GaAs wafer (run#3-2418) as shown in Figure 5.

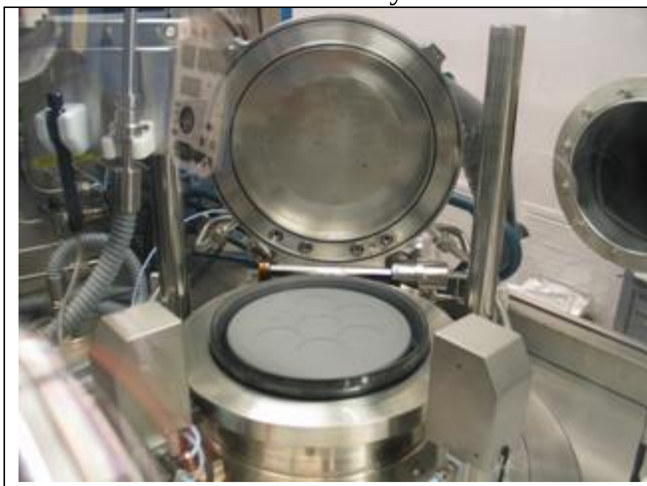

Figure 2: Photograph of Thomas Swan 6x2 CCS MOCVD system used for As+P+Sb growth with chamber lid open for wafer loading.

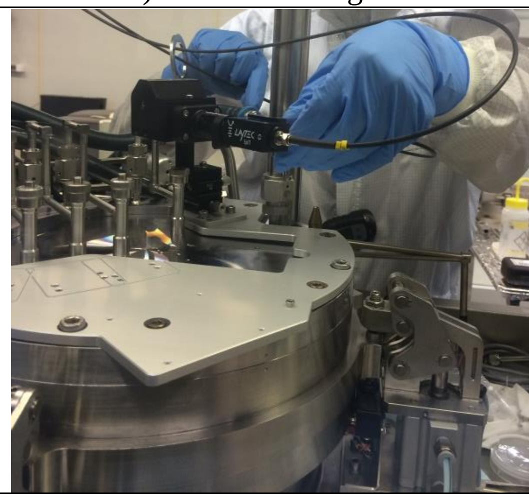

Figure 3: Photograph of the MOCVD growth chamber lid closed. The optical head of the new LayTec EpiTT was fitted on the designated view port.

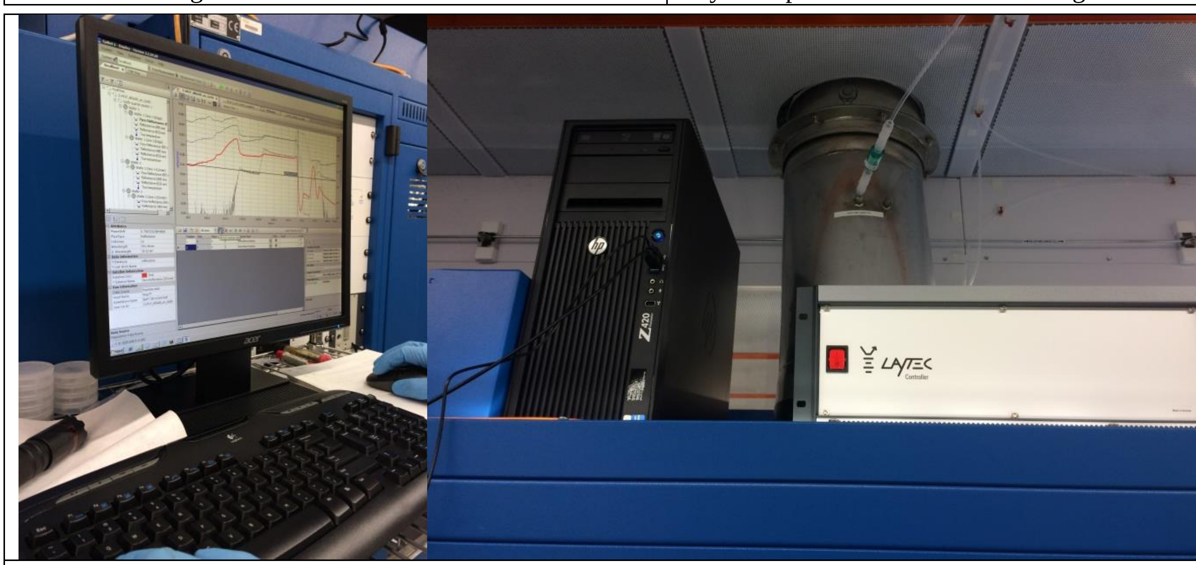

Figure 4: Operating software (left), control PC server (black middle box) and optical signal processing unit (white box with LayTec logo) of the LayTec EpiTT 3W system.

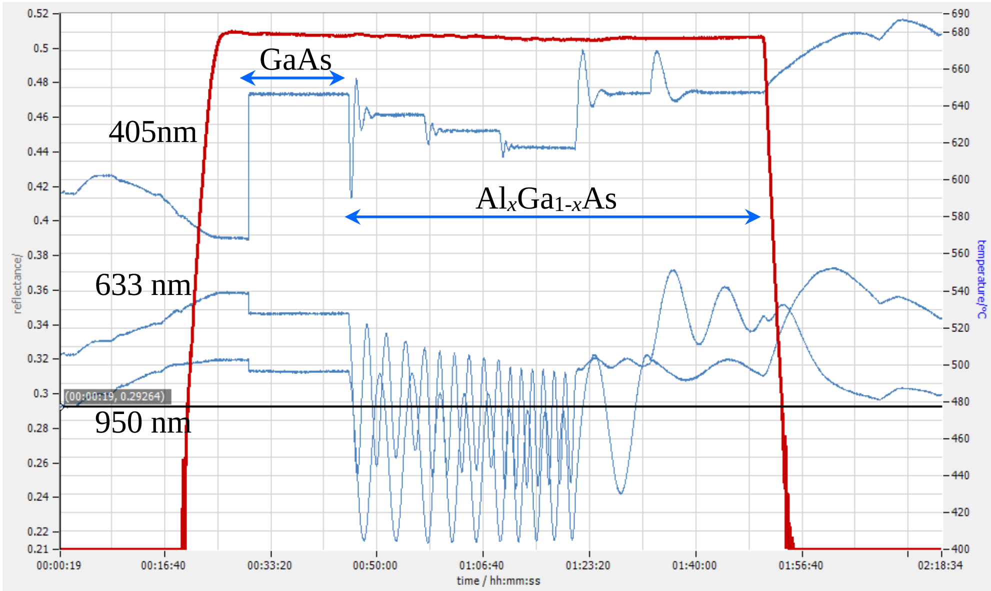

Figure 5: EpiTT scan of reflectance as marked by probing wavelengths, and temperature profile (red) for an MOCVD growth of a step-graded-composition heterosturcture of AlGaAs layers on GaAs wafer. Due to strong optical absorption during GaAs growth, constant reflectance from all 3 wavelengths assured the smooth surface was maintained. Osciallation of reflectance was observed during $Al_xGa_{1-x}As$ growth with x ~0.25-0.90 when absorption was less except for 405nm which exhibited rapid damping of such oscillation due to strong absorption.

3 Application of LayTec EpiTT 3W to the growth of InAs/InAsSb T2SL growth and characterization

3.1 GaSb buffer growth optimization and preparation of wafer and MOCVD chamber

In Dupuis' lab, InAs/InAsSb T2SL structures are typically grown by MOCVD on a GaSb buffer layer using optimized growth conditions However, we had investigated defect formation at the interface of GaSb buffer and GaSb wafer. As analyzed by SIMS, the contamination of As (~1% - required further quantitative analysis), and Al (~10%) including O (mid 1E18cm^{-3}) was the major concern for any subsequently grown T2SL of interest. We implemented EpiTT as well as reconditioning chamber step to minimize such contamination issue. As shown in the upper row of Figure 6, there was a slight decrease in reflectance with high density surface bumps and XRD fringes for a GaSb buffer grown during a typical MOCVD operation without proper cleaning and conditioning. In this case the shoulder peak on the higher angle side and fringes shown in (004) XRD confirmed the contamination of As at the ineterface between GaSb buffer and substrate. When chamber cleaning and GaSb coating was implemented, the surface morphology degraded significantly as shown in the middle row of this figure possibly due to the drift of Sb partial pressure in the growth chamber. However, after optimizing the V/III ratio, *i.e.*, increasing from 1.19 to 1.4, the reflectance stayed constant over the course of the GaSb buffer growth while surface defect was removed as well as the XRD fringes. This pregrowth preparation procedure and optimized growth conditions have been adopted for our current T2SL growth.

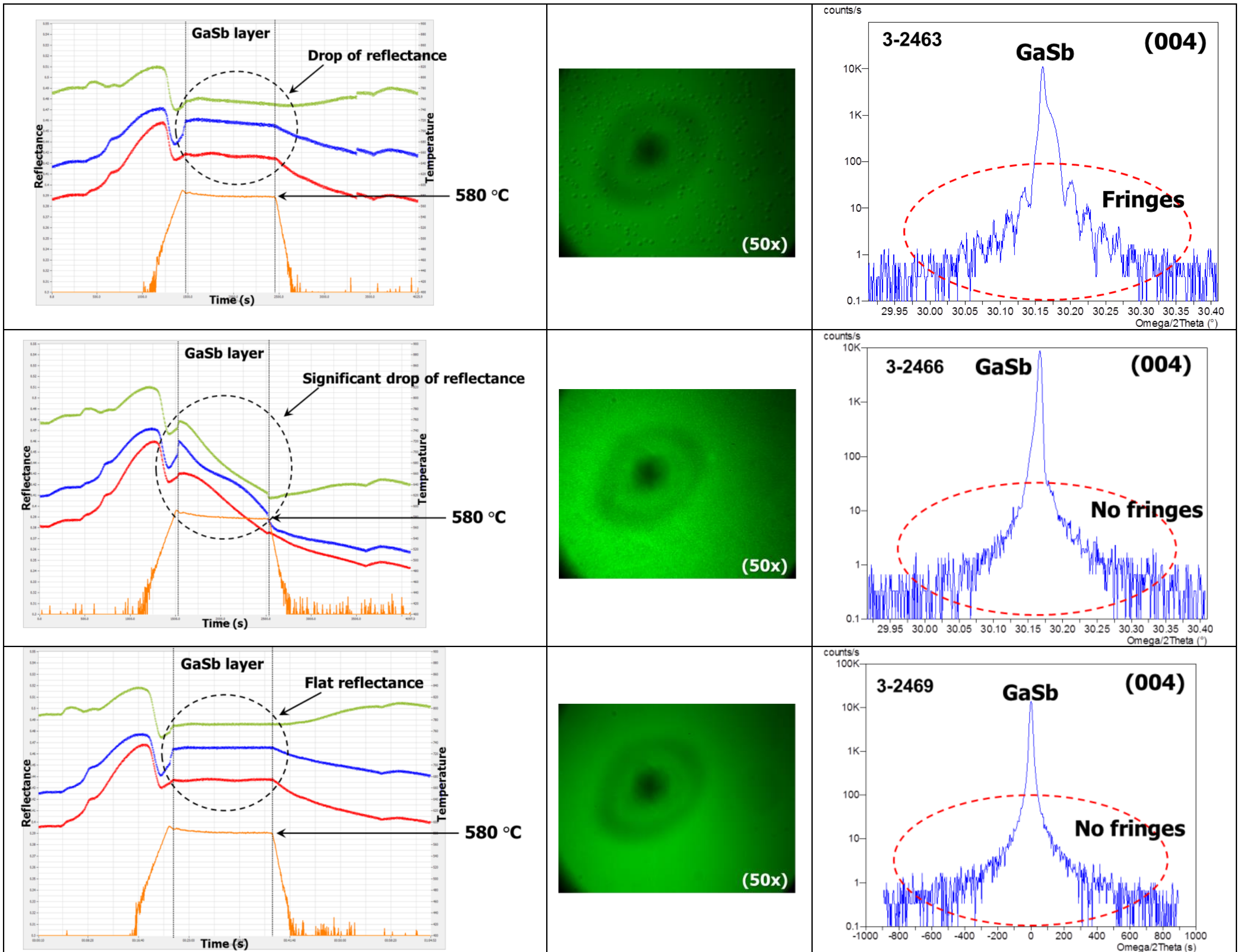

Figure 6 Reflectance & temperature profile (left column), surface morphology (middle column), and (004) XRD (right column) of GaSb buffer on GaSb wafer for before optimization (upper row), after platter cleaning & Sb coating (middle row), after V/III ratio optimization (lower row).

3.2 AlAs/AlAsSb T2SL Growth

The InAs-InAsSb T2SLs were grown at 100 Torr with a wafer rotation of 100 rpm. The carrier gas used is H_2 with the group III precursors being trimethylindium (TMIn) and triethylgallium (TEGa) and the group V precursors being trimethylantimony (TMSb) and arsine (AsH_3). A typical SL test structure is shown in Figure 7 with a 210 nm GaSb buffer layer on a GaSb (100) ± 0.04° substrate. Layer compositions and thicknesses were verified with (004) X-ray diffraction (XRD) scans and the corresponding simulation (Figure**Figure 8**). Additionally, atomic force microscopy (AFM) images were taken to measure surface roughness.

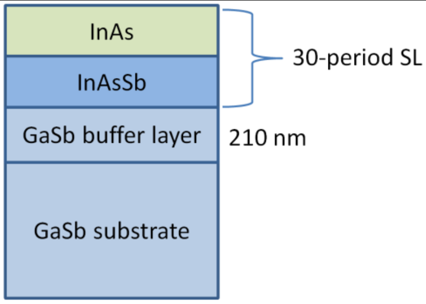

Figure 7: General structure of all the InAs/ InAsSb T2SL samples grown.

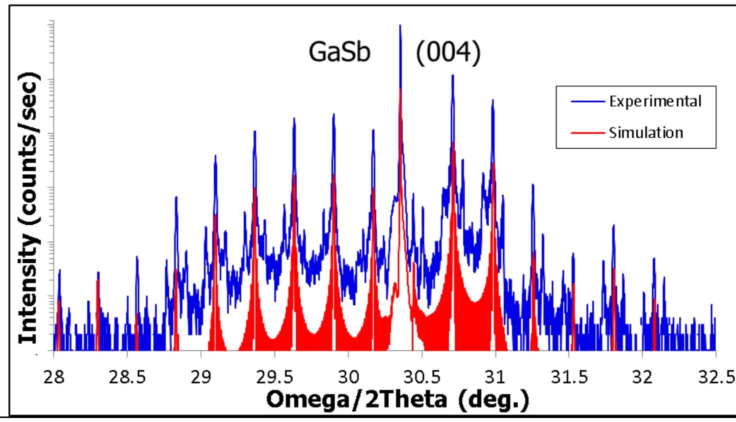

Figure 8: (004) XRD scan of 30-period InAs-InAsSb SL sample 3-2287 (blue) and simulation of the intended structure (red).

Prior to the implementation of pregrowth procedure mentioned in 3.1, material quality of T2SL structure was prone to structural degradation as more number of SL pairs increased as shown in the left plot of Figure Figure 9. With proper sequence of bake and coating, the uniformly grown T2SL can be achieved (Figure Figure 9 (right)).

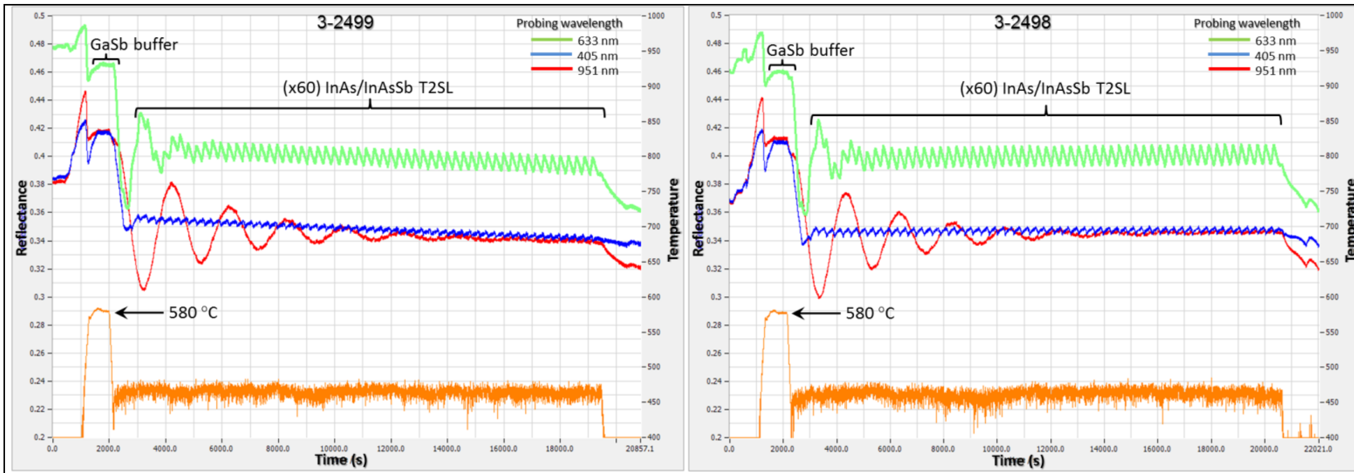

Figure 9: Reflectance and temperature profile (orange) of 60pairs of InAs/InAsSb T2SL on GaSb without (left) and with (right) proper chamber preparation..

For post growth characterization, AFM and XRD scan had been performed. Though smooth surface with atomic steps was observed for the T2SL without proper pregrowth conditioning, number of pits and trenches formed by pits were detected (Figure Figure 10 (a)). In case of T2SL growth with the proper chamber conditioning, no such defect was revealed (Figure Figure 10 (b)). The XRD intensity was found to be much stronger for this kind of sample ((Figure Figure 10 (d)). These results confirmed the improvement of material quality with the help of this newly installed LayTec EpiTT 3W in situ monitoring unit.

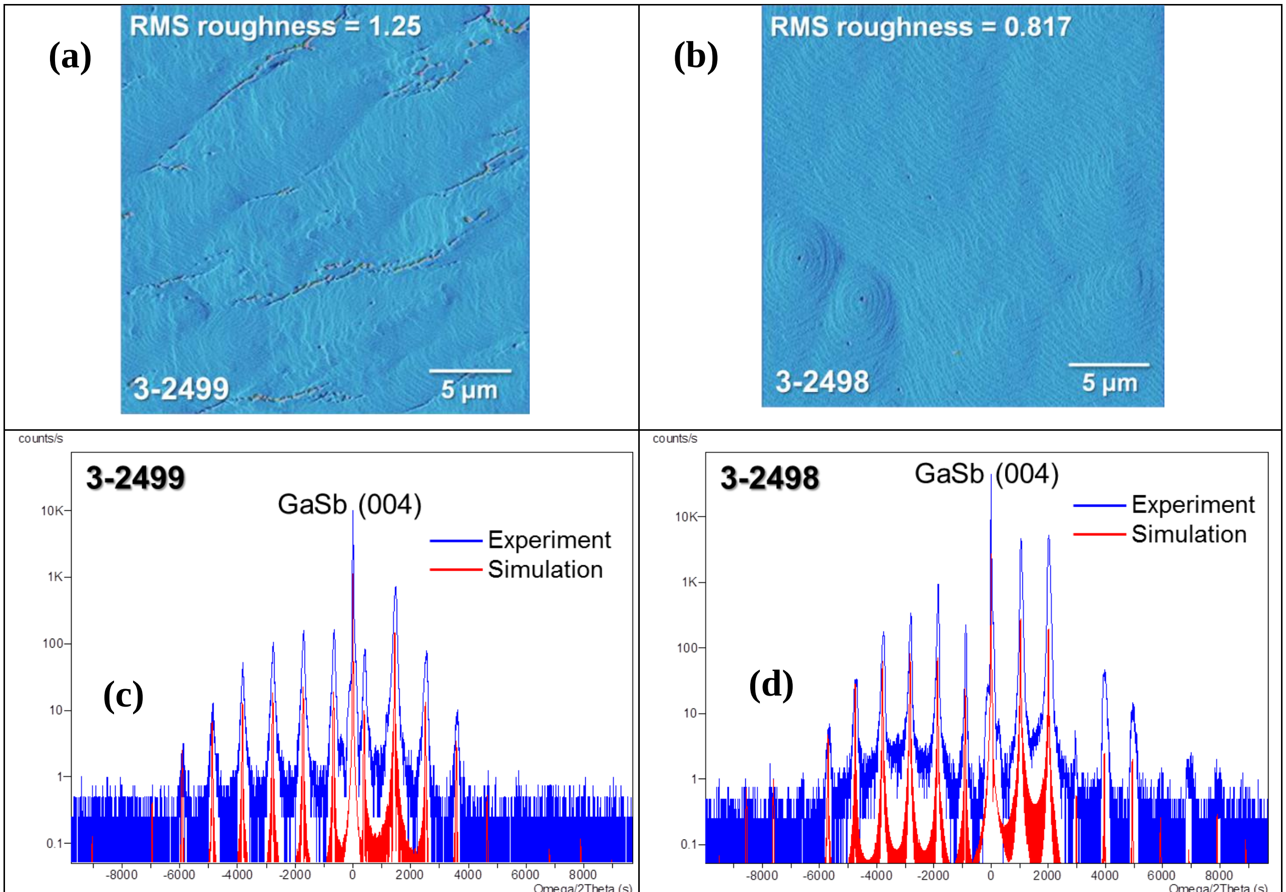

Figure 10: Atomic force microscope (AFM) images(a&b) and (004) XRD .scans (c&d) of of 60pairs of InAs/InAsSb T2SL on GaSb without (left column) and with (right column) proper chamber preparation.

Summary

The LayTec Epi TT 3W triple-wavelength in-situ emissivity corrected pyrometer for real-time growth temperature monitoring system has been installed on our III-V MOCVD system. The application of this in situ system has enabled us to establish a much more stable growth process as well as proper prepration of the growth chamber for T2SL structure. We are expecting that this implementation will enhance and improve MOCVD grown T2SL material quality which is an importance step toward any future T2SL device applications.

Conference Report

1. James D. Justice, Jeomoh Kim, Theeradetch Detchprohm, Russell D. Dupuis, Honggyu Kim, Jian-Min Zuo, Zhi-Yuan Lin, and Yong-Hang Zhang, "Study of MOCVD growth of InAs/InAsSb type-II superlattices using a LayTec EpiTT in-situ optical monitoring system", The 57th Electronic Material Conference, Ohio State University, Columbus, OH, USA, 24-26 June 2015.