

RESEARCH ON MANUFACTURING QUADRUPLE-JUNCTION SOLAR CELLS

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Prepared By:

University of California, Los Angeles
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Governor

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**ENERGY INNOVATIONS SMALL GRANT
(EISG) PROGRAM**

INDEPENDENT ASSESSMENT REPORT (IAR)

**RESEARCH ON MANUFACTURING
QUADRUPLE-JUNCTION SOLAR CELLS**

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PREFACE

The Public Interest Energy Research (PIER) Program supports public interest energy research and development that will help improve the quality of life in California by bringing environmentally safe, affordable and reliable energy services and products to the marketplace. The PIER Program, managed by the California Energy Commission (Commission), annually awards up to \$62 million of which 5% is allocated to the Energy Innovation Small Grant (EISG) Program. The EISG Program is administered by the San Diego State University Foundation through the California State University, which is under contract to the Commission.

The EISG Program conducts up to four solicitations a year and awards grants for promising proof-of-concept energy research.

PIER funding efforts are focused on the following seven RD&D program areas:

- Residential and Commercial Building End-Use Energy Efficiency
- Energy Innovations Small Grant Program
- Energy-Related Environmental Research
- Energy Systems Integration
- Environmentally-Preferred Advanced Generation
- Industrial/Agricultural/Water End-Use Energy Efficiency
- Renewable Energy Technologies

The EISG Program Administrator is required by contract to generate and deliver to the Commission an Independent Assessment Report (IAR) on all completed grant projects. The purpose of the IAR is to provide a concise summary and independent assessment of the grant project in order to provide the Commission and the general public with information that would assist in making follow-on funding decisions. The IAR is organized into the following sections:

- Introduction
- Objectives
- Outcomes (relative to objectives)
- Conclusions
- Recommendations
- Benefits to California
- Overall Technology Assessment
- Appendices
 - Appendix A: Final Report (under separate cover)
 - Appendix B: Awardee Rebuttal to Independent Assessment (awardee option)

For more information on the EISG Program or to download a copy of the IAR, please visit the EISG program page on the Commission's Web site at:
<http://www.energy.ca.gov/research/innovations>

or contact the EISG Program Administrator at (619) 594-1049, or email at:
eisgp@energy.state.ca.us.

For more information on the overall PIER Program, please visit the Commission's Web site at
<http://www.energy.ca.gov/research/index.html>.

Research On Manufacturing Quadruple-Junction Solar Cells

EISG Grant # 00-14

Awardee: University of California, Los Angeles
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Grant Funding: \$75,000
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Introduction

Photovoltaic (PV) technology could provide clean renewable electrical power distributed throughout California. Capital cost, sometimes known as first cost, for solar technology remains high and limits its widespread use. To reduce capital cost, the research community could develop solar cells with increased efficiency, reduce process costs, or reduce cells' material costs. This project focused on increased efficiency. It has been shown (See Appendix A) that if solar-cell efficiency of 40% were available, it would be competitive with traditional methods of power generation and would make large-scale implementation of solar power a reality.

By efficiently converting California's abundant sunshine into electricity, photovoltaic devices can provide electric power at a cost competitive with conventional generation. PV does not create atmospheric pollutants or any other byproducts from the generation of electricity. If located near the point of use, a PV generating system reduces the load on the power grid and eliminates most of the electrical losses associated with long-distance transmission lines. The installation of 1,000 PV local-area systems, each generating 2,700 MWh annually (totaling about 1,250 MW of capacity), would ease demand on the nation's diminishing supply of natural gas. It would also eliminate the annual generation and release of nearly 95 tons of oxides of nitrogen and over 135 tons of carbon monoxide gas, based on California's 2007 air-emission standards for natural-gas-burning distributed generation. Displacing older, more heavily polluting generators would result in even greater environmental benefits.

This project proposed to fabricate an advanced, high-efficiency solar cell. The researchers performed simulations on quadruple-junction, compound-semiconductor solar cells and demonstrated that terrestrial efficiencies of 42% could be achieved (See Appendix A). The solar-cell design proposed by the researchers consisted of a stack of lattice-matched thin films of indium gallium phosphide (InGaP), gallium arsenide (GaAs), indium gallium arsenide nitride (InGaAsN), and germanium (Ge). The researchers used a tunnel junction, separating each cell, to minimize electrical and optical losses. The bandgap of each p-n junction is optimized for collecting the solar spectrum: $E_g = 1.88$ eV for $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$, $E_g = 1.42$ eV for GaAs, $E_g = 1.05$ eV for $\text{In}_{0.07}\text{Ga}_{0.93}\text{As}_{0.98}\text{N}_{0.02}$, and $E_g = 0.66$ eV for Ge. Fabrication of the quadruple-junction solar cell represented an engineering challenge because each semiconductor film must be grown lattice-matched to a neighboring film to produce single crystals with a minimum number of defects. Moreover, the interfaces between each layer should be sharp, with minimal intermixing of the elements. Figure 1. illustrates a new, state-of-the-art reactor purchased in 2002 and used by the investigators in the performance of the project.

Figure 1. The new vertical rotating-disk reactor, an Emcore Discovery-125 MOCVD system, includes a loadlock for sample entry and transfer into ultrahigh vacuum (for *in situ* characterization). Sample platters hold three 2-inch wafers, one 3-inch wafer, one 4-inch wafer, or four 1x2 cm² custom sleds. During growth the carrier rotates at 1000 rpm and is heated radiatively. A total hydrogen flow of 25 standard liters per minute is used. Thickness and composition uniformity of <1% 1 σ has been demonstrated by this reactor on a 2- μ m-thick GaAs sample.



Objectives

The goal of this project was to determine the feasibility of developing a viable metalorganic chemical-vapor-deposition (MOCVD) process for manufacturing multi-junction devices. This would produce smooth and atomically abrupt In_{0.48}Ga_{0.52}/GaAs interfaces and the deposition of InGaAsN lattice-matched to GaAs with the desired nitrogen content. The researcher established the following project objectives:

1. Achieve lattice-matched InGaP on GaAs substrate with a smooth and atomically abrupt interface. Determine process conditions for optimum transition from GaAs growth to In_{0.49}Ga_{0.51}P growth. Minimize surface roughness of the In_{0.49}Ga_{0.51}P films.
2. Attain lattice-matched InGaAsN on GaAs substrate by metalorganic chemical-vapor deposition. Determine process conditions to deposit high-quality In_{0.08}Ga_{0.91}As_{0.97}N_{0.02} thin films. Optimize the indium and nitrogen distribution in the quaternary epitaxial layer to eliminate defects (<10⁴ cm⁻²) and strain. Incorporate 2% nitrogen in the InGaAsN film. Realize a bandgap/wavelength of 1.18 μ m.
3. Fabricate a simple quadruple-junction solar cell.

Outcomes

1. The researcher grew InGaP films lattice-matched to GaAs substrates. The feed ratio of the corresponding metalorganic sources determined the group III superlattice composition. The growth was mass transfer limited. Surface roughness was about 10 Angstroms. Exposing the GaAs surface to phosphorus sources did not cause the surface to roughen. The phosphorus atoms formed strained islands and induced strain in the substrate. Exposing an InP surface to arsenic caused the surface to roughen and form 3-D islands. To avoid this phenomenon, the surface must remain below 500 °C, or the gas-switching time must be below 10 seconds.
2. The researcher performed several growth runs to fabricate the InGaAsN layer. Annealing experiments found that N₂ annealing at 650°C for 30 minutes yielded the best material

quality in terms of PL measurements. The researcher found that a new source of nitrogen, TBHy, incorporated N more efficiently than the more widely used DMHy and achieved nitrogen incorporation of 1.65%.

3. The researcher did not fabricate a quadruple-junction solar cell.

Conclusions

The researcher solved several challenging issues in attempting to fabricate a quadruple-junction solar cell.

1. This project successfully grew high-quality InGaP and identified the conditions to produce the best material. The researcher learned how to grow interfaces between the arsenic-based layer and the phosphorus-based layer. The researcher also identified a new source of nitrogen, TBHy, for the InGaAsN layer and developed an MOCVD process to fabricate this layer with the desired composition.
2. More work remains to be done to improve the process. In particular, the cost of this method may preclude its use for terrestrial applications, although California industry could benefit from space applications.
3. It was not feasible to fabricate a quadruple-junction solar cell with the resources available.

The goal of this project was to determine the feasibility of developing a viable metalorganic chemical-vapor-deposition (MOCVD) process for manufacturing multi-junction devices. This project has made significant strides in developing the overall MOCVD manufacturing process. However, further research efforts must be undertaken to prove the feasibility of fabricating the targeted quadruple-junction solar cell.

Recommendations

1. Conduct further research to optimize the growth conditions for InGaAsN. The researcher has not incorporated enough N into the film. The N/GroupV percentage in the film should be increased to 2.0%.
2. Study the doping effects. In a quadruple-junction solar cell the four junctions -- the Ge, InGaAsN, GaAs, and InGaP layers -- are four p-n junctions with properly activated p-type and n-type dopants. During growth the dopant precursors, as well as the group III and group V sources, need to be fed into the MOCVD reactor. Further research must identify the growth conditions needed for the desired doping levels and then characterize the properties of the films.
3. Grow the device structure. Using the formula developed with this EISG grant, grow by MOCVD all the layers in the structure of a quadruple-junction solar cell. Test the efficiency of the cell, and optimize the entire fabrication process relative to capital costs for equipment and manufacturing.

After taking into consideration (a) research findings in the grant project, (b) overall development status, and (c) relevance of the technology to California and the PIER program, the Program Administrator has determined that the proposed technology should be considered for follow-on funding within the PIER program.

Receiving follow-on funding ultimately depends upon (a) availability of funds, (b) submission of a proposal in response to an invitation or solicitation, and (c) successful evaluation of the proposal.

Benefits to California

Public benefits derived from PIER research and development are assessed within the following context:

- Reduced environmental impacts of the California electricity supply or transmission or distribution system
- Increased public safety of the California electricity system
- Increased reliability of the California electricity system
- Increased affordability of electricity in California

The primary benefit to the ratepayer from this research will be reduced environmental impacts of the California electricity supply, transmission, or distribution system.

This project helped develop the manufacturing technology needed to produce high-efficiency photovoltaic cells. Photovoltaic cells can provide a solution to California's energy requirements. Further, it will reduce the demand on dwindling reserves of natural gas and eliminate the environmental and health hazards arising from combustion processes. Benefits will accrue to ratepayers only if the technology proves cost effective.

Overall Technology Transition Assessment

As the basis for this assessment, the Program Administrator reviewed the researcher's overall development effort, which includes all activities related to a coordinated development effort, not just the work performed with EISG grant funds.

Marketing/Connection to the Market

Activities related to marketing undertaken by this project include the publication of results in peer review papers and theses.

Engineering/Technical

This project solved two important technological challenges in fabricating quadruple-junction solar cells. One of them is to grow disordered, lattice-matched InGaP layers on GaAs substrates. The other is to successfully apply a new nitrogen source, TBHy, to the growth of InGaAsN. Once the manufacturing process for the targeted quadruple junction solar cell design is shown to be feasible, this knowledge can be transferred to industry to help establish the production process.

Legal/Contractual

There is no patent filed based on the results of this project. There are no legal/contractual issues foreseen at this point. The knowledge learned from this project is open to the public.

Environmental, Safety, Risk Assessments/ Quality Plans

The materials studied were grown in a commercial MOCVD reactor with adequate safety controls. All waste chemicals were trapped in a solid absorbent for hazardous-waste disposal later. Each experiment was reproduced to ensure quality and reproducibility. Until the complete manufacturing process has been developed, specific quality planning is unwarranted. Quality Plans include Reliability Analysis, Failure Mode Analysis, Manufacturability, Cost and Maintainability Analyses, Hazard Analysis, Coordinated Test Plan, and Product Safety and Environmental.

Production Readiness/Commercialization

This project developed new technology for growing the complex semiconductor hetero-structures used in quadruple-junction solar cells. Once cells with efficiencies greater than 40% are demonstrated, discussions can be initiated with various commercial enterprises for technology transfer.

Appendix A: Final Report (under separate cover)

Appendix B: Awardee Rebuttal to Independent Assessment (none submitted)

Appendix A to IAR 00-14
CEC-500-2006-019

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**RESEARCH ON MANUFACTURING
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Inquiries related to this final report should be directed to the Awardee (see contact information on cover page) or the EISG Program Administrator at (619) 594-1049 or email eisgp@energy.state.ca.us.

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Abstract

Quadruple-junction solar cells show great potential for achieving terrestrial conversion efficiencies above 40%. At these levels, photovoltaic devices could be competitive for large-scale electric power generation. Quadruple-junction solar cells consist of epitaxial thin films of indium gallium phosphide ($\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$), gallium arsenide and indium gallium arsenic nitride ($\text{In}_{0.08}\text{Ga}_{0.92}\text{As}_{0.98}\text{N}_{0.02}$) lattice-matched to germanium substrates. The compound semiconductor materials were deposited by metalorganic chemical vapor deposition (MOCVD) in a new Emcore D-125 reactor using trimethylgallium, trimethylindium, tertiarybutylarsine, tertiarybutylphosphine and tertiarybutylhydrazine (TBHy). Excellent quality materials were produced as evidenced by x-ray diffraction and photoluminescence spectra. In particular, InGaAsN containing 4.1% indium and 1.65% nitrogen was deposited at 550 °C, a group III to group V source ratio of 27.0, and a TBHy to group V source ratio of 0.7. The diffraction data indicated that the lattice mismatch was only 0.00045. The film exhibited a sharp photoluminescence peak at 1082 nm, equivalent to a bandgap of 1.15 eV. The standard deviation in the emission over a gallium arsenide 2" substrate was 0.9%. It was found that tertiarybutylhydrazine was a more efficient source of nitrogen than dimethylhydrazine (DMHy), the reagent used in previous studies of this process. To achieve the same nitrogen content, less than half as much TBHy was needed as compared to DMHy. Although quadruple junction solar cells were not prepared, each layer required in the device was successfully deposited. With more support, we are confident that an MOCVD process could be developed for the fabrication of high-efficiency solar cells.

Key Words:

MOCVD, InGaAsN, InGaP, Heterojunction

Executive Summary

Introduction

Solar energy is a renewable resource that has minimal impact on the environment, and therefore, offers several advantages over fossil fuel combustion. However, photovoltaic devices make a negligible contribution to U.S. power generation, because the efficiency of converting light to electricity is too low to make the technology competitive with other nonrenewable energy sources. The high-efficiency cells we are developing are best used under concentration in medium scale power plants (0.1 to 10.0 MW) [1]. According to Swanson [1], if a cell efficiency of 33% and a concentration ratio of 1000 are assumed, electricity can be generated at 6.6 ¢/kWh. If the cell efficiency exceeds 40%, the cost will be even lower, approaching 3.0 ¢/kWh [2]. This is comparable to the cost of generating power from fossil fuels. A significant capital investment is involved in the installation of high-efficiency solar systems, but this investment is no different than for any modern power plant. When environmental and waste disposal considerations are factored in, this option can be the most cost effective.

Hou and coworkers [3] have performed simulations on quadruple-junction compound semiconductor solar cells, and have demonstrated that terrestrial (AM0) efficiencies of 42% could be achieved. Development of these materials could possibly tip the balance towards photovoltaic power generation. The solar cell design proposed by Hou et al. [3] consists of a stack of lattice-matched thin films of indium gallium phosphide (InGaP), gallium arsenide (GaAs), indium gallium arsenide nitride (InGaAsN), and germanium (Ge). Separating each cell is a tunnel junction to minimize electrical and optical losses from the cell. The bandgap of each p-n junction is optimized for collecting the solar spectrum: $E_g = 1.88$ eV for $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$, $E_g = 1.42$ eV for GaAs, $E_g = 1.05$ eV for $\text{In}_{0.07}\text{Ga}_{0.93}\text{As}_{0.98}\text{N}_{0.02}$, and $E_g = 0.66$ eV for Ge [3]. Fabrication of the quadruple-junction solar cell represents a real engineering challenge, because all the semiconductor films must be grown lattice-matched to each other as single crystals without an appreciable number of defects. Moreover, the interfaces between each layer should be sharp with minimal intermixing of the elements.

Objectives

1. Achieve lattice-matched InGaP on GaAs substrates with smooth and atomically abrupt interfaces.
 - Determine conditions for optimum transition from GaAs growth to $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$ growth.
 - Minimize surface roughness of the $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$ films.
2. Attain lattice-matched InGaAsN on GaAs by metalorganic chemical vapor deposition.
 - Determine process conditions to deposit high-quality $\text{In}_{0.08}\text{Ga}_{0.91}\text{As}_{0.97}\text{N}_{0.02}$ thin films.
 - Optimize the indium and nitrogen distribution in the quaternary epitaxial layer to eliminate defects and strain.
 - Incorporate 2% nitrogen in the InGaAsN film.
 - Realize a bandgap/wavelength of 1.18 μm .

Outcomes

1. InGaP films lattice matched to GaAs substrates have been grown. The group III superlattice composition was determined by the feed ratio of the corresponding metalorganic sources. The growth was mass transfer limited.
2. Heterojunction formation was studied:
 - Exposing the GaAs surface to phosphorus sources did not cause the surface to roughen. The phosphorus atoms formed strained islands and induced strain in the substrate.
 - Exposing an InP surface to arsenic sources caused the surface to roughen and form 3-D islands. To avoid this phenomenon, one must keep the surface below 500 °C, or reduce the gas switching time below 10 seconds.
3. Several growth runs were done to fabricate the InGaAsN layer:
 - A new source, tertiarybutylhydrazine, was tested shown to incorporated N more efficiently than the more widely used source, dimethylhydrazine.
 - Different annealing approaches were taken and it was found that N₂ annealing at 650°C for 30 minutes yielded the best material quality in terms of PL measurements.

Conclusions

We have solved several challenging issues in fabricating quadruple junction solar cells. We have successfully grown high quality InGaP and identified the conditions that will produce the best quality material. We have also studied the heterojunction formation between the arsenic-based layer and the phosphorus-based layer. Important information was obtained to guide the growth of these interfaces. As for the InGaAsN layer, a new source, TBHy, was tested and annealing methods were investigated. An MOCVD process was developed to fabricate this layer with the right composition, although more work needs to be done to optimize the overall growth process.

Recommendations

1. Conduct further research to optimize the growth conditions for InGaAsN. Right now we have not incorporated enough N into the film. The N/GroupV percentage in the film should be increased to 2.0%.
2. Study the doping effects. In a quadruple junction solar cell, the four junctions, i.e., the Ge, InGaAsN, GaAs, and InGaP layers, are four p-n junctions with properly activated p-type and n-type dopants. During growth, the dopant precursors as well as the group III and group V sources need to be fed into the MOCVD reactor. One needs to identify the growth conditions to achieve the desired doping levels and then characterize the properties of the films.

3. Grow the device structure. All the layers in the structure of a quadruple junction solar cell need to be grown by MOCVD using the recipe developed with this EISG grant. The efficiency of the cell should then be tested and the entire fabrication process optimized.

Benefits to California

This project has assisted in developing the manufacturing technology needed to produce high-efficiency quadruple junction solar cells. Photovoltaic cells that convert solar energy to electricity will provide a solution to California's current energy requirements. In addition, the implementation of photovoltaic technology will reduce the demand on dwindling fossil fuel reserves, and eliminate the damage to the environment caused by fossil fuel combustion.

It was shown that high concentration III/V solar cells are the most cost effective PV approach for medium power plants (0.1 to 10.0 MW) in high resource areas [1]. California has many regions with high solar resources; and a lot of investment in PV manufacturing has already been made. It is strategically important that these assets be put to work for terrestrial solar power generation.

Main Report

Introduction

Background and Overview

This project tackles an important energy challenge, the need to deliver a low-cost and renewable energy source to California consumers. It enables the manufacturing technology needed to produce high-quality photovoltaic cells, with a high enough solar energy conversion efficiency.

This project targets at least four of the Public Interest Energy Research (PIER) program areas including: Environmentally Preferred Advanced Generation; Renewable Generation; Energy-Related Environmental Research; and Strategic Energy Research.

The current energy crisis in California highlights the need for affordable, renewable, environmentally benign energy sources. Photovoltaics can provide the solution to California's energy problems. By minimizing the demand on dwindling fossil fuel reserves, photovoltaic technology can eliminate rolling blackouts and skyrocketing utility fees. In addition, solar technology can eliminate the environmental hazards caused by fossil fuel combustion.

However, photovoltaics make a negligible contribution to U.S. power generation, because the efficiency of converting light to electricity is too low to make the technology competitive with nonrenewable energy sources. The high-efficiency cells we are developing are best used under concentration in medium-scale power plants (0.1 to 10.0 MW) [1]. According to Swanson [1], if a cell efficiency of 33% and a concentration ratio of 1000 are assumed, electricity can be generated at 6.6 ¢/kWh. If the cell efficiency exceeds 40%, the cost will be even lower, approaching 3.0 ¢/kWh [2]. This is comparable to the cost of generating power by fossil fuel combustion. A significant capital investment is involved in the use of concentrator solar cells, but this investment is no different than for any modern power plant. When environmental and waste disposal considerations are factored in, this option can be the most cost effective.

Quadruple-junction solar cells show great potential for achieving conversion efficiencies greater than 40% [3]. At these levels, photovoltaic devices would be competitive for large-scale electric power generation. Quadruple-junction solar cells are a stack of compound semiconductor thin films. In particular, they consist of layers of indium gallium phosphide (InGaP), gallium arsenide (GaAs) and indium gallium arsenic nitride (InGaAsN) lattice-matched to germanium (Ge) substrates. The bandgap of each junction is optimized for absorbing the solar spectrum.

Fabrication of quadruple-junction solar cells represents a real engineering challenge, because all the semiconductor films must be lattice-matched to each other as single crystals without any appreciable defects (i.e., $<10^4 \text{ cm}^{-2}$). Moreover, the interfaces between each layer should be sharp with minimal intermixing of the elements.

The objective of this EISG project was to develop a process technology for growing quadruple-junction solar cells. These films are produced by a sophisticated process known as metalorganic chemical vapor deposition (MOCVD). This deposition technique is well suited for large-scale manufacturing since several Southern California companies, for example, Spectrolab, Inc., are already producing large quantities of these epitaxial materials.

The current bottleneck to using MOCVD to produce quadruple junction solar cells is that the layers are not sharp due to atomic diffusion across the interfaces. In addition, complications arise when alloying gallium arsenide with indium and nitrogen to form InGaAsN. It is difficult to incorporate enough nitrogen into the film to achieve the desired electrical properties. At UCLA, we have excellent facilities for solving this problem. We have attached a commercial-grade MOCVD reactor to an ultrahigh vacuum thin film analysis system. Through this research, we hope to hasten the day when photovoltaics are widely used for terrestrial electric power generation.

Report Organization

This report is organized to:

- Present the objectives of our research.
- Give an overview of metalorganic chemical vapor deposition (MOCVD).
- Describe the state-of-the-art MOCVD reactor recently installed at UCLA.
- Detail the growth and characterization of thin films of InGaP and InGaAsN.
- Present conclusions and recommendations for future work.

Project Objectives

The project objective was to develop a metalorganic chemical vapor deposition process for fabricating InGaP/GaAs/InGaAsN/Ge quadruple-junction solar cells with efficiencies over 40%.

Specific EISG project objectives:

1. Achieve lattice-matched InGaP on GaAs substrates with smooth and atomically abrupt interfaces.
 - Determine process conditions for optimum transition from GaAs growth to $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$ growth.
 - Minimize surface roughness of the $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$ films.
2. Attain lattice-matched InGaAsN on GaAs substrates by metalorganic chemical vapor deposition.
 - Determine the process conditions for depositing high-quality $\text{In}_{0.08}\text{Ga}_{0.91}\text{As}_{0.97}\text{N}_{0.02}$ thin films.

- Optimize the indium and nitrogen distribution in the quaternary epitaxial layer to eliminate defects and strain.
- Incorporate 2.0% nitrogen in the InGaAsN film.
- Realize a bandgap/wavelength of 1.18 μm .

Economic objectives:

1. Develop a reproducible MOCVD process for growing the most challenging layers in a multi-junction solar cell, so that the cell efficiency exceeds 40%.
2. Transfer the knowledge we obtained from this study to California-based solar cell companies for commercialization.

Project Approach

Compound semiconductor layers in quadruple junction solar cells were deposited by metalorganic chemical vapor deposition (MOCVD) [4]. In this process, volatile compounds, e.g., trimethylgallium, trimethylindium and tertiarybutylphosphine, together with the carrier gas, hydrogen, are passed over a hot semiconductor substrate, such as GaAs (001), where they decompose and deposit an epitaxial film, e.g., InGaP. The reactions that occur on the surface of the film during growth control the composition of the film, its growth rate and the doping levels achieved. Therefore, understanding the MOCVD process is crucial to developing a manufacturing technology for quadruple-junction solar cells.

At the first stage of this project, a home-built MOCVD reactor was used to grow binary compound semiconductors, GaAs. Excellent epitaxial films were obtained. When we moved on to more complicated materials, i.e., InGaP and InGaAsN, this machine was not able to deliver chemical sources accurately enough to achieve reproducible results. Therefore, midway through this project, the apparatus was upgraded from the old home-built reactor to a new state-of-the-art system purchased from Emcore Inc., for \$800,000. A picture of this tool is shown in Fig. 1.

The new vertical rotating-disk reactor includes a loadlock for sample entry and transfer into ultrahigh vacuum (for *in situ* characterization). Sample platters hold three 2-inch wafers, one 3-inch wafer, one 4-inch wafer, or four 1x2 cm² custom sleds. During growth the carrier rotates at 1000 rpm and is heated radiatively. A total hydrogen flow of 25 standard liters per minute is used. Thickness and composition



Fig. 1: The Emcore Discovery-125 MOCVD system at UCLA.

uniformity of $<1\% 1\sigma$ has been demonstrated by this reactor on a 2- μm -thick GaAs sample.

The reactor is equipped with a RealTemp™ system that combines an infrared pyrometer with a laser reflectometer for emissivity-corrected measurement of the substrate surface temperature and growth rate. The pyrometer measures temperature as a function of emissivity, and the reflectometer is needed since the emissivity may change during growth. The principle behind RealTemp™ is that as different layers are grown, changes in the refractive index of the semiconductor layers leads to interference between the light emitted from the interface and the surface, producing a sinusoidal variation in the signal intensity [5,6]. The period of the sine function varies inversely with the growth rate. Curve-fitting software provided by Emcore yields the growth rate.

Ultra-high purity hydrogen was used as the carrier gas after being passed through a heated getter column. The following precursors were used: tertiarybutylphosphine (TBP), tertiarybutylarsine (TBAs, 2 units), trimethylindium (TMIn, 2 units), trimethylgallium (TMGa, 2 units), tertiarybutylhydrazine (TBHy), and carbon tetrabromide (CBr₄). Tertiarybutylarsine and tertiarybutylphosphine were used in place of traditional hydride sources primarily for safety reasons. These metalorganics also have a higher cracking efficiency than the hydrides, thus enabling growth at lower temperatures.

The MOCVD reactor was interfaced to an ultrahigh vacuum (UHV), surface characterization system. At any step during MOCVD, the growth could be interrupted and the sample transferred to the UHV system for analysis. After analysis, the sample could be transferred back to the reactor and the growth process continued. A schematic of the apparatus is shown in Fig. 2. It is equipped with the tools necessary to characterize and manipulate matter on the atomic scale. In particular, the vacuum chambers house a scanning tunneling microscope (STM), x-ray photoemission spectrometer (XPS), infrared spectrometer (IR), low-energy electron diffractometer (LEED), and reflectance difference spectrometer (RDS). Scanning tunneling

microscopy is used to probe the surface with atomic resolution. This equipment is especially useful for studying the formation of heterojunctions. It provides information on how the surface roughens when treated under different process conditions. Low-energy electron diffraction is

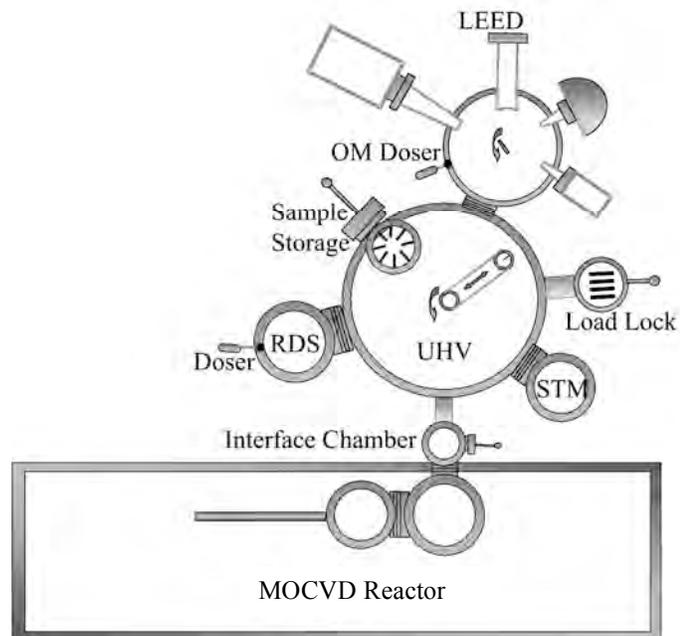


Fig. 2: Schematic of the combined MOCVD reactor and UHV surface analysis system.

used to characterize the long range ordering on the surface. Also, from the sharpness of the LEED spots, one can assess the surface quality. The x-ray photoemission spectrometer analyzes the surface composition and can identify the level of carbon and oxygen contamination. When studying interface formation, we can monitor the intermixing of elements using XPS. The reflectance difference spectrometer is an optical tool that enables us to observe the changes in surface structure in-situ.

After characterization of the films in the UHV chambers, the samples were removed from the apparatus and characterized by high-resolution x-ray diffraction (XRD), photoluminescence (PL), and Hall measurements. For XRD, the full-width-at-half-maximum of the triple-axis-rocking curve was used as an indicator of crystal quality of the epitaxial layers. The thickness and composition of the InGaP and InGaAsN films were assessed by double-axis XRD, based on an analysis of the peak splitting and fringe patterns [7]. Photoluminescence and Hall measurements were used to determine the bandgap energy and the carrier concentrations of the films. These results were correlated to the STM and XPS data and the growth conditions.

The InGaP epitaxial layers were grown at 575 to 600 °C, in 20 Torr of hydrogen, at V/III ratios between 10 and 100, using TMI_n, TMGa, TBP, TBHy and TBAs. The use of these alternative precursors allowed us to explore a wider range of process conditions, particularly in the low-temperature, non-equilibrium regime. At each stage of growth, i.e., the GaAs buffer layer, the interface, and the InGaP epilayer, the films were characterized by STM and XPS.

Thin films of InGaAsN were deposited using the same indium, gallium, and arsenic precursors as described above, and with the nitrogen source being tertiarybutylhydrazine. The InGaAsN layers were deposited at 550 °C in 60 Torr of hydrogen. A V/III ratio of 27.3 was used. Low growth temperatures were required to prevent alloy segregation within the film [8,9]. Throughout the deposition process, the composition and structure of the materials were characterized by scanning tunneling microscopy and x-ray photoemission spectroscopy. The electrical and crystal properties were analyzed by XRD, PL and Hall measurements.

Project Outcomes

InGaP/GaAs Deposition and Heterojunction Formation

Indium gallium phosphide films have been grown in the MOCVD reactor under the process conditions described in the previous section. X-ray photoelectron spectroscopy showed that the composition of the group III elements in this material was 49% In and 51% Ga. X-ray diffraction experiments indicated that the InGaP layer was lattice matched to the GaAs substrate with high crystallinity. It was found that the group III composition was controlled by the ratio of the metalorganic precursors fed into the reactor. This indicated that the growth rate was controlled by mass transport of the group III precursors to the wafer surface.

We have investigated the initial stages of GaAs/InGaP interface formation during film growth. In particular, the atomic structures of the phosphorus-treated gallium arsenide and arsenic-treated indium phosphide surfaces were determined [10,11]. After growing a buffer

layer on the GaAs substrate, the sample was annealed in vacuum, causing the surface to become gallium rich. Exposure to phosphine resulted in the growth of P-terminated islands on the GaAs terraces. As shown in Fig. 3, the islands grew anisotropically in the $[110]$ direction. This interface was formed because the phosphorous dimers strained the sub-surface gallium atoms, preventing further growth along the $[-110]$ direction. The surface did not go through a roughening process, but was simply covered with 2-dimensional islands. These results indicated that GaAs/InGaP interfaces should be atomically abrupt.

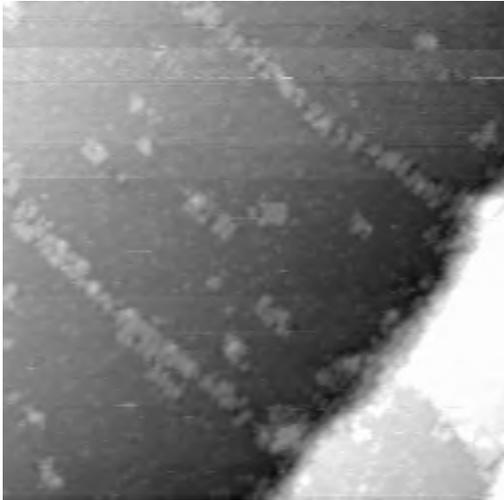


Fig. 3: Scanning tunneling micrograph of the phosphorus-rich islands on the GaAs surface (image size = $1160 \times 1160 \text{ \AA}^2$).

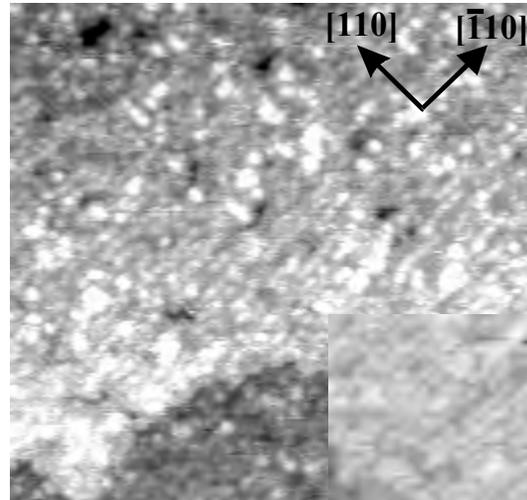


Fig. 4: Scanning tunneling micrograph of an arsenic-treated InP surface (image size = $540 \times 540 \text{ \AA}^2$, inset = $225 \times 225 \text{ \AA}^2$).

Conversely, exposing an indium phosphide film to arsine caused the surface to roughen. As shown in Fig. 4, the treated surface was disordered and contained several phases with different atomic structures. The surface roughness was about 10 \AA for the sample shown in the figure. Evidently, arsenic penetrated into the indium phosphide more easily than the phosphorus penetrated into the gallium arsenide. This explains why it is easier to obtain an abrupt interface for InGaP deposited on GaAs than vice versa.

X-ray photoelectron spectroscopy, low energy electron diffraction and reflectance difference spectroscopy have been used to investigate the surface and bulk structure of InGaP deposited on GaAs (001) substrates. Phosphorus-rich and group III-rich surfaces have been generated, and shown to be well ordered with either (2×1) or (2×4) lattices, respectively. The reflectance difference spectra of the (2×1) and (2×4) phases are presented in Fig. 5a. The former structure exhibits a sharp positive peak at 3.2 eV , while the latter structure exhibits an intense negative band at 2.1 eV . These features are indicative of high quality surfaces on epitaxial thin films.

Depending on the growth conditions, the indium gallium phosphide films grown on gallium arsenide were either disordered, or ordered with a CuPt structure superimposed on the group III sub-lattice. The ordered material exhibited a lower bandgap energy, which is undesirable in

solar cell applications. Both types of crystal lattices have been generated in our MOCVD system. Generally speaking, if the film is grown with a high growth rate, the ordering effect can be minimized. Shown in Fig. 5b are RDS line shapes for the oxide-covered disordered and ordered InGaP films. By oxidizing the surfaces, we observed the bulk contribution to the optical anisotropy of the material. The intense positive peak observed at 3.4 eV demonstrates the high degree of ordering obtained on one of the samples. Now that the process conditions responsible for these crystal transformations have been identified, we can proceed to grow the InGaP material required in solar cell structures.

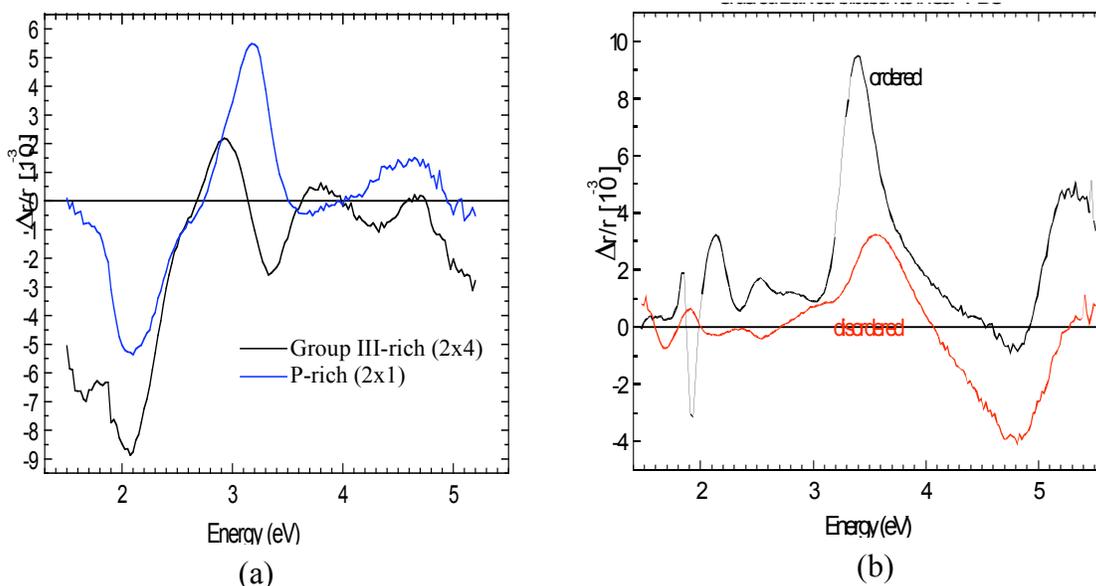


Fig. 5: (a) Reflectance difference spectra of phosphorus-rich and group-III rich InGaP films on GaAs substrates. (b) Reflectance difference spectra of oxidized InGaP/GaAs samples, revealing the bulk contribution to the anisotropy for ordered and disordered materials.

In summary, we have successfully finished the first task originally proposed. These include determining process conditions for optimum transition from GaAs growth to $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$ growth and minimize surface roughness of the $\text{In}_{0.49}\text{Ga}_{0.51}\text{P}$ films.

InGaAsN/GaAs Deposition

Lattice-matched indium gallium arsenide nitride films, 0.5 μm thick, were grown on gallium arsenide substrates by MOCVD using the tertiarybutylhydrazine (TBHy) source supplied by Epichem. The deposition conditions are described in Table 1. Shown in Fig. 6 is the RealTempTM output for the growth process. One can see a regular sinusoidal variation in the emissivity intensity which indicates that high quality InGaAsN layer was grown on the GaAs substrate. From the period of the sine function, the growth rate was calculated, which was 2.0 $\text{\AA}/\text{sec}$.

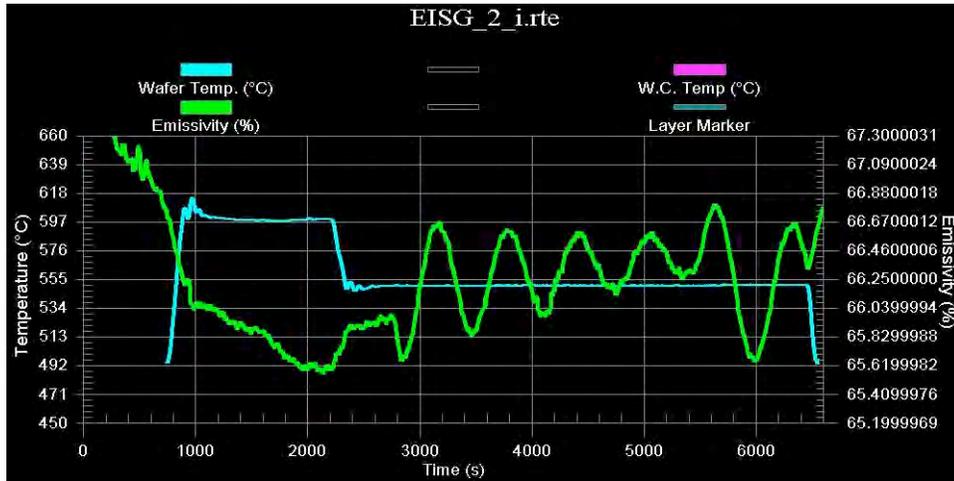


Fig. 6. RealTemp™ data file for the growth of the InGaAsN on a GaAs substrate

Table 1. InGaAsN growth conditions.

Precursors	TMIn, TBHy, TMGa, TBAs
Growth temperature	550 °C
V/III	27.3
TBHy/V ratio	0.67
TBIn/III ratio	6.6 %
Growth Rate	2.0 Å/sec

An x-ray diffraction rocking curve for this material is presented in Fig. 7. The peak labeled S is of the substrate, while the peak labeled L is of the InGaAsN. A peak separation of 110 arc-s is indicative of a small lattice mismatch with the GaAs substrate, equal to 0.00045.

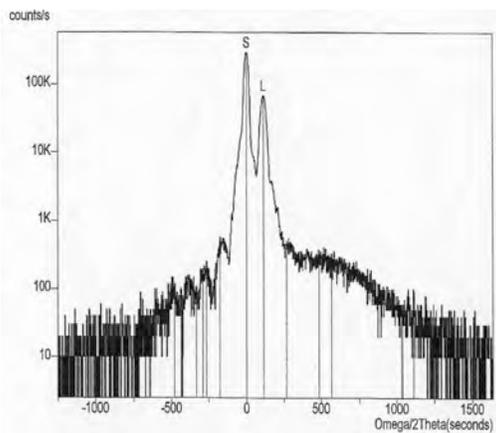


Fig. 7: High-resolution x-ray diffraction spectrum of the (004) reflection of the epitaxial InGaAsN film on GaAs.

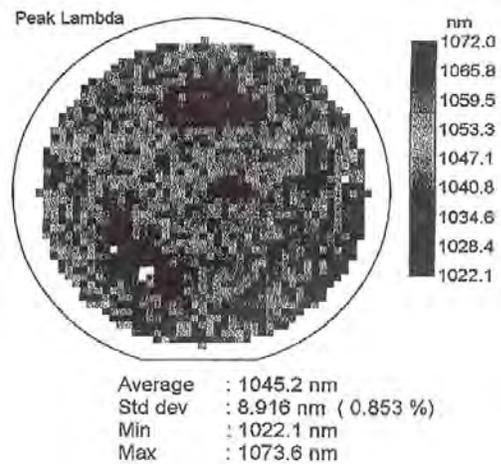


Fig. 8: Photoluminescence spectral map of the GaAs/InGaAsN/GaAs structure.

A photoluminescence spectral map of the InGaAsN layer is displayed in Fig. 8. The average wavelength is 1045 nm with a standard deviation of 8.9 nm (0.85%). Note that the as-grown InGaAsN material exhibited weak luminescence intensity, only 0.007 V. The intensity can be greatly increased by annealing at 500 to 700 °C. We have tried several treatment methods, including annealing in pure N₂ at different temperatures and annealing in a nitrogen plasma jet where the samples were exposed to activated N species. It was found that all these methods improve the material quality. The best result was achieved with N₂ annealing at 650 °C for 30 minutes, which increased the PL intensity to 1.265 V. In addition, the wavelength of the PL peak shifted to 1082 nm, corresponding to a bandgap of 1.15 eV. The photoluminescence data are summarized in Table 2.

Table 2. Effect of heat treatment on InGaAsN photoluminescence data.

	Wavelength (nm)	PL intensity (V)	FWHM (nm)
No post-growth treatment	1045.2	0.007	54.9
N ₂ annealed at 650 °C	1082.5	1.265	59.6

The composition of In_xGa_{1-x}As_{1-y}N_y film can be determined by solving the following equations simultaneously [12,13]:

Lattice constant (XRD):

$$a_{\text{InGaAsN}} = (1 - x)[(1 - y)a_{\text{GaAs}} + y \cdot a_{\text{GaN}}] + x[(1 - y)a_{\text{InAs}} + y \cdot a_{\text{InN}}] \text{ (\AA)}$$

Bandgap (PL):

$$E_{\text{g}}^{\text{InGaAsN}} = E_{\text{g}}^{\text{GaAs}} - \Delta E_{\text{g}}^{\text{InGaAs}} - \Delta E_{\text{g}}^{\text{GaAsN}} \text{ (eV)}$$

$$\text{where, } \Delta E_{\text{g}}^{\text{InGaAs}} = 1.416 - [1.416(1 - x) + 0.324x - 0.4x(1 - x)]$$

$$\Delta E_{\text{g}}^{\text{GaAsN}} = 1.416 - [1.416(1 - y) + 3.4y - 15y(1 - y)].$$

Based on this, the InGaAsN film after annealing in nitrogen at 650 °C was composed of 4.1% indium (x) and 1.65% nitrogen (y). The target composition was 8.0% indium and 2.0% nitrogen. Nevertheless, the crystalline quality of the material was excellent, and with more time, we would have been able to achieve the target composition.

In summary, for task 2, we finish subtask 2a which is to determine the conditions for depositing high-quality In_{0.08}Ga_{0.91}As_{0.97}N_{0.02} thin films. Many trial runs were performed before

we successfully incorporated nitrogen into the film. For subtask 2b, due to the time and budget limit, we did not achieve an N incorporation of 2.0%. It should only have taken a few more runs to optimize the growth conditions for this film composition. So for subtask 2b, we can claim 90% completion.

Conclusions and Recommendations

Conclusions

In this EISG project, we have addressed several challenging issues in fabricating a quadruple-junction solar cell. The results we obtained are promising and should advance the knowledge of solar cell fabrication techniques. With further work, a quadruple junction cell could be grown and tested. The key conclusions are drawn from the results presented in the “Project Outcomes”:

a) We successfully grew lattice-matched InGaP on GaAs substrates. It was found that the group III composition is determined by the feed ratio of the corresponding metalorganic sources, i.e., the ratio of TMIn and TMGa.

b) Heterojunction formation was studied. We found that when the GaAs surface was treated with TBP, the surface stayed smooth and did not roughen. On the contrary, when the InP surface was treated with TBAs sources, depending on the purging time and temperature, the surface became three dimensional with an RMS roughness of 1.0 to 3.0 nm. These are important technical issues that crystal engineers need to consider when growing heterojunctions.

c) We successfully grew lattice-matched InGaAsN on GaAs substrates. A new nitrogen source, tertiarybutylhydrazine, was used. This source was purchased from Epichem and is still in the development stage. According to our research, TBHy incorporated more efficiently than DMHy into the quaternary alloy. We performed several runs to find out the best growth conditions for growing InGaAsN. In this report, we have shown one sample that was close to the ideal material properties.

Recommendations

The recommendations for the next stage of this project are as follows:

a) Optimize the growth conditions for InGaAsN. Right now we have not incorporated enough N into the film. The ratio of TBHy to TBAs fed to the reactor should be increased further.

b) Study the doping effects. In a quadruple junction solar cell, the four junctions, i.e., the Ge, InGaAsN, GaAs, and InGaP layers are four p-n junctions with p-type and n-type dopants. One needs to identify the growth conditions to achieve the desired doping levels, and study the effect of dopants on the film composition.

c) Grow the device structure. All the layers in the structure of a quadruple junction solar cell need to be grown by MOCVD using the recipe developed during this project. The efficiency of the cell should then be tested and the entire fabrication process optimized.

Public Benefits to California

This project assisted in the development of a manufacturing technology for high-efficiency photovoltaic cells. Photovoltaic cells can contribute a substantial part of California’s future energy requirements. In addition, this technology will reduce the demand on dwindling fossil fuel reserves, and eliminate the environmental and health hazards associated with their combustion. It was shown that III/V compound semiconductor solar cells under high concentration are the most cost effective PV approach for medium-scale power plants (0.1 to 10.0 MW) in high resource areas. California contains many regions with high solar resources; and a lot of investment in PV manufacturing has already been made. It is crucial that we deploy these assets so that solar power will emerge as an important part of California’s energy future.

The MOCVD technology we are using to fabricate the solar cells has high throughput and deposition rate. The bottleneck here is the efficiency. If the cell has over 40% efficiency, the power generation can be achieved at a similar cost to the fossil fuel power production, i.e., ~3.0 ¢/kWh. Here one must also recognize the economies of mass production. The cost of MOCVD is high today because the manufacturing output is so low. As production is scaled up to meet the demands of many generating facilities, the costs will fall dramatically. We do not see these photovoltaics being sold to consumers and installed in homes. Instead, they would be deployed as medium-scale plants, possibly on the roofs of commercial buildings, hospitals and schools.

Development Stage Assessment

Table 3 displays the Stage Activity of our EISG project, which has been completed to Stage 3 (Research and Bench Scale Testing).

Table 3. Project development stage activity matrix

Stages	1 Idea Generation	2 Technical & Market Analysis	3 Research	4 Technology Develop- ment	5 Product Develop- ment	6 Demon- stration	7 Market Transfor- mation	8 Commer- cialization
Marketing								
Engineering/ Technical								
Legal/ Contractual								
Risk Assess/ Quality Plans								
Strategic								
Production. Readiness/								
Public Benefits/ Cost								

Marketing

The concept of a high efficiency solar cell in terrestrial use has been marketed widely in publications, magazines and government reports [1-3]. The public is aware of the benefit of this potential product. In particular for the EISG project, we have attended national conferences and published peer-reviewed articles presenting the research discoveries found here at UCLA. The marketing activity has mainly been on the research stage.

Engineering/Technical

The EISG project solved two important technological challenges in fabricating quadruple junction solar cells. One of them is to grow disordered, lattice-matched InGaP layers on GaAs substrates. The formation of the heterojunction InGaP/GaAs was also studied. Process conditions that ensure smooth interfaces were identified. The other technological challenge we solved is that we successfully applied a new nitrogen source, TBHy, to the growth of InGaAsN. Much better nitrogen incorporation was found as compared to more frequently used DMHy. This knowledge can be transferred to industry to help establish the production process.

Legal/Contractual

There is no patent filed based on this EISG project. We do not see any legal/contractual issues at this point. The knowledge learned from this project is open to the public and could be transferred to industry when necessary.

Risk Assessment/Quality Plans

The materials studied were grown in a commercial MOCVD reactor with adequate safety controls. All waste chemicals were trapped in a solid absorbent for hazardous waste disposal later on. Each experiment was reproduced to ensure quality and reproducibility.

Strategic

This project developed new technology for growing the complex semiconductor heterostructures used in quadruple junction solar cells. Once cells with efficiencies greater than 40% are demonstrated, the University can initiate discussions with Spectrolab, Inc., for technology transfer.

Production Readiness

A commercial reactor was used in the project so that the procedures developed at UCLA can be easily transferred to industry. These processes should be straightforward to incorporate into industrial practice.

Public Benefits/Costs

This project has helped develop the manufacturing technology needed to produce high-efficiency photovoltaic cells. Photovoltaic cells can provide a solution to California's energy requirements. This technology will reduce the demand on dwindling fossil fuel reserves and eliminate the environmental and health hazards arising from combustion processes. Through this research, we have helped foster the use of photovoltaics in electric power generation.

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