Progress of coating development at NTHU

(National Tsing Hua University in Taiwan)

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Ring-down measurement error analysis

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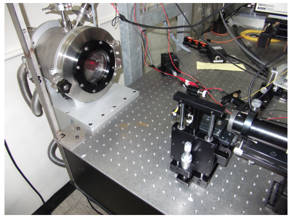
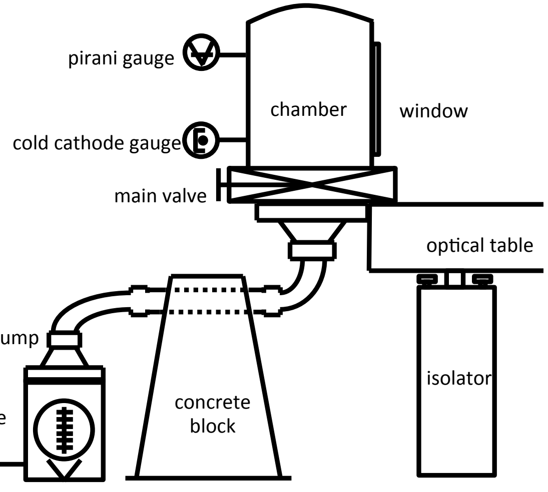
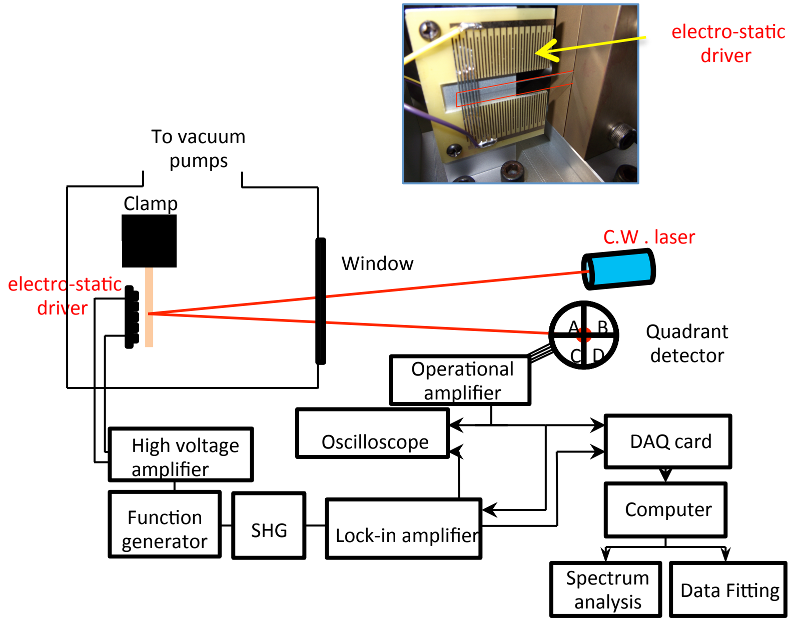
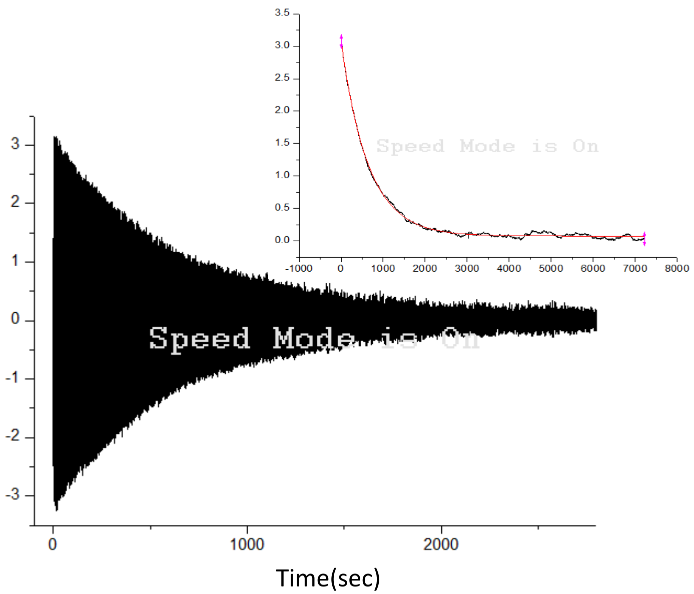
Ring-down measurement setup





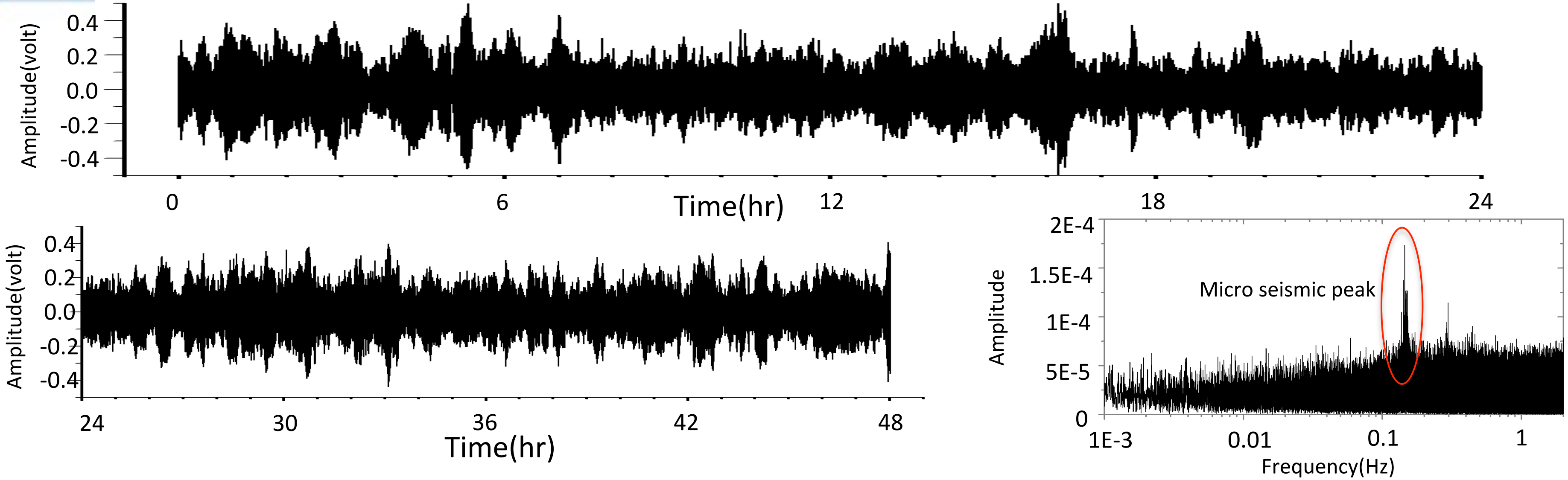




A typical ring-down pattern of Si cantilever

For the first bending mode (~ 100Hz) of a 3~5cm high quality cantilever, background low frequency noises dominate the uncertainty of the ring-down time determination

Error analysis (I)



48 hours background noise (measured) and its low frequency spectrum

Error simulation method

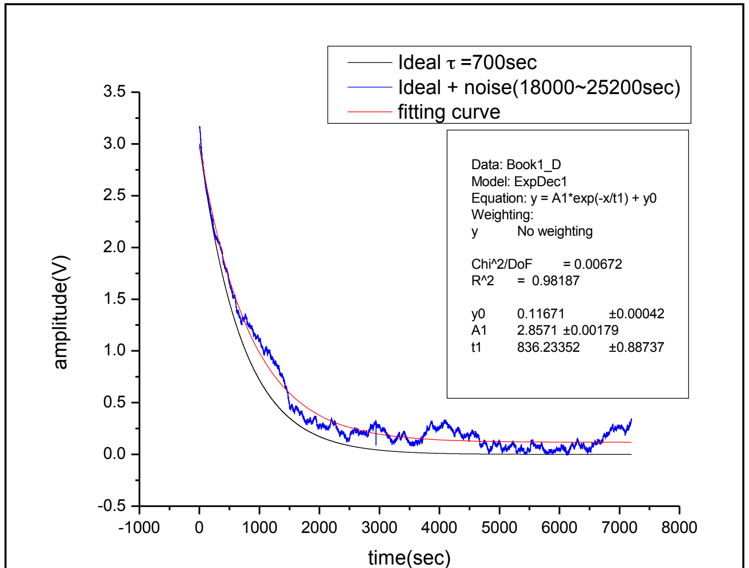
(1) Pick up a segment of continuous 2 hours background noise from the measurement

(2) Add the noise to an ideal noiseless ring-down curve

(3) Fit the curve to find the decay time

(4) Repeat the simulation many times

(5) Obtain the statistics for the decay time



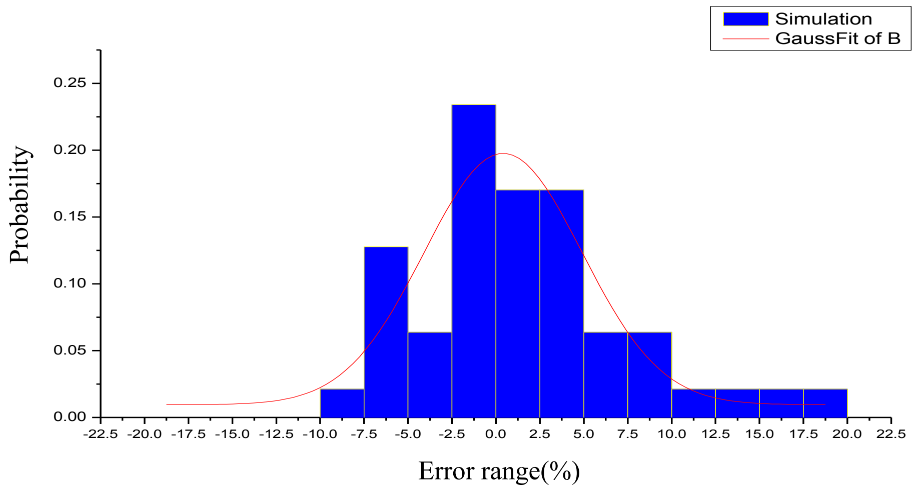
A typical simulation result

Error analysis (II) – simulation results – expected error from background noise

Sampling range(sec)	Decay time(sec)	Error %
Ideal	700	
0~7200	639.1	-8.70
3600~10800	687.8	-1.74
7200~14400	720.7	+2.96
10800~18000	669.1	-4.41
14400~21600	759.3	+8.47
18000~25200	836.2	+19.46
21600~28800	697.5	-0.36
25200~32400	674.8	-3.60
28800~36000	677.1	-3.27
32400~39600	707.4	+1.06
36000~43200	699.1	-0.13
39600~46800	712.6	+1.80
43200~50400	660.3	-5.67
46800~54000	714.3	+2.04
50400~57600	702.2	+0.31

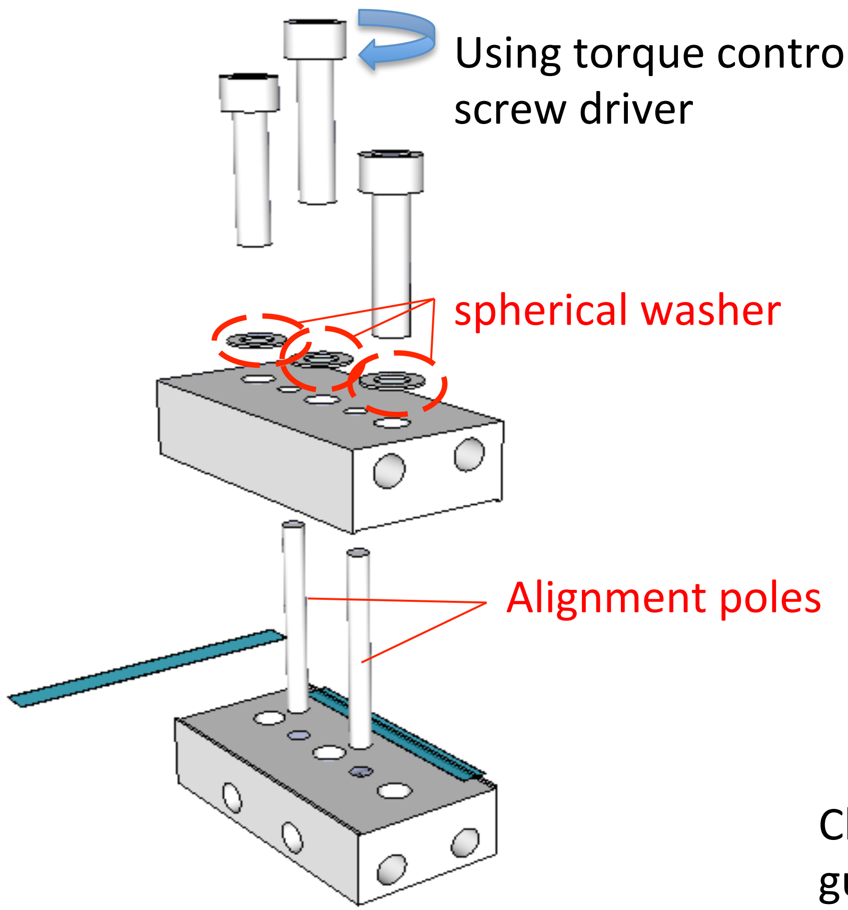
Sampling range(sec)	Decay time(sec)	Error%
54000~61200	656.6	-6.20
57600~64800	787.7	+12.53
61200~68400	719.6	+2.80
64800~72000	665.0	-5.00
68400~75600	690.9	-1.30
72000~79200	696.9	-0.44
75600~82800	710.9	+1.56
79200~86400	655.5	-6.36
82800~90000	736.5	+5.21
86400~93600	700.9	+0.13
90000~97200	698.7	-0.19
93600~100800	760.6	+8.66
97200~104400	722.4	+3.20
100800~108000	715.2	+2.17
104400~111600	692.6	-1.06
108000~115200	776.0	+10.86

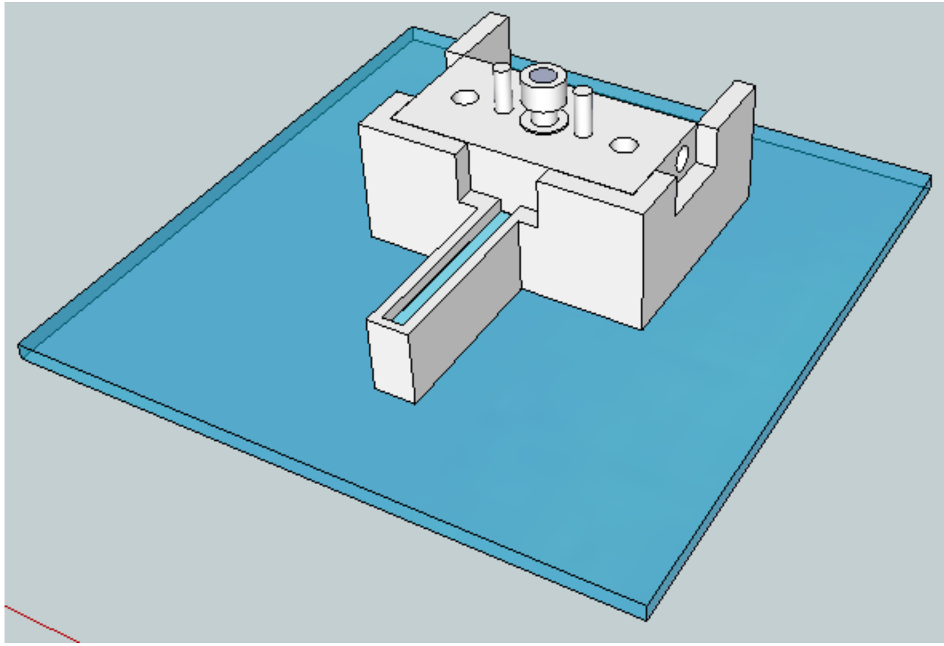
Sampling range(sec)	Decay time(sec)	Error%
111600~118800	663.6	-5.20
115200~122400	747.5	+6.79
118800~126000	727.6	+3.96
122400~129600	754.9	+7.84
126000~133200	705.9	+0.84
129600~136800	718.1	+2.59
133200~140400	724.6	+3.51
136800~144000	693.8	-0.89
140400~147600	735.7	+5.10
144000~151200	689.2	-0.26
147600~154800	687.1	-1.84
151200~158400	814.8	+16.40
154800~162000	698.0	-0.29
158400~165600	728.2	+4.03
162000~169200	655.4	-6.37
165600~172800	729.7	+4.24



Average= 711.0 (sec) ± 5.74%

Error analysis (III) -- precautions for reducing re-clamping error





Clamping aligner for assembling the cantilever to guarantee the repeatability of the clamping position

Error analysis (IV) -- experimental results – effect of re-clamping

Ring-down time and loss angle of a particular silicon cantilever subjected to 5 times re-clamping and 29 times ring-down test in total

Re-clamp / Measurement	1	2	3	4	5	Average of total 29 values
1	737.5 (3.84×10^{-6})	679.4 (4.26×10^{-6})	741.2 (3.86×10^{-6})	682.5 (4.16×10^{-6})	635.0 (4.55×10^{-6})	699.5±7.39% (4.15×10^{-6} ±7.99%)
2	737.8 (3.87×10^{-6})	690.8 (4.20×10^{-6})	724.9 (3.96×10^{-6})	676.8 (4.23×10^{-6})	673.0 (4.29×10^{-6})	
3	811.6 (3.51×10^{-6})	711.4 (4.08×10^{-6})	752.3 (3.82×10^{-6})	774.7 (3.71×10^{-6})	681.6 (4.25×10^{-6})	
4	730.2 (3.94×10^{-6})	674 (4.32×10^{-6})	740.2 (3.90×10^{-6})	676.0 (4.30×10^{-6})	616.4 (4.72×10^{-6})	
5	642.1 (4.50×10^{-6})	588.5 (4.96×10^{-6}	750.8 (3.85×10^{-6})	649.0 (4.48×10^{-6})	680.7 (4.42×10^{-6})	
6	784.4 (3.67×10^{-6})		677.4 (4.40×10^{-6})	706.7 (4.12×10^{-6})	648.3 (4.50×10^{-6})	
Average of each clamping (x ± y)	740.6 ±7.82% (3.89×10^{-6} ±8.71%)	668.8 ±7.05% (4.36×10^{-6} ±7.97%)	731.1 ±1.50% (3.95×10^{-6} ±1.37%)	696.0±6.20% (4.18×10^{-6}±6.90%)	660.0±4.52% (4.43×10^{-6}±4.76%)	
Average of re-clamping (Average of x)	699.3±5.16% (4.16×10^{-6} ±5.79%)					

*Loss angle from gas damping was substracted

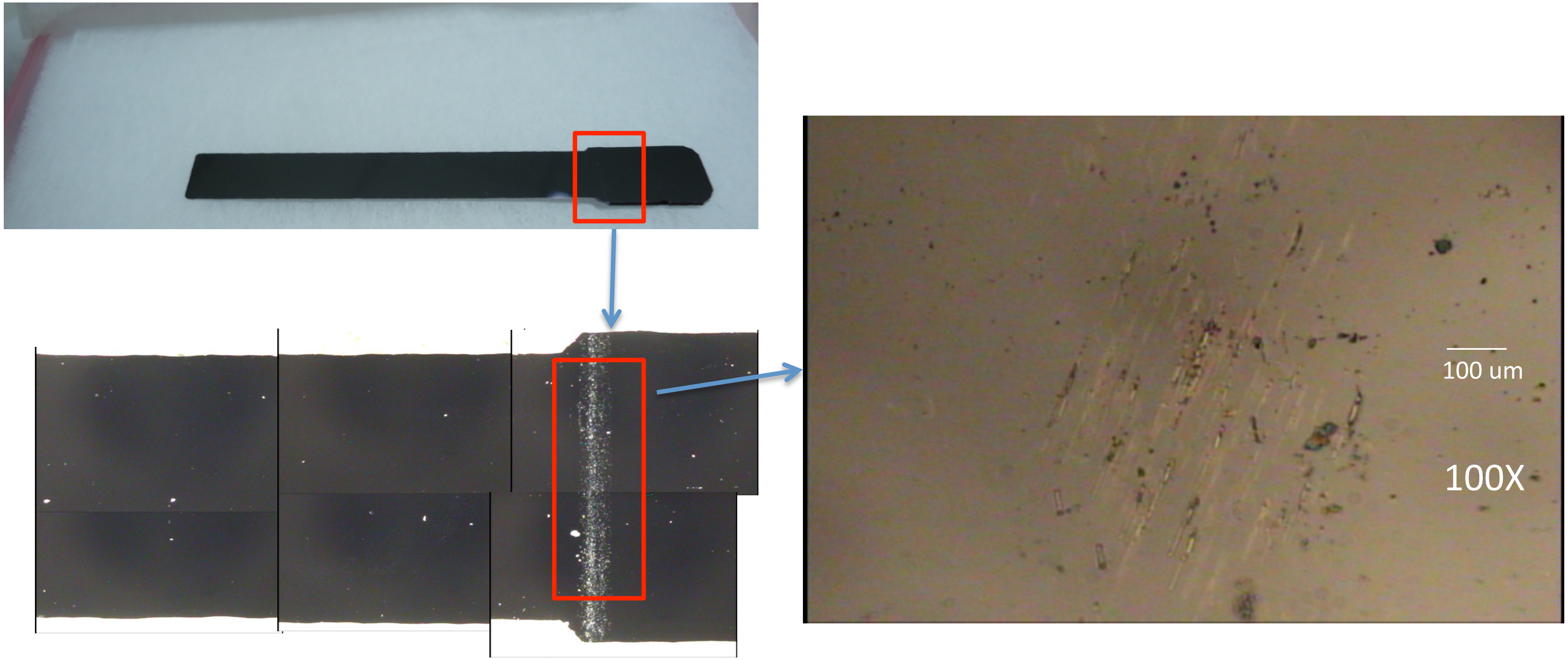
Resonant frequency : 106.73±0.01 Hz

Conclusion

The measured errors are approximately at the same level as the error from the background noise simulation results (5.74%, slide 6) implies that the errors are dominated by the background-noise.

Re-clamping error, if there is any, should be comparable or smaller than the error produced by the background-noise.

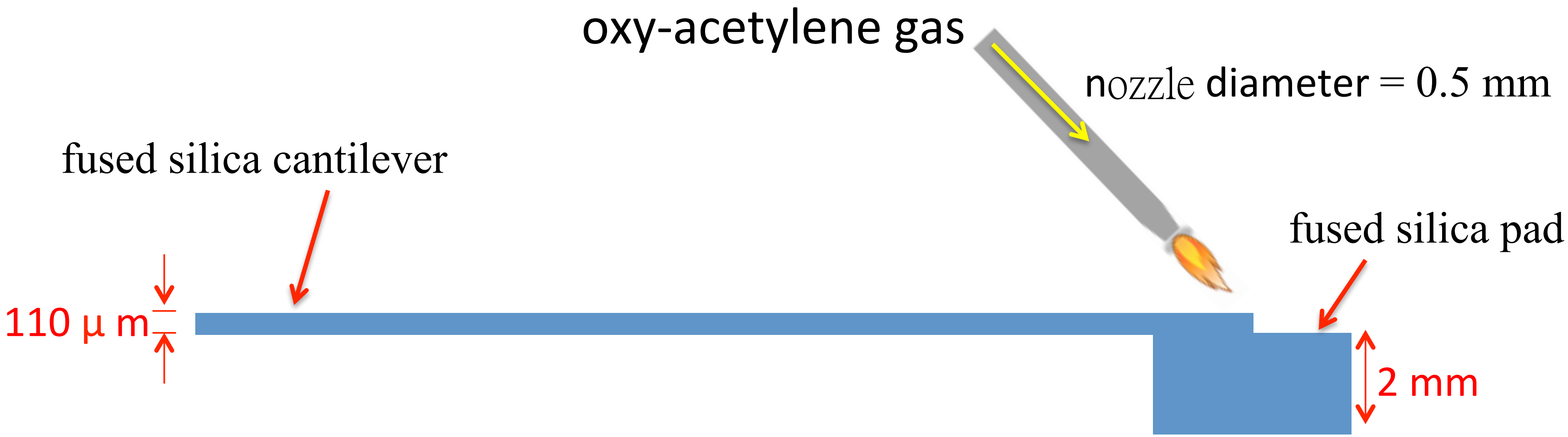
Effect of clamping scratches on silicon cantilever



This sample has been subjected to 5 times re-clamping, total of 29 times ring-down testing (data on previous slide). After all these tests, the decay time was still within the level of background-noise-dominated range. The scratches did not produce noticeable error above that of background noise and re-clamping.

Effect of thick clamping pad – add-on silica cantilever

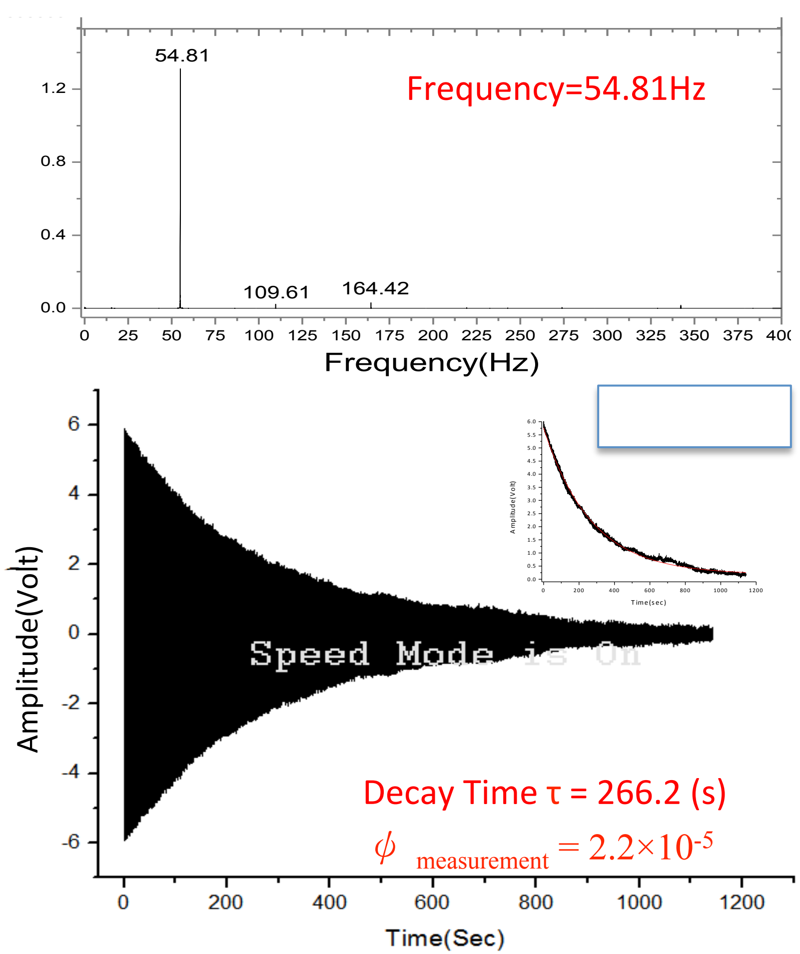
Fused silica cantilever flame welding

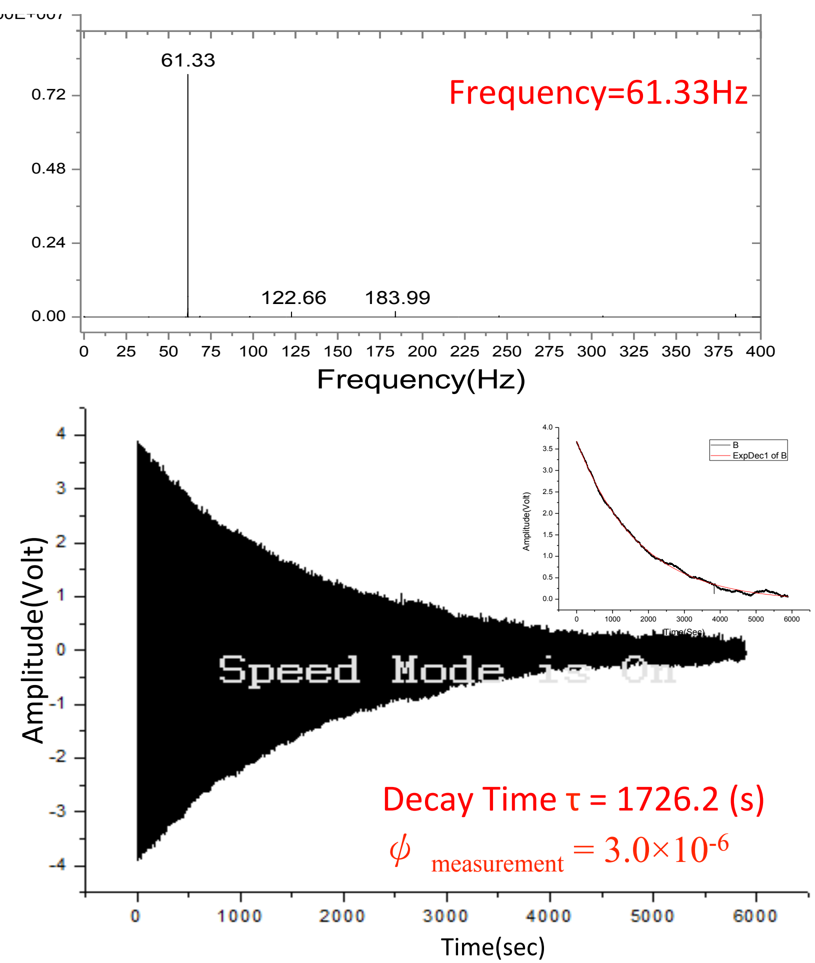


Sample: Corning 7980 fused silica 50mmx5mmx110um
Pad: 2mm thickness fused silica
Flame-welding: oxy-acetylene gas
Vendor: Good Harvest Technology, Hsinchu, Taiwan (proprietary know-how)

Fused silica cantilever flame welding

Without clamping pad | **with oxy-acetylene flame-welded pad**

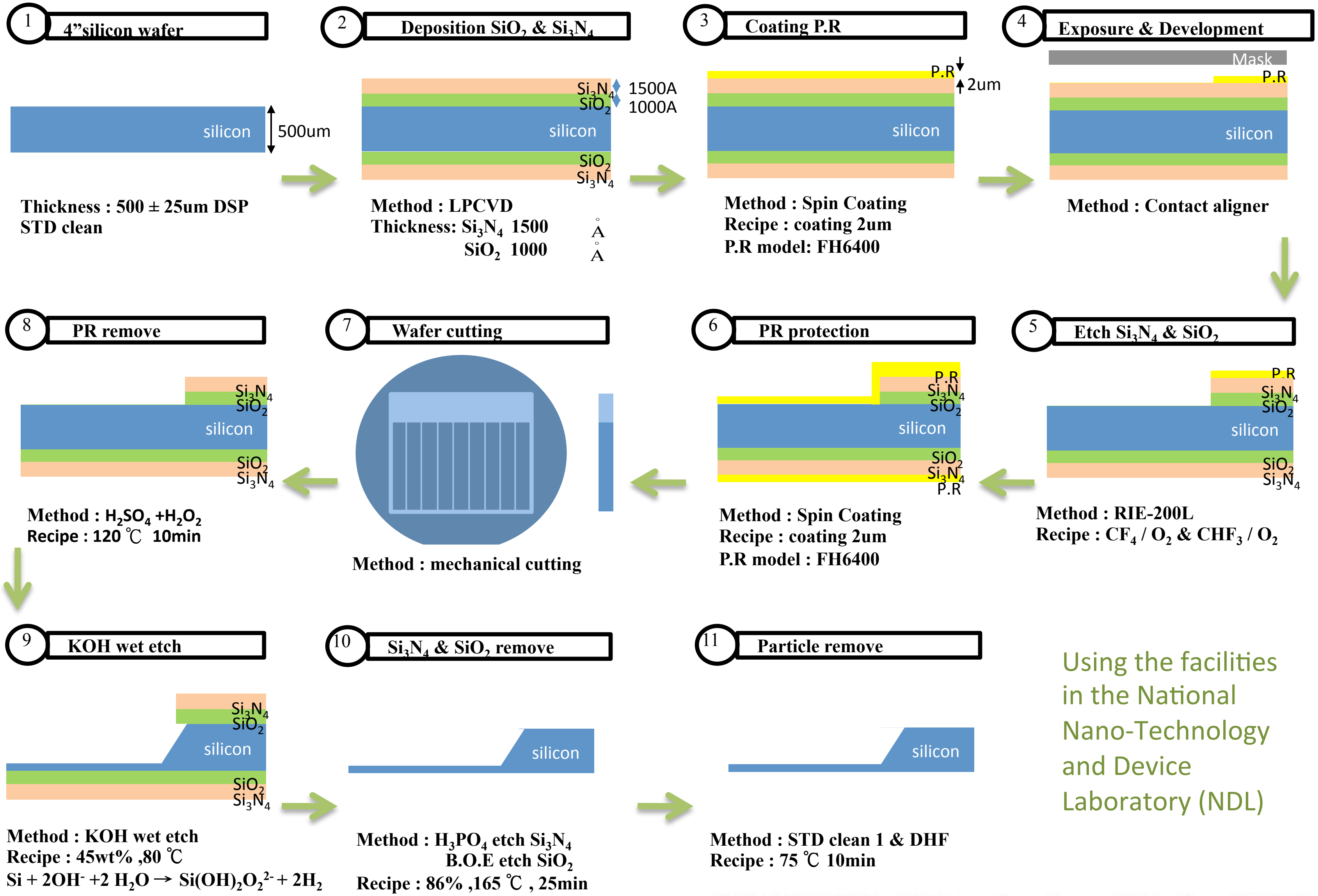




Loss angle decreased ~10 times from $2.2\text{x}10^{-5}$ (without pad) to $3.0\text{x}10^{-6}$ (with pad)

Fabrication of silicon cantilever and test results

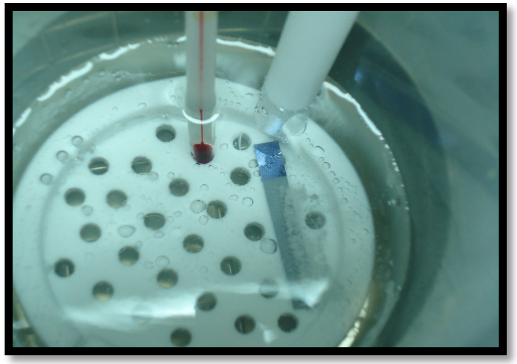
Silicon cantilever fabrication process (KOH wet etching)



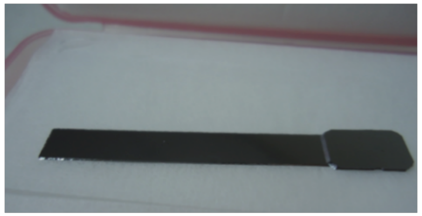
Using the facilities in the National Nano-Technology and Device Laboratory (NDL)

KOH wet etching:

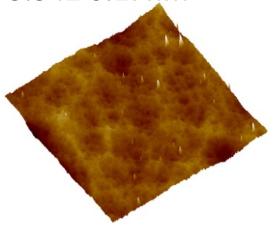
$$Si_{(s)} + 2(OH)^- + 2H_2O \rightarrow Si(OH)_2O_2^{2-} + 2H_{2(g)}$$

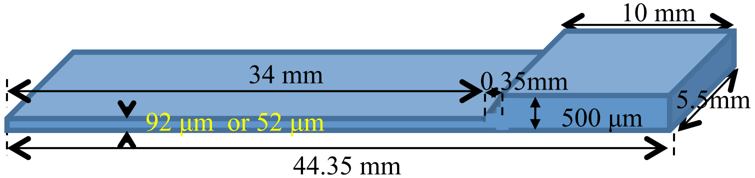


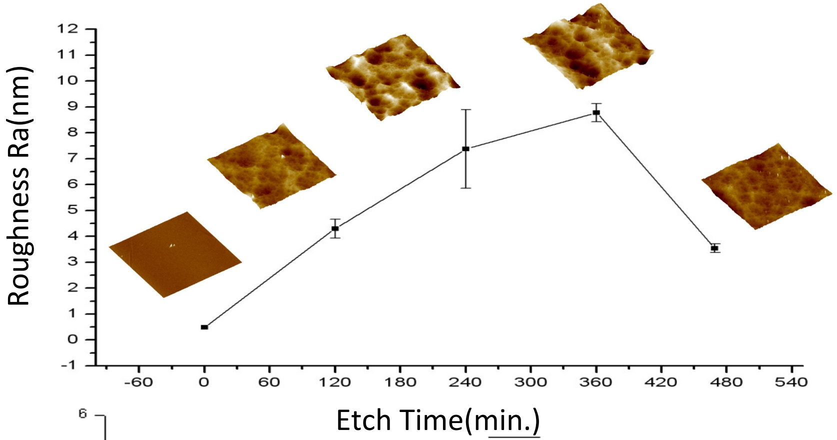
8hrs in KOH (92um)

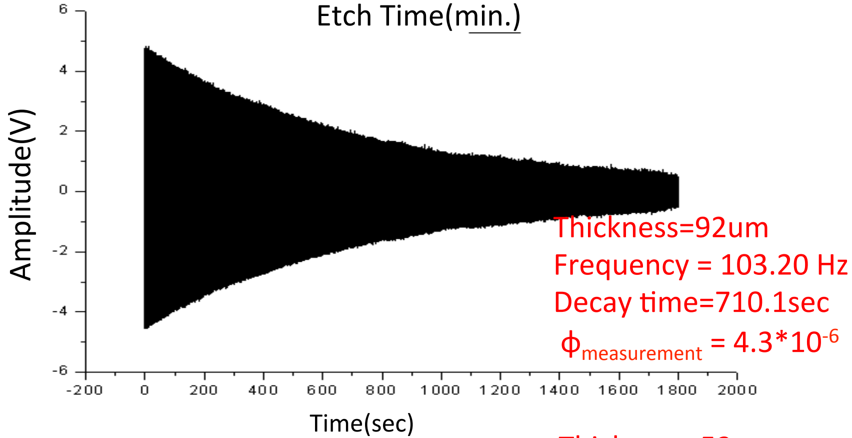


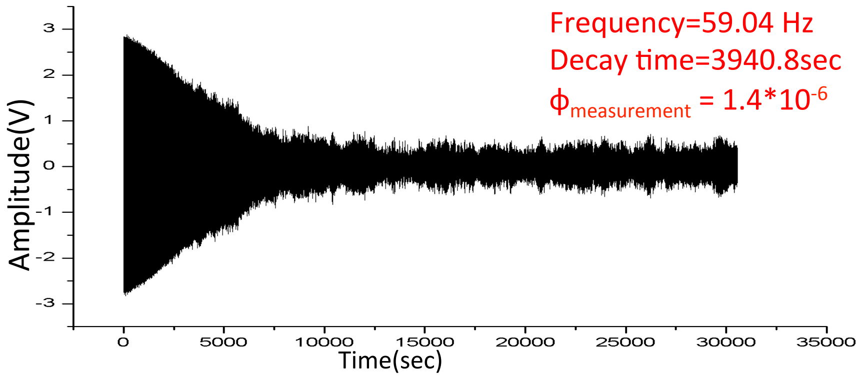
Ra=3.54± 0.17nm











Silicon cantilever fabricated from different wafers

CZ method : Czochralski method
FZ method : Floting- Zone method

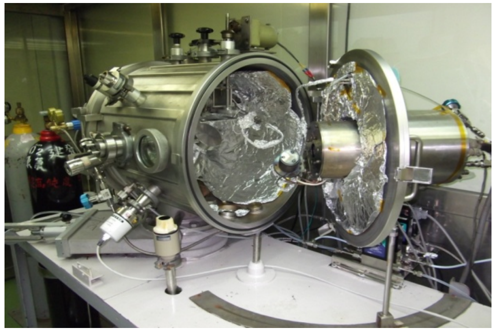
	Single Side Polish CZ Boron Doped wafer		Double Side Polish FZ Un-doped wafer	
	Cantilever No3	Cantilever No5	Cantilever No1	Cantilever No2
Size	4 inch		4 inch	
Polish	single side polish		double side polish	
Growth	CZ method		FZ method	
Orientation	(100)		(100)	
Doping	Boron		Un-doped	
Resistivity (ohm-cm)	1-100		10,000-15,000	
Length (mm)	34.4	34.3	34.5	34.2
Width (mm)	5.15	5.00	4.8	4.90
Thickness (um)	92±1	92±1	92±1	92±1
Clamping block thickness (um)	525±1	525±1	509±1	509±1
Etched side roughness Ra.(nm)	7.08±0.43	5.27±1.04	3.54±0.17	8.48±0.53
Un-etched side roughness Ra.(nm)	169.24±39.34(un-polished)	169.24±39.34(un-polished)	0.47±0.08 (polished)	0.47±0.08 (polished)
Fundamental Freq. (Hz)	103.36	96.66	106.73	104.12
Decay Time (sec)	690.6±2.95%	799.7±3.26%	697.8±5.75%	679.9±8.14%
Mechanical Loss Angle	$4.36*10^{-6}$±2.67%	$3.96*10^{-6}$±3.24%	$4.15*10^{-6}$±7.99%	$4.48*10^{-6}$±13.28%

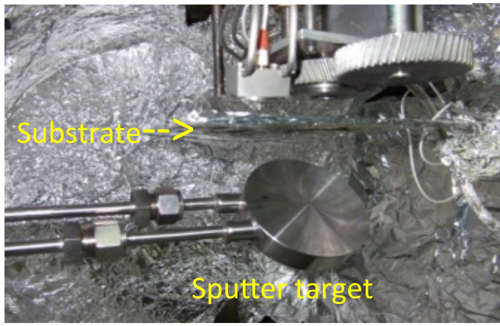
Conclusion

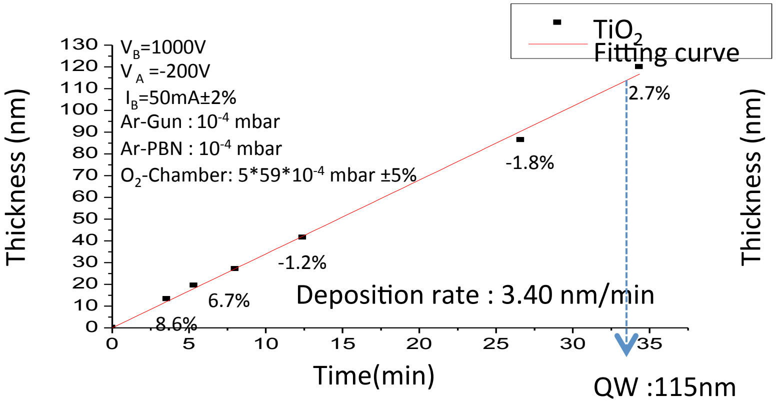
Despite of the differences between these two wafers, i.e. large differences in surface roughness (loss from surface), CZ vs. FZ(loss from defect), and doping level (loss from impurity), however, deviations of the loss angles for silicon cantilevers fabricated from these two wafers are still approximately within the range of background and re-clamping error.

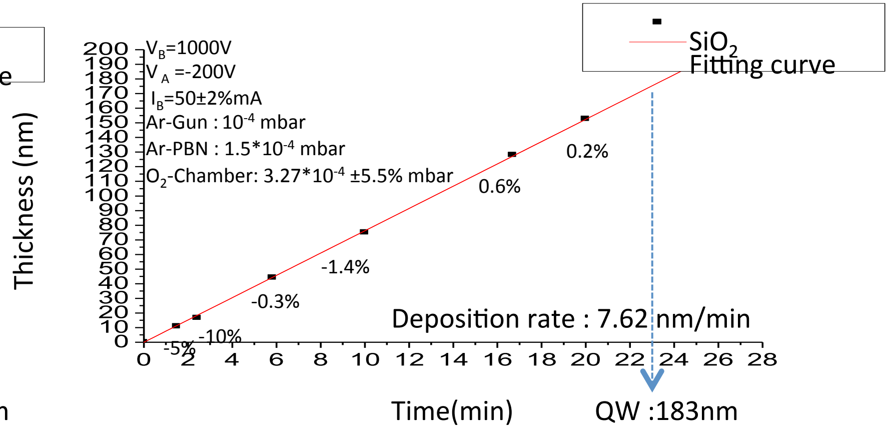
Nano-layer Deposition

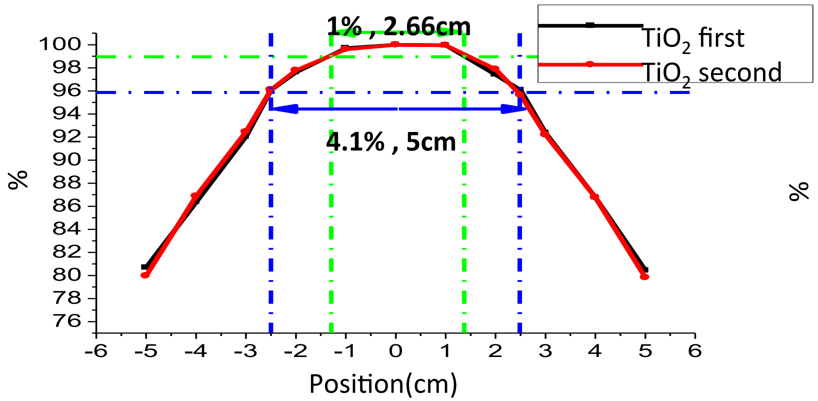
Coating uniformity and deposition rate calibration for TiO_2 and SiO_2 films

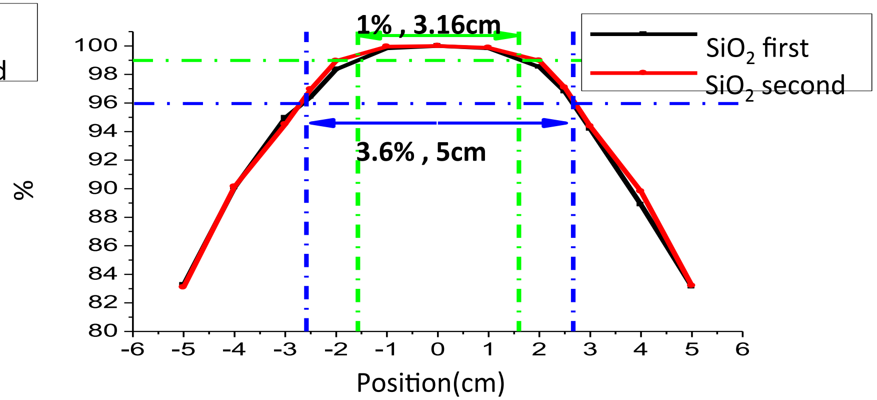




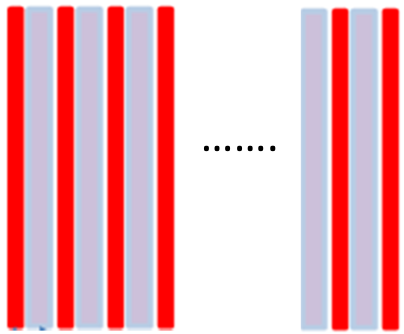








Nanolayered QWL TiO_2::SiO_2 prototypes (designed thickness)

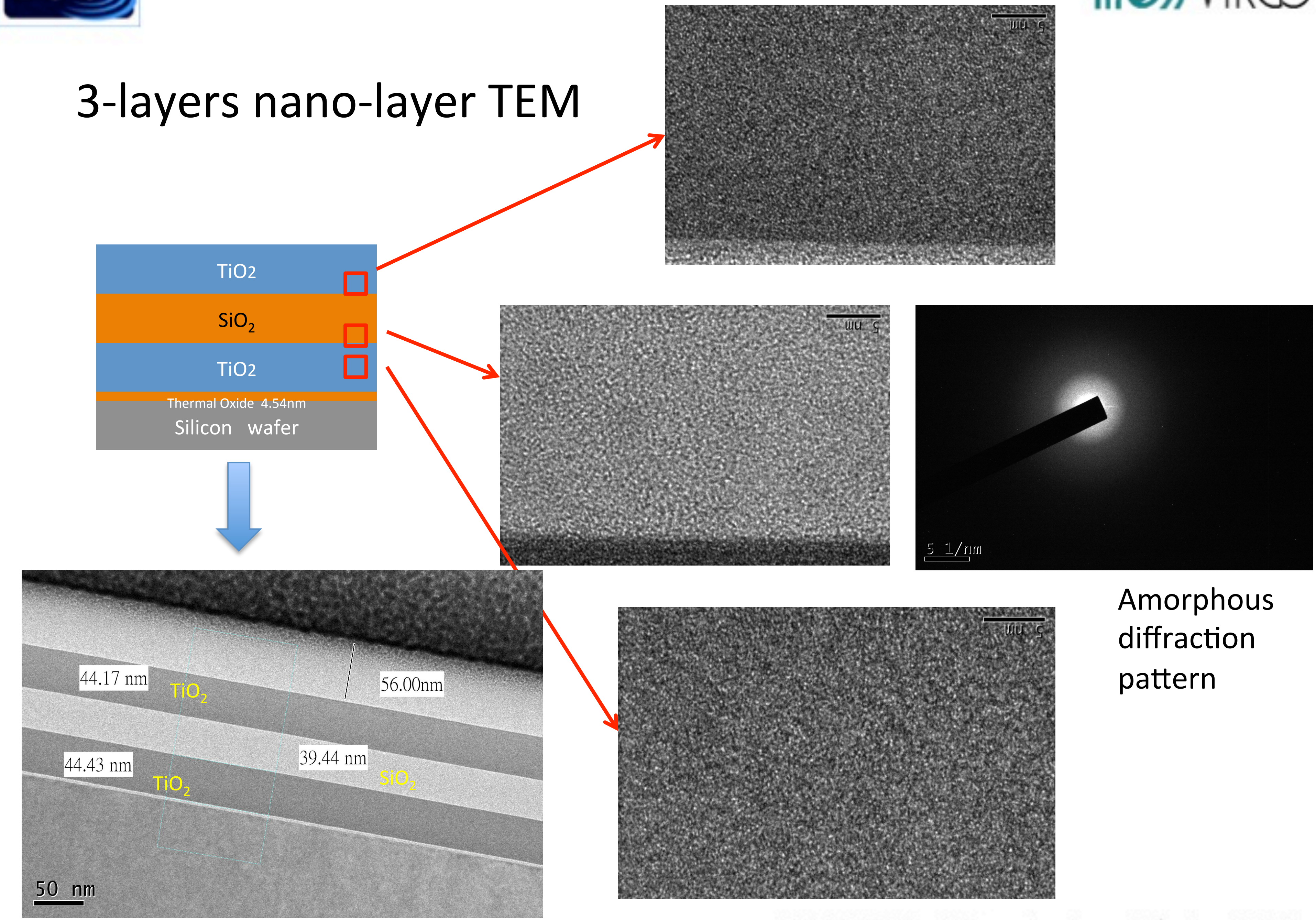


N TiO2	Thickness TiO2 [nm]	N SiO2	Thickness SiO2
2	42.56	1	42.15
3	28.37	2	21.07
4	21.28	3	14.05
5	17.02	4	10.54
6	14.19	5	8.43
7	12.16	6	7.02
8	10.64	7	6.02
9	9.46	8	5.27
10	8.51	9	4.68

Total physical thickness: 127.27nm, total optical thickness: 266.00nm(QW of 1064nm)

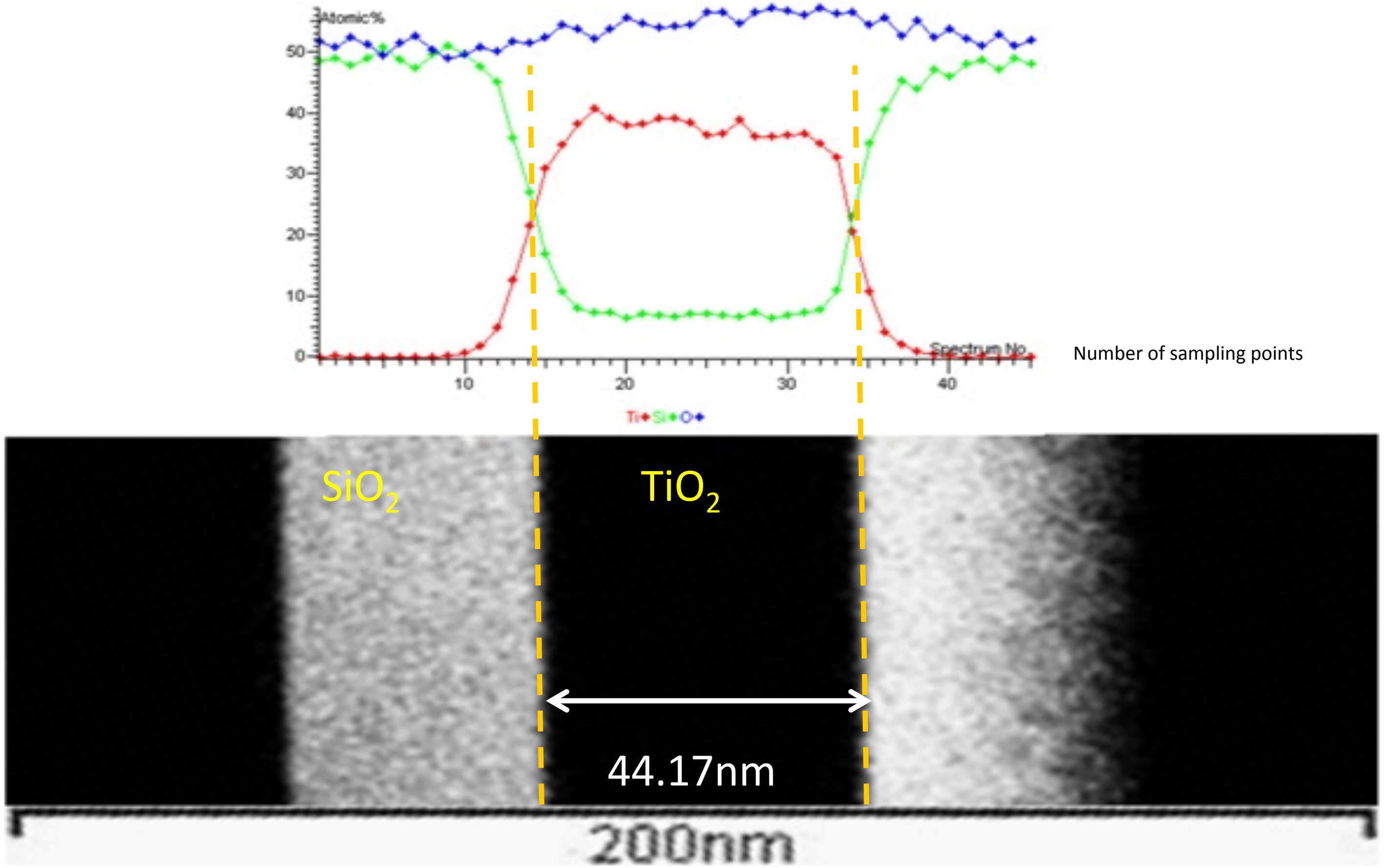
Number (N) of TiO_2 and SiO_2 nanolayers and corresponding thicknesses of each nanolayer (nm). Total optical thickness is QWL for all; equivalent index is 2.09 [Ref: Designs from Prof. Innocenzo Pinto]

3-layers nano-layer TEM



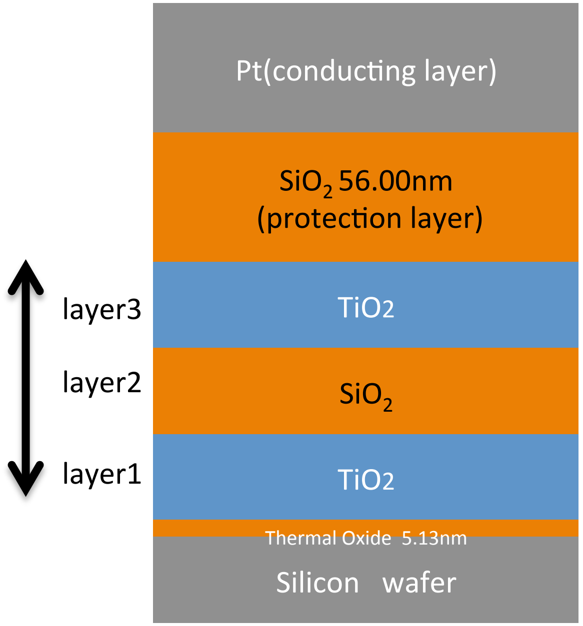
Amorphous diffraction pattern

Energy-dispersive x-ray spectroscopy (EDX) depth profile of the TiO_2 layer and the interface



Depth resolution of EDX (~1-2nm) is not sufficient to resolve the small interface mixing. It may work for large interface diffusion for samples subjected to high temperature annealing.

Precision of layer thickness control for the 3-layers nanolayer



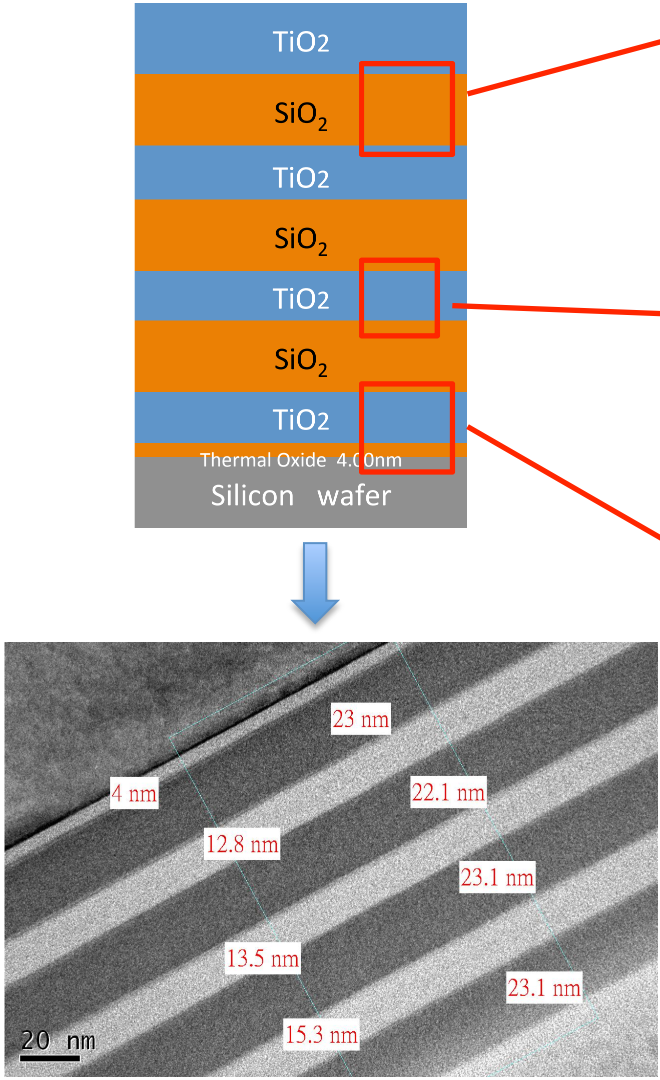
Deposition rate: TiO_2 3.40 nm/min SiO_2 7.79 nm/min

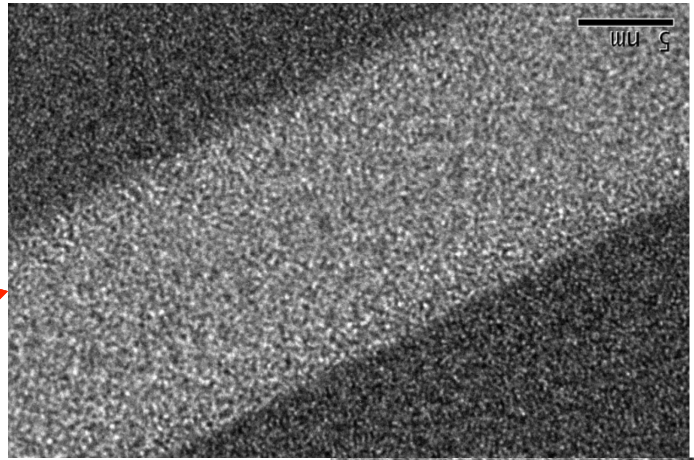
layer	Designed thickness	Deposition time	Measured thickness (TEM)	Thickness error	Measured thickness (Ellipsometry)	Thickness error
3	42.56 nm	12'31"	44.39 nm	4.3%	45.10 nm	6.0%
2	42.15 nm	5'32"	39.38 nm	6.6%	38.52 nm	8.6%
1	42.56 nm	12'31"	44.43 nm	4.4%	43.11 nm	1.3%

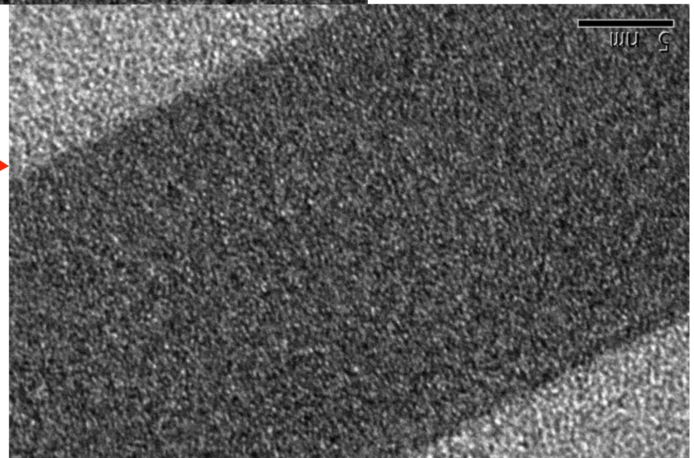
Tio_2 average thickness for layer 3 : 43.39nm Standard deviation : 0.47nm
SiO_2 average thickness for layer 2 : 39.38nm Standard deviation : 0.36nm

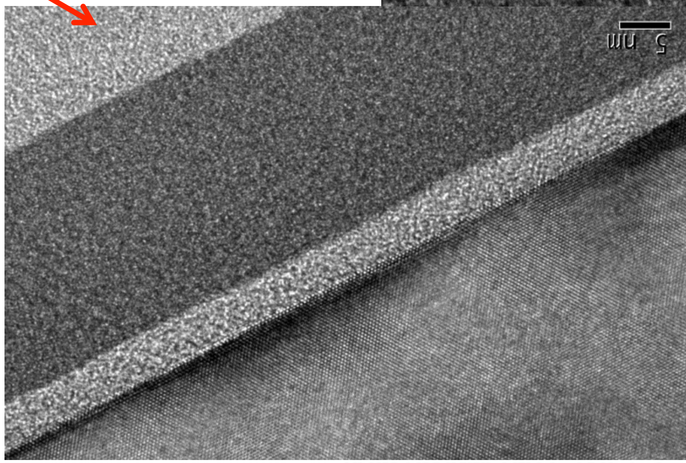
TiO_2 was over-deposited, SiO_2 was under-deposited

7-layers nano-layer TEM

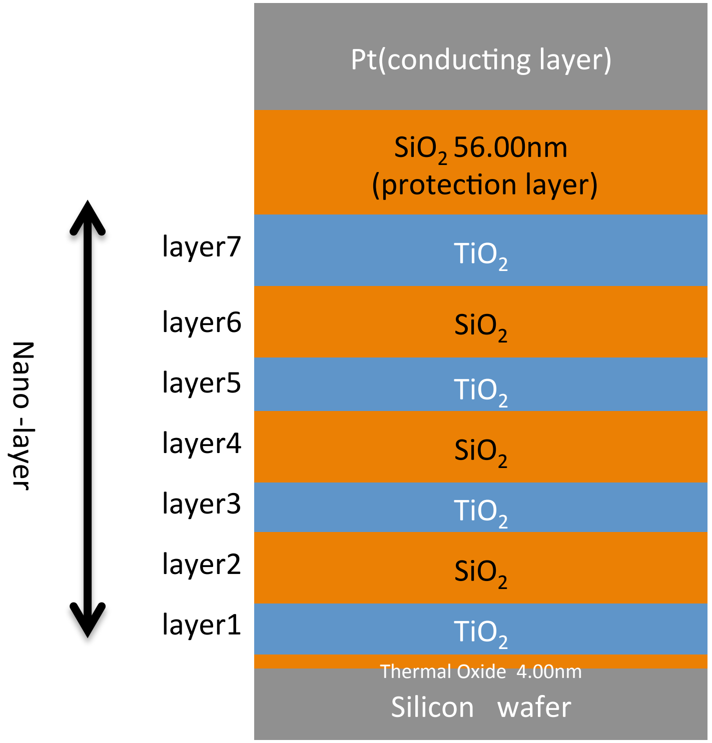








Precision of layer thickness control for the 7-layers nano-layer



Deposition rate: TiO_2 3.40 nm/min SiO_2 7.79 nm/min

layer	Designed thickness	Deposition Time	Measured thickness (TEM)	Thickness error	Measured thickness (Ellipsometry)	Thickness error
7	21.28 nm	6’16”	23.10nm	8.6%		
6	14.05 nm	2’11”	15.30nm	8.9%		
5	21.28 nm	6’16”	23.10nm	8.6%		
4	14.05 nm	1’48”	13.50nm	3.9%	Not available	
3	21.28 nm	6’16”	22.10nm	3.9%		
2	14.05 nm	1’48”	12.80nm	8.9%		
1	21.28 nm	6’16”	23.00nm	8.1%		

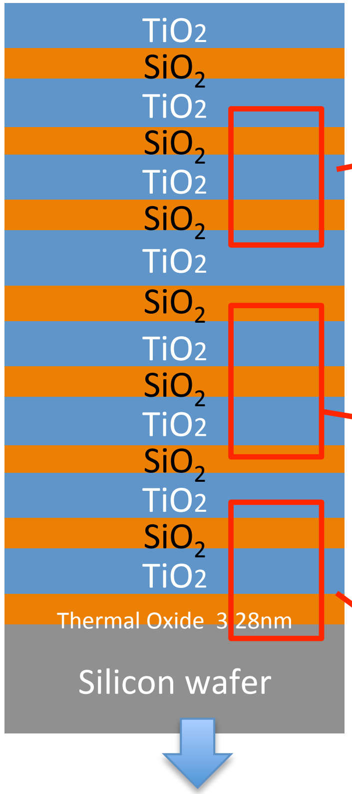
Tio$_2$ average thickness for layer 3：22.78nm Standard deviation：0.84nm
SiO_2 average thickness for layer 2：12.56nm Standard deviation：0.11nm

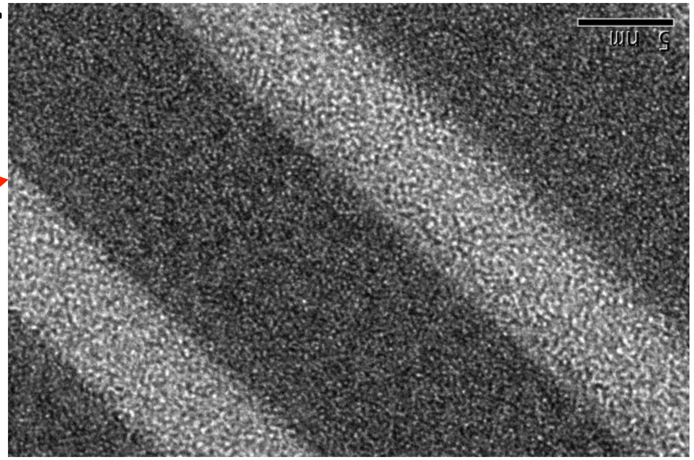
*layer 6 bad time control

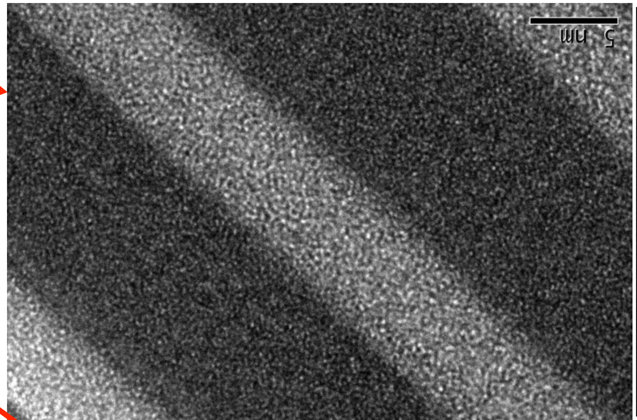
TiO2: 3.9~8.6%
SiO_2: 20.7~32.3%

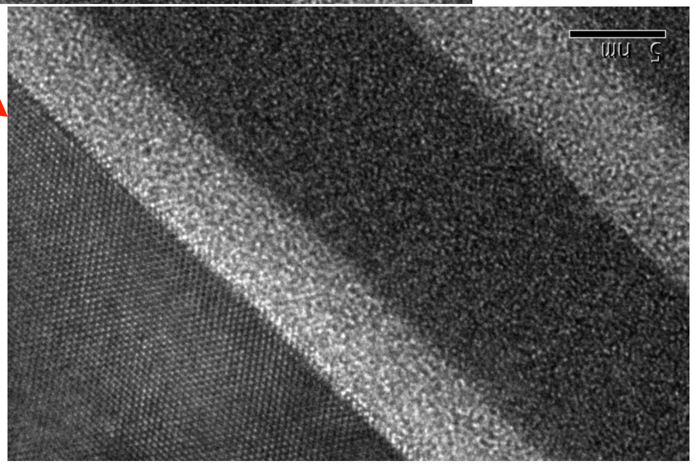
TiO_2 was over-deposited, SiO_2 was under-deposited

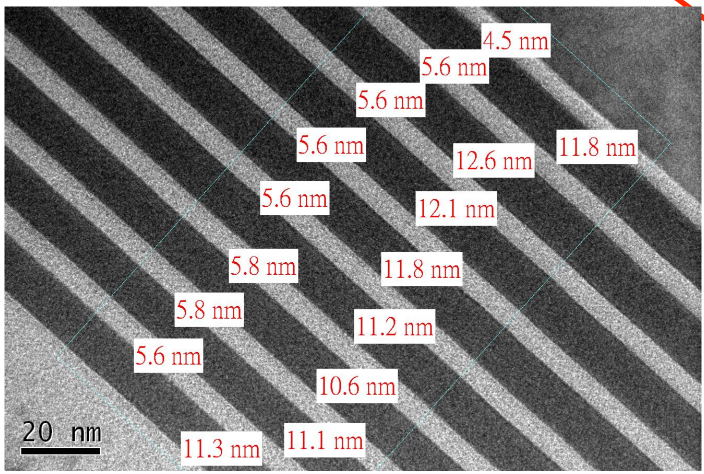
15-layers nano-layer



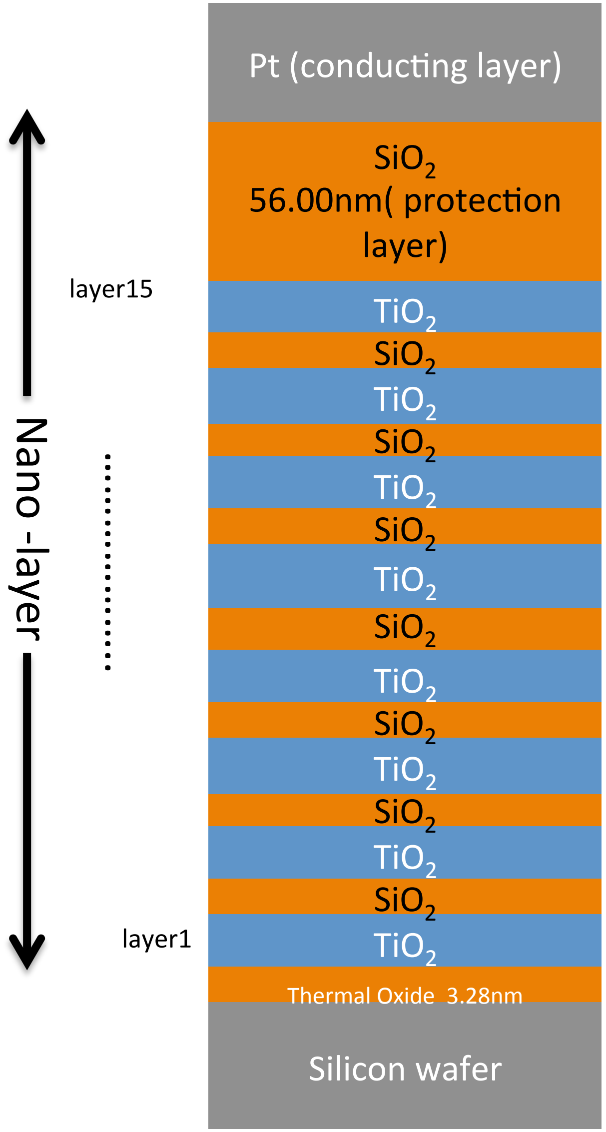








Precision of layer thickness control for the 15-layers nano-layer



Deposition rate: TiO_2 3.40 nm/min SiO_2 7.79 nm/min

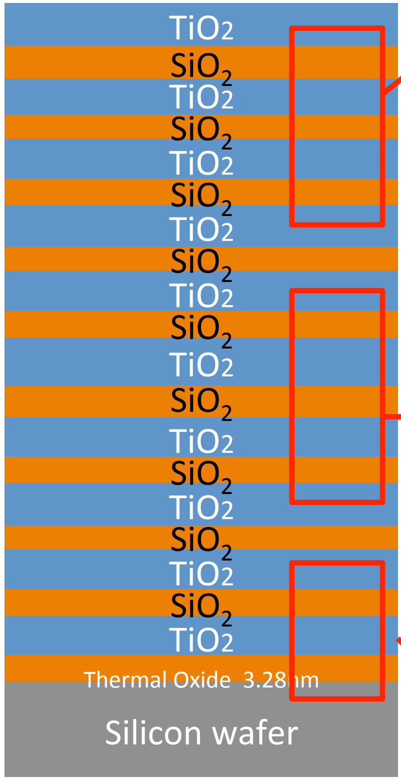
layer	Designed thickness	Deposition time	Measured thickness (TEM)	Thickness error	Measured thickness (Ellipsometry)	Thickness error
15	10.64nm	3'8"	11.30 nm	6.2%		
14	6.02nm	46"	5.60 nm	7.0%		
13	10.64nm	3'8"	11.10 nm	4.3%		
12	6.02nm	46"	5.80 nm	3.8%		
11	10.64nm	3'8"	10.60 nm	0.4%		
10	6.02nm	46"	5.80 nm	3.7%		
9	10.64nm	3'8"	11.20nm	5.3%	Not available	
8	6.02nm	46"	5.60nm	7.0%		
7	10.64nm	3'8"	11.80nm	11%		
6	6.02nm	46"	5.60 nm	7.0%		
5	10.64nm	3'8"	12.10 nm	13.7%		
4	6.02nm	46"	5.60nm	7.0%		
3	10.64nm	3'8"	12.60 nm	18.4%		
2	6.02nm	46"	5.60 nm	7.0%		
1	10.64nm	3'8"	11.80 nm	10.9%		

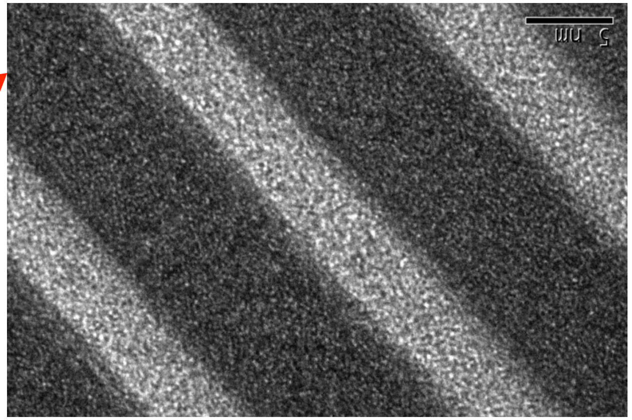
Tio_2 average thickness for layer1 : 12.06 nm Standard deviation : 0.14 nm
SiO_2 average thickness for layer 2 : 5.24 nm Standard deviation : 0.20 nm

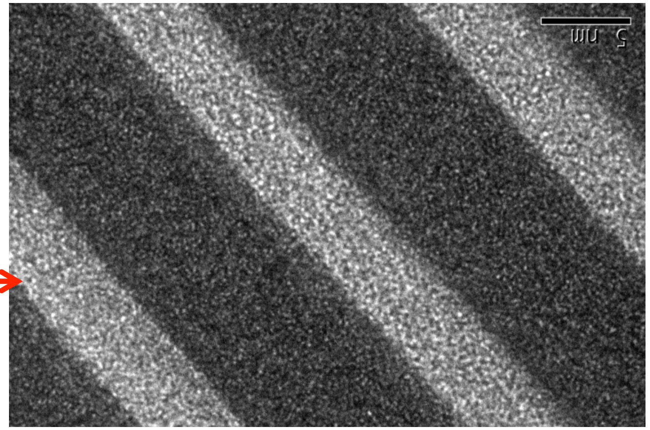
TiO2: 0.4~18.4%
SiO_2: 3.7~7.0%

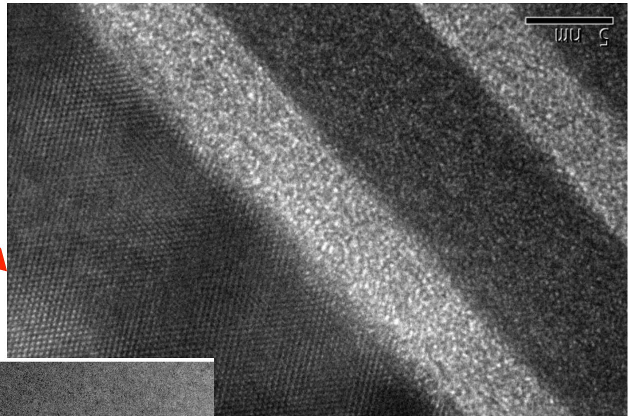
TiO2 was over-deposited, SiO2 was under-deposited

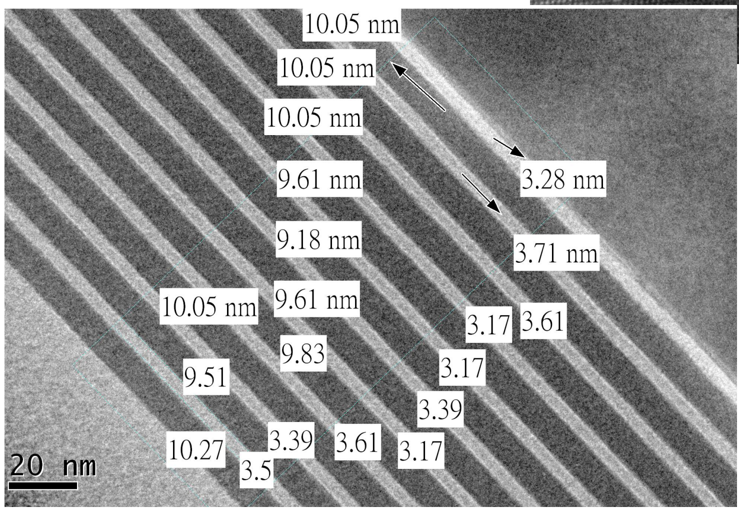
19-layers nano-layer

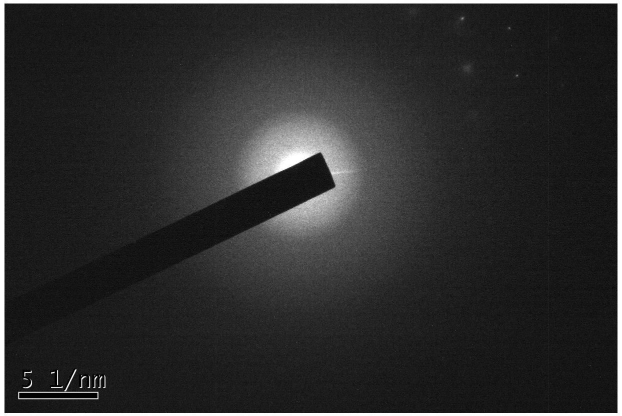




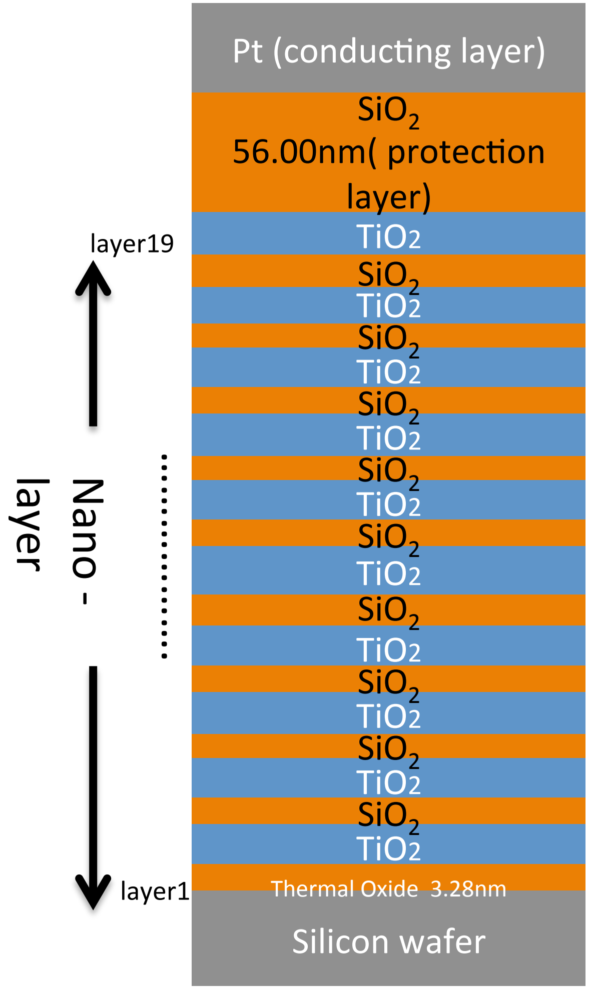








Precision of layer thickness control for the 19-layers nano-layer



Deposition rate: TiO_2 3.40 nm/min SiO_2 7.79 nm/min						
layer	Designed thickness	Deposition time	Measured thickness (TEM)	Thickness error	Measured thickness (Ellipsometry)	Thickness error
19	8.51nm	2'30"	10.27 nm	20.7%	Not available	
18	4.68nm	36"	3.5 nm	25.2%		
17	8.51nm	2'30"	9.57 nm	11.8%		
16	4.68nm	36"	3.39 nm	27.6%		
15	8.51nm	2'30"	10,05 nm	18.1%		
14	4.68nm	36"	3.46 nm	22.8%		
13	8.51nm	2'30"	9.83 nm	15.5%		
12	4.68nm	36"	3.17 nm	32.2%		
11	8.51nm	2'30"	9.61 nm	12.9%		
10	4.68nm	36"	3.39 nm	27.6%		
9	8.51nm	2'30"	9.18 nm	7.8%		
8	4.68nm	36"	3.17 nm	32.3%		
7	8.51nm	2'30"	9.61nm	12.9%		
6	4.68nm	36"	3.17 nm	32.3%		
5	8.51nm	2'30"	10.05 nm	18.1%		
4	4.68nm	36"	3.61 nm	22.9%		
3	8.51nm	2'30"	10.05 nm	18.1%		
2	4.68nm	36"	3.71 nm	20.7%		
1	8.51nm	2'30"	10.05 nm	18.2%		
Tio_2 average thickness for layer 17 : 9.57nm Standard deviation : 0.14nm SiO_2 average thickness for layer 13 : 3.46nm Standard deviation : 0.11nm						

TiO2: 11.8~20.7%
SiO_2: 20.7~32.3%

TiO_2 was over-deposited, SiO_2 was under-deposited, larger deviation ~20%, for thickness control

Conclusion

Measurements

- Thick clamping pad significantly reduces errors of loss angle measurement.
- With the clamping aligner, the decay time error from re-clamping is comparable or smaller than that from the background noise.
- We have developed capability to produce low loss angle silicon and fused silica substrates suitable to measure mechanical losses for our future mirror coating research.

Nano-layer coating

- Nanolayer coating is in progress, thinner nanolayer needs better thickness control.
- Measurement of the loss angle for the nanolayer coatings with different layer designs are on going.

Thanks for your attention