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# EE412 Spring 2014

## SiN<sub>x</sub> Deposition and Etching recipe Development

Yusi Chen & Muyu Xue

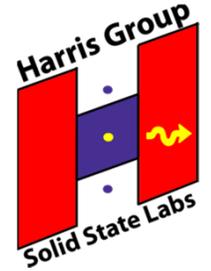
SNF Mentor: Jim McVittie

Faculty Mentor: James Harris



# Outline

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## 0. Motivation

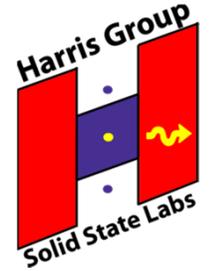
1. High stress SiN deposition using CCP and STS

2. SiN nanostructure etching using PT-OX



# Outline

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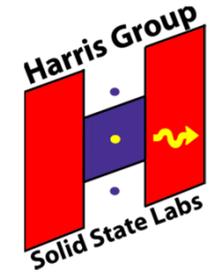


## 0. Motivation

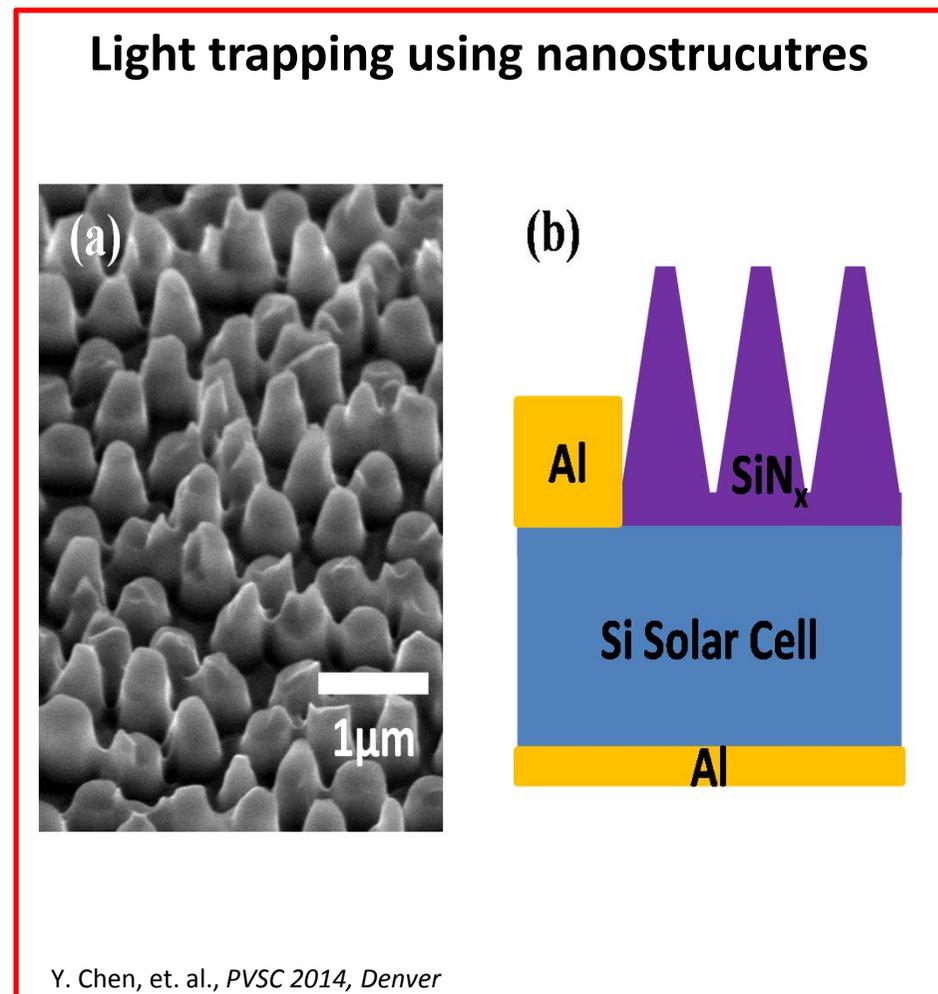
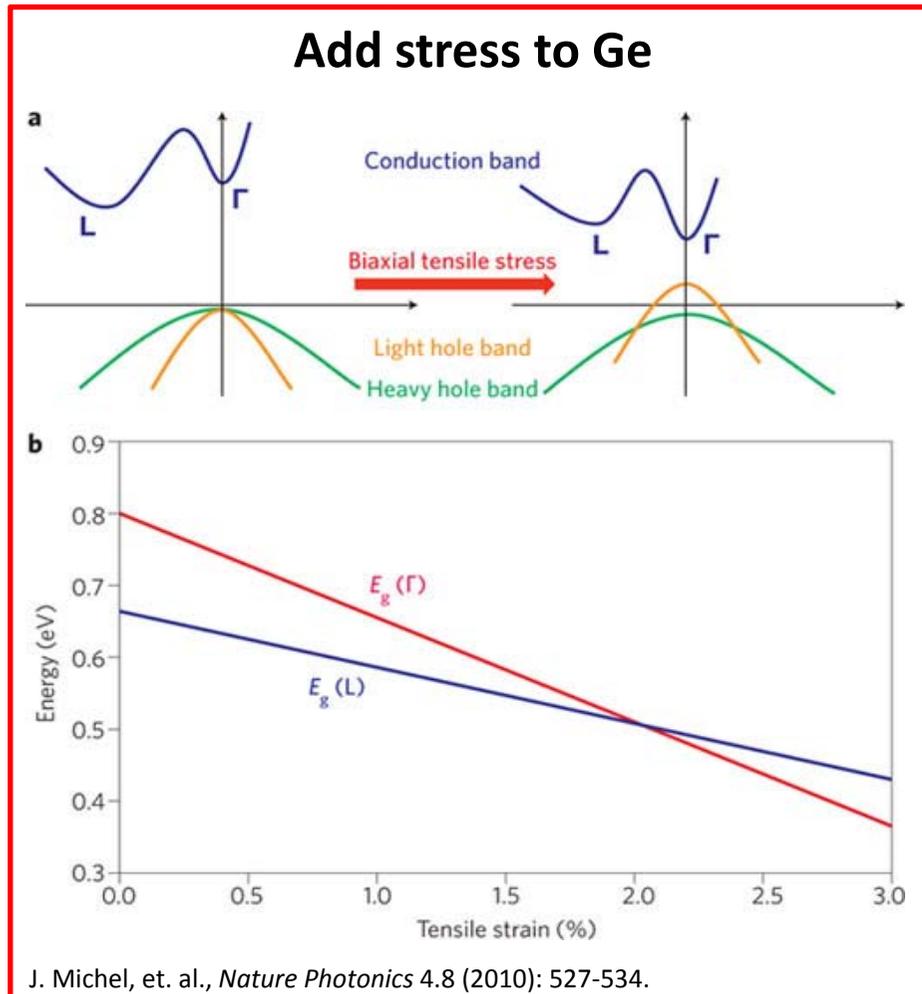
1. High stress SiN deposition using CCP and STS
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# Motivation



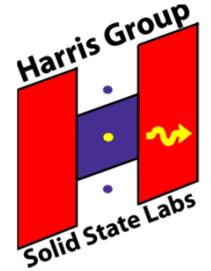
- $\text{SiN}_x$  has been widely using in micro/nanoelectronics
- More applications in optoelectronics/photronics





# Outline

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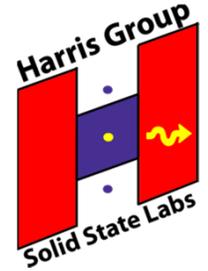
0. Motivation

**1. High stress SiN deposition using CCP and STS**

2. SiN nanostructure etching using PT-OX



# Stress Control Physics



## Target:

Highly compressive stress SiN<sub>x</sub> layer.

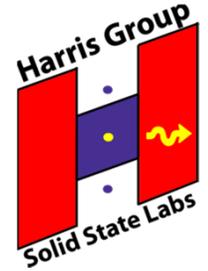
## Method:

1. Increase Si content. (Si atom volume larger than N atom)
  - NH<sub>3</sub>/SiH<sub>4</sub> ratio ↓.
2. Increase SiN<sub>x</sub> film density
  - Low frequency % ↑. (ion bombardment ↑)
  - Chamber pressure ↓. (mean free path ↑, ion bombardment ↑)
  - Power ↓. (growth rate ↓, more time for atoms to move)
3. Change ion species.
  - Inert gas dilution, add He. (↑ the creation of N<sup>+</sup> ions, hydrogen bond density)



# CCP Deposition Method

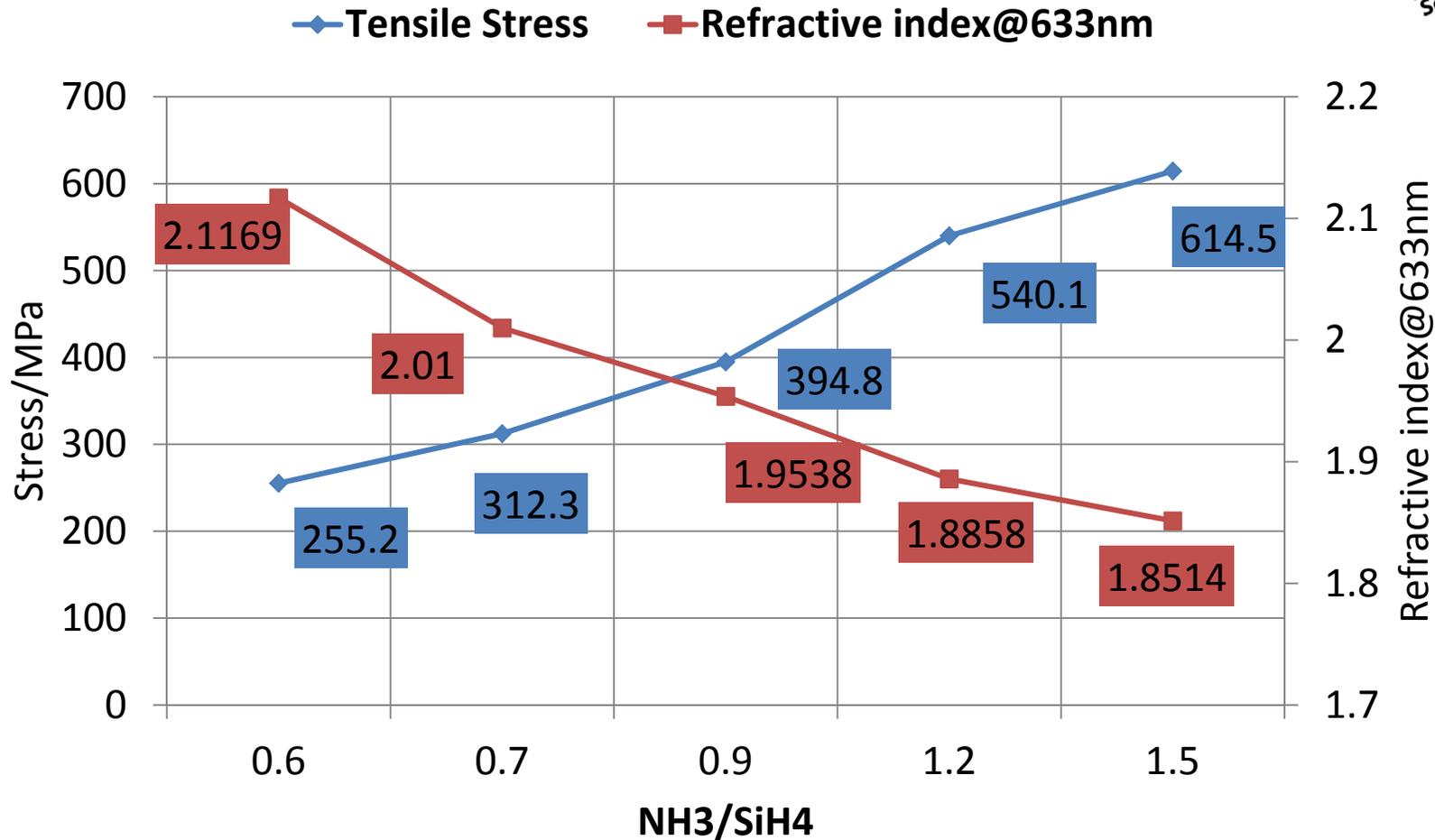
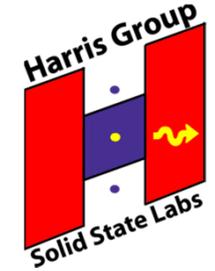
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- Temperature 350° C, Pressure 950mTorr, Deposition time 10min
- $N_2 / (N_2 + He) = 0.8$  (tensile limit),  
= 0.6 (intermediate case),  
= 0 (compressive limit)
- Change ratio of  $NH_3 / SiH_4$



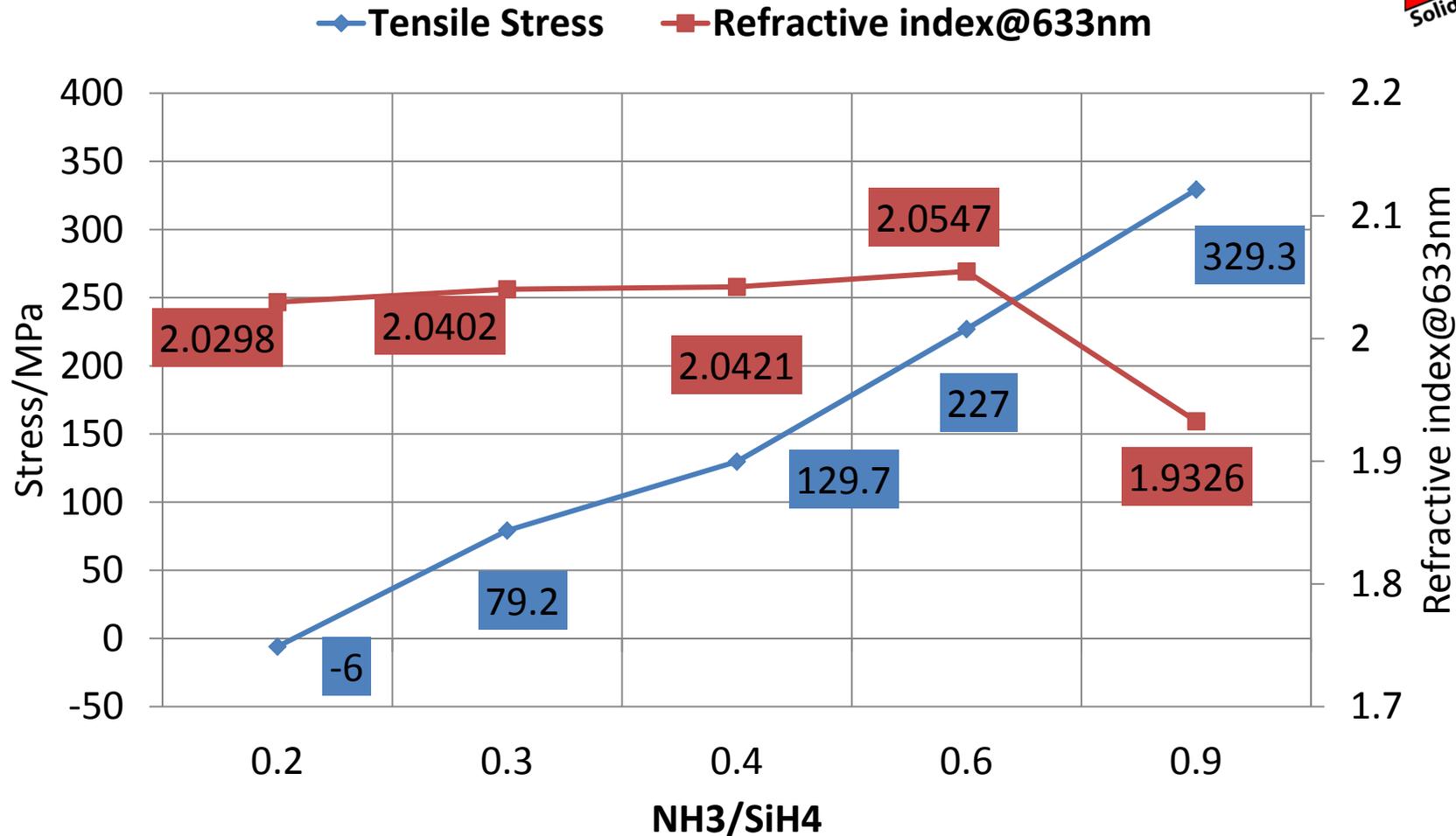
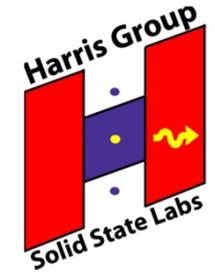
# CCP Tensile Limit



- $N_2/(N_2+He)=80\%$  ( $N_2=800$  sccm,  $He=200$  sccm)
- Change ratio of  $NH_3/SiH_4$ : 0.6, 0.7, 0.9, 1.2, 1.5



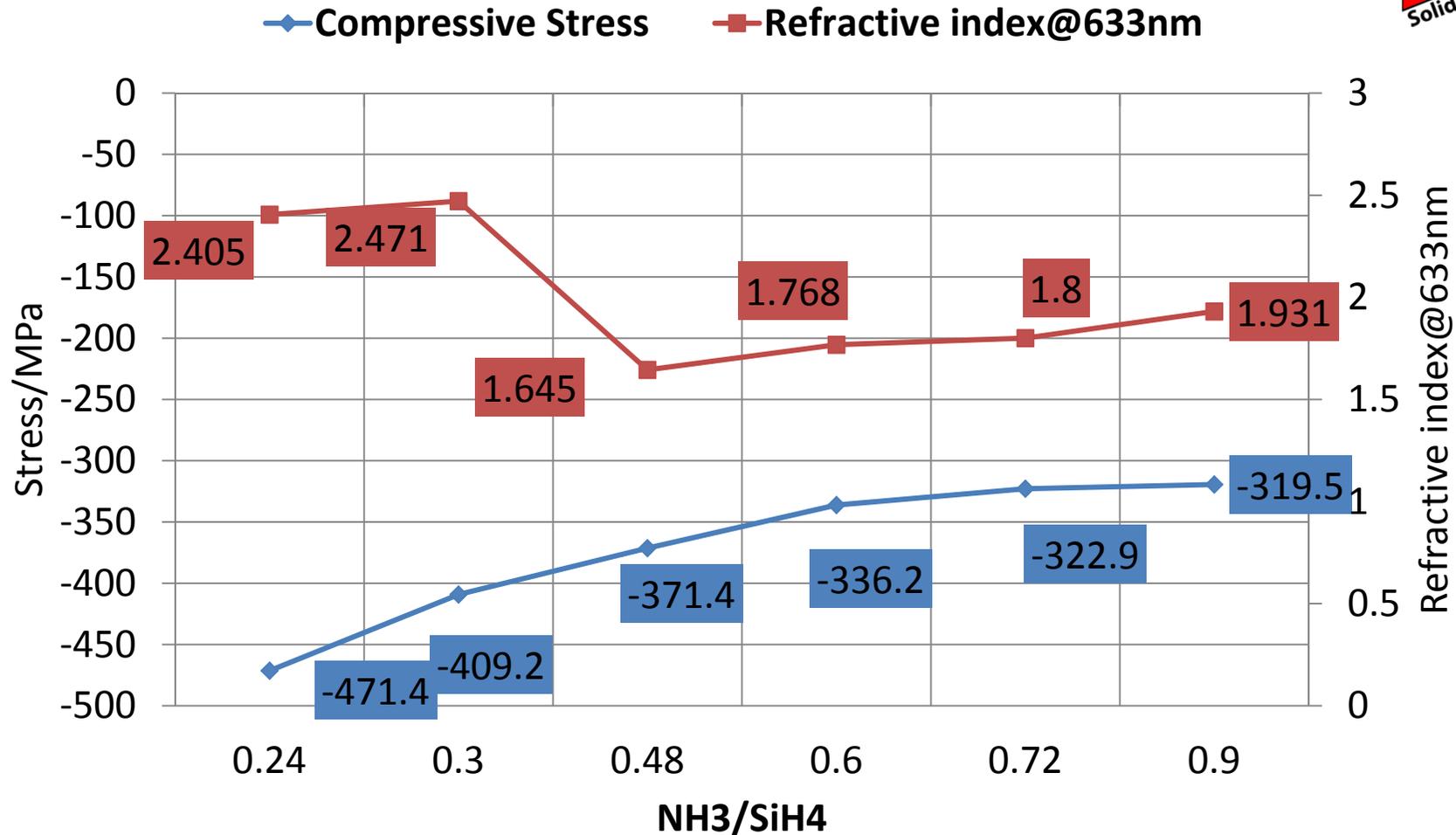
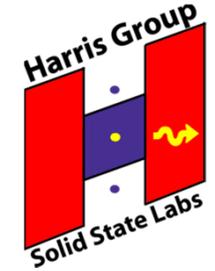
# CCP Intermediate Case



- $N_2/(N_2+He)=60\%$  ( $N_2=600$  sccm,  $He=400$  sccm)
- Change ratio of  $NH_3/SiH_4$ : 0, 0.2, 0.3, 0.4, 0.6, 0.9



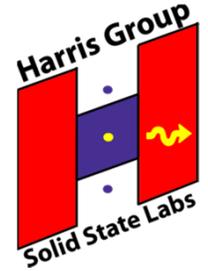
# CCP Compressive Limit



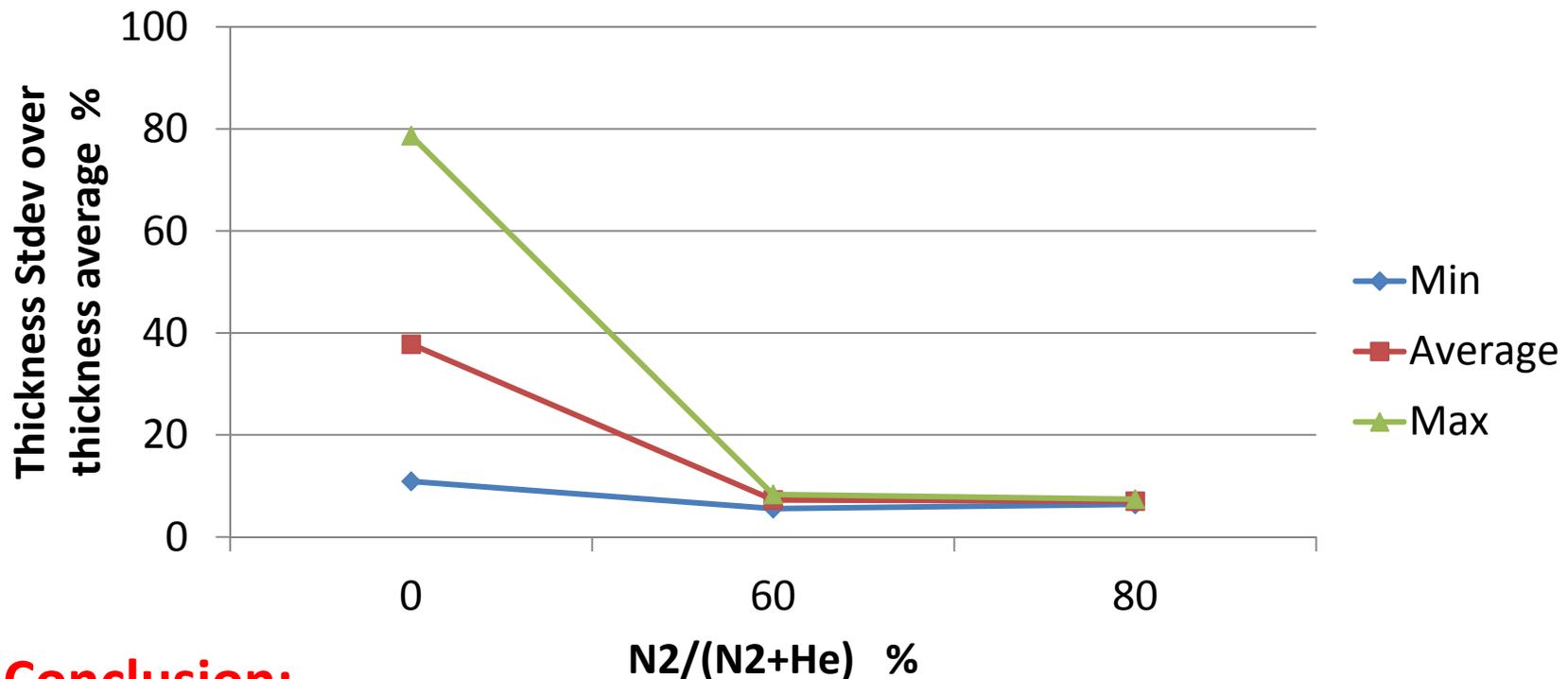
- $N_2/(N_2+He)=0$  (He=1300 sccm)
- Change ratio of NH3/SiH4: 0.24, 0.30, 0.48, 0.60, 0.72, 0.9



# Effect of N<sub>2</sub> on Uniformity



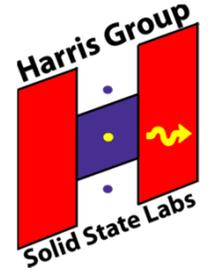
- Using Woollam 9 point mapping
- Characterize thickness standard deviation over thickness average



**Conclusion:**  
**N<sub>2</sub> is critical for SiN<sub>x</sub> uniformity!**



# STS Deposition Method



## Target:

Highly compressive stress SiN<sub>x</sub> layer.

## Steps:

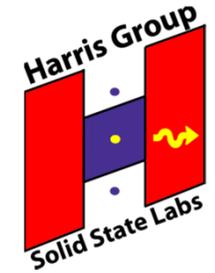
**Fixed: Percentage of low frequency power (100%)**

1. Process power ↓: 60w, 25w, 10w
2. Chamber pressure ↓: 650mTorr, 500mTorr
3. NH<sub>3</sub>/SiH<sub>4</sub> ↓: 0.8, 0.38

**Try thicker SiN<sub>x</sub> (600nm)**

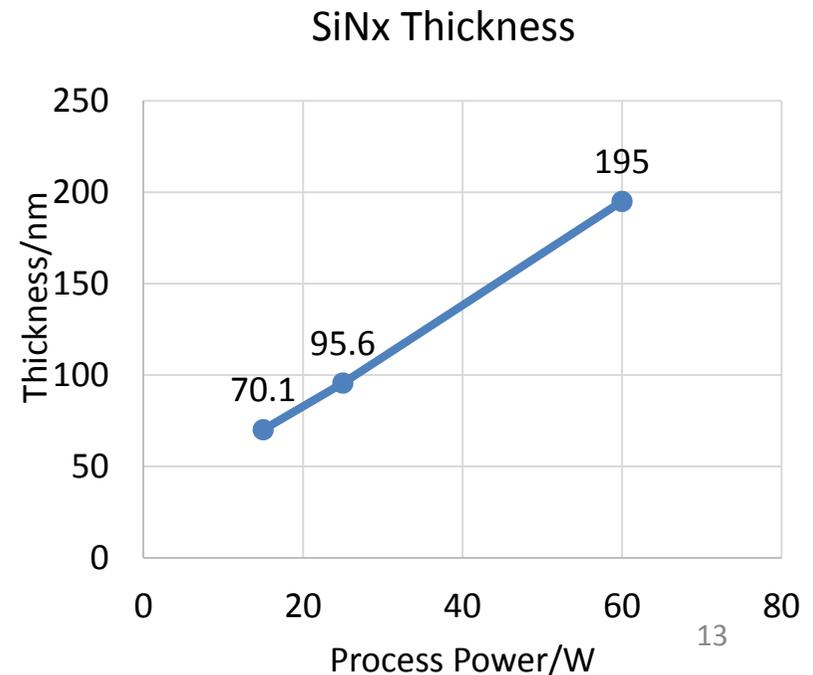
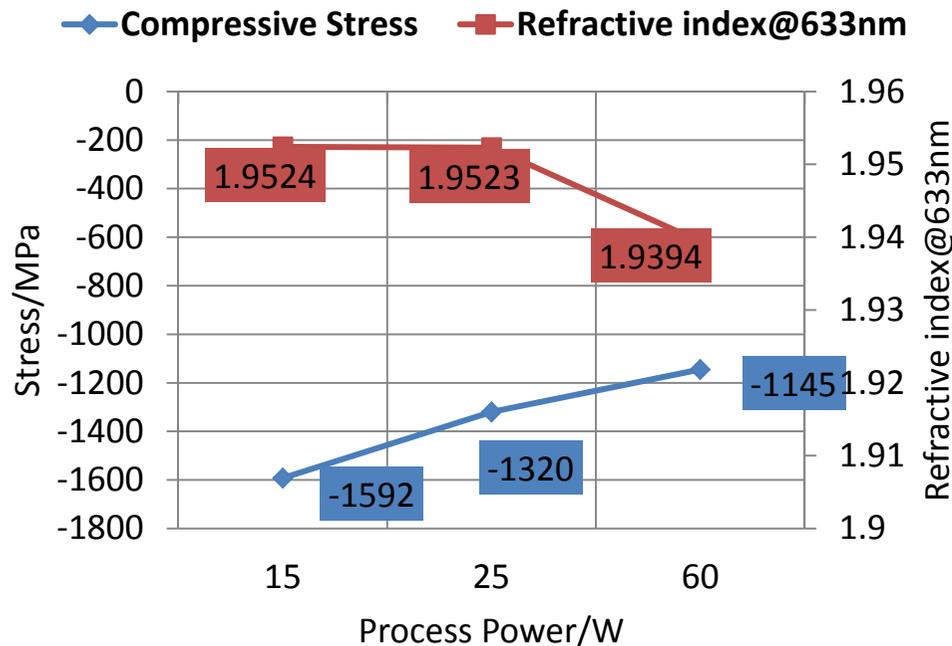


# STS Deposition Result



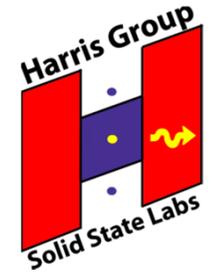
Fixed: Percentage of low frequency power (100%)

Step 1	Process power/w	Pressure/mTorr	NH <sub>3</sub> /SiH <sub>4</sub>	Deposition time/min	Thickness/nm	Stress/MPa
S1	60	650	0.84	10	195	-1145
S2	25	650	0.84	10	95.6	-1320
S3	15	650	0.84	10	70.1	-1592





# STS Deposition



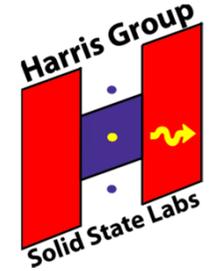
Step 2	Process power/w	Pressure/mTorr	NH <sub>3</sub> /SiH <sub>4</sub>	Deposition time/min	Stress/MPa	n @ 633nm
S21	25	650	0.84	10	-1320	1.9523
S22	25	500	0.84	10	-1770	1.9488

Step 3	Process power/w	Pressure/mTorr	NH <sub>3</sub> /SiH <sub>4</sub>	Deposition time/min	Stress/MPa	n @ 633nm
S31	10	500	0.84	10	-1711	1.9404
S32	10	500	0.38	10	-2095	N/A

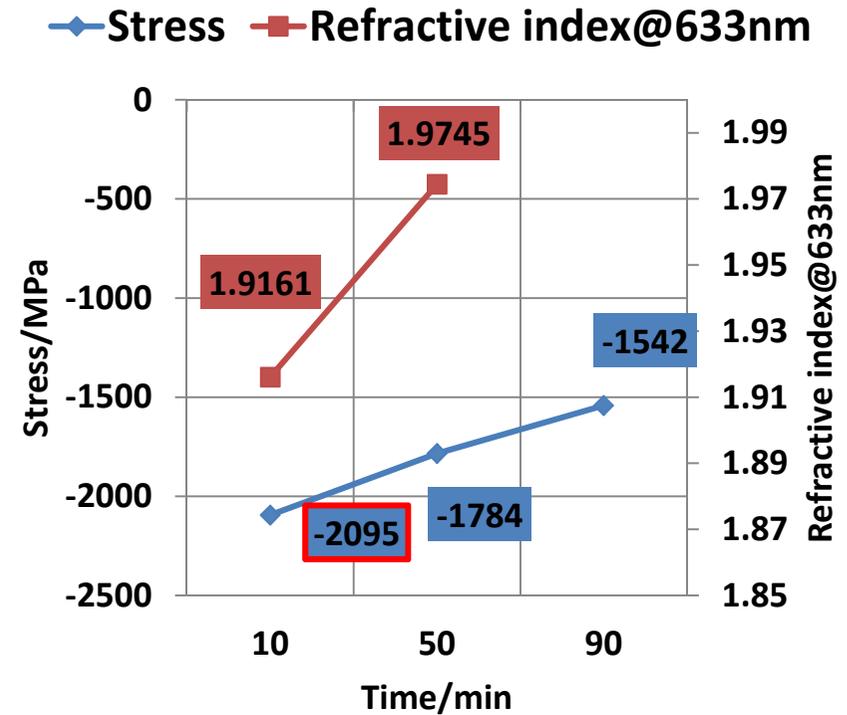
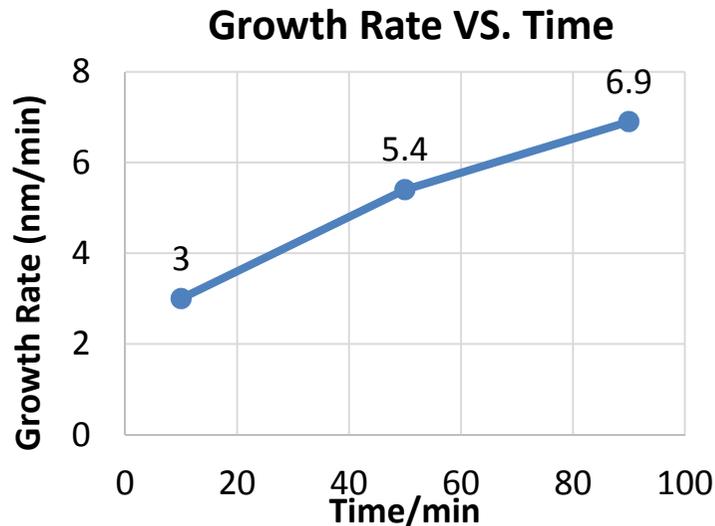
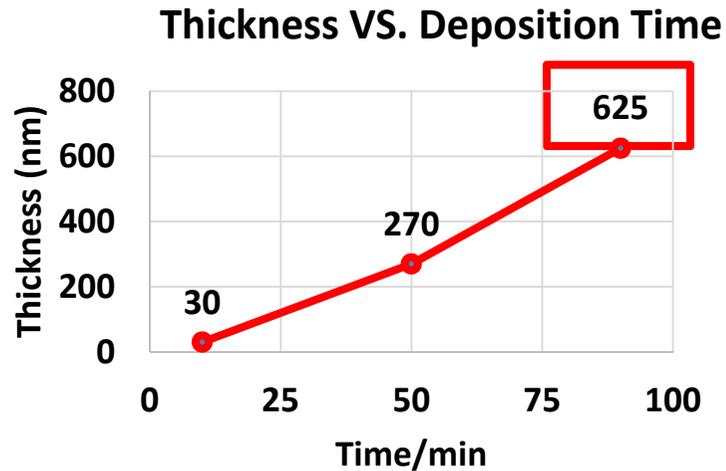
**-2 Gpa!**



# Thicker SiN<sub>x</sub> ( 600nm)



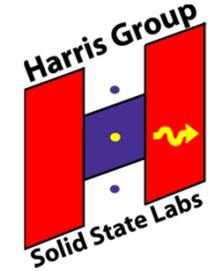
PF power=10w, NH<sub>3</sub>/SiH<sub>4</sub>=0.37, t=10/50/90min



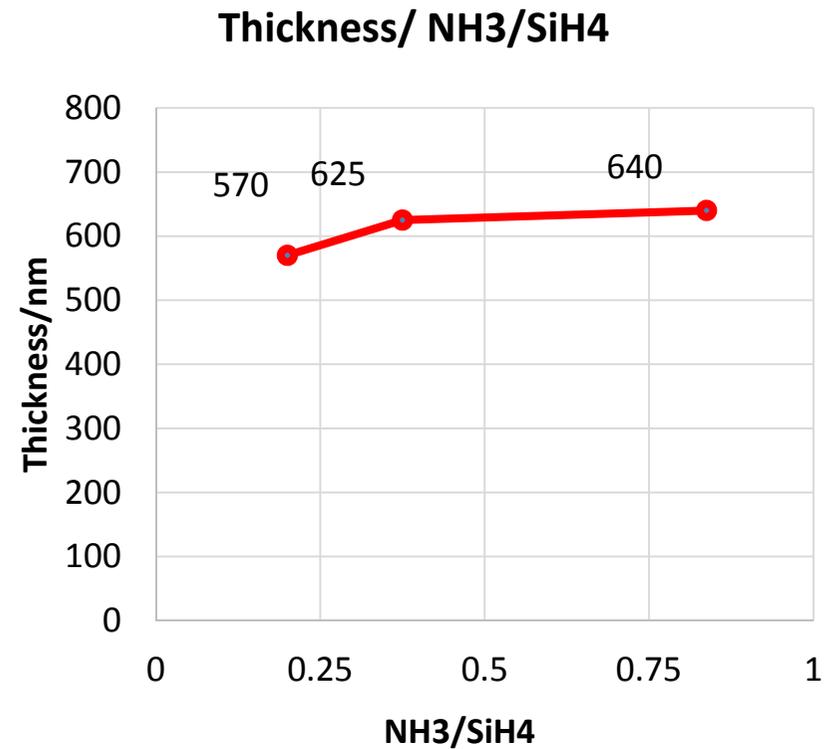
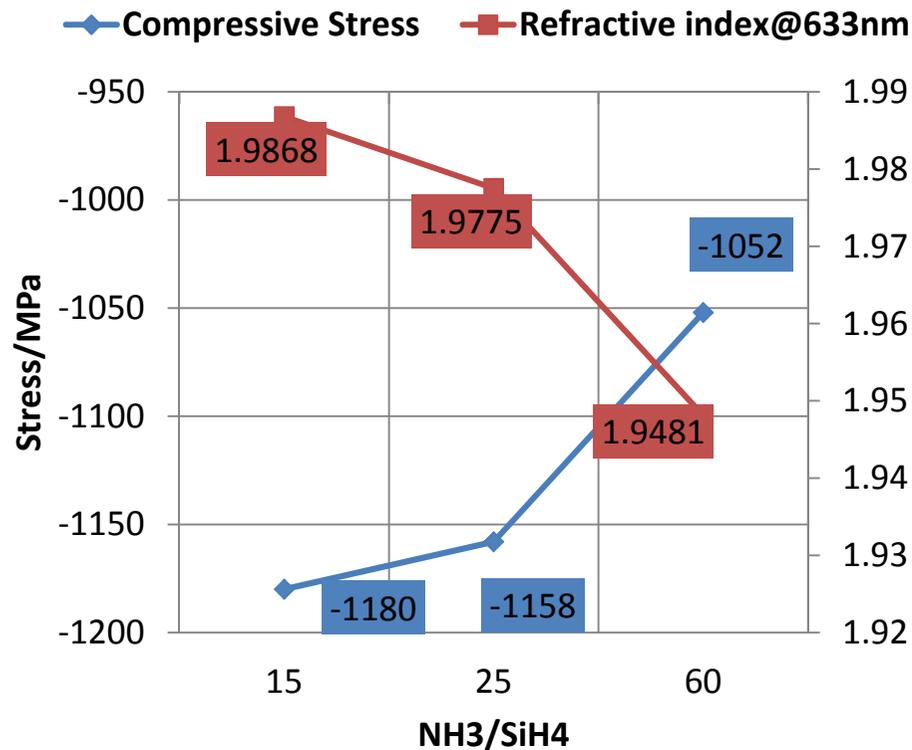
1. Get 600nm thick SiN<sub>x</sub> in STS
2. Get -1.5Gpa compressive stress



# Thicker SiN<sub>x</sub> (600nm)



RF power=60w, t=30min,  $\text{NH}_3/\text{SiH}_4=0.2/0.37/0.84$

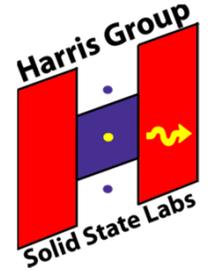


Conclusions:

$\text{NH}_3/\text{SiH}_4$  ratio is secondary for SiN<sub>x</sub> stress & thickness



# Conclusions



1. Stress range on CCP:

-470MPa ----- 614MPa

2. Achieved largest compressive stress using STS:

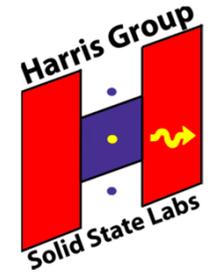
Process power/w	Pressure/mTorr	NH <sub>3</sub> /SiH <sub>4</sub>	Deposition time/min	Stress/MPa
10	500	0.38	10	-2095

3. Thick SiN<sub>x</sub> may reduce stress due to anneal effect

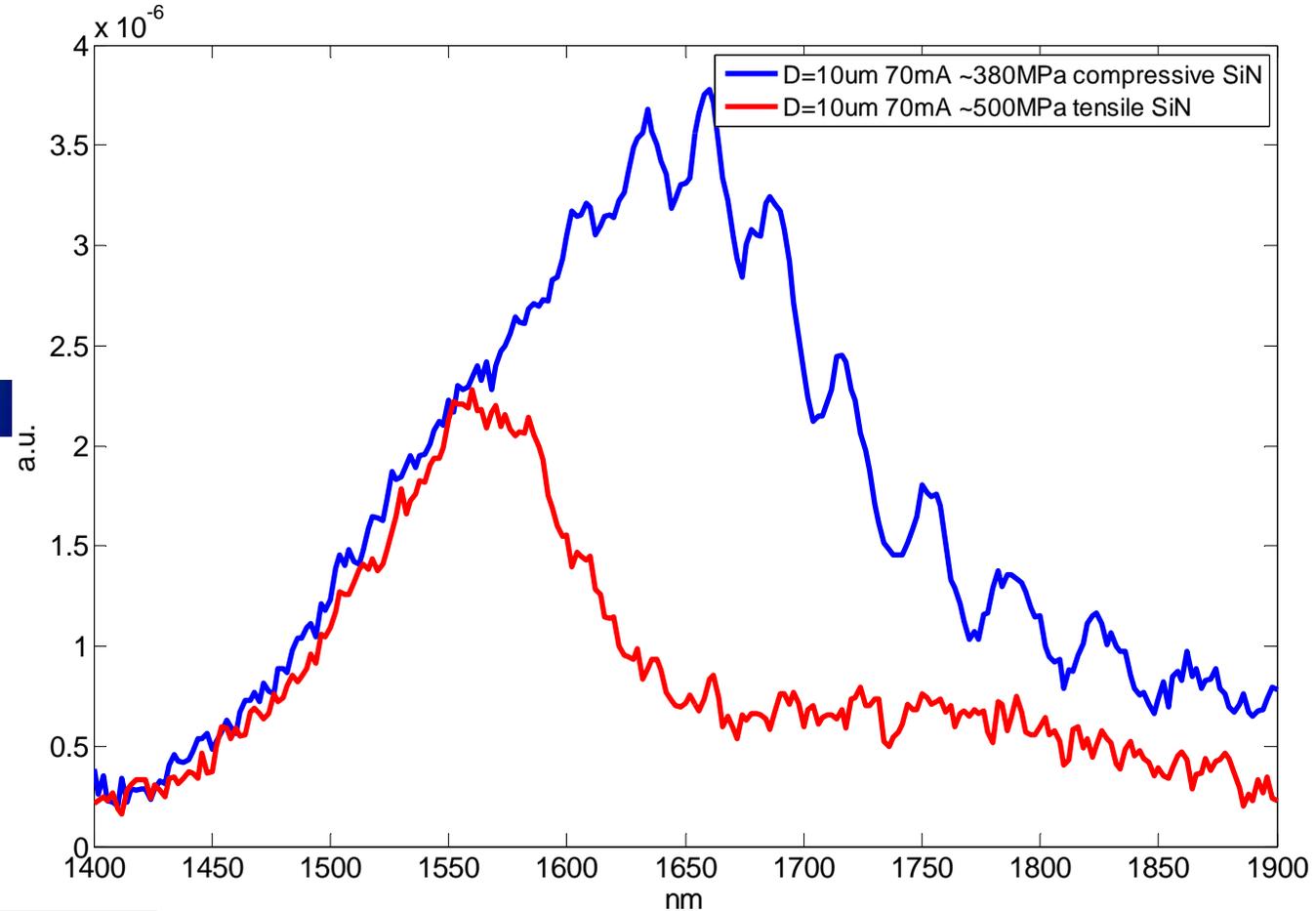
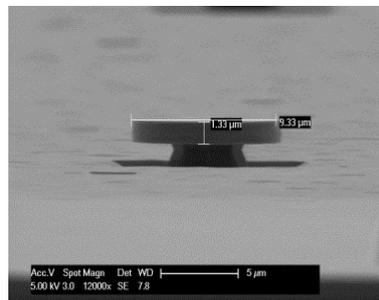
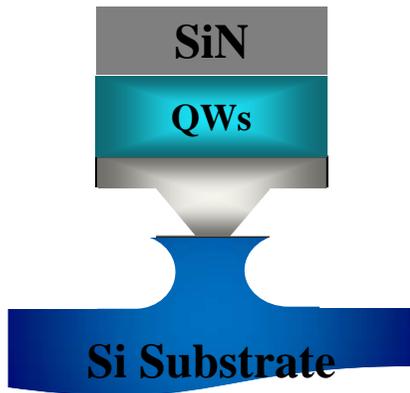
4. Add N<sub>2</sub> is important for SiN<sub>x</sub> uniformity!



# Application



## High stress SiN<sub>x</sub> on SiGe multi-quantum-well ring resonator

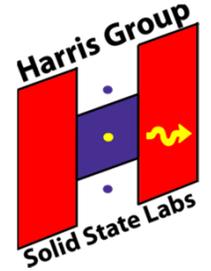


- Peak shift ~90nm.
- Intensity ~x2



# Outline

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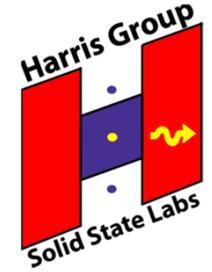
## 0. Motivation

1. High stress SiN deposition using CCP and STS

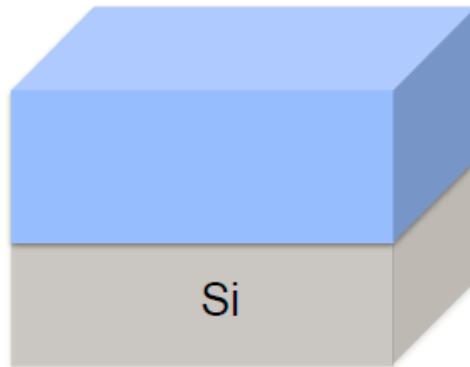
**2. SiN nanostructure etching using PT-OX**



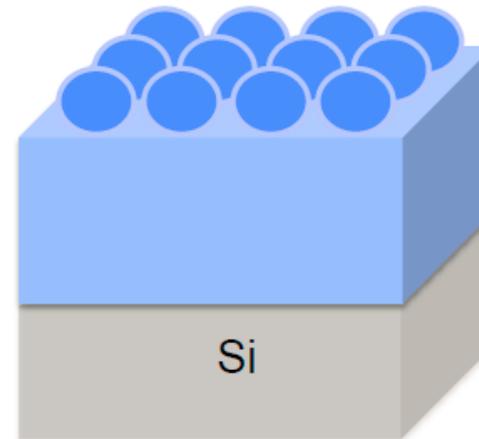
# Nanosphere lithography



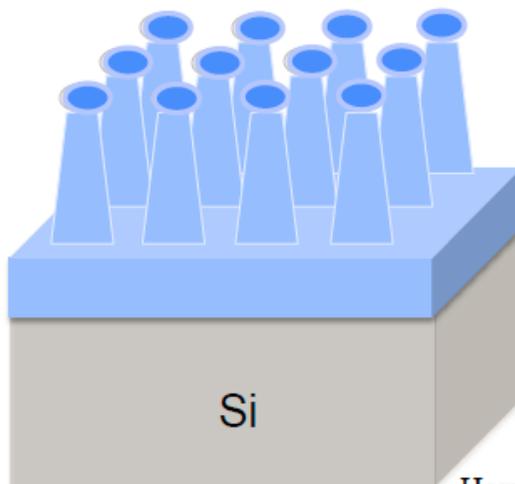
**Film Deposition**



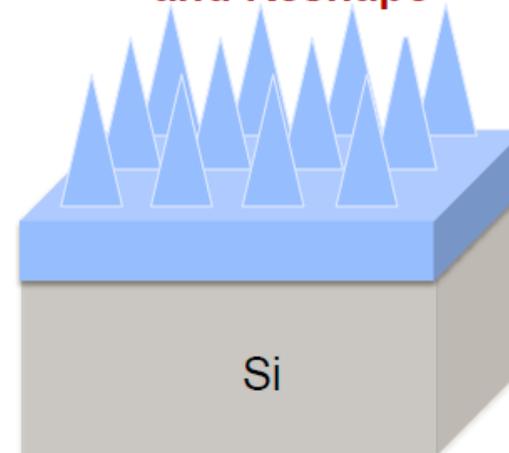
**Nanosphere LB Coating**



**Reactive Ion etch**



**Remove Nanosphere and Reshape**

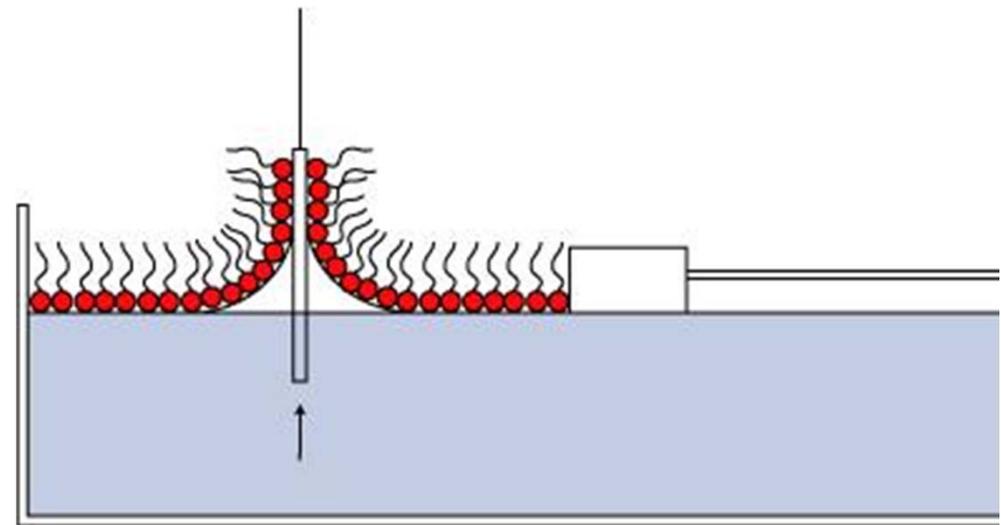
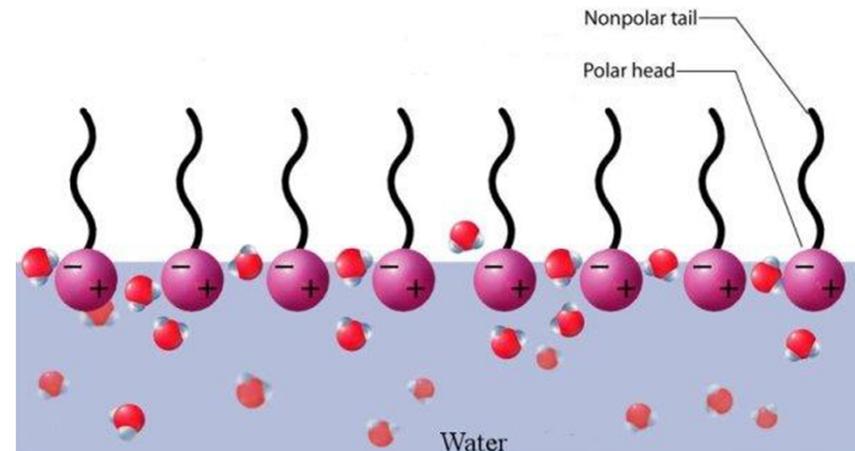




# Langmuir–Blodgett Method

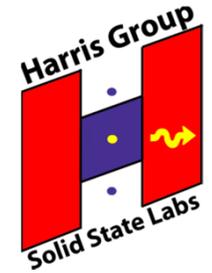


- Molecules with hydrophobic 'tails' and hydrophilic 'heads'.
- In water, amphiphilic molecules interact with air at an air–water interface.
  - Tails exposed to air
  - Heads stay inside water
- When compressed, one compact film will be formed
- Then transfer monolayer onto a substrate after film compression.

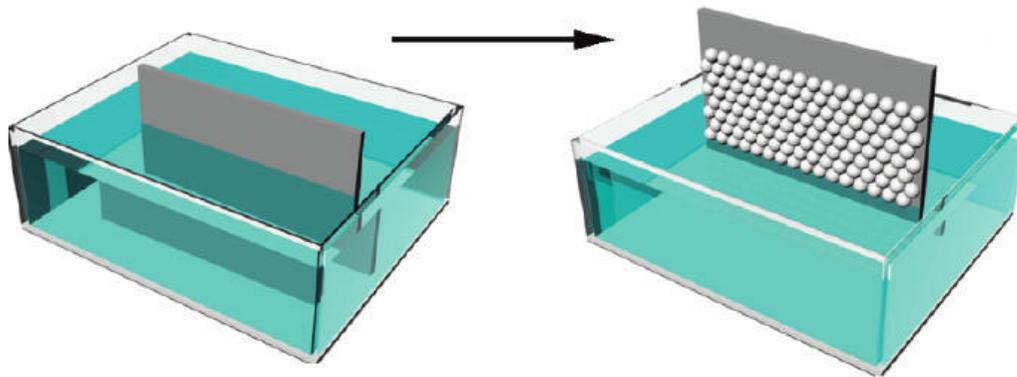




# Coating in LB Trough

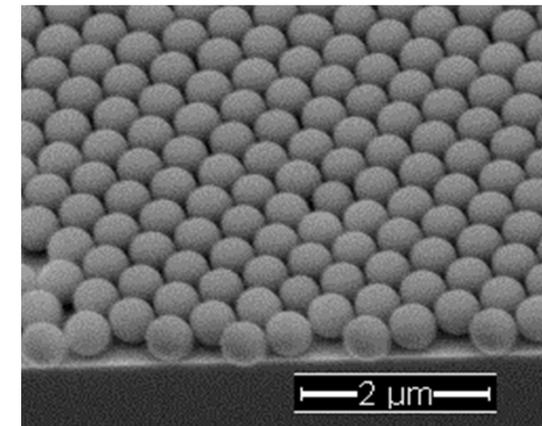
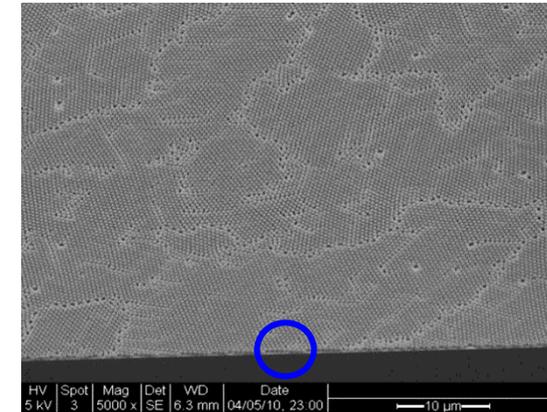


LB coating in silica  
nano-sphere solution



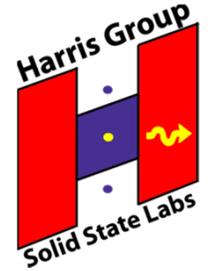
Erik Garnett, et al. Nano Lett. 2010

- Feedback control system
  - Use surface tension pressure of compact film as parameter
- Pull up substrate from water

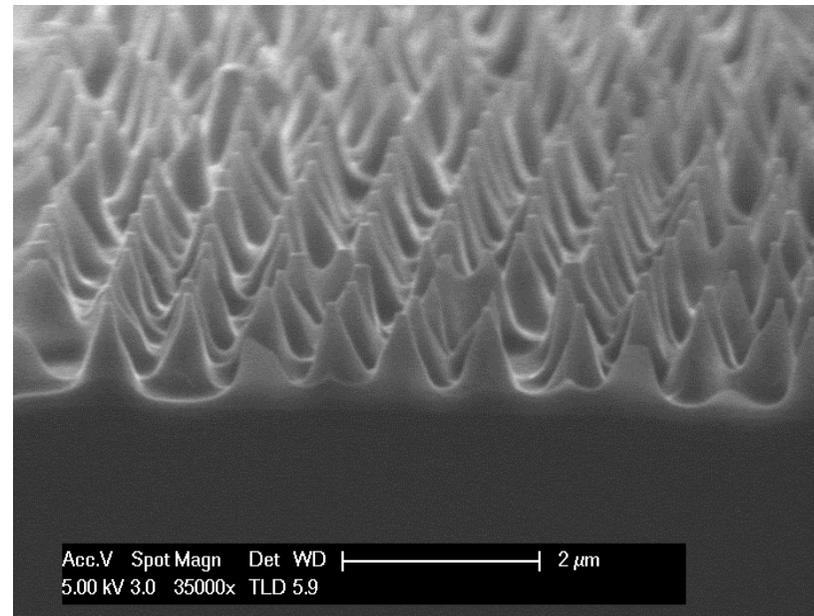
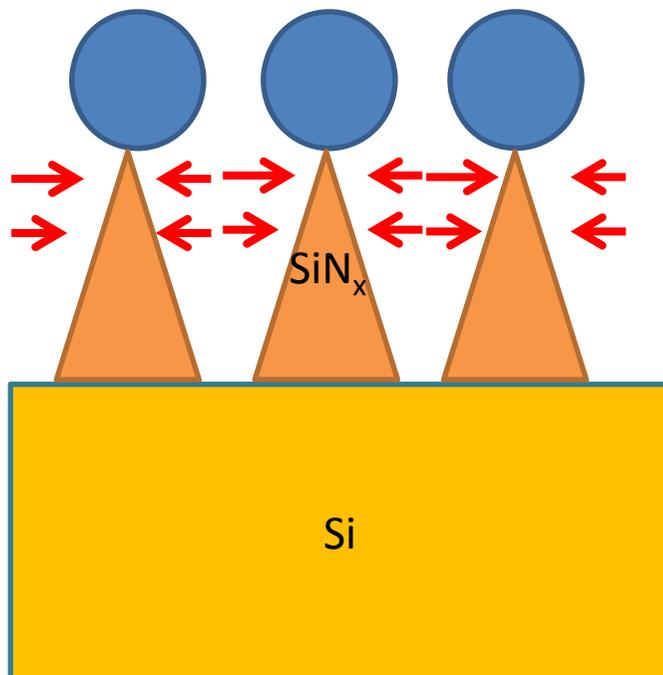




# Nano-cone Structures



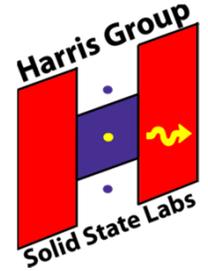
- Using PT-OX: ICP RIE etcher
- High gas pressure: 40-50 mtorr, high scattering, more isotropic





# Etching recipe development

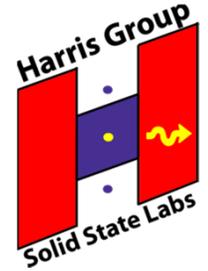
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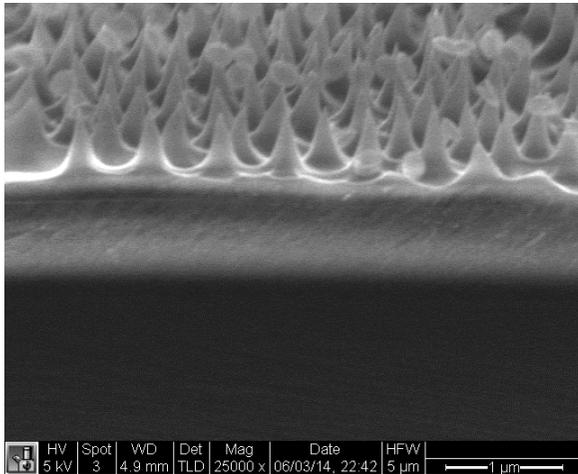
- Fixed parameters:
  - 600nm nanospheres
  - 900nm thick  $\text{SiN}_x$  using CCP SiN300 recipe
  - 10 degree C temperature
  - 1000W ICP power
  - 50V RF DC bias
- 3 main parameter tuning:
  - Etching chemistry
  - Etching pressure
  - Etching time



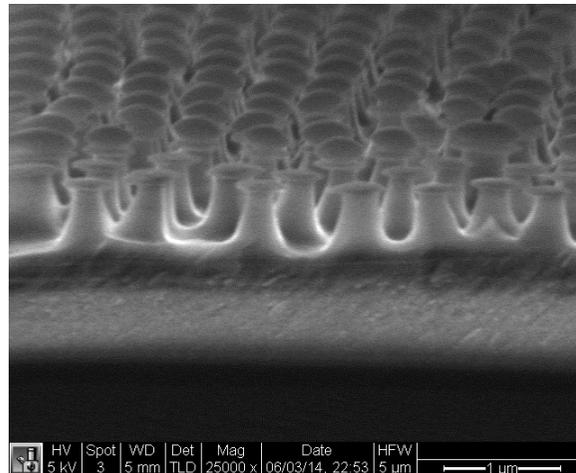
# Etching chemistry



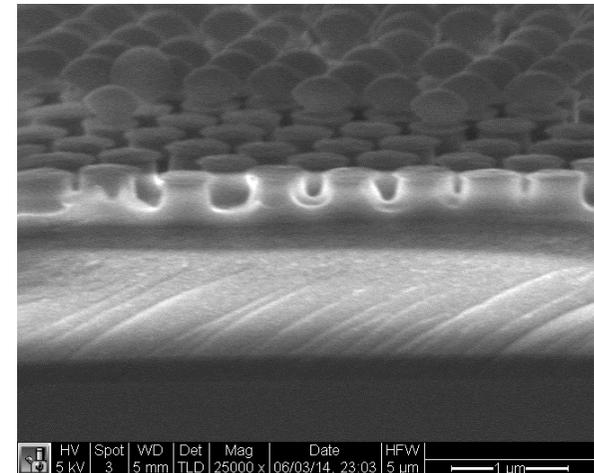
- Tried CF<sub>4</sub>, CHF<sub>3</sub> and O<sub>2</sub>
- 240s etching, 30 mtorr pressure
- 5sccm O<sub>2</sub>, CF<sub>4</sub>+CHF<sub>3</sub>=50sccm



5% CHF<sub>3</sub>



50% CHF<sub>3</sub>

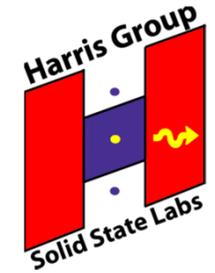


95% CHF<sub>3</sub>

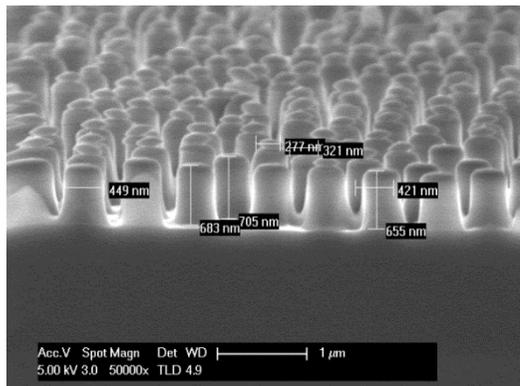
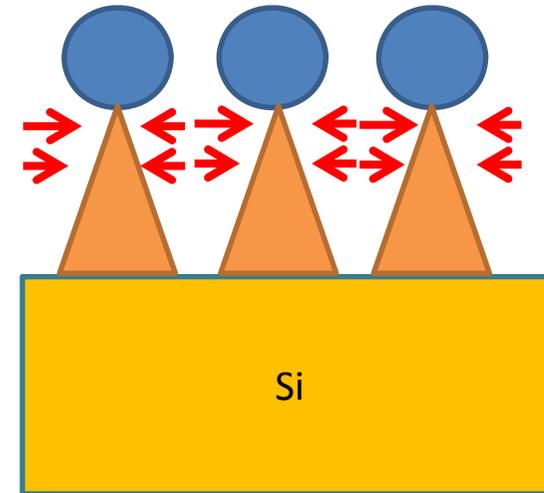
- **More CF<sub>4</sub>, more isotropic**



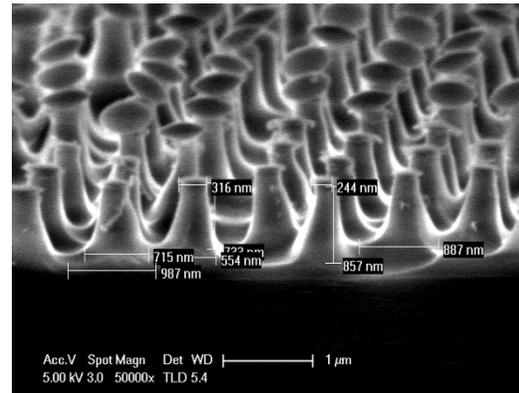
# Etching pressure



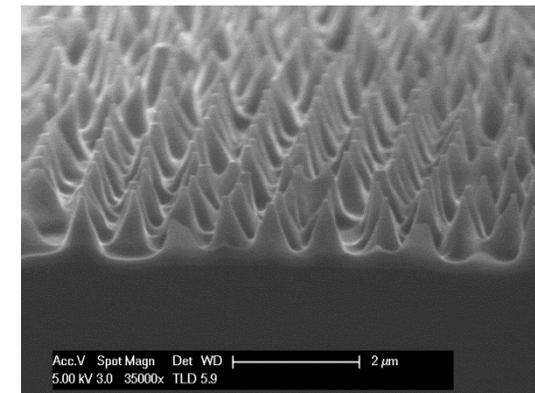
- Larger pressure, more isotropic etching
- Pressure cannot be too high: plasma not working



10mtorr, 120s



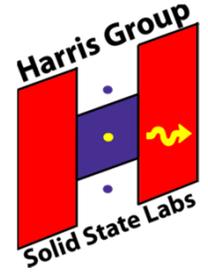
30mtorr, 240s



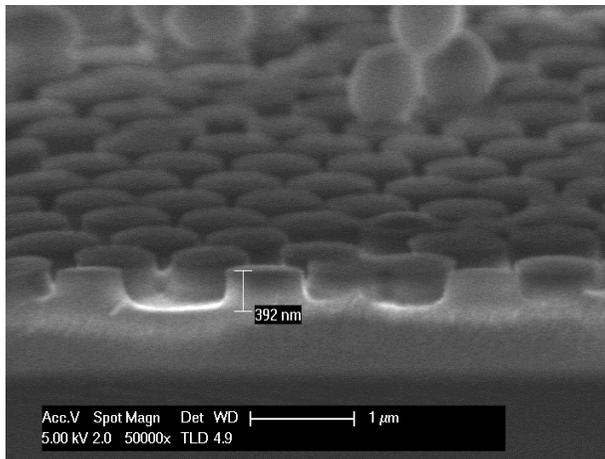
50mtorr, 240s



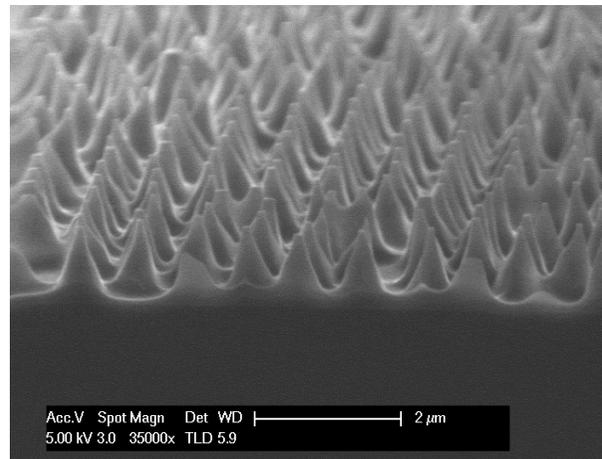
# Etching time



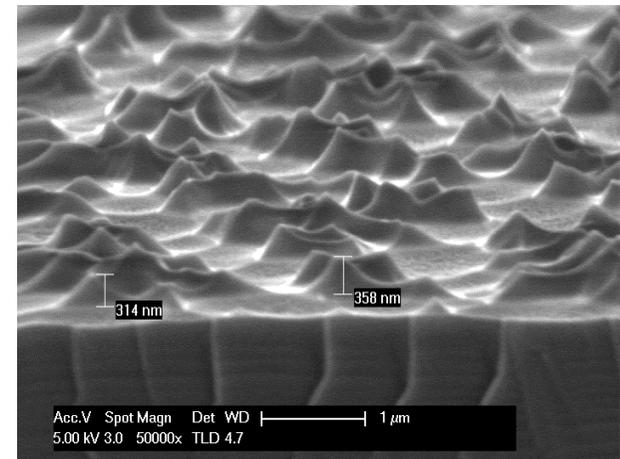
- Need to be carefully tuned
- 50sccm CF4 + 5sccm O2, 50 mtorr pressure



**120s, not enough**



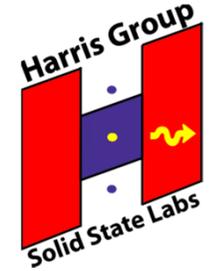
**240s, good**



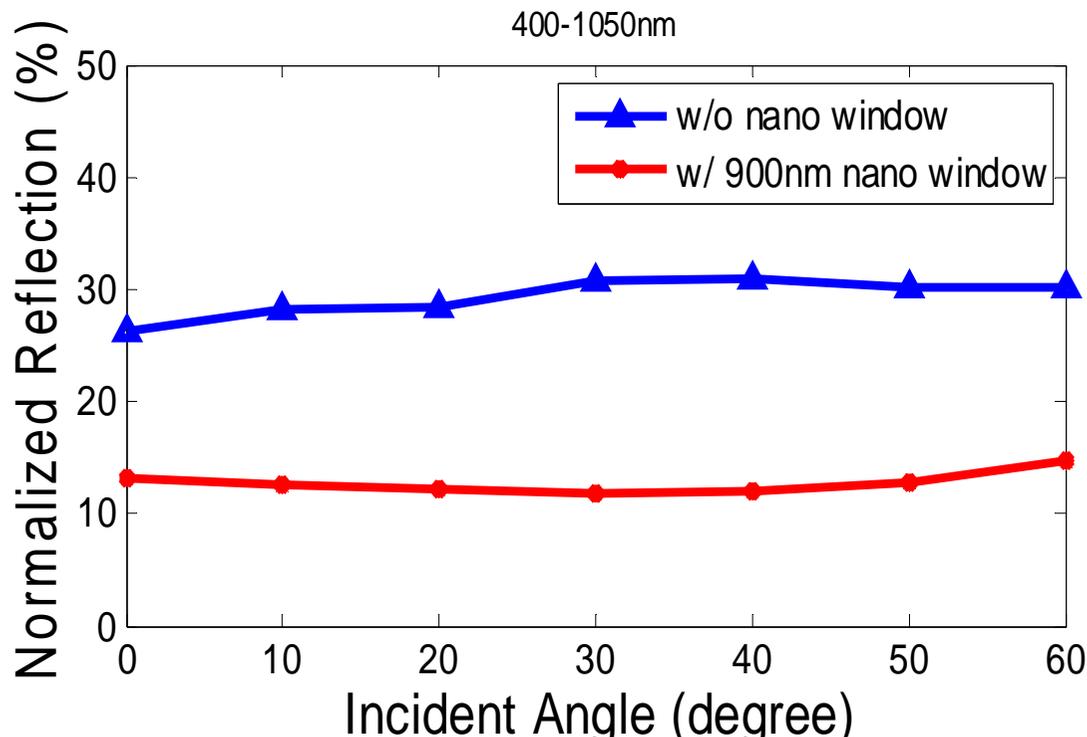
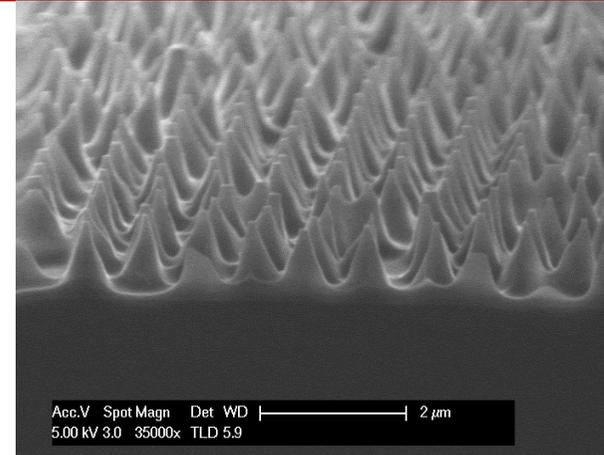
**360s, over-etch**



# BKM Recipe and Result



- 50sccm CF4 + 5sccm O2
- 50 mtorr pressure
- 240s

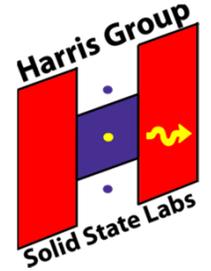


- Reflection measurement
- Integrated range of wavelength 400 -1050nm



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