

# Growth and characterization of III-V epitaxial mirror coatings on silicon

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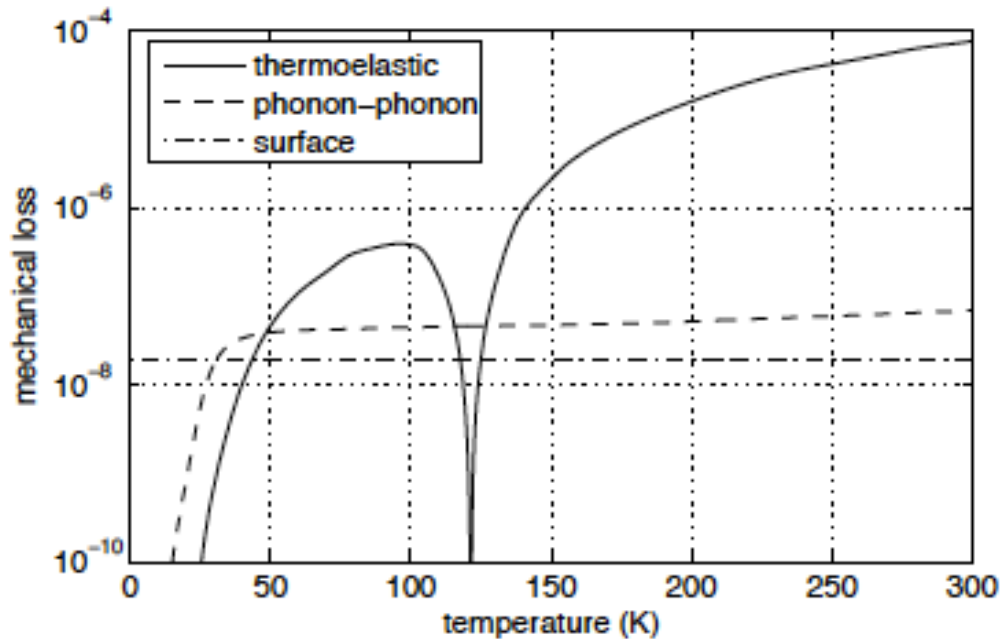
Gravitational Wave Advanced Detector Workshop

May 14, 2012



# Single crystalline coatings

Loss sources in a Si flexure<sup>1</sup>



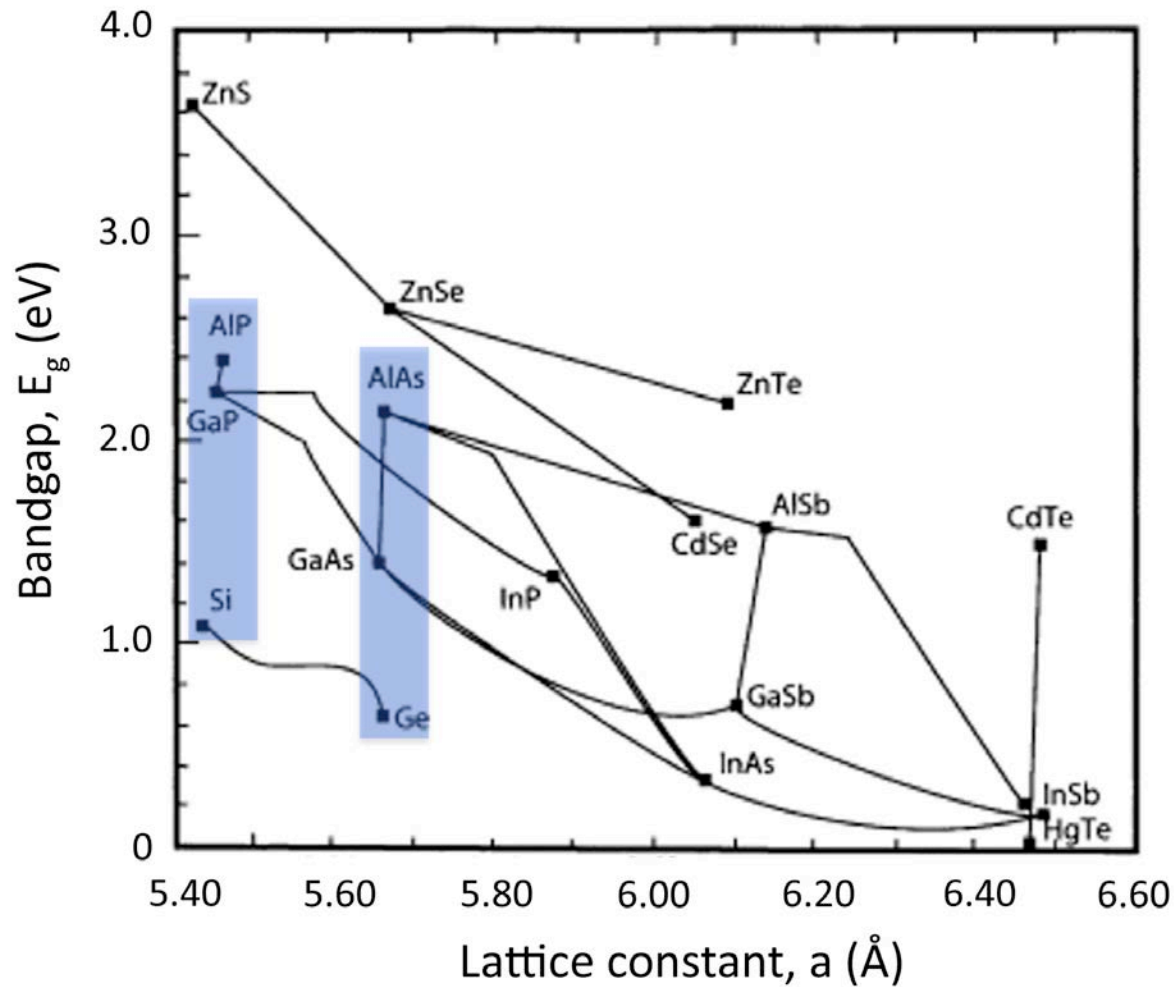
For third generation detectors operating at cryogenic temperatures:

- Low mechanical loss in silicon at low T
- Silicon is available in large sizes:
  - Commercially available: 300 mm (12")
  - Research: 450 mm (18")
- Single crystalline coatings have already been shown to have low mechanical loss<sup>2</sup>

[1] Nawrodt et al. arXiv:1003.2893v1 (2010). [2] Cole et al. Appl. Phys. Lett. 92, 261108 (2010).



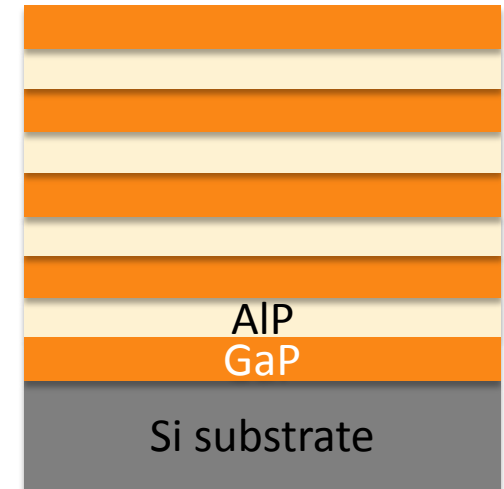
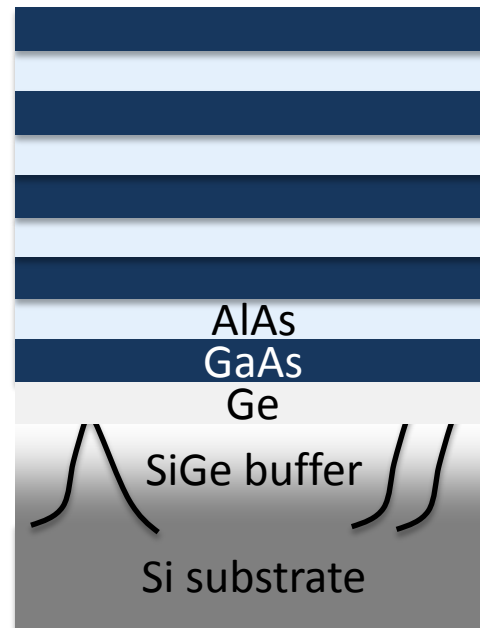
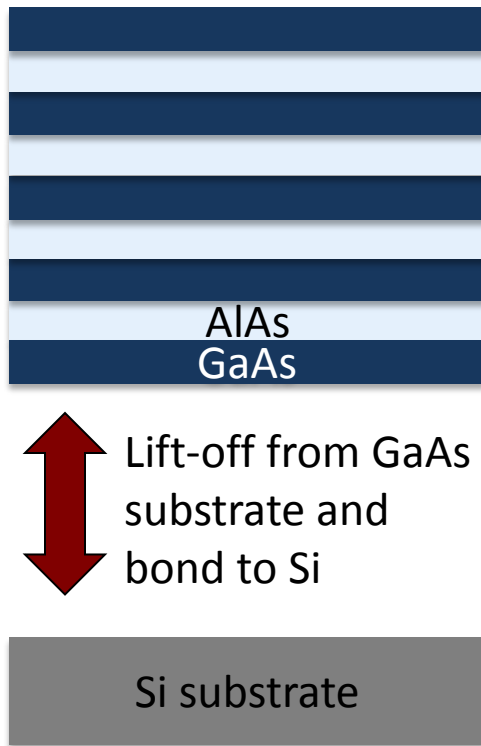
# Direct integration of mirrors onto Si



- Materials systems with index contrast but no change in lattice constant



# Integration approaches



- Commercially available GaAs: 6"

- Dislocations in SiGe arise from 4% mismatch between Si and Ge

- Lattice-matched system, but less studied



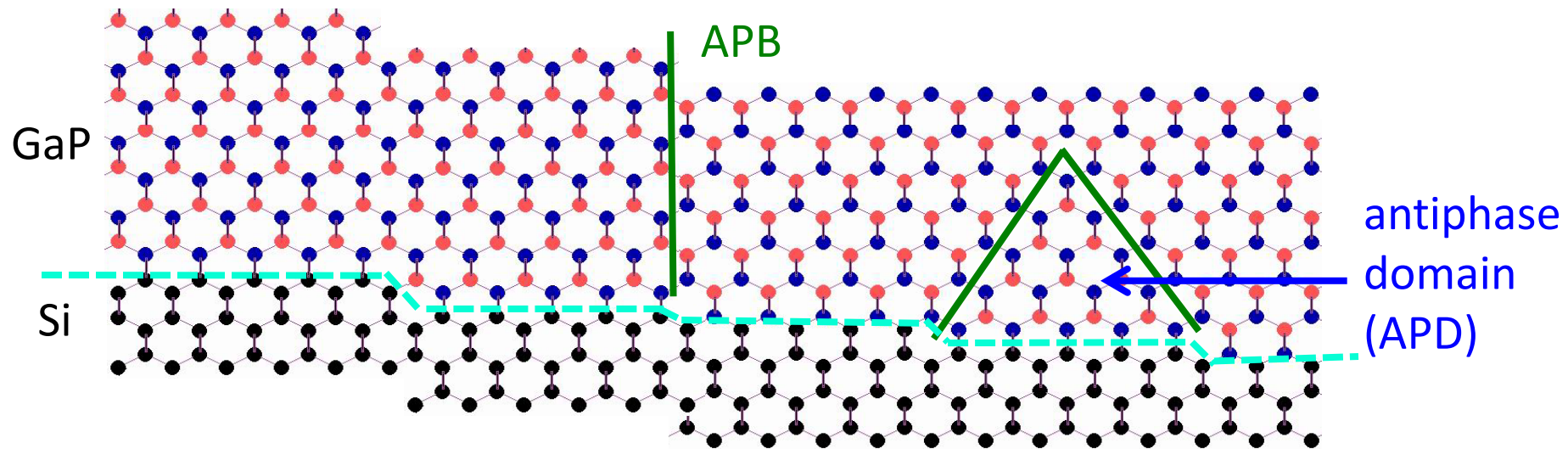
# Outline

- Epitaxial integration of III-V coatings on Si
- Anticipated challenge: antiphase domains
  - Growth technique: molecular beam epitaxy
  - Film characterization techniques
  - Reducing antiphase domains through growth conditions
- GaP/AIP mirror coating on Si: initial results
  - Materials characterization
  - Optical absorption
  - Mechanical loss
- Summary and future work



# Anticipated challenge: antiphase defects

Antiphase boundary (APB): defect with incorrect bonds (P-P or Ga-Ga)

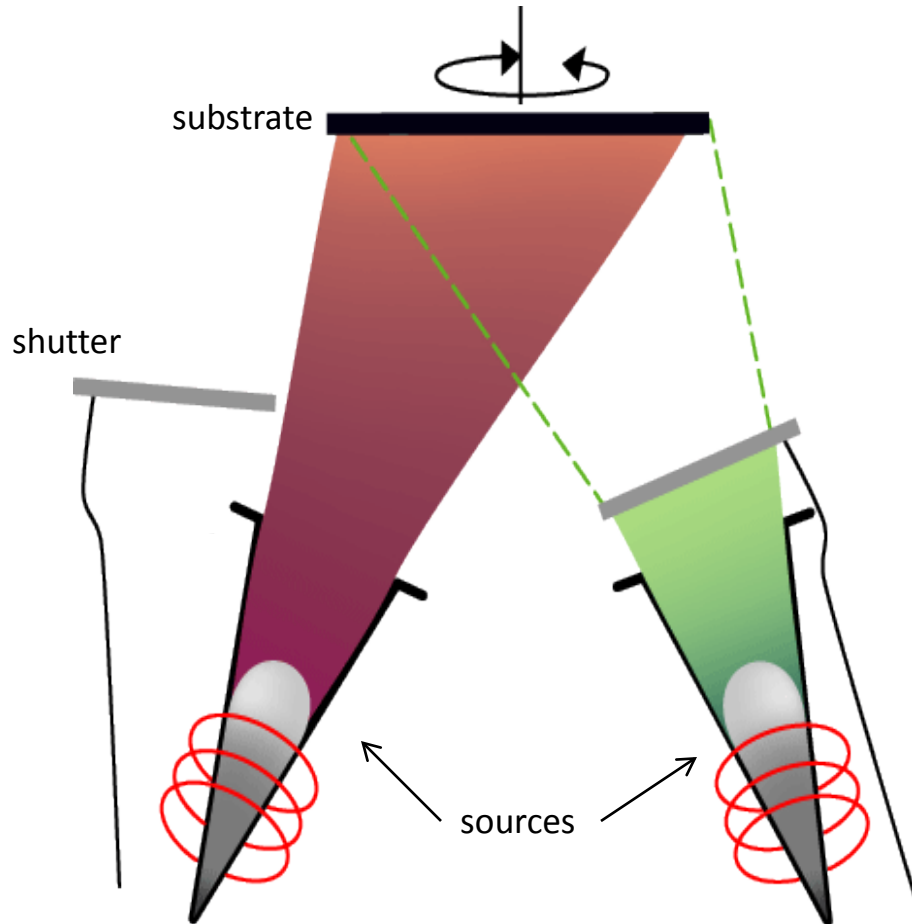


Double atomic steps on Si surface  
→ correct bonds

Single atomic steps on Si surface  
→ wrong bonds



# Growth by molecular beam epitaxy



## Sources and components:

- Group III: Ga, Al, In
- Group V: As, P
- Group IV: Ge, Si
- In-situ reflection high-energy electron diffraction (RHEED)

## Advantages of using MBE:

- Low impurity incorporation (UHV)
- Monolayer control
- Growth rate and substrate temperature decoupled

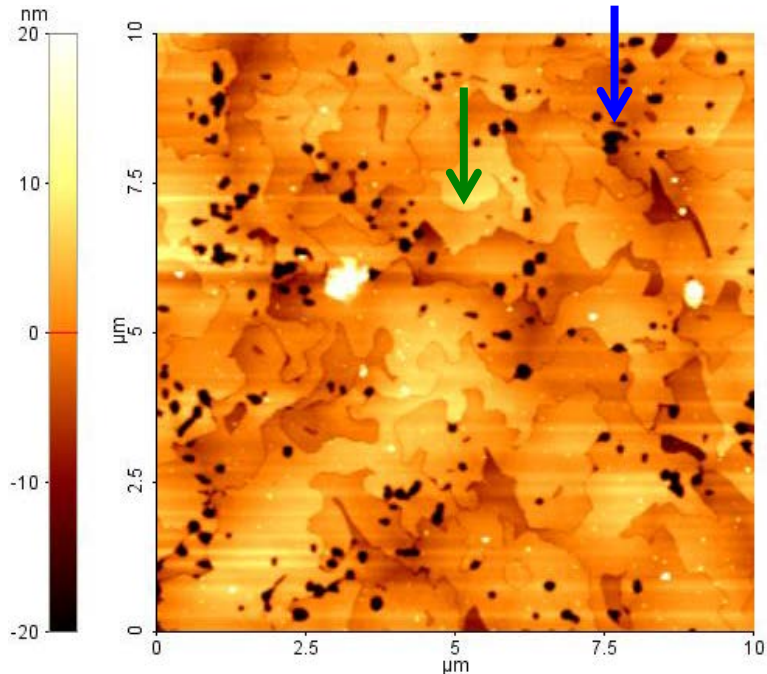
Control over growth rate, substrate temp, V/III flux ratio



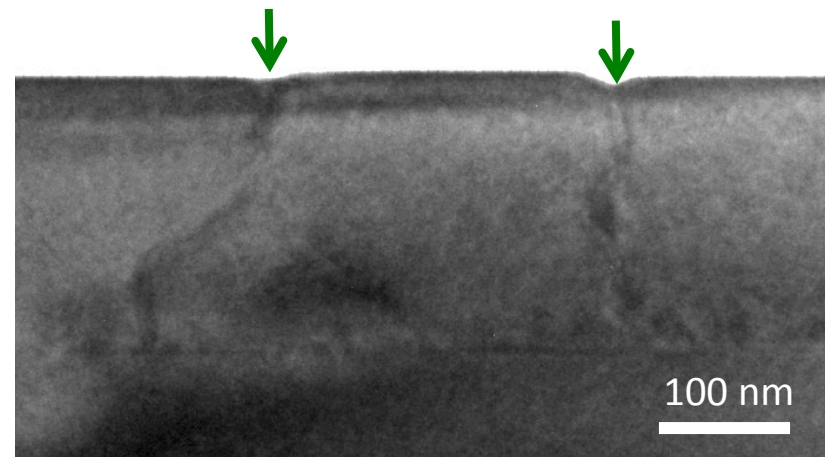
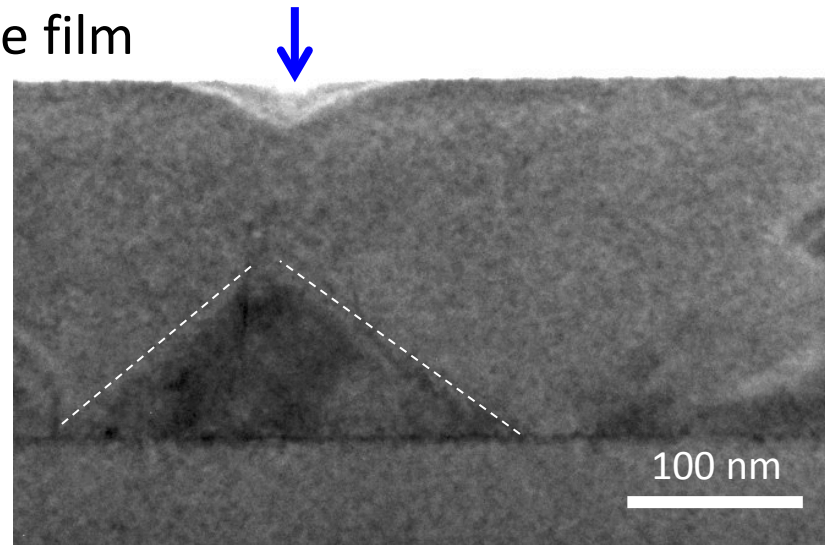
# Ex-situ characterization techniques

Detect APDs with atomic force microscopy (AFM) and transmission electron microscopy (TEM)

- AFM: surface pit density
- TEM: how APBs propagate in the film



A film with many antiphase domains



Sample courtesy of D. Liang, Y. Kang (Stanford)

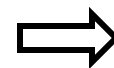


# Achieving high quality GaP on Si

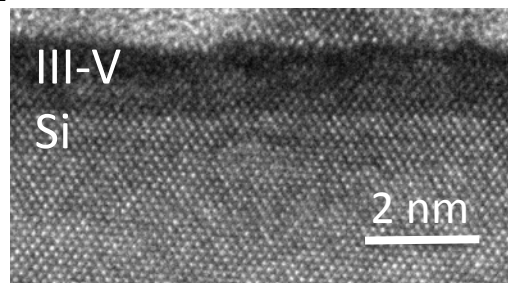
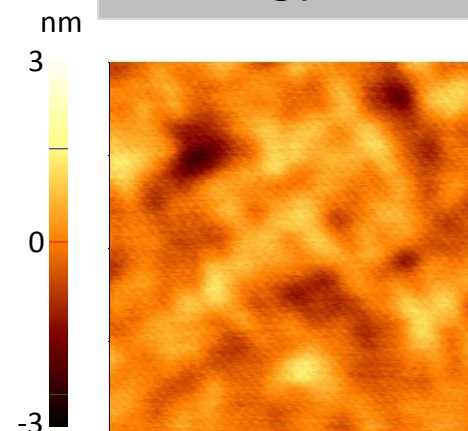
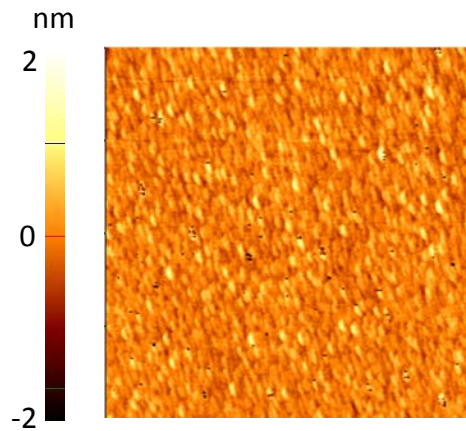
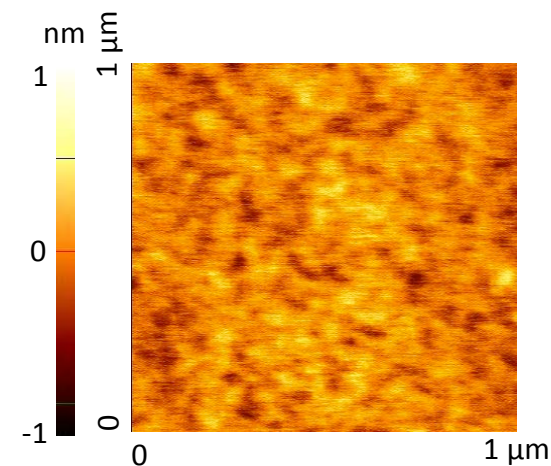
Si



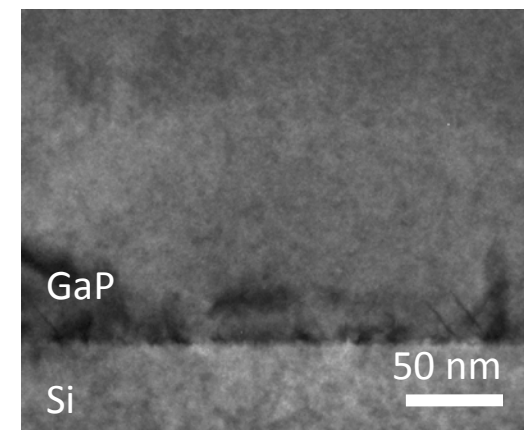
Si



GaP  
Si



III-V nucleation:  
• 2D growth



Si surface prep:

- Standard clean
- Si buffer
- High T anneal

GaP overgrowth:

- Annihilate defects

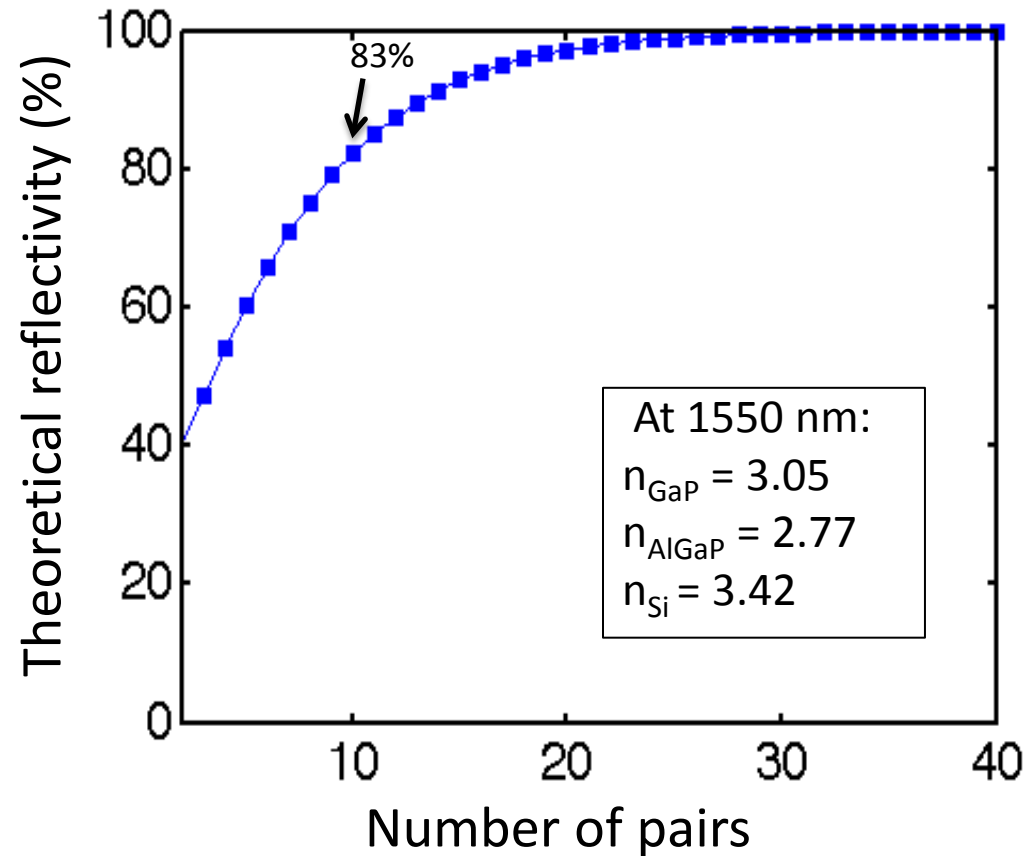
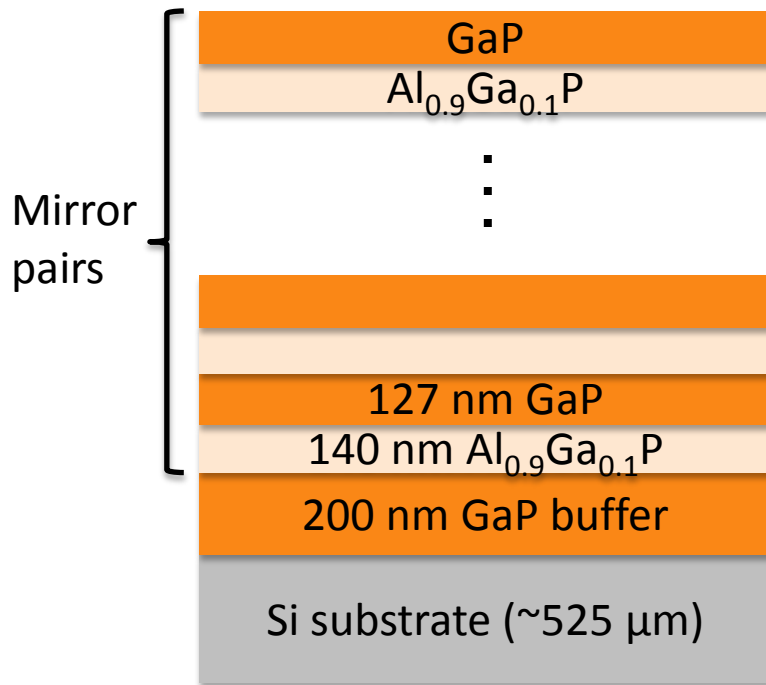


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  - Growth technique: molecular beam epitaxy
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  - Materials characterization
  - Optical absorption
  - Mechanical loss
- Summary and future work



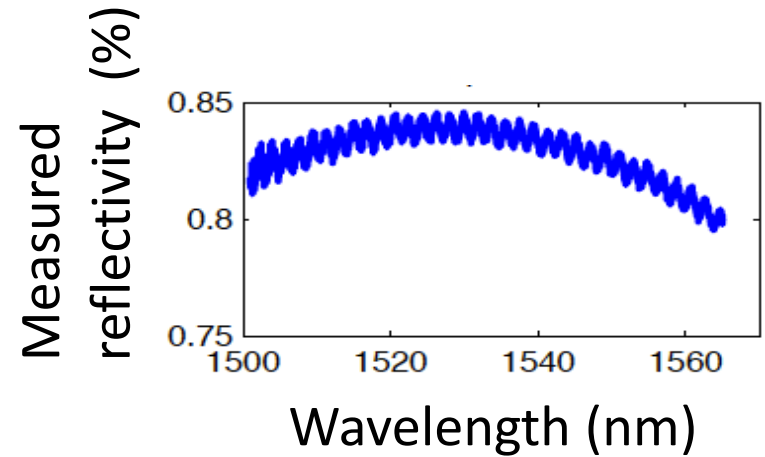
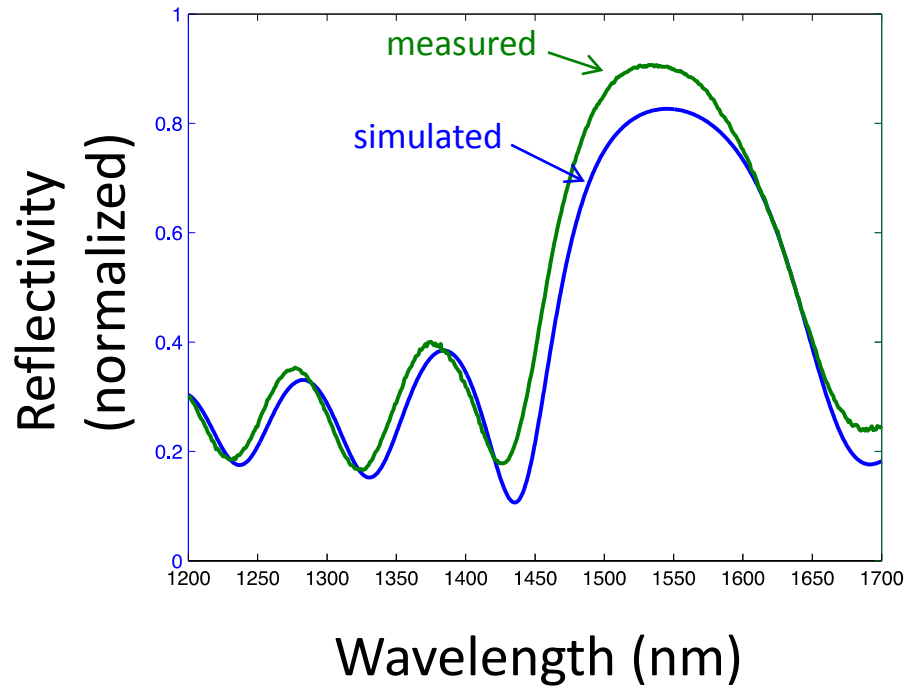
# Epitaxial GaP/AlGaP layers on Si



- Index contrast  $\Delta n = 0.28$
- Pure AlP oxidizes easily



# Achieved expected reflectivity

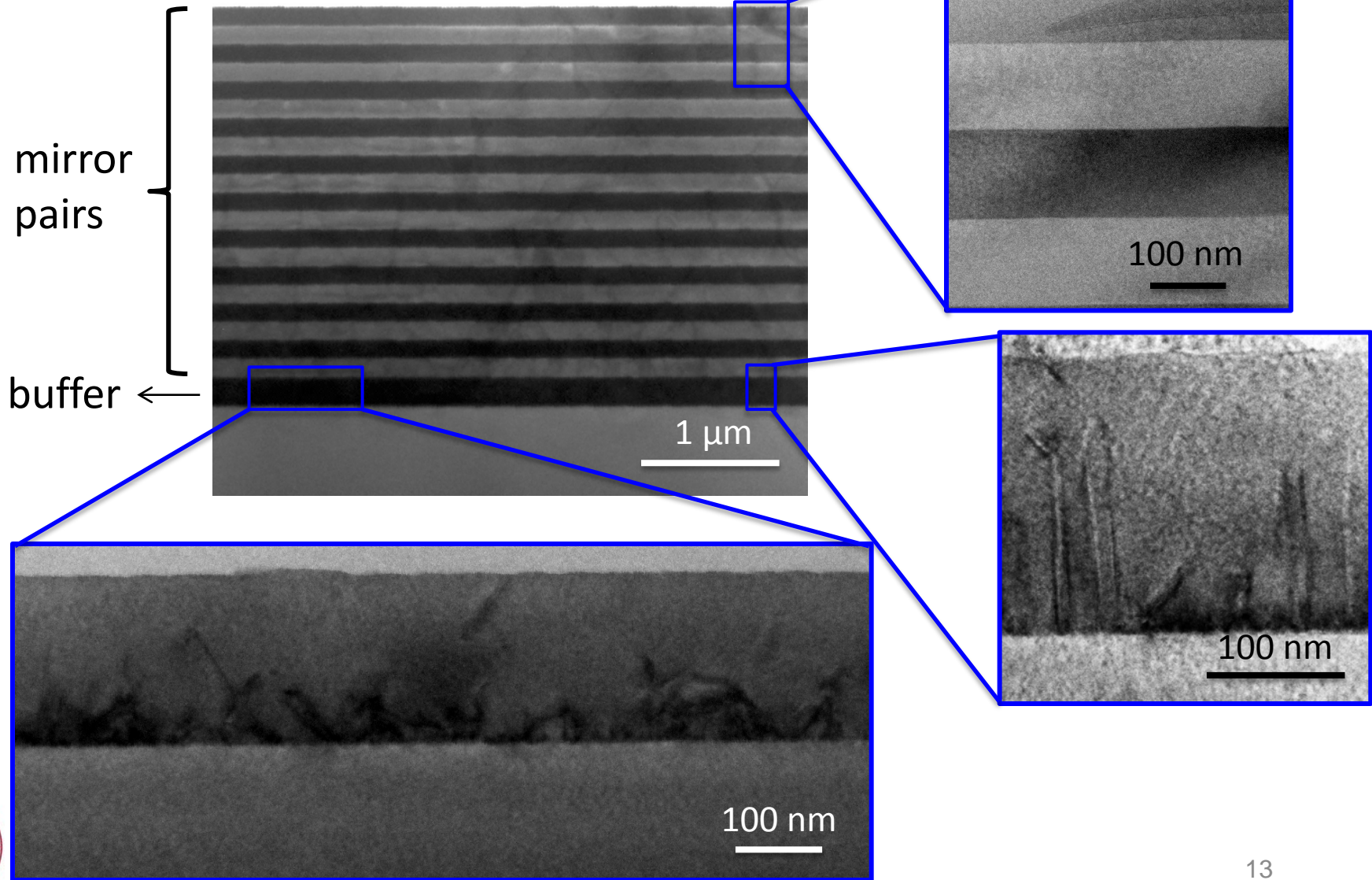


For 10 pairs: measured reflectivity matches theoretical reflectivity (83%)



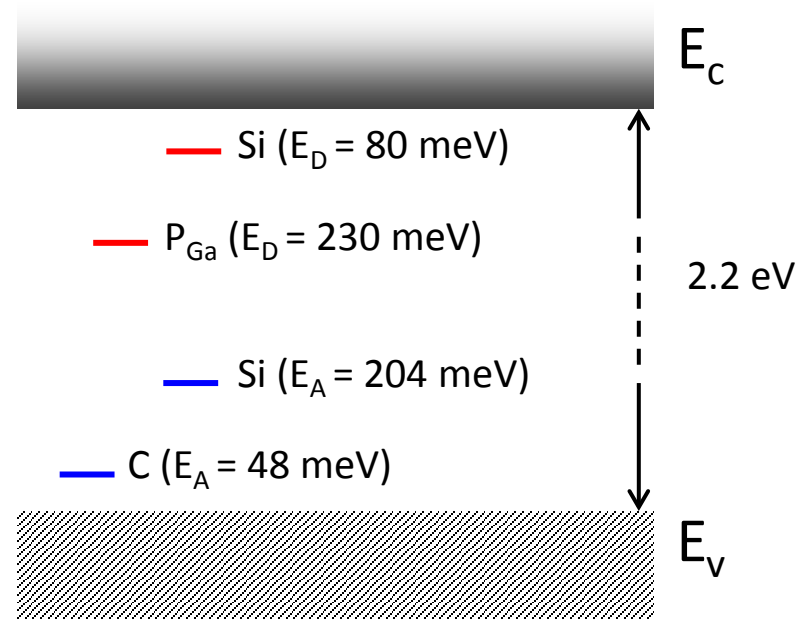
# Transmission electron microscopy

Bright-field 110 TEM: mass contrast



# Optical absorption

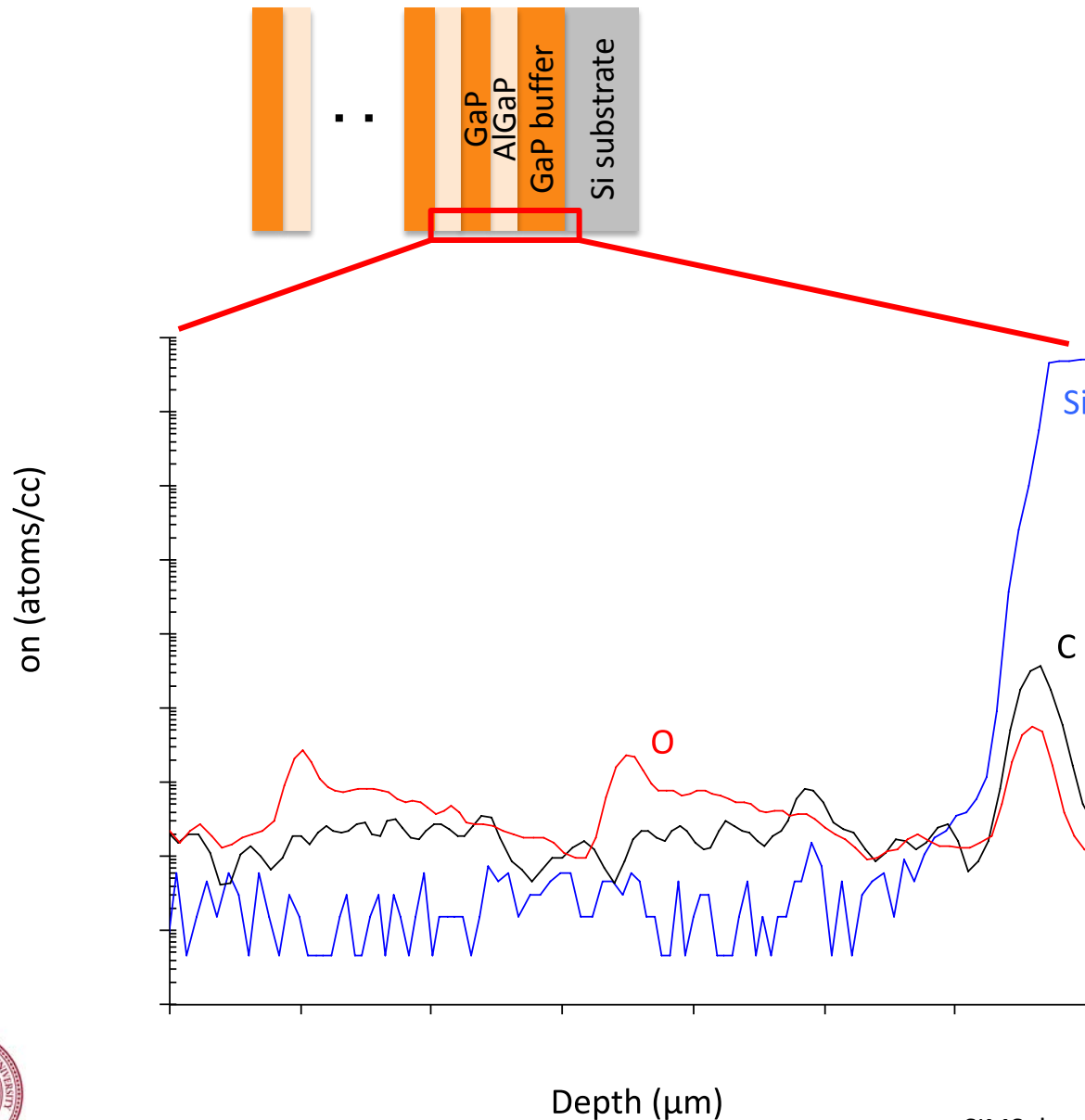
- Using photothermal common-path interferometry (PCI) to measure absorption
- Possible source of absorption: free carriers in GaP<sup>1,2</sup>
  - Si outdiffusion from substrate
  - Carbon, oxygen incorporation
  - Antiphase domains (Ga-Ga, P-P)
- Low temperature will freeze out the carriers



[1] P.J. Dean et al. J. Appl. Phys. 39, 5631 (1968).

[2] K.W. Nauka, Imperfections in III/V Materials (1993).

# Secondary ion mass spectroscopy

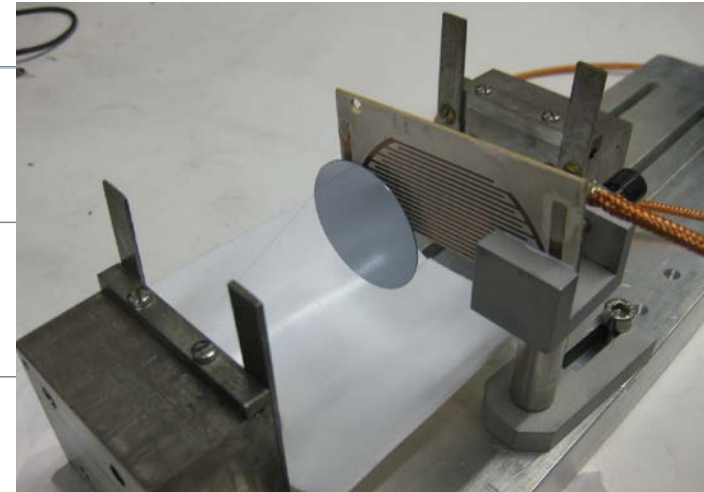
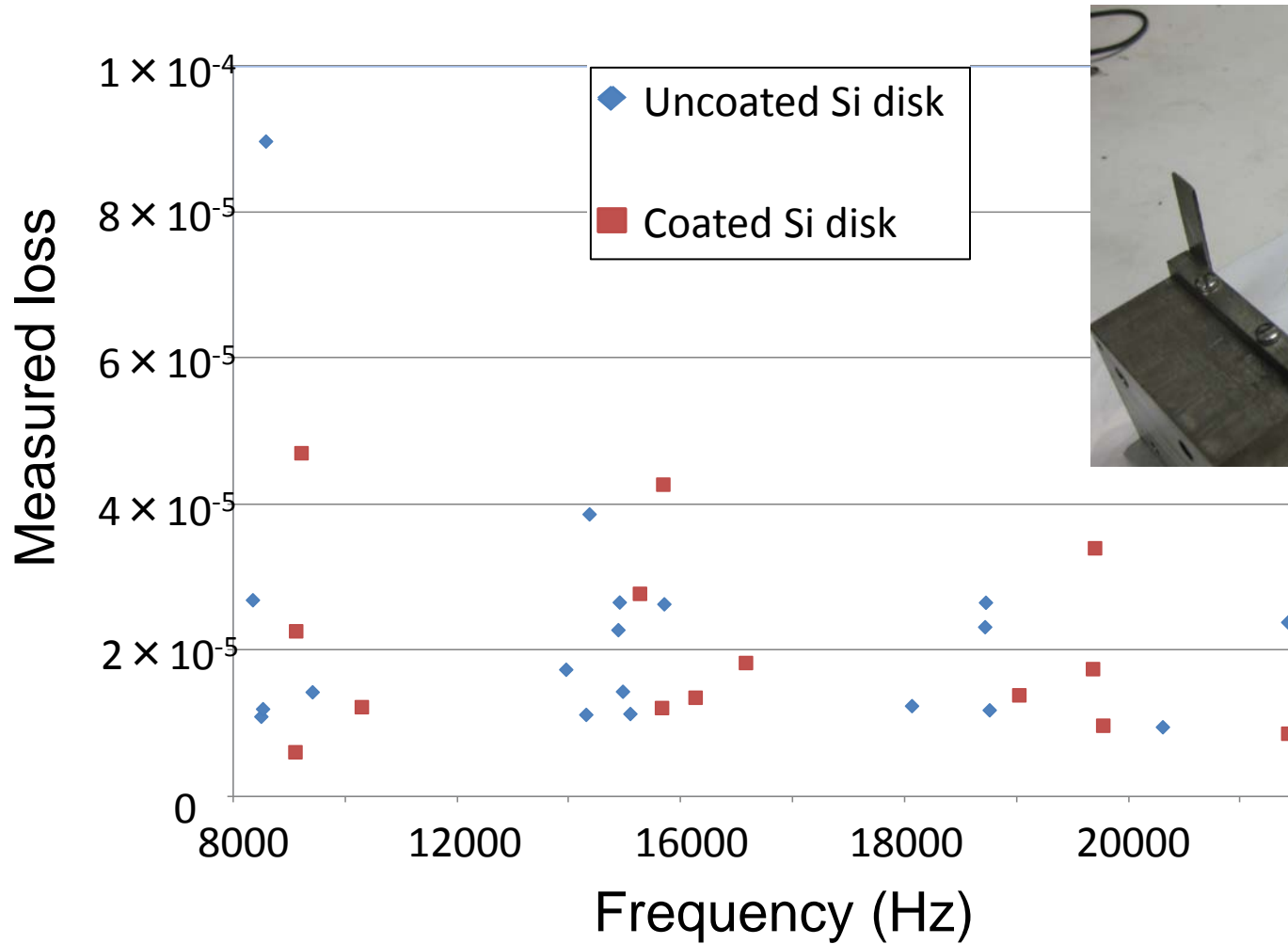


Is there a source of free carriers?

- C, O present at Si/GaP interface
- Si diffusion limited to GaP buffer layer



# Initial results for mechanical loss



No discernible difference between lowest measured losses in coated and uncoated samples





# Summary and future work

- GaP/AlP mirrors can be grown directly on Si
- Challenges with epitaxial integration onto Si can be addressed by nucleation and growth conditions
- Preliminary structural, optical absorption, and mechanical loss measurements have been done on GaP/AlGaP mirrors

## Future work:

- Low temperature absorption measurements
- Low temperature mechanical loss measurements
- Investigating other growth conditions to further reduce defects and impurities

Acknowledgements: This work was supported by NSF grant PHY-10 68596.



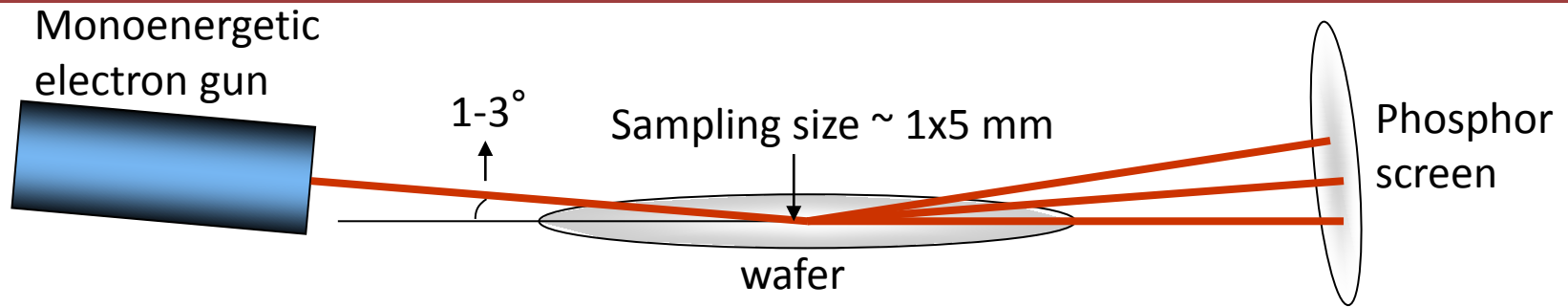


# Material properties

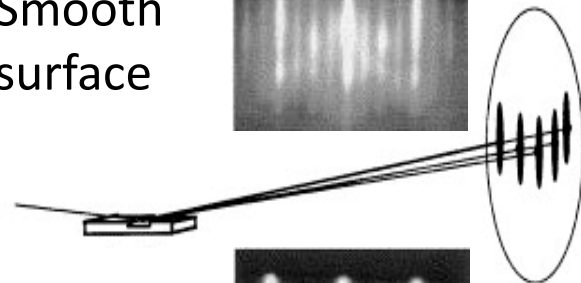
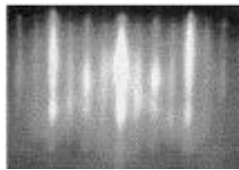
Material	Lattice constant (Å)	Mismatch (%)	Refractive index (near-IR)	Linear thermal expansion coefficient ( $^{\circ}\text{C}^{-1}$ )	
AlP	5.4510	} 0.4	2.75	$6.10 \cdot 10^{-6}$	
GaP	5.4505		} 0.08	3.02	$4.65 \cdot 10^{-6}$
Si	5.4310			3.42	$2.60 \cdot 10^{-6}$
AlAs	5.6611	} 0.08	2.86	$5.20 \cdot 10^{-6}$	
GaAs	5.6533		} 0.08	3.30	$5.73 \cdot 10^{-6}$
Ge	5.6580			4.00	$5.90 \cdot 10^{-6}$



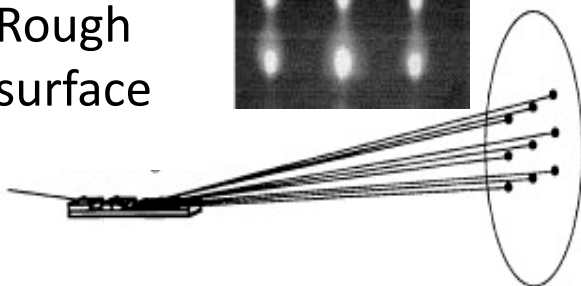
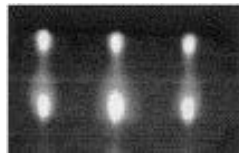
# In-situ characterization: RHEED



Smooth surface

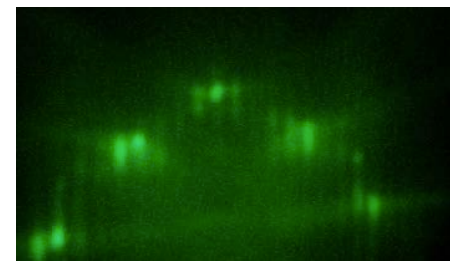
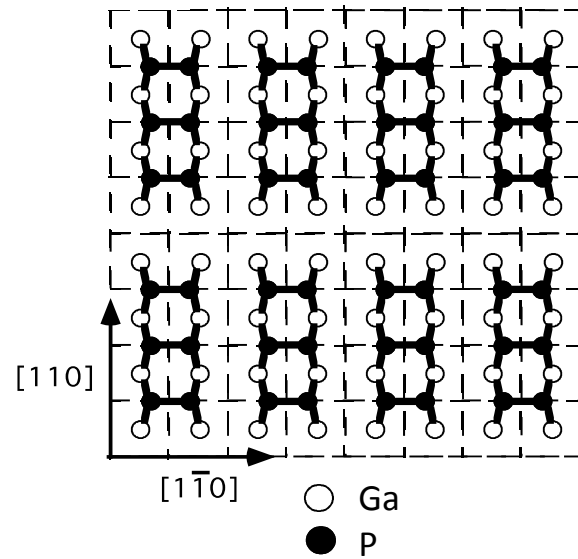


Rough surface

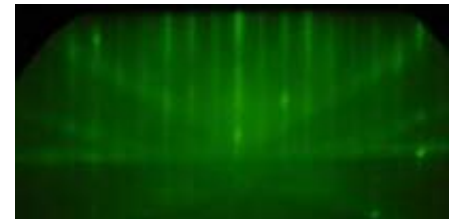


S.A. Chambers, Surf. Sci. Rep. 39, 105 (2000).

GaP (001) 2x4 surface



Beam along [1 $\bar{1}$ 0]



Beam along [110]

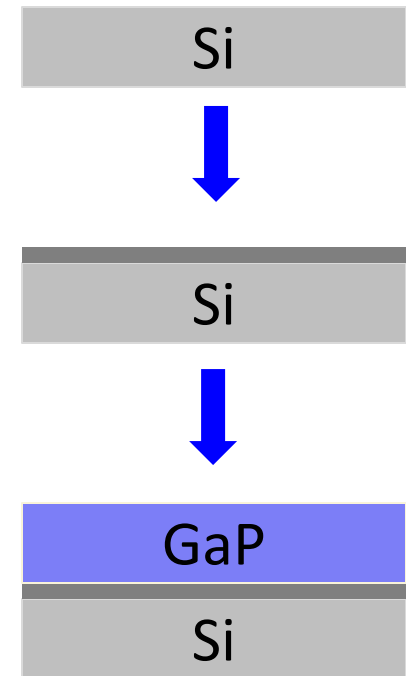
Observe how the film is growing: island or 2D growth mode



# Achieving high-quality GaP on Si

In order of growth sequence:

1. Start with a smooth, double-stepped Si surface
  - High temp anneal under H<sub>2</sub> flow <sup>1</sup>
1. Reduce APD formation
  - 2D nucleation of GaP on Si <sup>2-5</sup>
2. Encourage APD annihilation
  - Control APB propagation <sup>6</sup>



<sup>1</sup>B. Kunert et al. Thin Solid Films 517 (2008) 140.

<sup>2</sup>K. Yamane et al. J. Crys. Growth 311 (2009) 794.

<sup>3</sup>T.J. Grassman et al. Appl. Phys. Lett. 94 (2009) 232106.

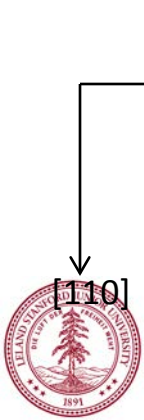
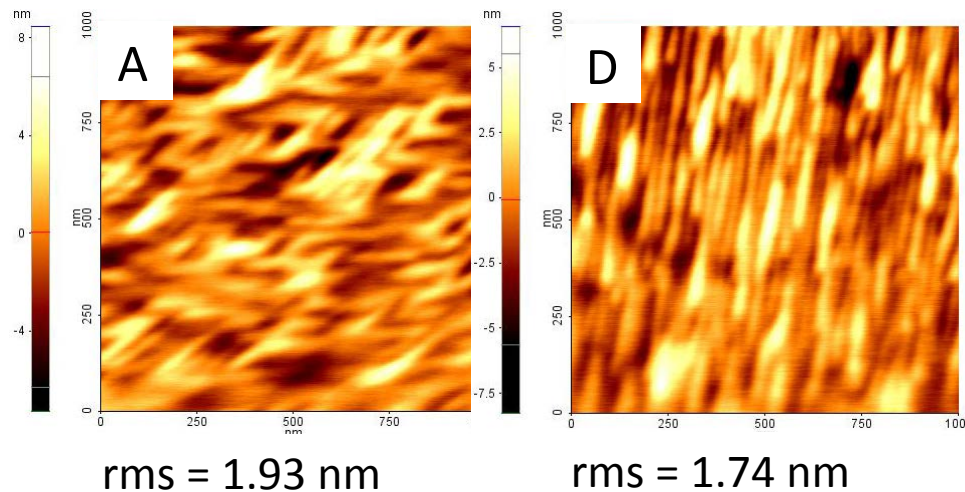
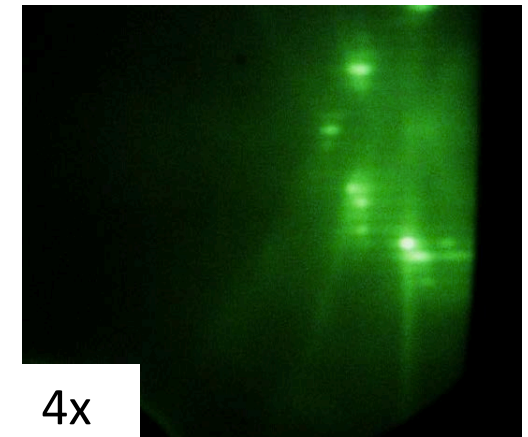
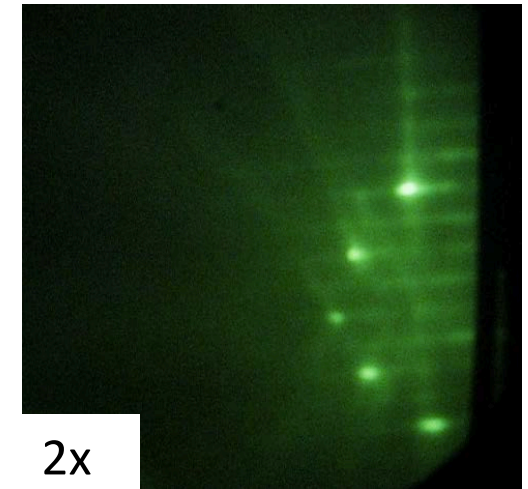
<sup>4</sup>I. Nemeth et al. J. Crys. Growth 310 (2008) 1595.

<sup>5</sup>A.C. Lin et al. J. Vac. Sci. Technol. B 29 (2011) 1201.

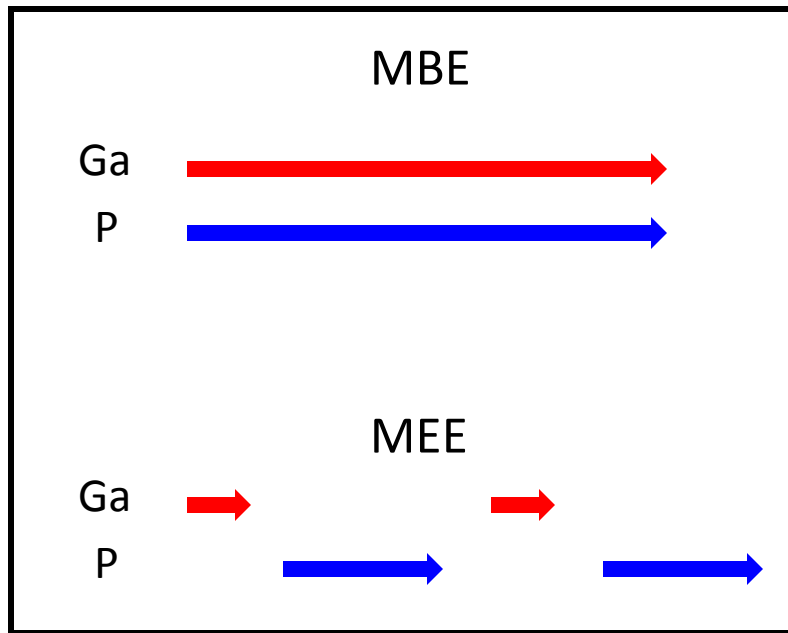
<sup>6</sup>X. Yu et al. J. Crys. Growth 301-302 (2007) 163.

# Control GaP orientation with temperature

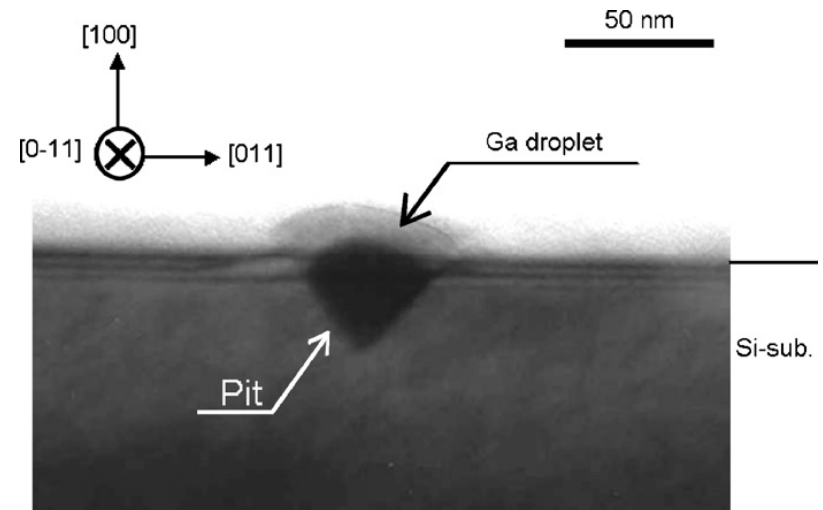
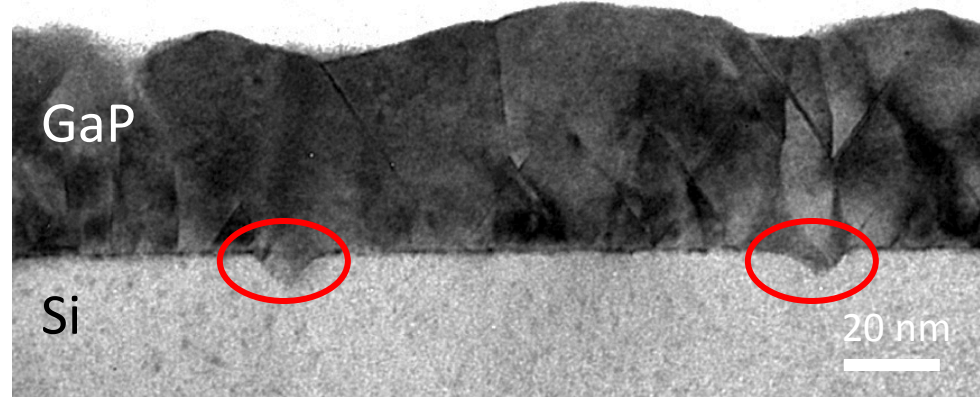
	High T nucleation		Low T nucleation	
Sample	A	B	C	D
Nucleation layer	525° C	500° C	325° C	325° C
GaP film	525° C	550° C 600° C	550° C	550° C 600° C
RHEED	4x2	4x2	2x4	2x4
orientation	-	-	+	+



# Need well-controlled nucleation



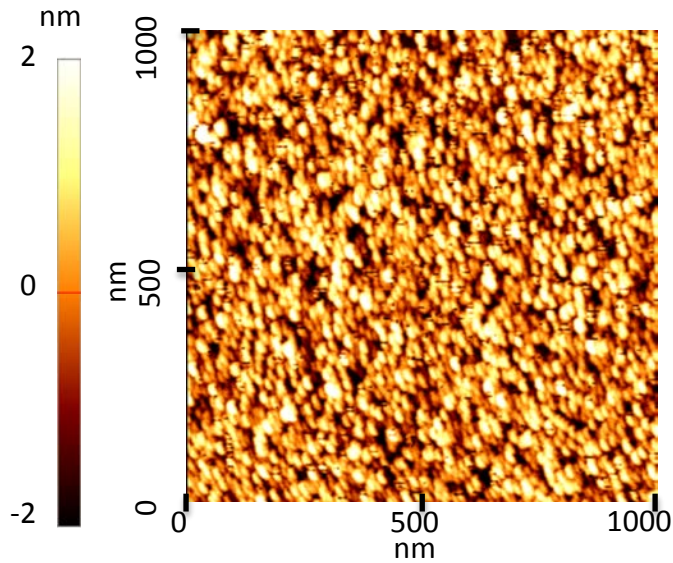
GaP film nucleated with migration enhanced epitaxy (MEE)



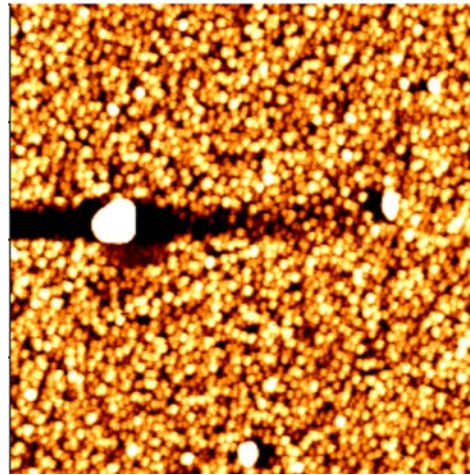
K. Yamane et al. J. Crys. Growth 311 (2007) 794-797.



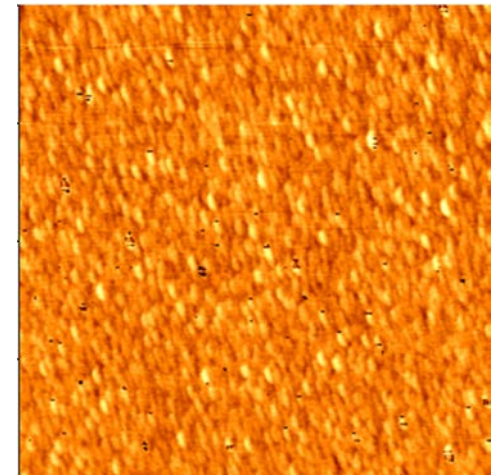
# Two-dimensional III-V film on Si



rms = 0.961 nm

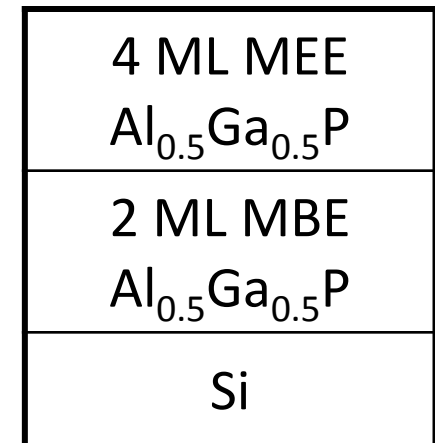
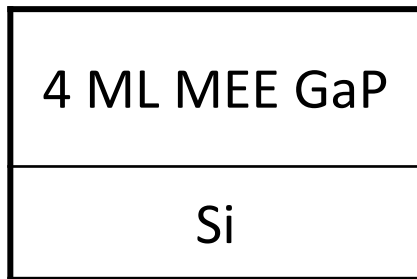


rms = 0.863 nm



rms = 0.279 nm

↑  
growth direction

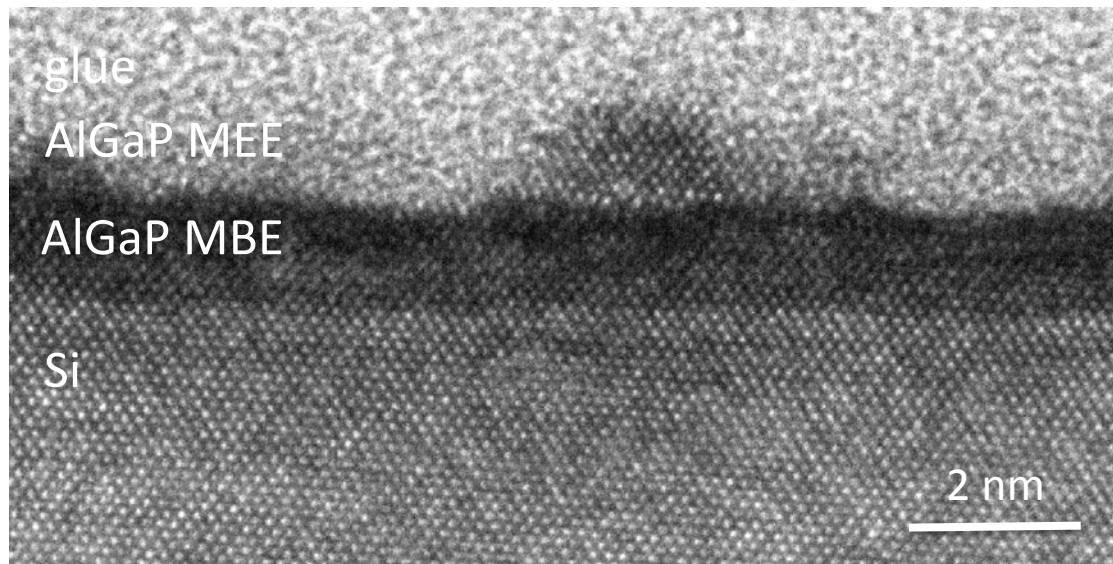
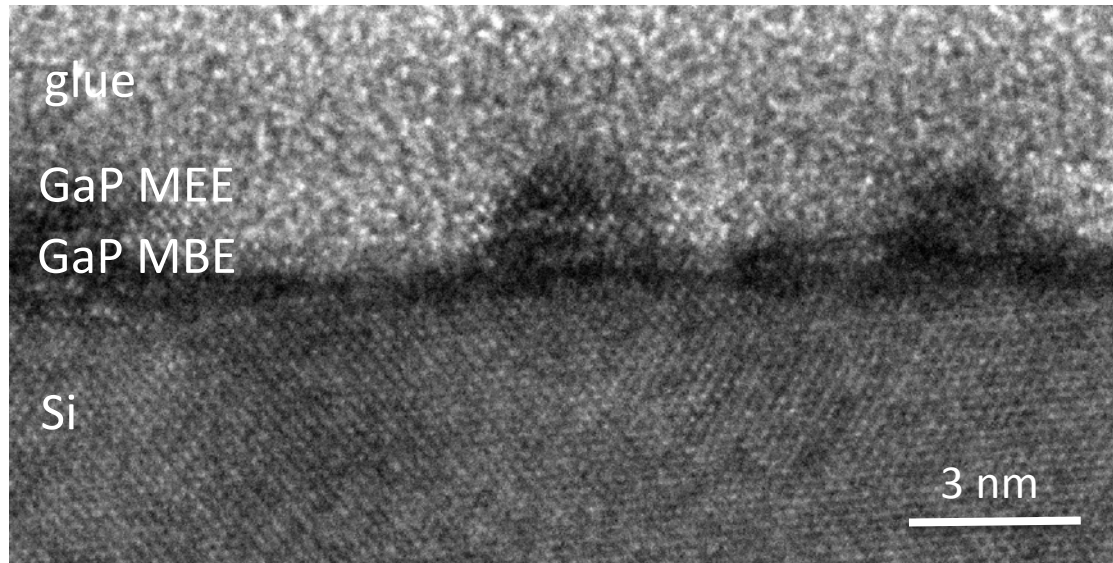


3x reduction in rms roughness with addition of Al

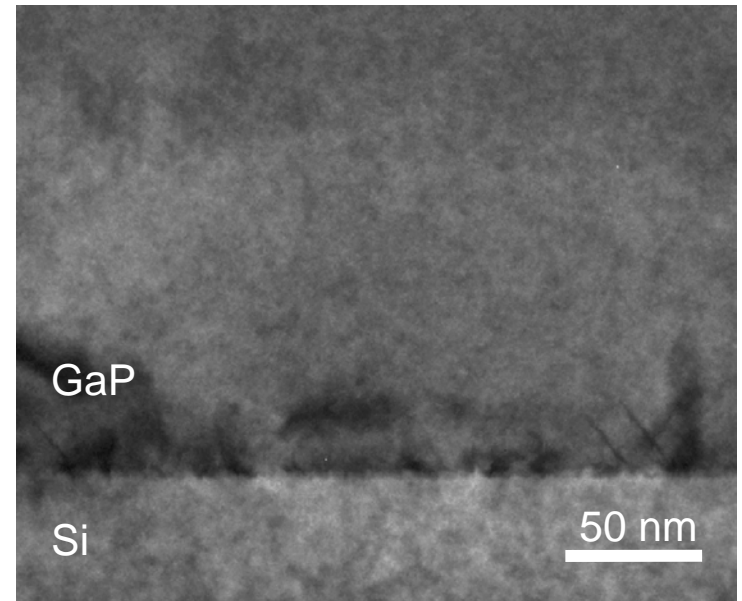
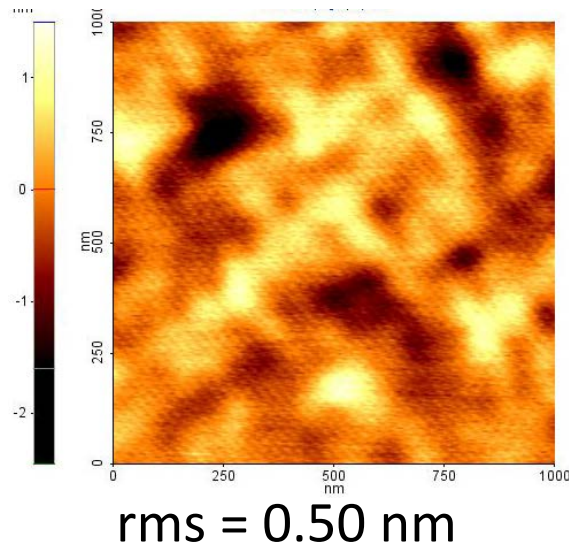
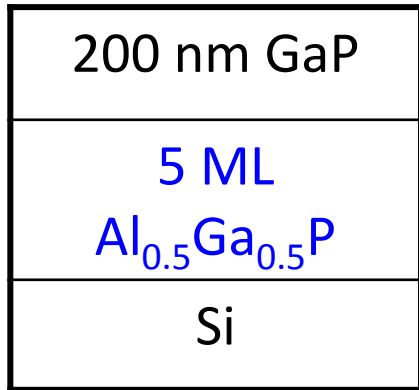
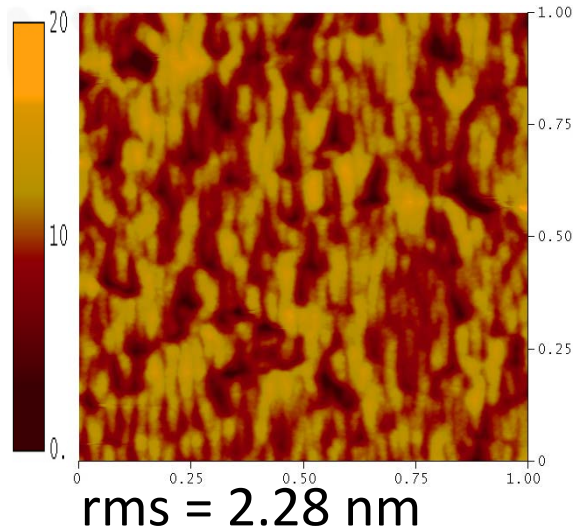
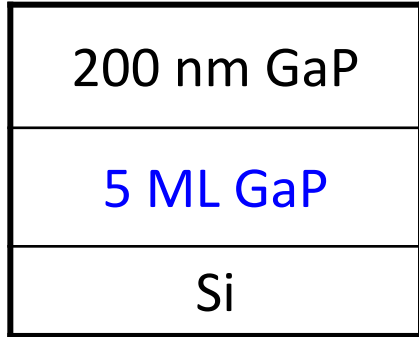




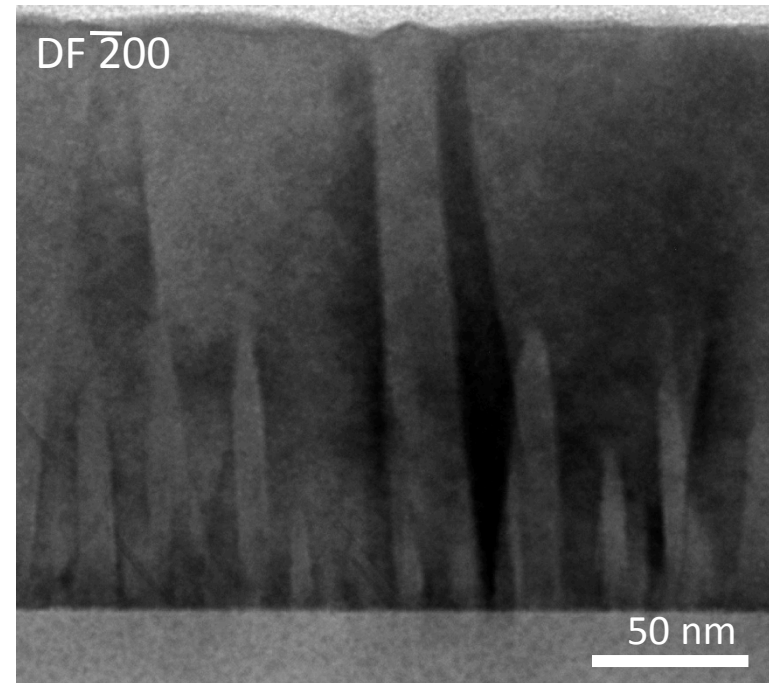
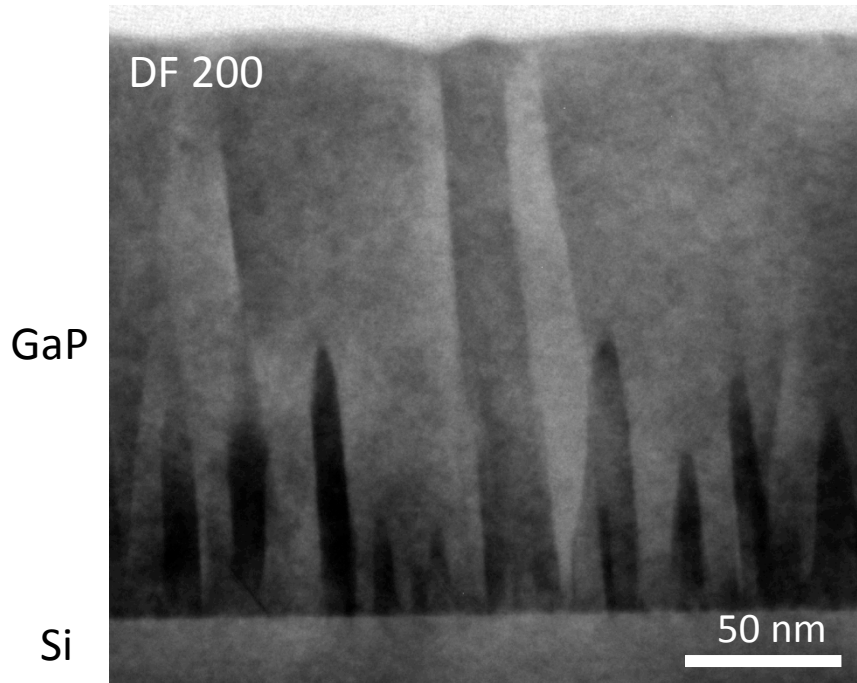
# Abrupt interface between III-V and Si



# Effect of the first 5 monolayers



# Using TEM to view APDs



APDs of opposite polarity have contrast under certain diffraction conditions  $\rightarrow$  distinguish antiphase boundaries from other defects (stacking faults, dislocations)

