

Vol 31 | Issue 2 | April 2023 | ISSN No. 0971 - 4413

www.drdo.gov.in

ADVANCED SEMICONDUCTOR MATERIAL TECHNOLOGIES

1000-00-00-000

CZT SUBSTRATES

Wafer HEMT epi-structure



Technology Focus focuses on the technological achievements in the organization covering the products, processes and technologies.

April 2023

Vol. 31 | Issue 2 | ISSN No. 0971-4413

Editor-in-Chief: Dr K Nageswara Rao Assoc Editor-in-Chief: Alka Bansal Managing Editor: Ajay Kumar

Editor: Dipti Arora Prepress: Raj Kumar

Printing: SK Gupta



Wafer HEMT epi-structure

Readers may send their suggestions to

The Editor, Technology Focus DESIDOC, Metcalfe House Delhi-110 054 Telephone: 011-23902403, 23902472 Fax: 011-23819151; 011-23813465 E-mail: director.desidoc@gov.in; techfocus.desidoc@gov.in; technologyfocus@desidoc.deldom https://www.drdo.gov.in/technology-focus

Laboratory Correspondents

Agra	:	Shri SM Jain, ADRDE		
Ahmednagar	:	Col Atul Apte, Shri RA Shaikh, VRDE		
Ambernath	:	Dr Susan Titus, NMRL		
Bengaluru		Shri Satpal Singh Tomar, ADE		
	:	Smt MR Bhuvaneswari, CABS		
	:	Smt Faheema AGJ, CAIR		
	:	Shri R Kamalakannan, CEMILAC		
	:	Dr V Senthil, GTRE		
	:	Dr Sushant Chhatre, MTRDC		
Chandigarh	:	Dr Pal Dinesh Kumar, TBRL		
	:	Dr Anuja Kumari, DGRE		
Chennai	:	Smt S Jayasudha, CVRDE		
Dehradun	:	Shri Abhai Mishra, DEAL		
		Dr SK Mishra, IRDE		
Delhi	:	Shri Sumit Kumar, CFEES		
	:	Dr Dipti Prasad, DIPAS		
	:	Shri Santosh Kumar Choudhury, DIPR		
	:	Shri Navin Soni, INMAS		
	:	Shri Anurag Pathak, ISSA		
	:	Dr Rupesh Kumar Choubey, SSPL		
Gwalior	:	Dr AK Goel, DRDE		
Haldwani		Dr Atul Grover, DIBER		
	:	Dr Ranjit Singh		
Hyderabad		Shri ARC Murthy, DLRL		
	:	Dr Manoj Kumar Jain, DMRL		
Jodhpur	:	Shri DK Tripathi, DL		
Kanpur	:	Dr Mohit Katiyar, DMSRDE		
Kochi	:	Smt Letha MM, NPOL		
Leh	:	Dr Tsering Stobden, DIHAR		
Pune		Dr Ganesh Shankar Dombe, HEMRL		
	:	Shri AK Pandey, ARDE		
	:	Dr Anoop Anand, R&DE(E)		
Tezpur	:	Dr Sibnarayan Datta, DRL		
Mysuru	:	Dr M Palmurugan, DFRL		





From the Desk of Guest Editor

The microelectronic technology is driving the modern defence systems as much as the comforts of civilian life. All advancements that we observe today in the microelectronic applications are the outcome of the evolutions either in the material growth or in device processing technologies. Solid State Physics Laboratory (SSPL), Delhi that was born from the semiconductor arm of the Electronics Research Wing of DRDO about six decades ago has steadily emerged as a unique laboratory in India covering the complete spectrum from semiconductor material growth to device design and fabrication, transfer of technology to production, and product delivery.

It's a great privilege for SSPL, that DESIDOC is bringing out the areas of core competence of SSPL compiled in two issues of Technology Focus, the first issue in your hand covers the Material Growth and Characterization and the second issue would be bringing out Semiconductor Devices, Sensors and the Emerging Technology areas being pursued at SSPL.

This issue of Technology Focus namely 'Advanced Semiconductor Material Technologies' covers the State-of-the-art Material Growth Technology of Gallium Nitride heterostructures essential for Monolithic Mircrowave Integrated Circuits used in the transreceive modules for radars. The Gallium Nitride heterostructures are grown on Silicon Carbide Substrates, earlier sourced through import only. Now, SSPL is on the path of complete self-reliance through the development of indigenous Silicon Carbide single crystal growth technology which is open for ToT.

Materials required for IR imaging are highly specialised and are totally denied to our country. SSPL has received Agni Award for self-reliance for the growth of IR sensitive materials already transferred in production. The IR detectors technology, at a mature stage in SSPL, is developed on our indigenous material. The most advanced material technology for IR detectors involving Type II superlattices is also matured at SSPL. The SSPL has also developed materials for THz generation and detection exhibiting high sensitivities not even observed in the imported counterparts. 

This issue also covers the efforts and achievements of the laboratory in the growth of 2-Dimensional films of graphene and MoS_2 as a potential futuristic application in wearable electronics.

Development of any material system is highly dependent on the characterization of its properties. SSPL has a sophisticated Material Characterization facility, one of its kind in the nation. This issue also covers the detailed account of the facilities available for material characterization at SSPL. This facility is also being used by other R&D institutions of the country.

The dream of marching from RLD onto production for SSPL has borne fruit in the form of a product range completely developed indigenously. GaN MMIC, High Power Laser Diodes, critical sub-technologies associated with IR detection and sensors based on MEMS, Acoustic Emission and SAW devices are the flagship areas of development at SSPL. The products that have emerged from these technology verticals include Power Amplifiers, Low-Noise Amplifiers, SPDT Switches in chip form, SiC Single Crystal Wafer, Single Emitter Fibre-coupled Laser Diodes, Stirling Cryocooler, IR sensitive materials, MEMS g-switch, e-Nasika CWA Detector and CNT-based n-Nose for explosive detection. Several technologies and products developed by SSPL are accepted and are in use by DRDO laboratories as well as for space applications.

I do hope that the compilation of this issue on 'Advanced Semiconductor Material Technologies' provides a clear picture of the achievements of the laboratory in the field of Material Growth and Characterization to all the readers.

> Dr Seema Vinayak OS & Director, SSPL



ADVANCED SEMICONDUCTOR MATERIAL TECHNOLOGIES

Solid State Physics Laboratory (SSPL), Delhi is involved in the growth of bulk and epitaxial materials of some of the most strategically important material for defence applications. R&D efforts over the years have led to vast experience and knowledge base in the laboratory in this niche area.

Modern devices require very sophisticated, complex hetero epi-structures, which are composed of stacks of thin layers with varied compositions, thicknesses varying from few mono layers to microns, controlled doping profiles with atomically abrupt interfaces. Efforts were put-in for indigenous development of such epi-structures for different microelectronic devices being required for various DRDO projects. Material epi-structures with such stringent requirement can only be achieved by epitaxial growth techniques like Molecular Beam Epitaxy (MBE), Metal Organic Chemical Vapor Deposition (MOCVD), etc.

Technology Focus टैक्नोलॉजी फोकस

The state-of-the-art facilities were created for bulk and epitaxial growth of various advanced III-V and II-VI semiconductor materials. SSPL has indigenously developed the technology for II-VI material which are strategic in nature and has led their production in the country through ToT to achieve self-reliance. Device quality heterostructures epi-material developed in the laboratory have been used for the development of various devices like IRFPA, GaAs and GaN-based HEMT/MMICs, High Power Laser Diodes, Solar cells, etc.

The significant developments accomplished in epimaterial and bulk crystal growth technologies, during the past few years are described below:

AlGaN/GaN HEMT Epitaxial Growth Technology

GaN-based High Electron Mobility Transistor (HEMT) heterostructures are required for the fabrication of high performance HEMT devices/ MMICs. A typical GaN HEMT heterostructure, consists of various epitaxial layers with strict control over composition as well as thicknesses at nanometer scale.

The desired smoothness/abruptness of various interfaces at sub-nanometer level requires atomic

scale control over growth process. Metal Organic Chemical Vapour Deposition (MOCVD) was used for developing AlGaN/GaN HEMT epiwafer growth process due to its capability of scaling up for volume production and low manufacturing cost. MOCVD facility established at SSPL employs a Close-coupled Showerhead (CCS) reactor with growth capability of $3\times2"$, $1\times3"$ and $1\times4"$ wafers (Fig. 1). The in-situ tools allow the real-time growth process monitoring along with complete susceptor temperature mapping.



Figure 1. III-Nitride MOCVD System



GaN material growth is very challenging, mainly due to lack of native GaN substrates. A variety of crystalline substrate options, namely Sapphire, SiC and Si, are used for growing the desired epitaxial heterostructures. However, for defence applications the technology development on SiC is used due to its least mismatch in lattice constant and thermal expansion coefficient with GaN. Further, a high thermal conductivity of SiC renders it most suitable for high power RF applications.

The main challenge in developing the GaN HEMT material technology includes achieving the desired two dimensional electron density and mobility with reduced dislocation density and control over impurities acting as deep electron traps.

With sustained R&D efforts, a production worthy GaN HEMT growth technology was developed on Sapphire and SiC substrate. The developed GaN HEMT epi-wafers (Fig. 2) showed characteristics (Table 1) comparable to the state-of-the-art GaN HEMT material technology.



Figure 2. Grown HEMT Epi-wafers on Sapphire and SiC

AlGaN/GaN HEMT specifications on SiC substrate				
Mobility (cm²/Vs)	~ 2000			
Sheet conc. (cm ⁻²)	~ 1E13			
R _s uniformity, (Std. dev.)	~5%			
Thickness uniformity, (Std. dev.)	~1%			
AlGaN, Al content uniformity	~1%			

Epi-structures with AFM RMS roughness $(5\mu m \times 5\mu m)$ of ~ 0.34 nm having step flow growth morphology (Fig. 3) and thickness uniformity with Std dev. < 1%, have been realized consistently. These AlGaN/GaN HEMT epi-wafers have been used for successful development of HEMT device fabrication technology.

Apart from the standard AlGaN-based HEMT structures, InAlN-based lattice matched HEMT structures were also grown and optimised for higher frequency devices.



Materials for IRFPA Technologies

For strategic material development required for IRFPA technologies, bulk crystals of semiconducting materials, e.g Cadmium Zinc Telluride (CZT, Zn = \sim 4%), epitaxial layers of HgCdTe or Mercury Cadmium Telluride (MCT) and Germanium (Ge, Sb doped) are grown and fabricated into required sizes and quality in the laboratory. Over the years several technological achievements, breakthroughs and product development has taken place in these areas.

CZT substrates are used for in-house growth of MCT epitaxial layers for the IRFPA development, a major thrust area of the laboratory. Due to strategic importance, device quality MCT epilayers and IRFPA grade CZT substrates are not available to India. Major players in HgCdTe-based IRFPA technologies



worldwide do mainly rely on in-house prepared material. Self-reliance has been achieved in both CZT and MCT, IRFPA materials required for the laboratory's IR device development. CZT substrates are regularly produced in india with industry partner using the SSPL developed technology and MCT epitaxial layers are grown at SSPL on these supplied substrates, which are then used for device fabrication.

The laboratory indigenously developed a production worthy technology for CZT crystal growth and fabrication of substrates and established a production facility in the country for their regular production. The baseline technology has been constantly upgraded by continuous R&D efforts of SSPL for further enhancing the material quality, yield, and substrate size and simultaneously incorporated in production line. The technology has matured to a level to produce large area (30 mm x 40 mm) CZT substrates of international standards.

MCT technology has been indigenously developed and established on CZT substrates produced in the country. Demonstration of the technology has been done by fabricating detector array on them. MCT epitaxial layers for Middle Wave Infrared (MWIR) applications are grown in the laboratory using Liquid Phase Epitaxy (LPE) technique and characterized. Device quality, fully characterised epilayers are regularly supplied and used for the detector array fabrication in the laboratory. IR thermal image has been obtained on this material thereby demonstrating the IR material technology of SSPL.

SSPL's IR programme solely depends on the CZT substrates produced at production facility developed in India and in-house MCT epilayers grown on them.

Cadmium Zinc Telluride (CZT) Bulk Crystal Growth

The major issues in the technological development of IRFPA grade Cadmium Zinc Telluride (CZT) substrate include size, crystallinity, and economics which are associated with the material's inherent properties. With extensive R&D efforts and modifications, up-gradations were made in the technology in terms of crystal diameter and substrate size along with enhanced crystalline quality and inducted in production line as shown in (fig. 4).

CZT substrates meeting international standards are regularly produced in the country (Fig. 5) using SSPL developed technology and used routinely for development of IRFPA in the laboratory. These CZT substrates are used in-house for MCT epitaxial growth for the IRFPA programme of the laboratory, making nation self-reliant in this strategic defence area.



(a)

(--)





Figure 5. Substrate produed in the Country (a) Specification, and (b) CZT Substrates ≤ 30 mm x 40 mm

Mercury Cadmium Telluride (MCT) Epitaxial Layer by LPE

Hg_{1-x}Cd_xTelayers of device quality are grown on (111) B oriented lattice matched CZT substrates by Liquid Phase Epitaxy (LPE) technique for the development of MCT-based IRFPA's. A complete facility consisting of LPE growth reactor, polishing of epilayers (Fig. 6) and characterization has been set up in the laboratory for the development of MCT epilayers for MWIR and LWIR waveband applications.



Figure 6. (a) MCT Epilayers Grown on CZT Substrates





Figure 6. (b) Microscopic Surface (1350 mm X 1010 mm) of asgrown Epilayer and Polished Device Epilayer Surface

Device quality IRFPA epilayer growth technology has been established on CZT substrates produced in the country using SSPL's indigenous CZT technology. As on today, in-house developed MCT epilayers of device quality characteristics (Fig. 7) are regularly grown in the laboratory and used for IRFPA development programme.

This has led to complete indigenization of material development programme of the laboratory for IRFPA technology.



Figure 7. Device Quality MCT Epilayers Grown in the Laboratory

Sb-doped Ge Single Crystals

Antimony doped Germanium, owing to its high transmission in the infrared region (2-14 μ m), and large refractive index is being used extensively for making lenses and windows for IRFPAs. SSPL has developed the technology for growth of optical grade 4" diameter, <111> oriented Germanium single crystals (antimony doped) by Czochralski (CZ)

technique (GEPULLER 20-04) and fabrication of its blanks (Fig. 8 and Fig. 9).



r Figure 9. Ge Crystal of 4" Diameter and Ge Blanks

Improvements in growth system and parameters were made to grow optical grade 2" and 3" diameter Ge crystals, as per the user requirements.

Ge blanks of requisite quality and dimension have been supplied to various users (IRDE-Dehradun, BEL-Machilpatnam and SAC-ISRO Ahmedabad) for the indigenous product development. Ge lenses fabricated on SSPL supplied Ge blanks were found to be of optical grade (Fig. 10) by users and as per international standards upon integration with thermal imager. Upon successful user trial, the Ge technology is now ready for transfer, for production of Ge blanks in the country.





Figure 10. Ge Blank Characterisrtics and Fabricated Ge Lens



ZnTe Crystal Growth for TH_z Applications

High quality ZnTe crystals are a material of choice for THz generation and detection based applications and devices. Growth of ZnTe crystal is one of the recent endeavours of SSPL in which ZnTe crystal were grown using Temperature Gradient Solvent Method (TGSM) and ZnTe substrates in (110) orientation and required dimensions were fabricated by orientation cutting of crystal, dicing the substrates from cut wafers and their lapping/polishing (Fig. 11).

Pure ZnTe phase and strong band edge peak against weak defect luminescence band observed in the X-ray diffraction spectra and Photoluminescence spectra respectively indicated good quality and low defect in the grown ZnTe crystal (Fig. 12). This result was substantiated by the high resistivity of the material (>1E7 ohm-cm) and the infrared transmission (50-55 % in the wave number range 500-6000 cm⁻¹).

Evaluation of the fabricated (110) ZnTe substrates for THz applications was done by Tata Institute of Fundamental Research (TIFR), Mumbai (user). SSPL developed ZnTe substrates showed very high THz transmission and THz detection in comparison to the commercial ZnTe substrates (Fig. 13). TIFR found these substrates of very high quality and suitable for THz- based applications.





Figure 13. Evaluation for THz Applications



Type II Strained Layer Superlattice (T2SL)-based Materials

The current third generation infrared photon detectors, which provide enhanced capabilities like large number of pixels, higher frame rates, better thermal resolution as well as multi-colour functionality and other on-chip functions have been based on HgCdTe, Quantum Well (QWIPs) or Quantum Dot (QDIP)-based structures. IR sensors based on a new type of material known as Type II Strained Laver (T2SL) Superlattice structure are predicted to overcome several difficulties posed by these materials and demonstrate better performance.

These are artificially engineered materials consisting of hundreds of alternate thin lavers of InAs and GaInSb/GaSb with perfect interfaces and a unique type-II band alignment, which allows the band gap to be controlled entirely by the thickness of the constituent layers. The preferred technology to achieve this is Molecular Beam Epitaxy (MBE), by which perfect crystalline layers of materials with different composition or doping can be grown with atomic level resolution. SSPL has procured a Molecular Beam Epitaxy system with an aim to develop this material technology (Fig. 14).

several challenges needed to be overcome to achieve this such as controlling interface abruptness, perfect strain balancing through interface engineering to avoid defects, controlling segregation issues and group III and V atoms intermixing. Tight control over the growth conditions is paramount in achieving the desired specifications.

Completely strain balanced T2SL structures consisting up to 300 periods (~ 1.8 µm) with InSb interfaces have been successfully grown at SSPL. Schematic of a basic undoped T2SL structure grown in SSPL using MBE is shown in Fig. 15. Figure 16 shows the image of a grown InAs/GaSb SL on a 2 inchGaSb wafer. The High Resolution X-ray Diffraction (HRXRD) and TEM results show excellent crystalline quality with sharp interfaces and a small lattice mismatch of the superlattice with the substrate (Fig. 17). Smooth morphology of the sample surface with atomic steps has been revealed in the AFM measurements (Fig. 18). The surface is mirror like and without any macroscopic defects as observed in optical microscope. FTIR measurements on the samples reveal an absorption edge of 5.6 µm and also the higher quantum transitions (Fig. 19 and Fig. 20).



Figure 14. Molecular Beam Epitaxy System for T2SL Growth

The key to T2SL-based IR detector technology is growth of several microns thick perfect strain balanced superlattice layers, which act as the active component of the detector structure. There are





Figure 15. Schematic of a Standard Figure 16. Grown InAs/GaSb T2SL Laver

SL Epilayer





Figure 18. TEM Image of a 100 Period InAs/GaSb Superlattice Layer





The laboratory has also successfully grown the complete detector structure having a bariode type design, which is preferred due to better performance and availability of materials with nearly similar lattice constants and large difference in band offsets. The complete detector structure consisting of a thick active layer, contact layers and an AlGaSb-based barrier layer is shown in Fig. 21. The individual layers for the entire detector structure have been completely calibrated.



Figure 21. nbnbariode-based T2SL Detector Structure

Single element detectors based on the MBE grown T2SL bariode hetero-structures have been fabricated. Both dark and photo current characteristics of the single element detectors are demonstrated. The fabrication of T2SL-based MWIR is under process. This achievement is a significant milestone in the development of T2SL-based detectors.

Silicon Carbide (SiC) Wafer

Silicon Carbide (SiC) Wafer is a disruptive compound semiconductor material with intrinsic properties providing superior performance and efficiency over silicon. It offers several advantages over conventional silicon-based power devices, such as IGBTs and MOSFETs, which have long dominated the market by virtue of their cost-effectiveness and simplicity of the manufacturing process. The main advantages offered by SiC can be summarised as follows:

- Higher switching frequency
- Higher operating temperature
- Greater efficiency
- Lower switching losses
- High power density
- Reduced size and weight
- Better thermal management

SiC is also the substrate for growth of GaN for highpower RF devices. The laboratory is engaged in the development of SiC bulk single crystal growth process and wafer fabrication technology indigenously. SSPL has set up the facilities for the development of 4H-SiC bulk growth technology (4" diameter, 4H-polytype) and also the complete infrastructure for the epi-ready wafer fabrication process. This is unique facility established in India for 4" SiC wafers.

SiC being strategic materials, the know-how is not available and the process steps are trade secret of the companies. The task becomes even more difficult due to limited knowledge sharing within the country.





Figure 22. PVT Growth Reactor

Figure 23. Grown SiC Single Crystal Boule



The commonly adopted technique for SiC growth is Physical Vapor Transport, (PVT) also known as seeded sublimation technique, in which the growth occurs at 2500-2700 K by the transport of subliming molecular species across a temperature gradient resulting in deposition upon a SiC seed held at relatively lower temperature as in Fig. 22 and Fig. 23.

After bulk growth of SiC, the crystal undergoes various processes like cylindrical grinding of grown boule, wafer slicing of ingot, edge grinding, wafer surface grinding and polishing followed by final Chemo-mechanical polishing of wafers. Each process steps is very challenging due to its high hardness (third hardest material) and chemical inertness. Wafer fabrication facilities established at SSPL are shown in Fig. 24 and Fig. 25.



Figure 24. (a) Diamond Wire Multi-wire Saw Machine, (b) Sliced SiC Wafers from SiC ingot, (c) Wafer Surface Grinder, (d) CMP Machine, (e) Wafer Edge Grinder

CA A MANA	SiC Polytype	4H
	Wafer Diameter	4 inch
	Wafer thickness	350-500 µm
	Resistivity	≥10 ⁵ ohm-cm
XIII/	Micropipe density	≤10 cm ⁻²
CHARLES & CHARLES	Surface roughness	<0.5 nm

Figure 25. Diameter 4H-polytype Semi-insulating SiC Wafers and Specifications

Graphene and other 2D Materials

2D materials are crystalline films formed by a single layer of atoms, it has width and length but have no depth. In these materials, the electrons or holes move freely in a two dimensional plane but restricted to move in the third direction. The unique electrical and optical properties of the 2D materials arise due to the quantum confinement effect in which electrons/holes are spatially confined in one or more dimensions within that material and have discrete electronic energy levels. The thickness of certain materials can be thin down to the scale of just one atom layer and in such materials various interesting property arises which is quite different from its bulk material.

The electronic structure of the 2D materials can be altered significantly, depending on the layer count, the relative orientation between layers, the electric field and strain. By layering different 2D materials on top of one another, we can produce hetero-structures, which have novel electrical, optical and thermal properties. Till today different classes of 2D materials have been discovered. Some of these materials are conducting (graphene), insulating (hBN) or semiconducting (TMDC-based 2D materials), etc. (Fig. 26 (a) shows different types of 2D materials. Transition Metal Dichalcogenides (TMDC)-based 2D materials are also widely popular because of their semiconducting nature with a distinct band gap.

Graphene is one atom thick layer of carbon atoms arranged in a hexagonal lattice structure. The lattice structure and band structure of graphene is shown in Fig. 26 (b) and (c). Graphene has various interesting electrical properties, e.g. very high carrier concentration (upto 1×10^{14} cm⁻²), very high mobility (> 20,000 cm²/V.s), ambipolar charge transport, high saturation velocity (10^6 m/s), negligible effective mass, symmetric conical band structure (the conduction band and valence band meet Dirac point). The electrons behave like massless Dirac Fermions as the electron effective mass is zero. Because of these interesting properties graphene can interact with electromagnetic radiation ranging from THz to visible radiation. In the infrared and visible







Figure 26. (a) Different 2D Materials (b) C Atoms in Graphene (c) Band Structure of Graphene

range absorption by graphene is only about 2.3 per cent at normal incidence, which is limited by the interband transitions. In the lower frequency range (THz region and near infrared), intraband transition dominates; in the THz range graphene can absorb up to 100 per cent of the incident THz photons.

TMDC-based 2D Materials are semiconducting 2D materials having non-zero band gap and high ON/ OFF ratios (~10⁴). TMDC 2D materials consists of hexagonal layers of M atoms sandwiched between two layers of chalcogen atoms X-forming the compound MX_2 . These materials have thickness dependent band gap properties. There is a transition of indirect to direct band gap when thickness of TMDC material is reduced from 1 micron to a monolayer of MX_2 . Interestingly bulk MOS_2 has an indirect band gap of 1.2 eV while single layer MOS_2 has a direct band gap of 1.8 eV. These materials have new opportunities for the optoelectronic application and also for the digital electronics.

Graphene and 2D Materials Activity

The laboratory is working on different types of 2D materials mainly graphene and TMDC materials (MoS_2) . Single layer graphene films are grown by using a Low-pressure Chemical Vapour Deposition (LPCVD) system and fabrication of graphene-based THz devices mainly, THz detectors, THz modulators, graphene FET is in progress. Figure 27 shows the



Figure 27. Flowchart for (a) Graphene Growth (b) Modified RCA Technique for Etching of Cu

flowchart for the deposition of graphene on Cu foils in the LPCVD and transfer of graphene on SiO₂/ Si substrate. Graphene is grown on copper-foil in presence of CH_4/H_2 gas mixture at temperature of ~1000 °C. Graphene is then transferred to silicon substrate for fabrication of various graphene-based devices.

Single layer/multilayers of MoS₂ are deposited by using 2-zone CVD system to develop MoS₂-based FET. Raman Spectra analysis confirms the growth of single layer bilayer/multilayer growth of graphene and TMDC-based 2D FILMS; characterisation techniques such as FESEM, AFM, XPS, STM are used for determining the micro-structural, optical and electronic properties of these 2D materials. The detail characterization of LPCVD grown graphene is shown in Fig. 28. The characterisation results of MoS₂ single layer/multilayers films are displayed in Fig. 29.



Figure 28. (a) Raman Spectra (b) FESEM and (c) Optical micrograph showing Graphene grains



Figure 29. (a) Raman Spectra (b) Photoluminiscence Spectra MoS₂ Single Layer/multilayers Films, and (c) Optical Micrographs MoS₂ Single Layer



Applications of Graphene and 2D Materials

Graphene THz devices has variety of applications, viz. spectroscopic identification of battlefield chemical warfare agents; non-invasive imaging for home-land security; and high throughput secure communication. Graphene THz devices can also play a major role in THz communication for aircraft to satellite; satellite to satellite; and within platoon communication as THz signal attenuates over a distance, so enemy

cannot eavesdrop from distant position.

TMDC-based 2D materials show strong photoluminescence emission and their light emitting properties may find widespread applications. Heterogeneous 2D-stacked materials may create enabling technology for future communication and computation applications..

CHARACTERISATION FACILITIES FOR MATERIAL RESEARCH AT SSPL

Material Characterisation plays an active role in the fabrication of devices. The material parameters such as composition, structure, morphology, defects, impurities, band gap, resistivity, mobility, lifetime etc. broadly define the performance of the devices fabricated out of it. This is accomplished by the material characterization using various techniques. In all the techniques, the probing is made of electrons, photons, ions that eventually interact with the sample. Analysis of the resultant beam such as electrons, photons, ions in various combinations is used to get various material parameters.

The elemental composition of any compound semiconductor is the most basic information required for the growth of bulk crystals and epitaxial films. The properties, like band gap, carrier concentration, etc. depend strongly on the composition and its variation. Also the compositional homogeneity is also one of the main requirements. The knowledge of concentration of dopants and unintentional impurities and their profile is key to the fabrication of devices. Energy dispersive (WDS/EDS) X-ray spectroscopy and Secondary Ion Mass Spectroscopy (SIMS), etc. are used for this purpose. The chemical state of bonding, adsorbed impurities on the surface, surface composition, etc. are probed using X-ray Photoelectron Spectroscopy (XPS), Auger Electron Spectroscopy (AES) and Ultra Violet Photoelectron Spectroscopy (UPS). The X-ray diffraction analysis is used for the analysis of structural information, crystalline imperfection and phase identification. It is well known that the defects in the semiconductor materials adversely affect their properties and can render a material unsuitable for the device fabrication. Transmission Electron Microscopy (TEM), is also used for this purpose. Optical techniques such as transmittance/reflectance and luminescence are used for the determination of the spectral variation of the complex refractive index determining the band gap, impurities, etc. The Raman spectroscopic measurements are also used in the determination of various lattice vibrations which is unique signature to any materials. The surface morphology roughness characterisation is required to monitor the quality of the surface prepared.

Some of the tools used for the material characterisation purposes are:

- Secondary Ion Mass Spectrometer (Cameca, IMS 7f)—(SIMS)
- Field Emission Scanning Electron Microscope (Zeiss SUPRA 55)—(FESEM)



- Multi-technique Surface Analysis System having XPS, Auger (AES) and UV spectroscopy (UPS) along with depth profiling (Omicron ESCA+)
- High Resolution X-ray Diffraction system (PANalytical X'Pert MRD System)—HRXRD
- Fourier Transform Infrared Spectrophotometer with a TGA facility (Varian 680 IR with 600 IR Microscope)—FTIR
- UV-Visible Spectrophotometer (Agilent: CARY-300)
- Deep Level Transient Spectroscopy-DLTS
- Low Temperature and High Magnetic Field Hall Measurement System
- Scanning Probe Microscope (SPM) (Keysight 5600 LS)
- Transmission Electron Microscopy (TEM)
- Raman Spectrometer/Photoluminescence (Horiba Jobine Yvon Lab RAM HR Evolution Micro-Raman system

Secondary Ion Mass Spectrometer (SIMS)

The specimen under study is sputtered by a beam of negative oxygen or positive cesium ion in the range of range of 0.5 keV to 20 keV. A small fraction of the sputtered material may be in the ionized form and hence the name secondary ion. These secondary ions carry the signature of the element when analysed using a mass spectrometer. The controlled layer by layer sputtering of the atoms and very good mass resolution makes this technique versatile in depth profiling. This is very useful in analysing the impurity profile in the material. All elemental masses from hydrogen to uranium can be analysed using this technique with ppb to ppm detection capability. This analysis is destructive and hence mainly useful in standardizing growth, device processing and fault finding. Figure 30 shows the SIMS system and Fig. 31 shows the doped Phosphorous and Arsenic depth profiling in Si Substrate by SIMS.

SIMS



Figure 30. SIMS System



Technical Specifications at a Glance

- ✤ Range of elements: H to U; all isotopes
- Destructive: Yes, material removed during sputtering
- Chemical bonding information: No
- & Quantification: Standards usually needed
- Detection limits: 10¹²-10¹⁶ atoms/cm³ (ppb-ppm)
- $\textcircled{\mbox{\sc b}}$ Depth probed: 2 nm-100 μm (depends on sputter rate and data collection time)
- Depth profiling: Yes, by the sputtering process; resolution 2 nm-30 nm
- Imaging/mapping: Yes
- Sample requirements: Solid conductors and insulators, typically
 1- 2.5 cm in diameter, 1- 6 mm thick, vacuum compatible
- Main use: Measurement of composition and of trace-level impurities in solid materials a function of depth, excellent detection limits, good depth resolution



Field Emission Scanning Electron Microscope

In this system a focused fine e-beam raster scans over the sample thereby various signals such as secondary electron, backscattered electrons, cathodoluminescence, characteristic X-rays, etc., are generated. These signals can be used to obtain information such as surface morphology, structure of the surface, chemical composition, defects etc. The system is capable of giving spatial resolution around 15 Å. The backscattered electrons are used in getting the elemental mapping. The characteristic X-rays are analysed using multichannel analyser to get qualitative and quantitative compositional analysis. Fig. 32 shows the FESEM system at SSPL and Fig. 33 shows the SEM image at 1 KV of sputtered gold particles The cathodoluminsescence is useful in analysing band to band and impurity emission in the UV, visible and near IR wavelength regions. By choosing different accelerating voltages the emission can be tuned at different depths, usually upto a μ m. This technique is useful to all elements from boron to uranium. This technique is generally non destructive. Figure 34 shows the CL image at GaN epitaxial layer, in which dark spots shows the dislocations present in the GaN sample.

Field Emission Scanning Electron Microscope



Figure 32. FESEM System



Figure 33. SEM Image at 1 KV of Sputtered Gold Particles



Figure 34. CL Image at GaN.

Technical Specifications at a Glance

FESEM

- Main use: High magni-fication imaging and composition (elemental) mapping
- Destructive: No, some cases electron beam may damage
- ✤ Magnification range: 10~-100000,
- Beam energy range: 100 eV-30 keV
- Sample Requirement: Conducting film; must be vacuum compatible
- & Sample Size: Less than 0.1 mm, upto 10 cm or more
- & Lateral resolution: 1.5 nm in secondary electron mode
- Bonding information: No

Energy Dispersive Spectrometer

- Range of elements: Boron to Uranium
- Destructive: No
- Quantification: Best with standards, although standard less methods are widely used
- ✤ Accuracy: Nominally 4-5 %, relative, for concentrations > 5 % wt
- Detection limits: 100-200 ppm for isolated peaks in elements with Z> 11, 1-2 % wt. for low-Z and overlapped peaks
- Sample requirements: Solids, powders, and composites; size limited only by the stage in SEM

Cathedo Luminiscence (CL)

Main use: Dislocation density estimation, band edge emission and defect estate determination



Multi-technique Surface Analysis System

Multi-technique surface analytical system consists of X-ray Photoelectron Spectroscopy (XPS), Ultraviolet Photoelectron Spectroscopy (UPS), and Auger Electron Spectroscopy (AES). The depth profiling is carried by sputtering the layers of atoms using an ion gun. The system is capable of providing information on the elemental composition and chemical bonding from the top most exposed surface.

XPS or ESCA and UPS

The XPS gives useful information about the bonding and the chemical state of the elements present at the surface. In XPS, the X-ray photon is absorbed by an atom of a molecule at the surface of the sample. This leads to emission of core electrons (inner shell. The kinetic energy of the electron is determined by energy dispersive analysis. The energy of the emitted electrons carries the information of the binding energy of the shell electron which is characteristic to an element. A small shift in the binding energy gives information about the chemical bonding of the particular element. XPS is best suited for the nondestructive chemical analysis of the surface. The information upto a depth of 50 Å typically can be obtained. Though the technique is non destructive but becomes destructive when ion beam sputtering is resorted for depth profiling. When low energy photon (Ultraviolet), the system becomes UPS. Here valence electrons are probed there by useful in the study of electronic structure. Figure 35 shows the XPS system at SSPL, and Figure 36 XPS depth profile of Ohmic contact metals on GaN/Sapphire.

AES

In this system a focused electron beam of energy range 2-25 keV is used to excite atoms. When the core level ionization due to the emission of electrons takes place, the relaxation process can take place by an electron from higher energy shell. In that process the energy gained by the electron that participates in the relaxation process can transfer to electron of the same or different level. The emitted electron will have a characteristic Auger energy. The Auger electron carries the signature of an element in the matrix. The exact energy of Auger electron can be used to get the chemical information.

Multi-technique Surface Analysis System (XPS, AES, UPS)



Figure 36. XPS Depth Profile of Ohmic Contact Metals on GaN/Sapphire

Technical Specifications at a Glance

- Range of elements: All except Hydrogen, and Helium
- Destructive: No, some beam may damage to X-ray sensitive materials
- Elemental analysis: Yes, semiquantitative without standards; quantitative with standards and not for trace elements.
- Chemical state: Yes information
- 🕸 Depth probed: 5-50 Å
- Depth profiling: Yes, over the top 50 Å; greater depths require sputtering
- Depth resolution: A few to several tens of Å, depending on conditions
- Sample requirements: All vacuum-compatible materials; flat samples best; size accepted depends on particular instrument
- Main uses: Determinations of elemental and chemical state compositions in the top 30 Å



High Resolution X-ray Diffraction System

High Resolution X-ray Diffraction is a powerful tool for the nondestructive investigations for crystalline quality of semiconductor bulk single crystals and epitaxial layers, thickness, composition and strain of multilayer epitaxial film. In this system incident X-ray beam (wavelength 1.54 Å Cu-Ka) falls on a 4 bounce 4-crystal Bartels monochro-collimator with divergence around 12 arc-seconds. The sample can be optimized with respect to the incident beam using the six axis movements x, y, z, ψ (rotation), ϕ (tilt) and ω (rocking angle) to satisfy the Bragg condition to get diffraction from a particular plane. The diffracted X-rays are collected by a detector. Figure 37 shows the XRD/HRXRD system at SSPL.

Following two types of arrangements are used to measure the diffracted beam:

Rocking curve arrangement for X-ray rocking

curve measurement, when X-ray falls on the sample and scanned along Bragg angle and diffracted X-rays detects by the detector with an acceptance angle of 1°. This measurement is useful in analyzing the crystalline quality of samples and composition and thickness of epitaxial films.

Triple axis measurement in which a single channel cut crystal Ge (220) is used as an analyser crystal in front of detector. This arrangement reduces the acceptance angle of the detector to 12 arc seconds, which improves the angular resolution in the diffracted beam.

Figure 38 shows the X-Ray Rocking Curve of GaAs substrate for crystalline perfection assessment. Figure 39 shows the Reciprocal space mapping of AlGaN/GaN HEMT structure, in the figure GaN and AlGaN Reciprocal space lattice contour are aligned vertically. It represents AlGaN layer is fully strained w.r.t. GaN buffer layer.







Optical Characterisation

Optical Characterisation is a non-destructive technique. The transmittance and reflectance of any material is governed by the optical constants namely refractive index and absorption coefficient. The spectral dependence of these optical constants is essential for making use of the materials in device fabrication applications in the required spectral range. The spectroscopic measurement can also be used to obtain the thickness of the epitaxial thin films. The optical studies are carried out from 0.2 to 200 μ m using two instruments, namely, Fourier Transform Infrared Spectrometer (0.5 μ m-200 μ m) and Dispersive UV-Visible spectrometer (0.2 μ m to 0.85 μ m). Micro-Raman measurements are carried out using a high resolution dispersive spectrometer.

FTIR, UV-Visible Dispersive Spectrometer

The Michelson interferometer consists of a fixed mirror, a movable mirror and a 60° beam-splitter. The beam-splitter transmits ideally half of the incident radiation to the fixed mirror and reflects the other half to the moving mirror. The beams are reflected by these mirrors back to the beam-splitter, where they superimpose. When the fixed mirror and moving mirror are equidistant from the beam-splitter, the amplitudes of all wavelengths are in phase and undergo constructive interference. The position of zero path difference is where the interferogram center burst occurs. As the moving mirror is moved away from the beam-splitter an optical path difference is generated. As the position of the moving mirror changes, the two beams travel different distances within the interferometer before recombining to form an interferogram. The interferogram is demodulated to get the spectrum using Fast Fourier Transformation. FTIR is very useful in the IR wavelength region. Figure 40 shows the FTIR spectrometer at SSPL.

A grating based dispersive UV-visible spectrometer isusedtotheopticalstudies in the visible and UV regions. Figure 41 shows the UV-visible spectrophotometer at SSPL. Figure 42 shows the Transmission spectrum of MCT epitaxial layer measured by FTIR system. Figure 43 shows the transmittance spectra of CZT substrate using UV-visible spectrophotometer.





Deep Level Transient Spectroscopy

Semetrol Deep Level Transient Spectroscopy (DLTS) uses a semiconductor structure in which there is a region that is depleted of mobile charges, and measurement conditions may be changed to allow trapping of carriers. The most straightforward method is to use a diode and decrease the width of the depletion region to fill traps that may be present, followed by an increase in the depletion width by applying a reverse bias, and measure the rate of emission of the trapped charges. The emission rate

is measured as it changes with temperature to obtain information on trap energy, capture cross section, and concentration.

The effect of DC stress, RF and optical excitation on defects can also be found by using DLTS system along with Keithley 2611B, 2651A SMU, Agilent E4980A LCR meter, and PXI VNA. The temperature variation of DLTS system is from 78K to 750K. Figure 44 shows the DLTS system at SSPL.



Figure 44. DLTS System



Low Temperature High Magnetic Field Hall/Resistivity

electrical characterization The implies measurements of properties like Hall coefficient, conductivity as a function of temperature and magnetic field. This is used to extract important material parameters like carrier type and concentration of charge carriers, mobility. Studying the variation of conductivity and Hall coefficient as a function of magnetic field gives information about the multicarrier present in narrow gap semiconductors and multi-layer structures.

The system at SSPL is capable of measuring the Hall/Resistivity data in the temperature range of 1.6-400 K. The variable temperature measurement data is useful for the analysis of the activation energy levels of the donors and acceptors in the material under analysis. This state-of-the-art system provides simultaneous controlled cooling of both the sample and the magnet using a single cryo-cooler.

For carrying out the Hall voltage measurements the system is equipped with Cryogen free magnet (CFM) up to 8 Tesla. The variable magnetic field measurement is useful for the analysis of multicarrier present in the material and for the multilayer structures. The cooling for the magnet is provided by a standard cryo-cooler with a base temperature of approximately 6K. The variable temperature insert (containing the sample) is built into the bore of the CFM, thus providing both variable temperature and magnetic field environment for the sample using a single cryo-cooler. An airlock is provided to change the sample without contaminating the sample space.

Figure 45 shows the Cryo-state with Electronic System for measurement of Magneto transport. Figure 46 shows the Shubnikov De-Hass (SdH) Oscillation in GaN-based HEMT structure

Low Temperature High Magnetic Field Hall/Resistivity



Technical Specifications at a Glance

- Main use: Quantum Hall Effect Measurement
- & Range of elements: Low resistive semiconducting materials
- ♥ Temperature range: 1.6 K-400K
- Magnetic field Range: 0-8 Tesla
- ♥ Sample requirement: less than 5 mm × 5 mm
- ✤ Nature of contacts : Perfect Ohmic contacts
- Properties can be measured:
 - Type of carriers present in the materials
 - Defects analysis
 - Carrier concentration in 2DEG system
- Specialized application: Study of Quantum Hall Effect and Spin Hall Effect

Scanning Probe Microscope

Scanning probe microscopy is a probe-based imaging technique for investigating the surface features of samples from micron to atomic level. In SPM techniques, a sharp probe tip interacts the sample surface and scans across the sample surface. Interactions between tip and sample are detected as a function of interaction force, friction, charge, conductivity, etc. Different techniques sense different interactions, which can be used to describe surface topography, interaction force, electrostatic charge, conductivity, etc.

Possible modes of SPM system are:

- Atomic Force Microscopy (AFM)
- Scanning Tunneling Microscopy (STM)
- Surface Spreading Resistance Microscopy (SSRM)



• Kelvin Force Microscopy (KFM)

AFM is highly used mode of SPM system. Interatomic forces between tip and surface of the sample are responsible for surface topography. The small size of probe tip is used for high resolution. The main applications are surface imaging, surface roughness determination, line profiling, Grain/ nano dot sizes determination, piezo coefficients for piezo-based materials, using electrical modes surface potential, local density of states, resistivity mapping for semiconductor samples, etc. The technique is generally non-destructive. Figure 47 shows SPM System and Fig. 48 shows the 2D AFM image of area $5 \,\mu m \times 5 \,\mu m$ of GaN layer on SiC substrate.

SPM



Figure 47. SPM System



Technical Specifications at a Glance

- ✤ Main use: High resolution probe-based imaging
- Magnification range: 1000000 x
- ✤ Modes: Contact, tapping for topography, electrical modes
- & Sample requirement: Any thin film/bulk flat sample
- & Sample size: Less than 1 cm, up to 100 cm or more
- & Lateral resolution: 0.1 nm in x-y
- & Vertical resolution: 0.05 nm in z
- Main use: Surface imaging, surface roughness determination, line profiling, Grain/nano dot sizes determination, using electrical modes surface potential, local density of states, resistivity mapping for semiconductor samples, piezo-coefficients for piezo-based materials





Transmission Electron Microscope

Transmission Electron Microscopy (TEM)-based techniques are suited for evaluating interfacial details, dimensions of device structures, and defects or flaws that arise during the fabrication process. Moreover, the features in semiconductor devices have rapidly shrunk to sub-nanometer size, therefore TEM has become an essential technique for process monitoring and failure analysis due to its superior spatial resolution and sensitive elemental analysis capabilities for material characterization.

TEM is the way to study the relationship between structural and electronic properties of the materials versus growth conditions. TEM can be used for imaging of dislocations, measurement of grain size, nano particles size, crystallite size, and atomic arrangement in material.

To extend the analytical capabilities to work in atomic dimensions, a TEM laboratory is recently established at SSPL.

The TEM laboratory comprises two sections, namely, (a) High Resolution Transmission Electron Microscope (HRTEM), and (b) TEM sample preparation lab.

Figure 49 shows the (a) TEM System, (b) Sample preparation instruments at SSPL, and Fig. 50 shows the TEM and EDS results of CdTe/MCT layers.





Raman Spectroscopy

Raman spectroscopy is a non-destructive characterization technique which provides the comprehensive information about the materials (i.e. molecules, compounds, semiconductors, etc.) through the Raman (phonon) modes. Raman spectroscopy characterizes the material for their molecular composition, the bonds, the chemical environment, the phase, stress/strain and crystal orientation, carrier concentration etc.

Raman spectroscopy is widely used technique because of easy operation and minimal sample

preparation. The elastically scattered Rayleigh line provides no information, where as the inelastically scattered anti-Stokes lines are generally of too weak in intensity. Stokes Raman lines is usually recorded by filtering dominant the Rayleigh light using suitable long pass edge filters. The spectrometer is also used to record the photoluminescence spectra.

Figure 51 shows the Raman spectrophotometer at SSPL. Figure 52 shows the Raman spectra of GaN epitaxial layer. Figure 53 shows the Raman Spectra of ZnTe/GaAs sample by varying temperature.



डेसीडॉक द्वारा प्रकाशित Published by DESIDOC

RNI No. 55787/93