Mechanical loss of silicon nitride films grown by plasma-enhanced chemical vapor deposition (PECVD) method

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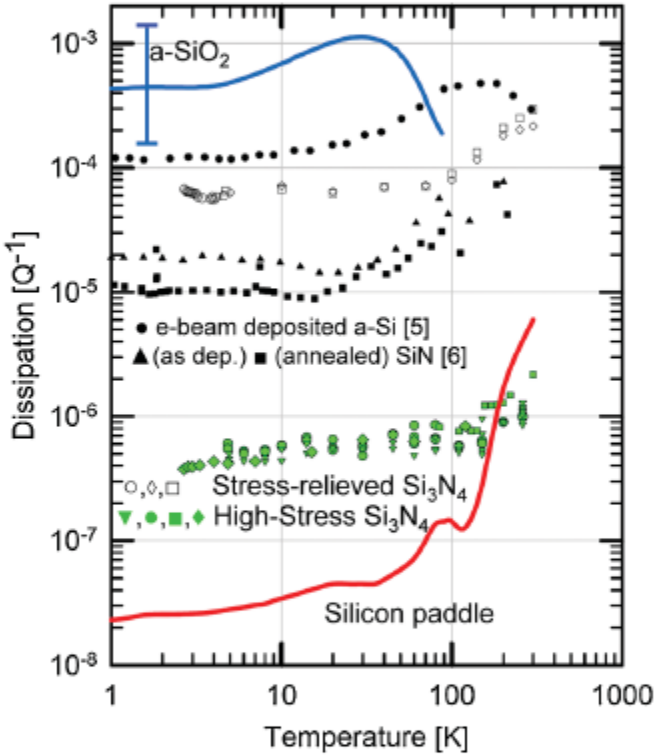
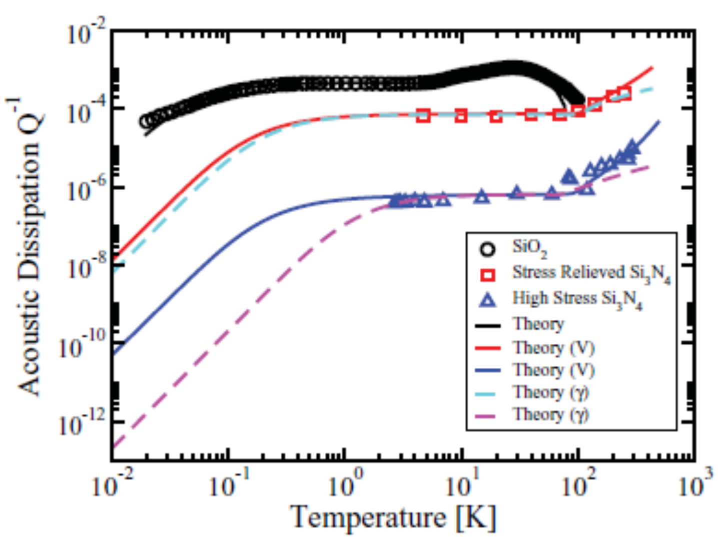
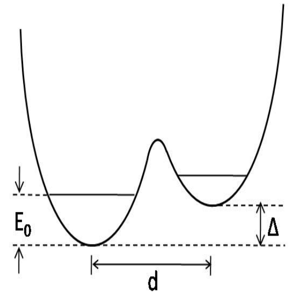
Motivation

- Amorphous silicon nitride films with high internal stress were reported [1]to have much lower loss angle (~ 2 order of magnitude) than most other amorphous materials that followed the “universality” behavior[2] in the 1~10°K temperature range. The loss reduction mechanism was modeled and attributed to the coupling between the stress and the double well potential in the amorphous structure to either reducing the tunneling probability or reducing the thermal excitation by the increased barrier height[3].



Ref. 1: D. R. Southworth , et al. PhysRevLett.**102**.225503(2009)





Ref. 3: Jiansheng Wu, et al. PhysRevB.**84**.174109(2011)

For the “universality” behavior of the amorphous solid in the low temperature region, see, for example: Ref. 2: Robert O. Pohl et al. Reviews of modern physics, **74** Oct.(2002)

Advantages for using silicon nitride film

- Adequate refractive index (~2, wide transparent range that covers 1064nm), could either be used as L layer together with amorphous silicon as H layer (~3.4, although proper transparent region need to be considered), or as H layer in combination with other L layer films to form multi-layer high reflector.

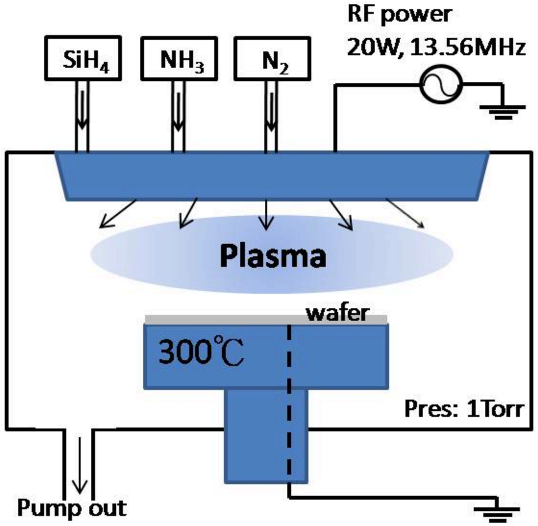
- The chemical vapor deposition (CVD) in various modifications, e.g. plasma-enhanced (PECVD), low pressure (LPCVD) for SiN deposition are mature and well-established in the silicon IC manufacture industry (including Si-photonics IC process) ➔ less coating vendor-selection constrain.

- It is compatible with the 16” silicon wafer process that is nowadays a common practice in the silicon IC manufacture industry, i.e. for the potential silicon mirror, all-silicon process is possible ➔ well-controlled process and less cost for mirror coating.

Fabrication of SiN film on Silicon Cantilever by PECVD

PECVD

Plasmalab system100, Oxford Inst.
Courtesy photo from National Nano-Device Lab (NDL) of Taiwan



$$SiH_4 + 4NH_3 \xrightarrow{\text{plasma}} Si(NH_2)_4 + 4H_2$$

$$3Si(NH_2)_4 \xrightarrow{\text{heat}} Si_3N_4 + 8NH_3$$

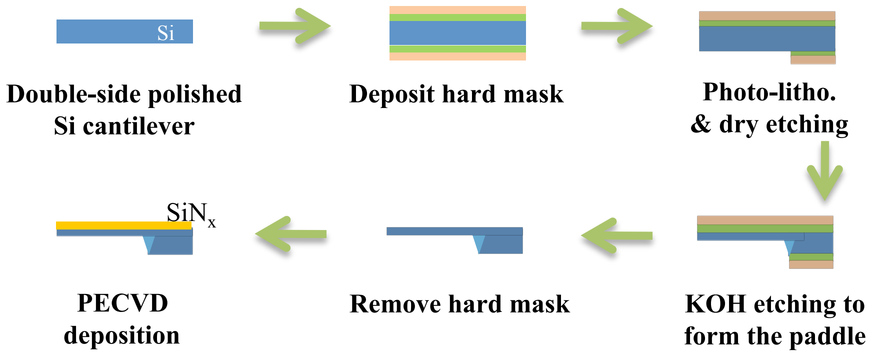
Ref : Donald L. Smith, et al." mechanism of SiN_xH_y Deposition from NH_3-SiH_4 plasma". J.Electrochem. Soc. **137**, 614-623(1990)

Ref : J. N. Chiang, et al **"**Mechanistic Considerations in the Plasma Deposition of silicon nitride film" J. Electrochem. Soc. **137**, 2222-2226.(1990)

Adjusting the ratio of the gas flow rate, the composition of the SiN film can be changed

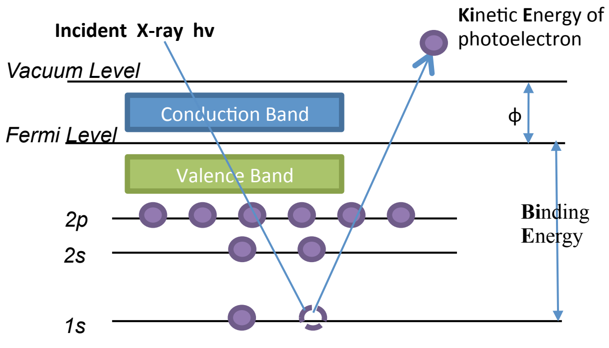
With fixed N_2 gas flow at 980 sccm,
We used two recipes with different gas flow rate :

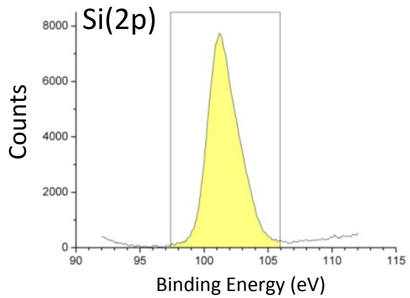
Gas flow rate SiH_4/ NH_3(sccm)	Deposition rate(A/s)	Thickness from TEM (nm)
25/30	3.13	182.7
10/60	2.69	201.3

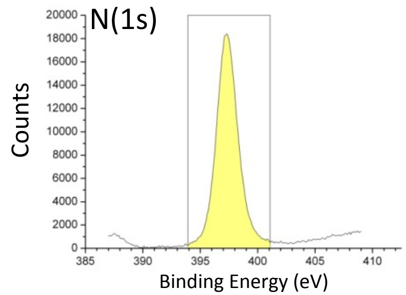


Measurement of the composition of SiN_x films

by Electron Spectroscopy for Chemical Analysis (ESCA)







Binding Energy = hv – $KE_{photoelectron}$ – ϕ

Sensitivity Factor (SF) = f σD λ

f = x-ray flux

σ = photoelectron cross-section

D = detector efficiency

λ =electron mean free path

$$\frac{n_N}{n_{Si}} = \frac{A_N / SF_N}{A_{Si} / SF_{Si}}$$

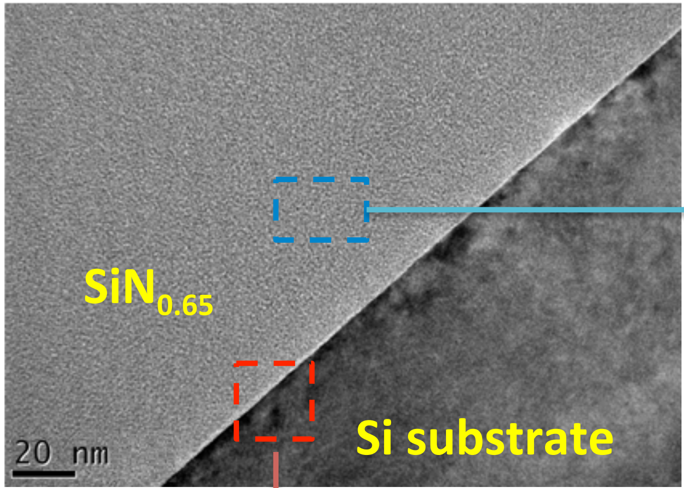
A= peak area

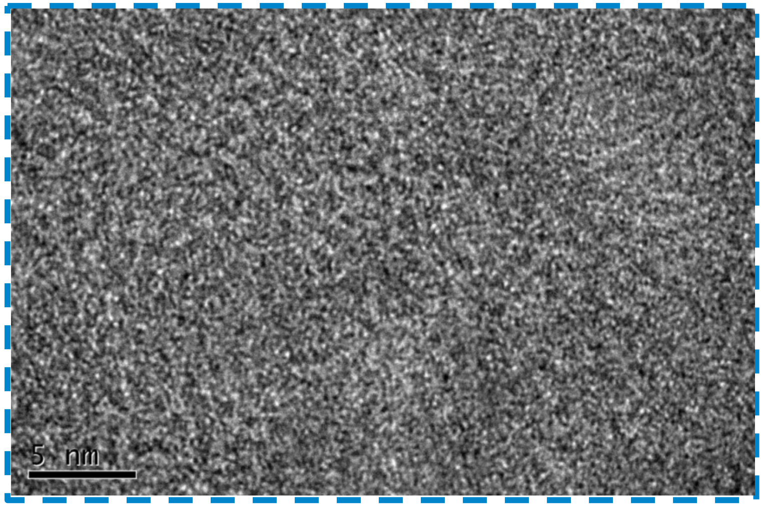
n = number of atoms, the ratio is conventionally expressed as **SiN_x**

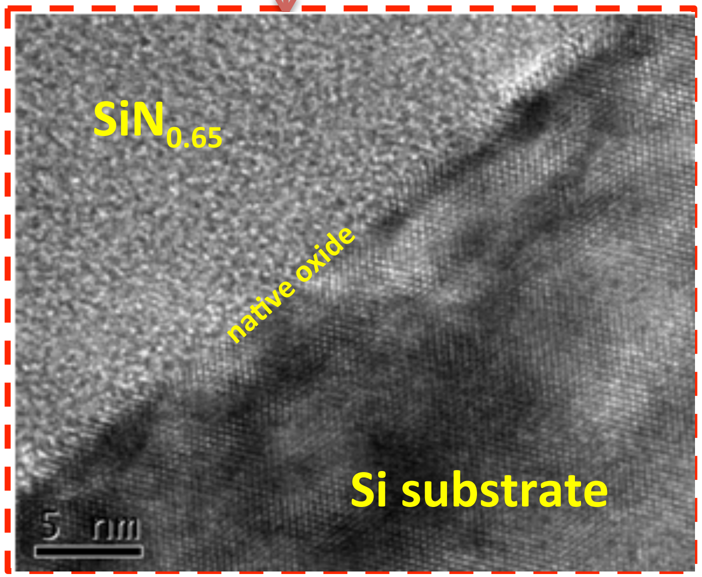
Gas flow rate SiH_4/NH_3(sccm)	Si/N	SiNx
25/30	60.64/30.36	$SiN_{0.65}$
10/60	53.47/46.53	$SiN_{0.87}$

Structure (from TEM)

$SiN_{0.65}$



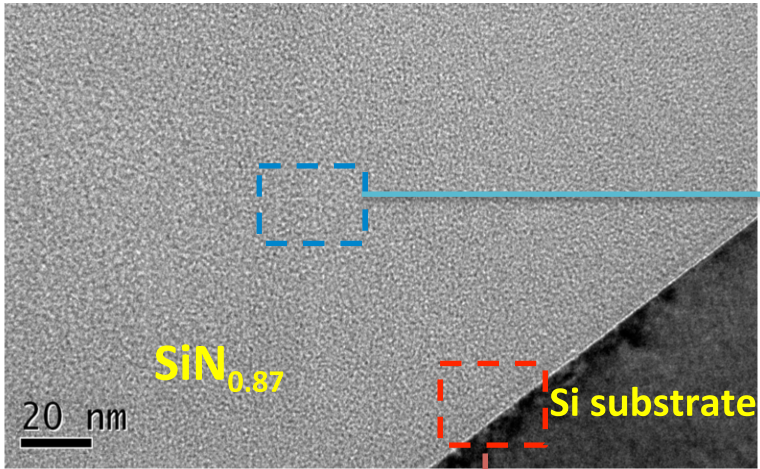


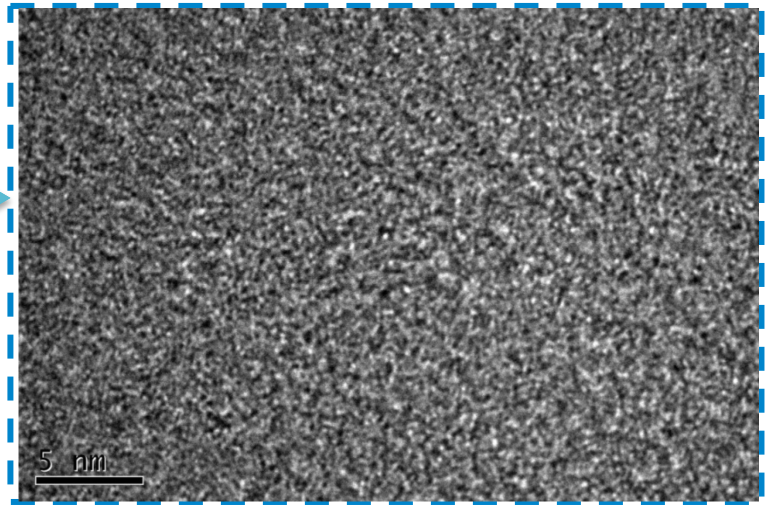


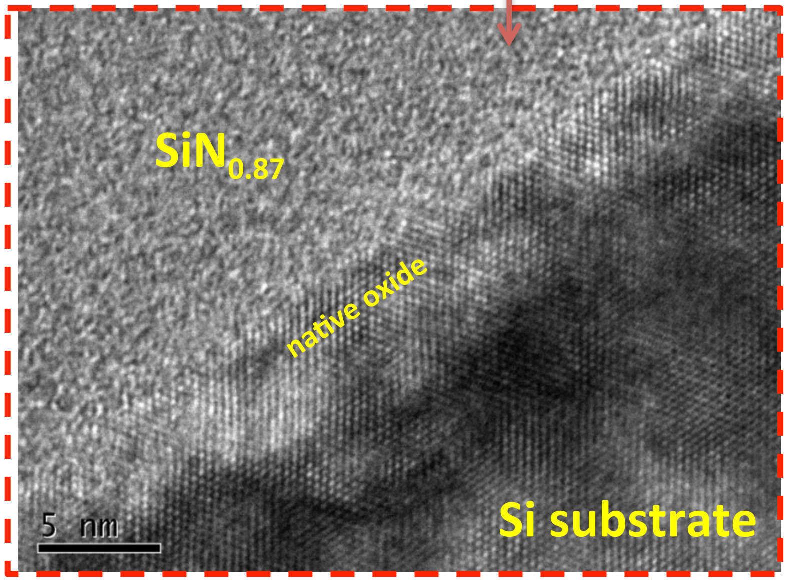
$SiN_{0.65}$ Structure : amorphous

Structure (from TEM)

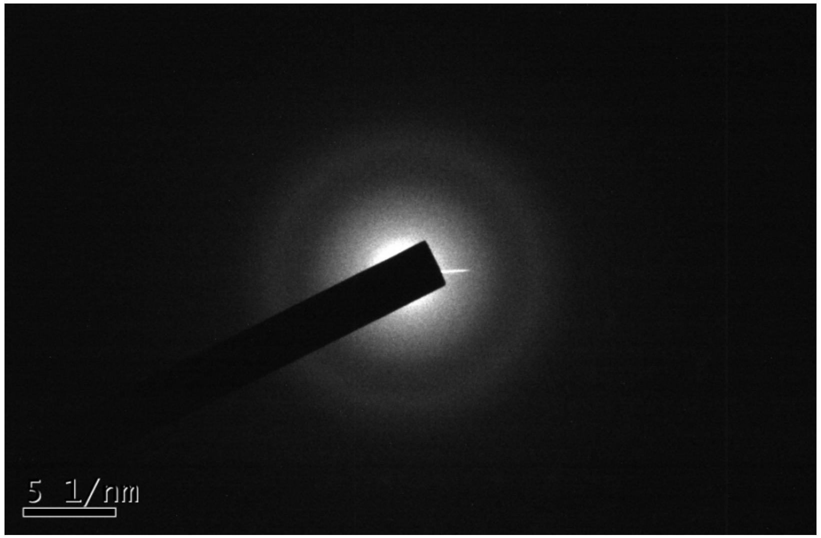
$SiN_{0.87}$





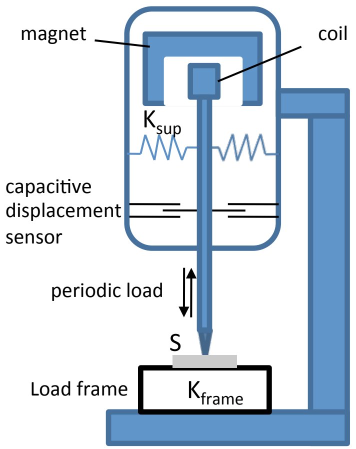


$SiN_{0.87}$ Structure : amorphous

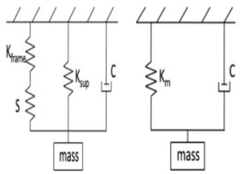


Young's modulus measurement by nano-indentation method

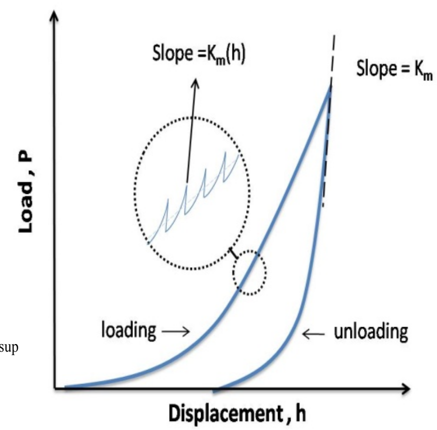
(illustrations from operational manual of Nano Indenter XP System ,MTS company, USA)

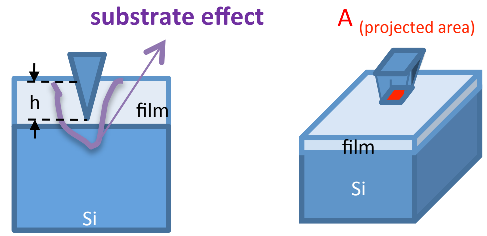


S : stiffness of sample



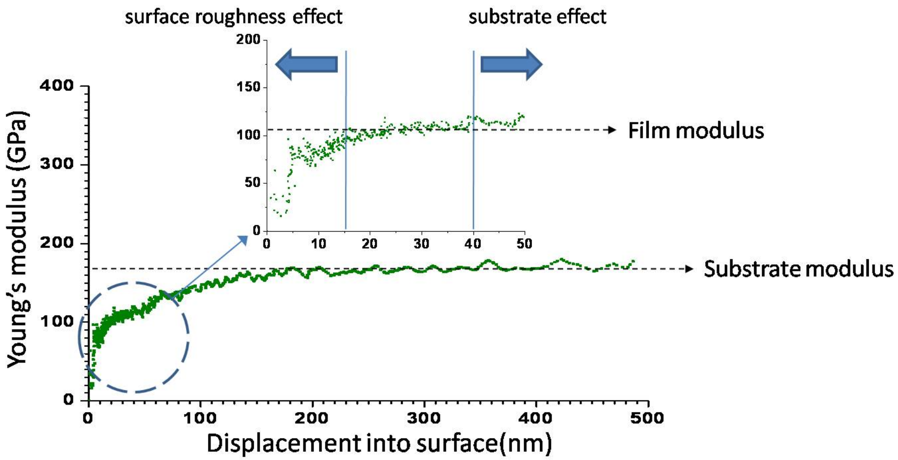
$$K_m(h) = [S(h)^{-1} + K_{frame}^{-1}]^{-1} + K_{sup}$$





$$E = S(h)\frac{\sqrt{\pi}}{2\beta\sqrt{A}}(1-\nu_{film}^{2}) - \frac{1-\nu_{film}^{2}}{(1-\nu_{indenter}^{2})}E_{indenter}$$

•$E_{indenter}$, $\nu_{indenter}$, ν_{film} , A and β are known.
•β :geometrical factor of the indenter.

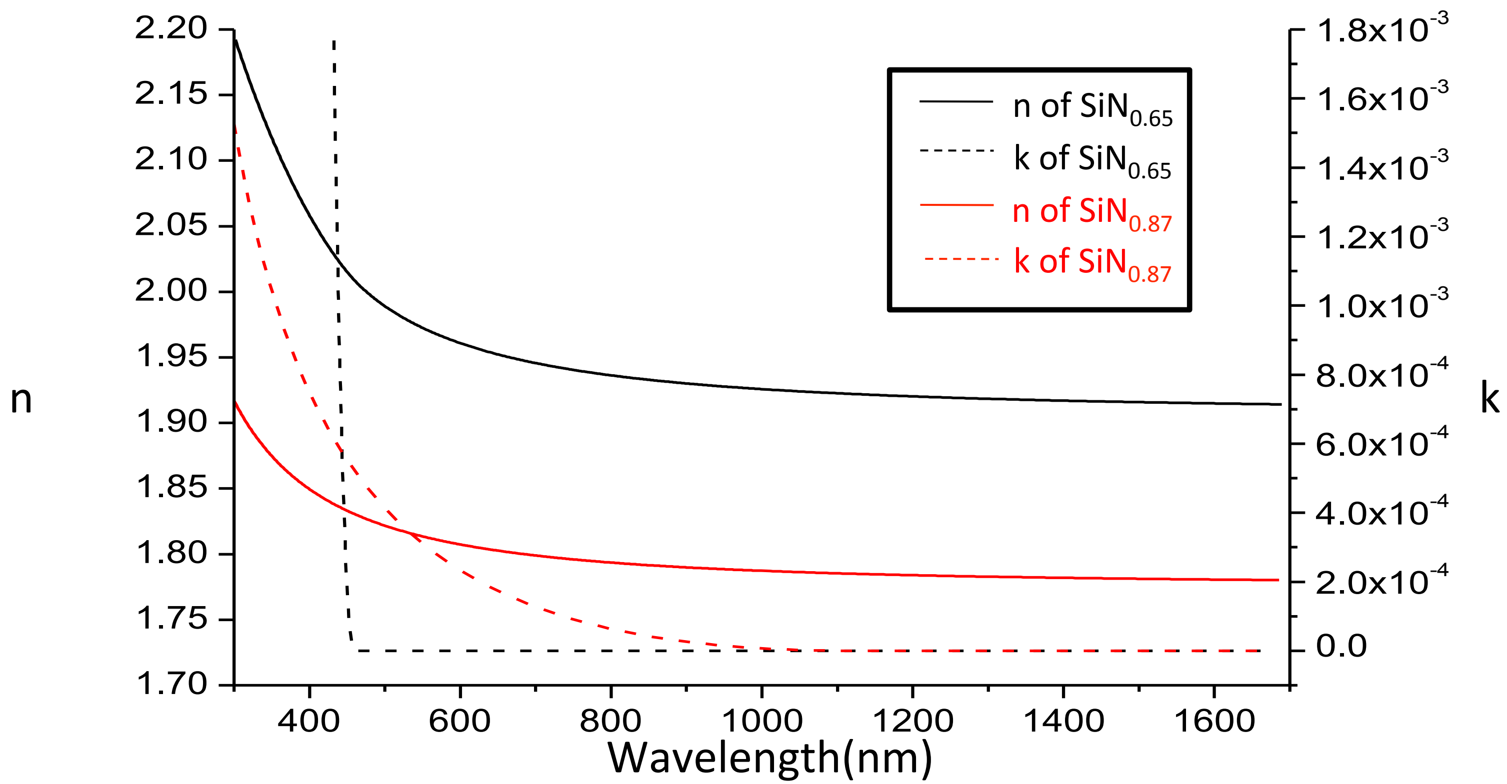


Results:
$SiN_{0.65}$: (100.8±7.8) GPa
$SiN_{0.87}$: (123.3±4.6) GPa

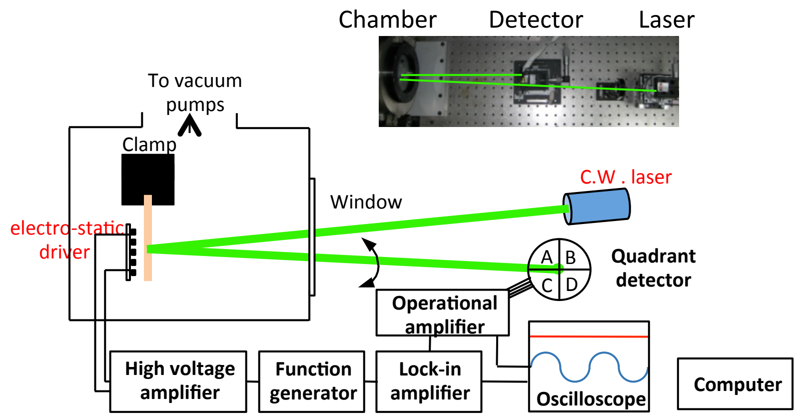
Optical properties (measured with ellipsometer)

Fitted by Tauc Lorentz dispersion model:

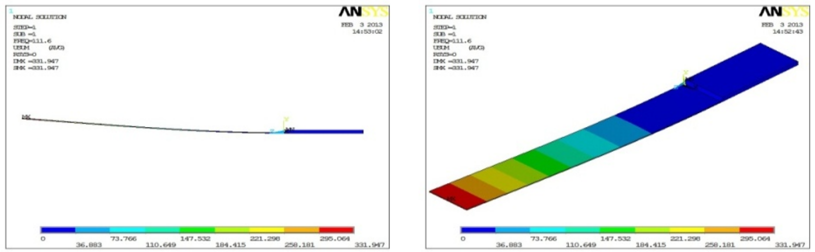
	SiN0.65		SiN0.87	
wavelength	1064nm	1550nm	1064nm	1550nm
n	1.923 ±0.003	1.915 ±0.003	1.785±0.001	1.781±0.001
k	0.0000	0.0000	0.0000	0.0000



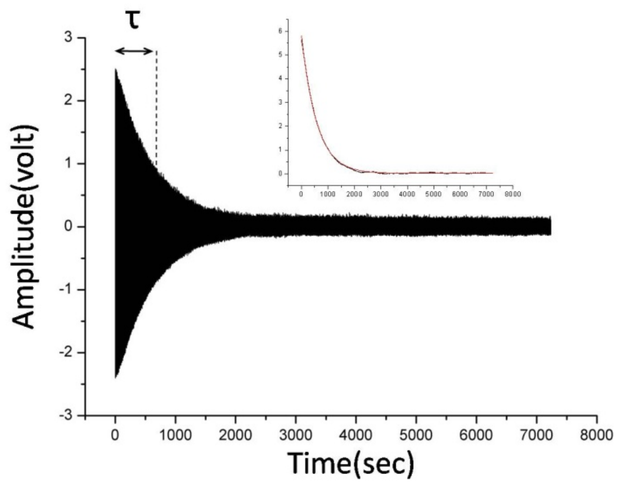
Mechanical loss measurement by cantilever ring-down

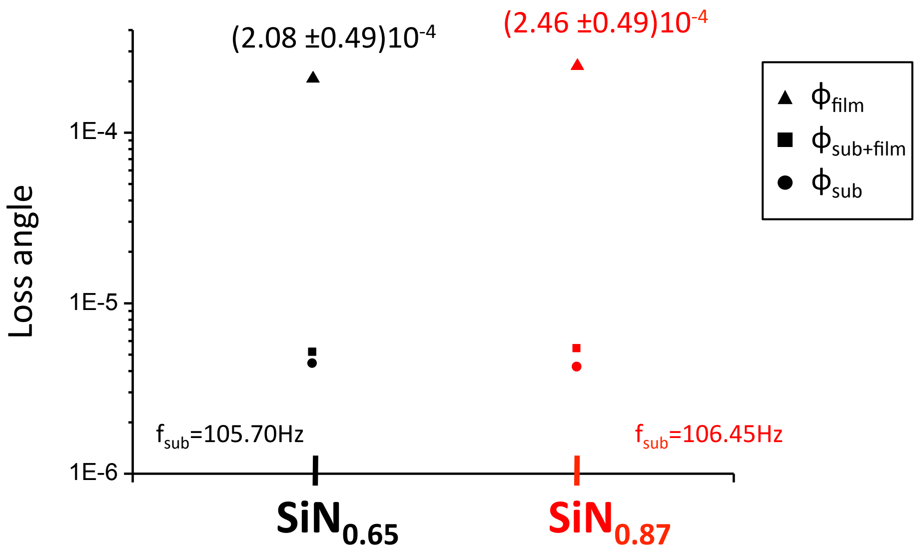


$$\phi_{film} = \frac{Y_s d_s}{3Y_f d_f}(\phi_{sub+film} - \phi_{sub}) \quad , \phi_{sub+film} = 1/\pi\, f_0'\tau' \quad , \phi_{sub} = 1/\pi\, f_0\tau$$



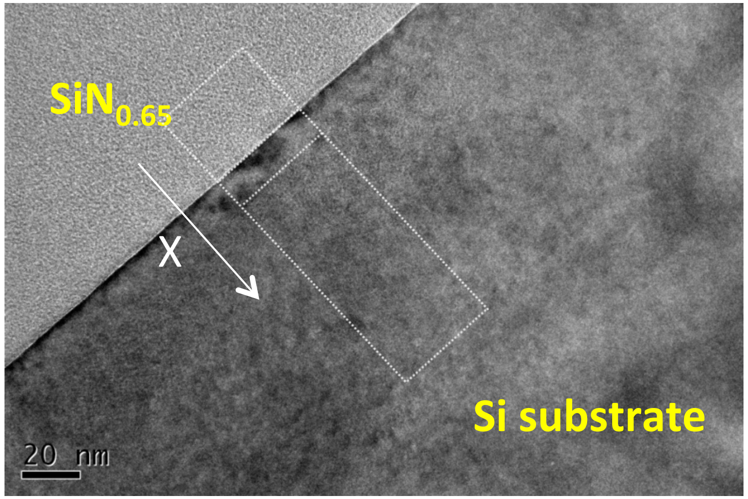
First bending mode (~100Hz) pattern from ANSYS simulation of a 34mmx5mmx92um Si cantilever.

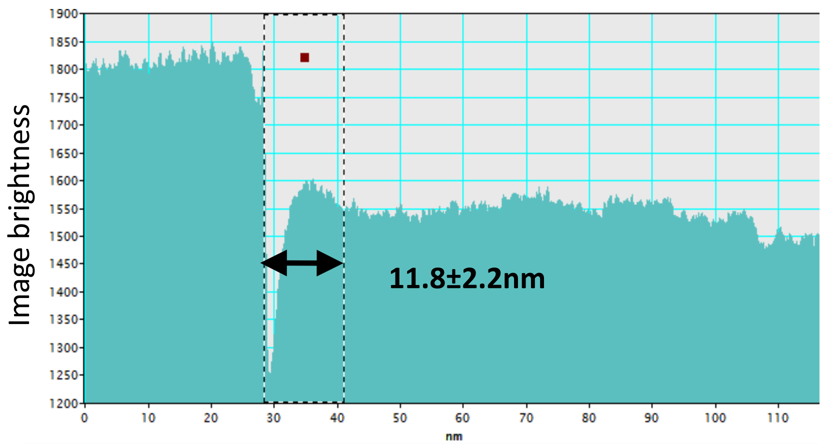




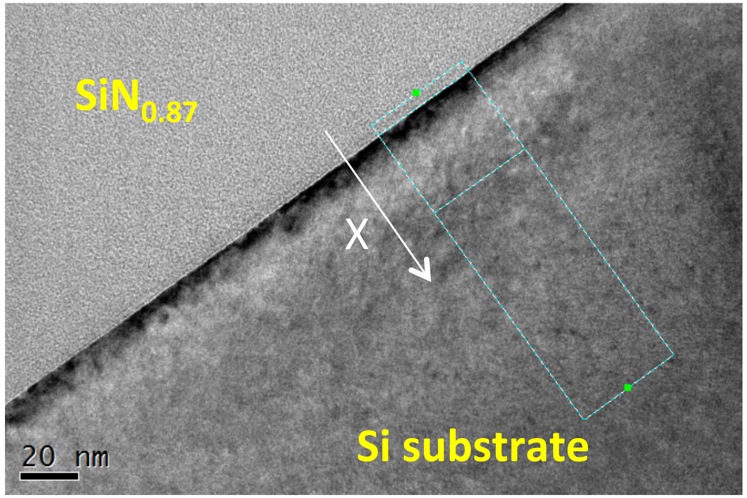
Effect of Stress (TEM)

$SiN_{0.65}$





$SiN_{0.87}$



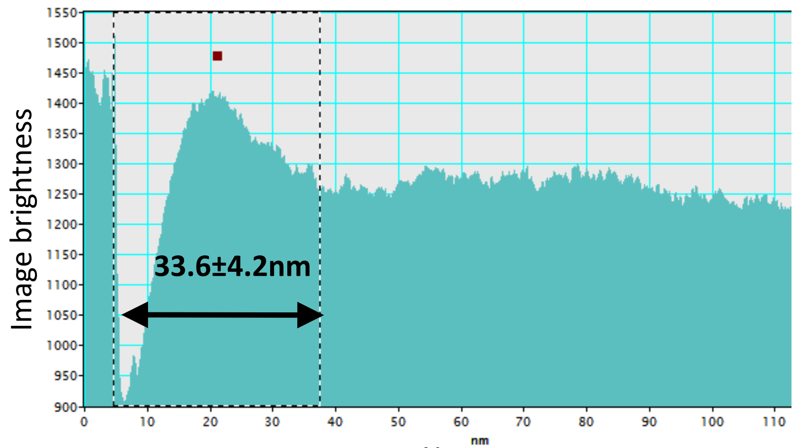
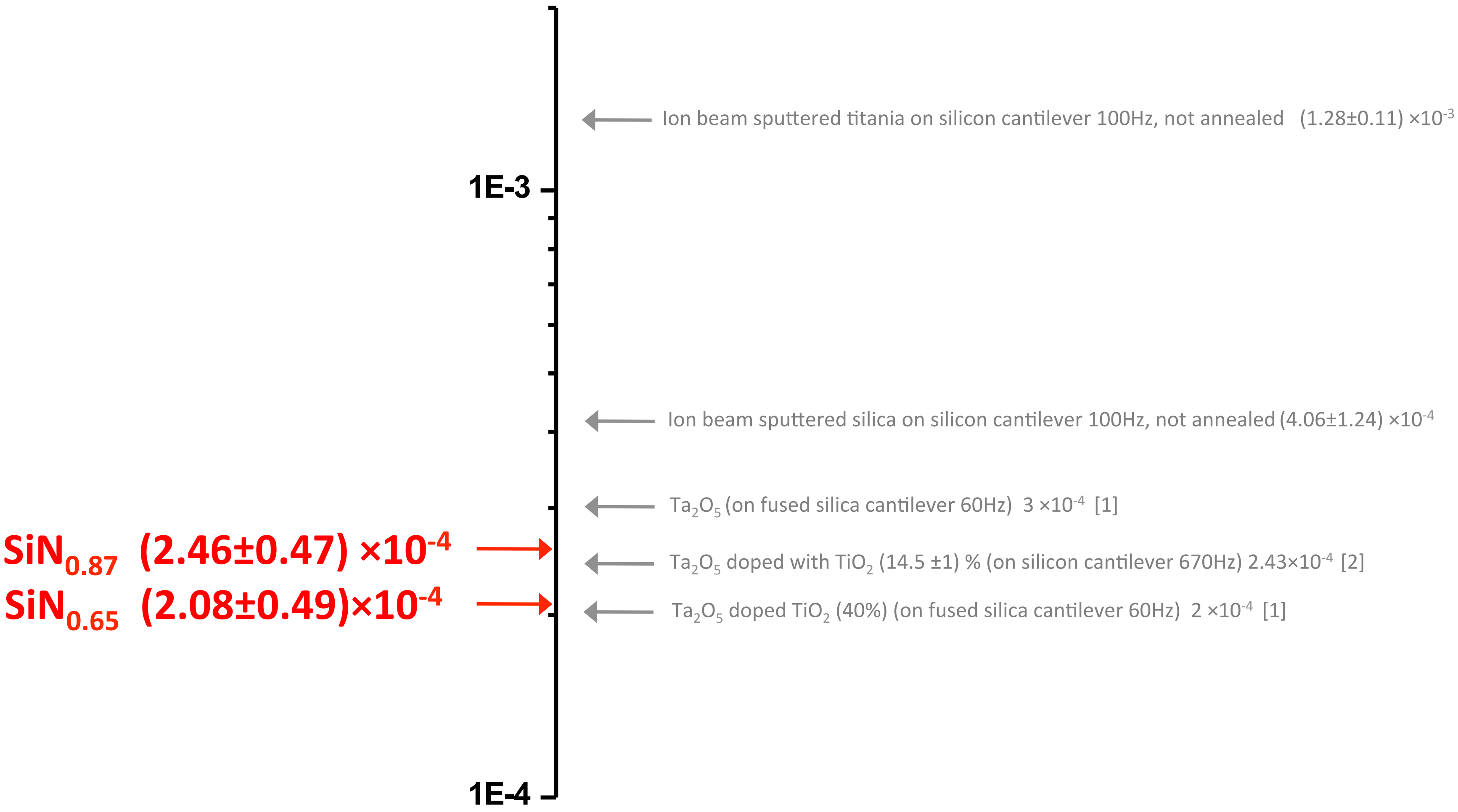


Image brightness distribution along X. (Average were taken within the dashed frame, ten frames were then averaged again)

Results Summary

Property	Method	Sample	
		$SiN_{0.65}$	$SiN_{0.87}$
Si/N ratio	ESCA	60.64/30.36	53.47/46.53
Refractive index	Ellipsometer	1.923 ±0.003 @ 1064nm 1.915 ±0.003 @ 1550nm	1.785 ±0.001 @ 1064nm 1.781 ±0.001 @ 1550nm
Extinction coefficient	Ellipsometer	$<10^{-4}$ @ 1064nm & 1550nm	$<10^{-4}$ @ 1064nm & 1550nm
Young's modulus	Nano - Indentation	(100.8±7.8) GPa	(123.3±4.6) GPa
Structure	TEM	amorphous	amorphous
Stress	1. Curvature scanner in NTHU 2. Curvature scanner in NDL (Tencor FLX-2320) 3. Phase shift interferometry in NCU	TBD	TBD
Loss angle	Ring down system	$(2.08\pm0.49)\times10^{-4}$	$(2.46\pm0.47)\times10^{-4}$

Loss angle comparison (room temperature)



[1]R Flaminio,et al "A study of coating mechanical and optical losses in view of reducing mirror thermal noise in graviational wave detectors." Class. Quantum Grav. **27** (2010) 084030

[2]Iain William Martin "Studies of materials for use in future interferometric gravitational wave detectors" presented as a thesis for degree of Ph.D Feb. 14, 2009

LSC LIGO Scientific Collaboration

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Composition-dependent internal stress of SiN films

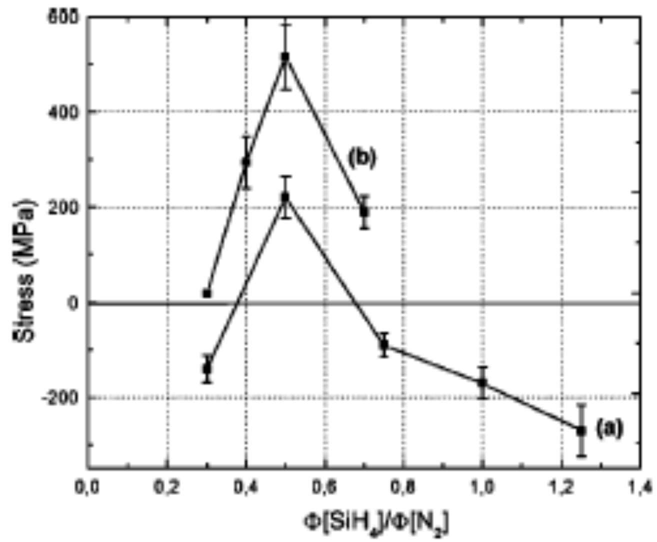

FIG. 7. Intrinsic stress in silicon nitride films vs SiH_4/N_2 flow ratio (a) and with helium dilution (b).

Ref 1. E. Cianci, F. Pirola, and V. Foglietti. "Analysis of stress and composition of silicon nitride thin films deposited by electron cyclotron resonance plasma-enhanced chemical vapor deposition for microfabrication processes". J. Vac. Sci. Technol. B 23, 168 (2005);

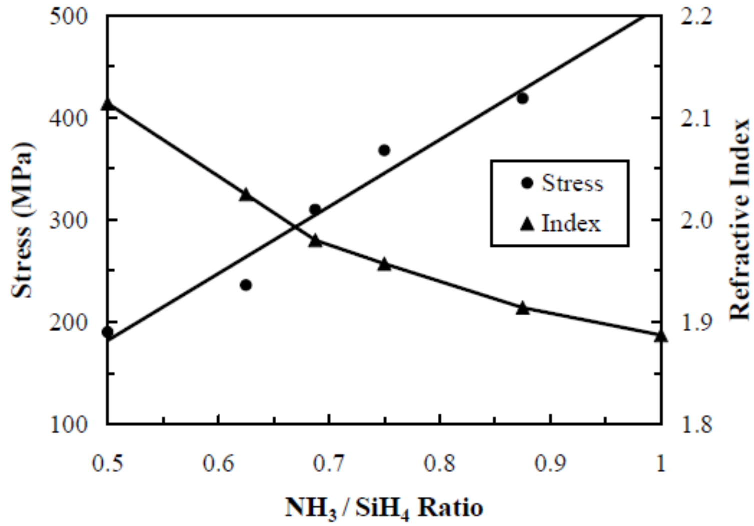

Figure 3. Tensile stress and refractive index of PECVD SiN_x films deposited at 250°C as a function of NH_3/SiH_4 gas flow ratio. Deposition rates were about 120 Å/min.

Ref 2. K.D. Mackenzie, et al. "silicon nitride and silicon dioxide thin insulating films & other emerging dielectrics VIII, PV2005-01, 148-159, ElectroChemical Society, Pennington, NJ (2005)".

Our sample: NH_3/SiH_4=1.2 for $SiN_{0.65}$,
NH_3/SiH_4=6.0 for $SiN_{0.87}$

Conclusion and future works

1. $SiN_{0.65}$ and $SiN_{0.87}$ films fabricated by PECVD on Si cantilever showed comparable loss angle to the Tantala and Tantala/Titania mixed films at room temperature.

2. Stress measurement is on-going.

3. Samples of different NH_3/SiH_4 flow rate ratio are in fabrication.

4. Correlation between the stress, composition and the loss angle will be established.

5. Effect of annealing shall be investigated

6. Fabrication of SiN films by LPCVD (reported to have higher stress) is on-going.

7. Low-temperature loss angle measurements need to be done.

Stress in the film may play positive or negative roles in various aspects, let's keep it open at this early stage.