Annual NP Accelerator R&D + Data Science AI/ML virtual PI Exchange meeting - November 30, 2021

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MBE, GSMBE, CBE and MOCVD

MBE

Gas Source Molecular Beam Epitaxy

elemental As, P, Ga

- Pressure ~10⁻⁸
 mbar
- Growth rates
 ~ 1 µm/hr
- Very precise control

Molecular beams

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GSMBE

Gas Source Molecular Beam Epitaxy

AsH₃, PH₃, elemental Gallium

CBE

Chemical Beam Epitaxy

AsH₃, PH₃, triethyl gallium (TEGa) or elemental Gallium

Pressure <10⁻⁴
 mbar

Molecular

and gas sources

 Growth rates 0.5-1 µm/hr

MOCVD

Metal organic chemical vapor deposition

AsH₃, PH₃, trimethylgallium (TMGa)

- Pressures >100 mbar during growth
- Growth Rates 10 µm/hr
- Traditionally difficult to get sharp interfaces

Gas sources

Objective

- SPIRE/Bandwidth Semiconductor used MOCVD to grow single-strained-layer GaAs/GaAsP photocathodes (good)
- SVT used MBE to grow strained-superlattice GaAs/GaAsP photocathodes (better)
 - MBE, for purity and precision
 - Beryllium doping
 - Slow growth rate
- In this work JLab, ODU and BNL: focus on MOCVD
 - Strained Superlattice photocathodes
 - Strained Superlattice photocathodes with DBR
- We are about 3 months behind schedule



Tasks Year 1	Q1	Q2	Q3	Q4
Calibration of p-GaAs _{0.65} P _{0.35} (ODU)				
Calibration of metamorphic grade from GaAs to GaAs _{0.65} P _{0.35} (ODU)			
Calibration of GaAs/GaAs _{0.65} P _{0.35} strained superlattice (ODU)				
Mott system: assemble and pump down (BNL)				
System commissioning and calibration (BNL & JLab)				
Fabrication runs strained-superlattice (ODU & JLab & BNL)				
Strained-superlattice Photocathodes Evaluation (JLab)				

	Tasks Year 2	Q1	Q2	Q3	Q4
	Calibration of p-AlAs _{0.6} P _{0.4} (ODU)				
	Calibration of GaAs _{0.65} P _{0.35} /AlAs _{0.6} P _{0.4} DBR (ODU)				
	Strained-superlattice Photocathodes Evaluation (BNL)				
	Fabrication runs strained superlattice with DBR (ODU & JLab & BNL)				
	Measure CsTe/CsI coated SL-GaAs (BNL)				
<	Strained superlattice/DBR Photocathodes Evaluation (JLab & BNL)				



Budget

FY20 loaded budget = 180k\$

- Support for ODU
- Graduate student, summer term
- Procurements: sample evaluation, Mott apparatus upkeep
- 1.7 weeks for Matt, 2.5 weeks for Marcy

	FY20 (\$k)	FY21 (\$k)	Totals (\$k)
a) Funds allocated	180,000	180,000	360,000
b) Actual costs to date	100,703	0	100,703



- a. Metamorphic grading from GaAs to GaAsP_{0.35} to create relaxed layer
- b. Superlattice has strain to increase polarization
- c. Highly p-doped surface to inhibit surface charge limit

GaAs	5 nm	$\rm p=5\times10^{19}\rm cm^{\cdot3}$		
GaAs/GaAsP SL	(3.8/2.8 nm) ×14	p=5 $ imes$ 10 17 cm $^{-3}$		
GaAsP _{0.35}	2750 nm	p=5 $ imes$ 10 ¹⁸ cm ⁻³		
Graded GaAsP _x (x = $0 \sim 0.35$)	5000 nm	$\rm p=5\times10^{18}\rm cm^{\cdot3}$		
GaAs buffer	200 nm	p=2 \times $10^{18}\text{cm}^{\text{-3}}$		
p-GaAs substrate (p>10 ¹⁸ cm ⁻³)				



(b)

(a)



MOCVD growth parameters

- Key Precursors
 - Trimethyl Gallium (Ga(CH₃)₃)
 - Arsine (AsH₃) and Phosphine (PH₃)
 - Diethyl Zinc (Zn(CH₃CH₃)₂)
 - Carbon Tetrachloride (CCl₄)
 - Lower diffusivity of carbon in GaAsP should improve lifetime of device surface
- Substrate: 2" GaAs wafers with either 0 or 2° offcut in the 110 direction. Offcut improves growth quality
- Growth rate range: 3-8 µm/hr
- Temperature: 650-750°C



MOCVD system at Rochester Institute of Technology



Characterization: Nomarski Microscopy

- Microscopic technique with very high resolution in highly contrasting samples
- Shows surface quality indicated by faceting, pitting, and other irregularities



10 μ m/hr 650°C Growth Temp with no Superlattice With a 2° (110) offcut on GaAs



10 $\mu m/hr$ 650°C Growth Temp with no Superlattice With no offcut



Characterization: Reciprocal Space Mapping (RSM)

- Specialized high resolution XRD technique to show relaxation of deposited films
- Key to determining quality of the deposited metamorphic grading



3µm/hr 730°C Growth Temp Close to vertical line: more strain



10µm/hr 730°C Growth Temp Close to diagonal line: less strain



- Numerous sample growth studies performed at Rochester Institute of Technology, with different temperatures and growth rates
- Three types of growth studies
 - Metamorphic Grading
 - Superlattice
 - Full Devices

Results: Metamorphic Grading

- High-quality strain relaxation of underlying layers is key to getting intended GaAs strain in the emitting region
- Necessary because of growing on lattice mismatched substrate.
- RSM used to characterize extent of relaxation in metamorphic layers
- Key parameters changed:
 - Growth Rate
 - Growth Temperature
 - Arsine/Phosphine Ratio





Results: Metamorphic Grading

- Slower Growth Rate resulted in highest strained films (left)
- High Temperature with smaller composition change between steps is more promising (right)





10µm/hr 650°C lower phosphorous grade

Jefferson Lab 12



Results: Superlattice

- Alternating GaAs/GaAsP maintains the strain in the GaAs regions
- Initially grown on GaAs wafers to ensure sufficient doping and layer thicknesses
- Later grown on metamorphic grading so the GaAs is the strained layer
- Interference fringes in XRD show high quality superlattice structure





- Superlattice grown on metamorphic layer
- Not fully relaxed metamorphic layer causes uniformity issues as seen in photoluminescence (PL) mapping







Results: Device Continued

- Surface quality initially poor but subsequent runs improved surface quality
- Refining runs to improve relaxation

Surface completely matted, RSM not performed







7.223

7.207 -

7.19 -

qx, h[110] 4.995 5.002 5.01 5.017 5.025 5.033 5.04 5.048 5.055 5.06

PL Map taken on this sample

1.621E2

1.376E2 1.167E2

9.906E1

8.407E1

7.134E1

6.054E1

4.36E1

3.7E1

3.14E1

2.665E1

2.261E1 1.919E1

1.629E1

1.173E1

9.953E0

8.446E0

168E0

6.083E0

5.162E0

4.381E0

3.155E0 2.677E0 2.272E0 1.928E0 1.636E0

1.389E0

22

7.21

7.13

7.12

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Conclusion

- Multiple devices and superlattice structures have been grown
- Successful doping and compositional calibration of all layers
- Key Remaining Challenge: Relaxation of the metamorphic layer
- Solution: Change offcut of the wafer to facilitate more relaxation, modify substrate temperature and growth rate
- Characterization
 - Surface analysis (SIMS, TEM) planned, working with JLab Procurement Department and NCSU to make this happen
- Ready for first polarization measurements
 - Marcy verifying microMott functionality
 - Student Ben Belfore to work at JLab, using the microMott polarimeter, some training required







MOCVD: Photocathode progress





Marcy Stutzman 10 Nov 2021 P3 Workshop

Results: MOCVD



Higher temperatures yielded improved surface with moderate relaxation throughout

730°C growth temperature

Optimizing temperatures, graded layer profile

100 um

Marcy Stutzman 10 Nov 2021 P3 Workshop

MOCVD monitoring: graded layer optimization



Results: MOCVD photocathode progress

- Graded layer "metamorphic" test runs
 - Optimizing parameters for highest relaxation
 - Hall effect measurements for dopant characterization
- Superlattice runs
 - Growing superlattice on each metamorphic run
 - Optimizing parameters with zinc dopant
- Characterization
 - Surface analysis (SIMS, TEM) planned
 - Ready for first polarization measurements
 - JLab: MicroMott Polarimeter
 - BNL: Specs Mott Polarimeter
 - Operational, testing various samples



X-ray reciprocal space mapping

Crystal growth by Ben Belfore under the supervision of Sylvian Marsillac, Old Dominion University MOCVD system at Rochester Institute of Technology

Photocathodes with 90% polarization and QE > 1% for DOE NP

Matt Poelker , JLab Erdong Wang and Wei Liu, BNL

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Project description and tasks (BNL part)

- Commission a new low voltage Mott polarimeter.
- Evaluate samples provided by Prof. Marsillac, comparing results with those obtained by TJNAF
- Tasks year 1: (All completed)
 - Mott system: assemble and pump down
 - System commissioning and calibration
 - Strained-superlattice photocathode evaluation



BNL Budget summary

	Baseline Total Cost	Costed &	Estimate	Estimated
Item/Task	(۵۷/۲)		IO Complete	
	(AYŞ)	(AYŞ)	(AYŞ)	(AYŞ)
Photocathodes with 90% polarization and QE > 1% for DOE NP	100,000	50,975	49,025	100,000
Total	100,000	50,975	49,025	100,000

	FY2021
a) Funds allocated	100,000
b) Actual costs to date	40,464
c) Uncosted commitments	10,511
d) Uncommitted funds	49,025
(d=a-b-c)	



Drawing of Mott polarimeter system



The system has 3 parts:

- Load-lock manipulator (BNL)
- Preparation chamber (BNL)
- Polarimeter (Specs)

Feature of the system:

- The load-lock system is matched to the polarized gun load-lock.
- Use the same cathode puck as the gun puck.
- The Mott system is light source II beamline compatible.

Specs Mott polarimeter system Overview

• Laser: 450 -850 nm, 1-5 mW/nm

SuperK Laser

- Voltage: up to 25 kV
- Vacuum: low 10⁻¹¹ torr
- Photocurrent: <1 mA

NKTPhotonics

Superk EXTREME



Operating of the system

- Load a fresh photocathode sample into the system. Then, bake the system up to 200 °C to obtain vacuum pressure of low 10^{-11} torr
- Heat clean the photocathode sample at 550 °C
- Activate the photocathode with Cs,(Te) and O₂
- Measure QE and ESP

Process	Laser power	Laser wavelength	Voltage on cathode	Voltage on detector	Voltage on lens	Vacuum pressure
Activation	20-40 uW	532 nm	-200 V	0 V	0	<1e-9 torr
Measure QE	10-100 uW	500 -800 nm	-200 V	0 V	0	~1e-11 torr
Measure ESP	0.1-10 uW	700-800 nm	-200 V	25 kV	0-2000 V	~1e-11 torr



Measured QE

- All photocathode samples are heated clean and activated at same condition.
- The bulk GaAs was firstly tested to confirm the system can get reasonable QE compared with our other activation chamber.
- Tested samples:
 - Bulk GaAs(AXT)
 - > SVT SL (SVT)
 - ➢ SVT DBR (SVT)
 - Luca's DBR (Sadia)



QE of GaAs photocathodes



Polarimeter performance

- The initial energy of electron from photocathode is 200 eV
- The Sherman function is almost linear for ΔE<200 eV
- The theoretical effective Sherman function is 0.27





Measured ESP

- Several GaAs samples have been measured.
- Reasonable ESP for bulk GaAs and SSL GaAs/GaAsP photocathodes are obtained with error < 2% of the value





Conclusion

- We purchased *Specs* Mott polarimeter system.
- Cathode activation system and Mott system have been integrated and commissioned.
- Reasonable QE and ESP of GaAs photocathodes have been measured.
- The polarimeter has a good performance, ESP error is smaller than 2% of the ESP value.
- We achieved all the 1st year goals.



Back up



Activation of GaAs photocathode

- Starts activation when the temperature goes down below 40 °C.
- Activation is performed by yo-yo technique with Cs and O_{2.}
- Photocurrent usually reach peak value after 8-12 yo-yo cycles.
- Photocurrent has a slightly decrease after stopping activation.





