Developing the TiO_2/SiO_2 mixed films for low optical loss dielectric mirror --- A retrospect

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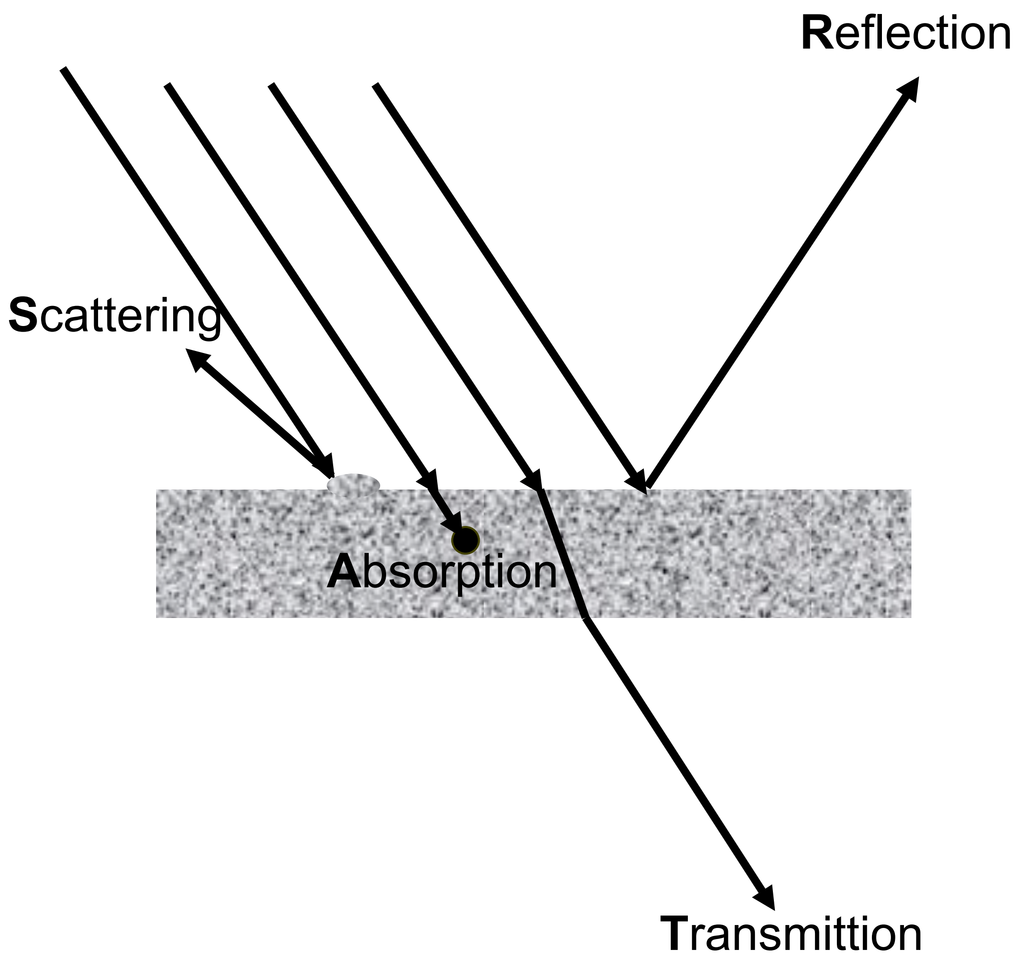
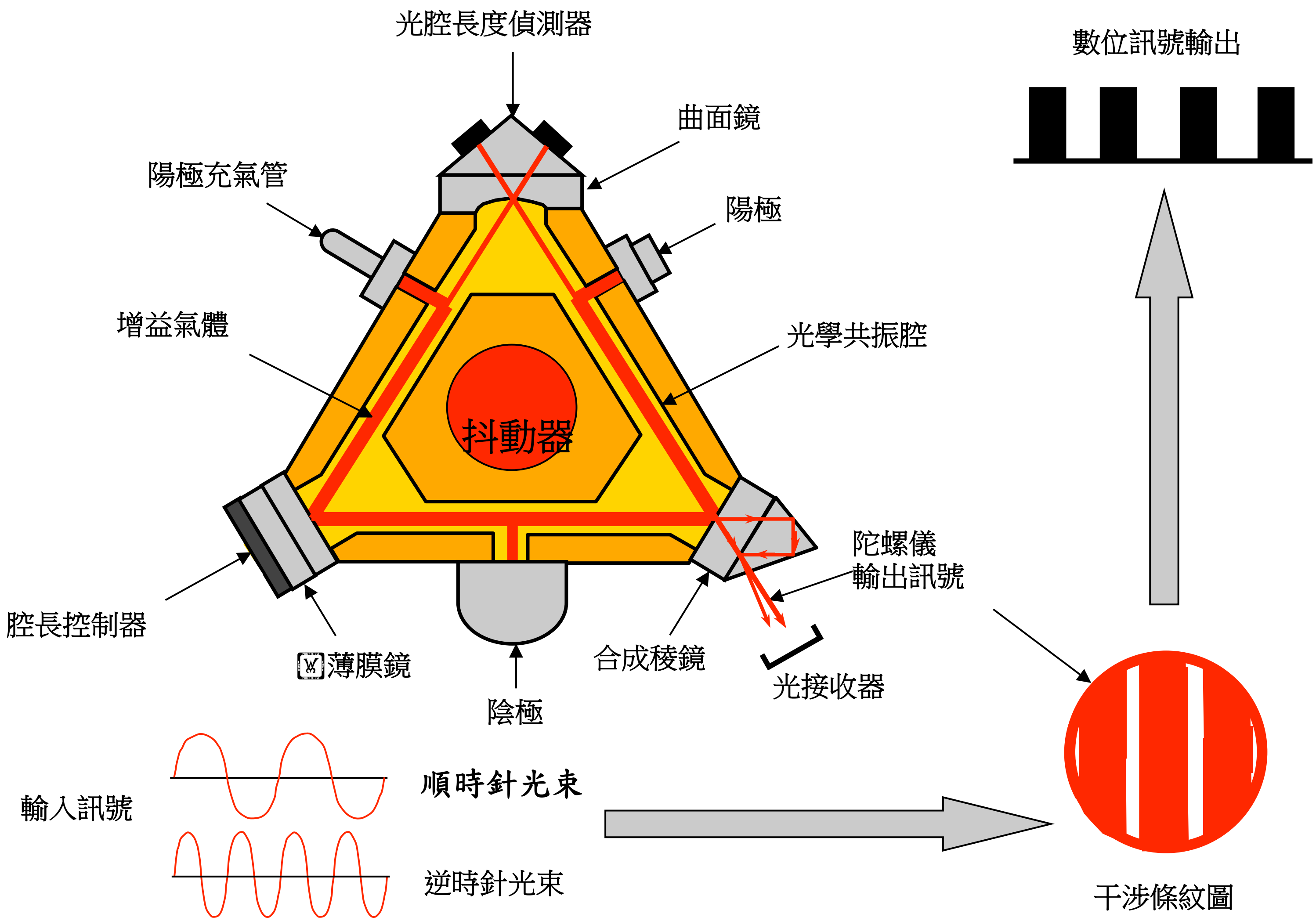


1=R+T+S+A

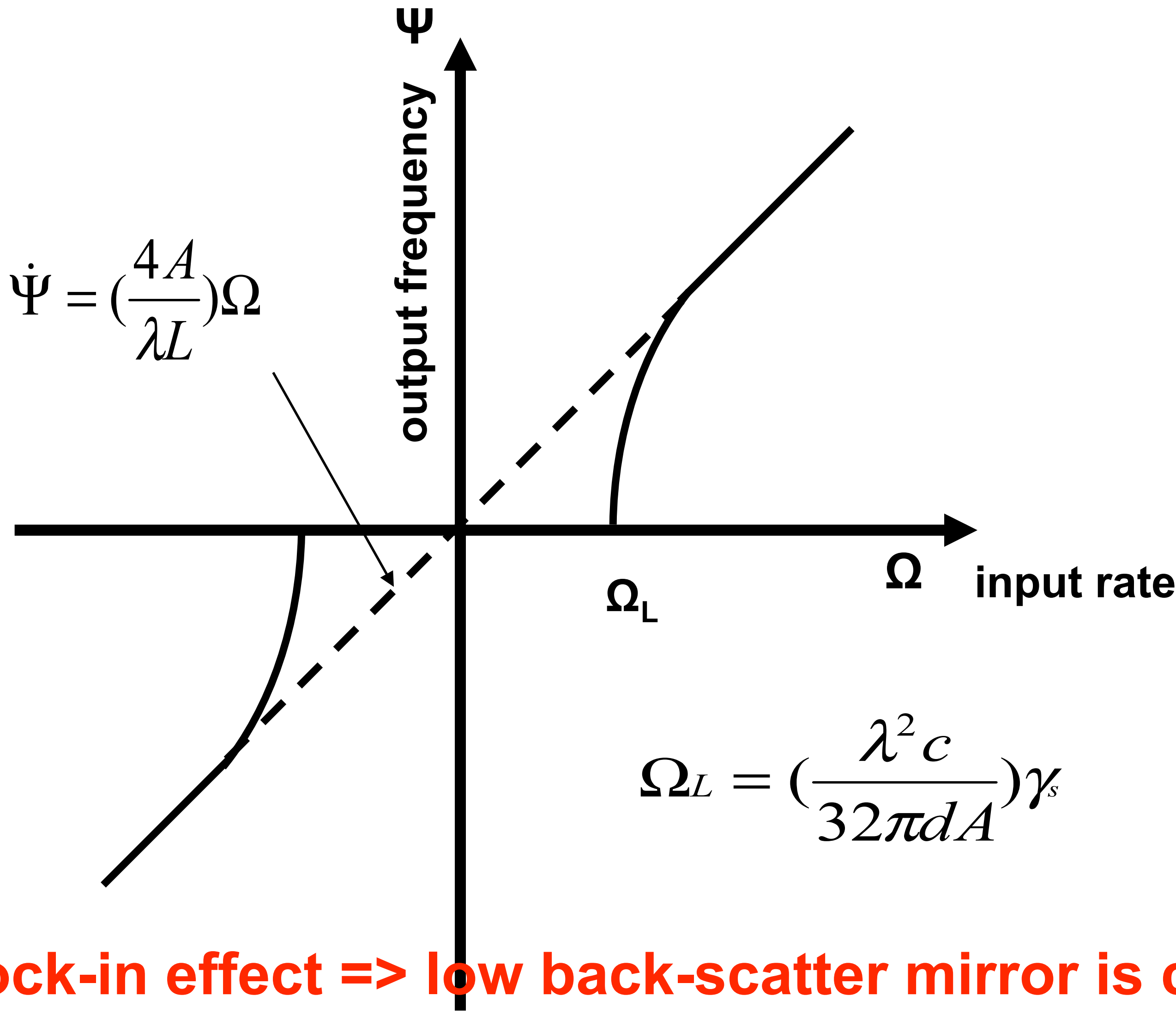
Total Loss ≡ 1-R-T = S+A

RING LASER GYROSCOPE



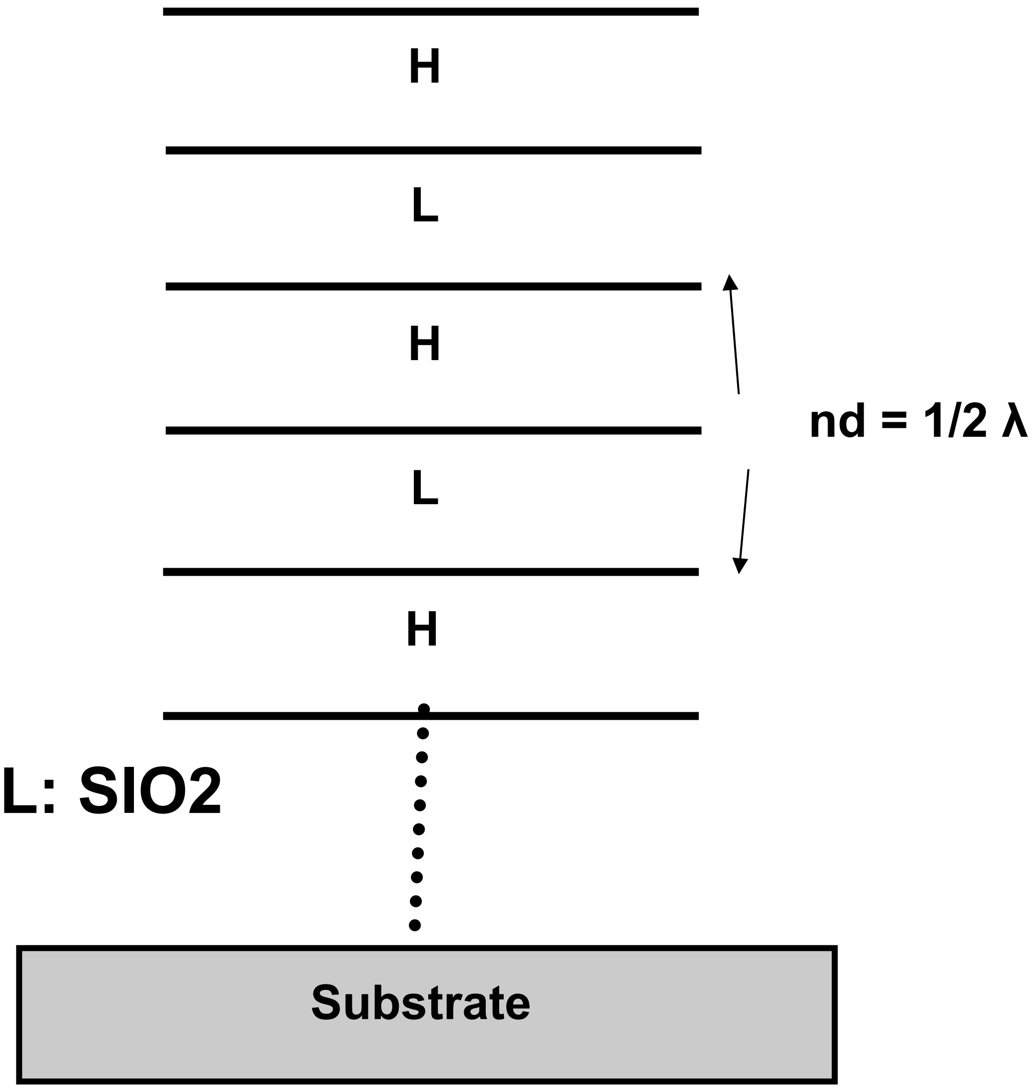
High Q resonator ➔ low optical loss for the mirrors

LOCK-IN EFFECT IN RLG



To avoid lock-in effect => low back-scatter mirror is critical

Multi-layer dielectric mirror



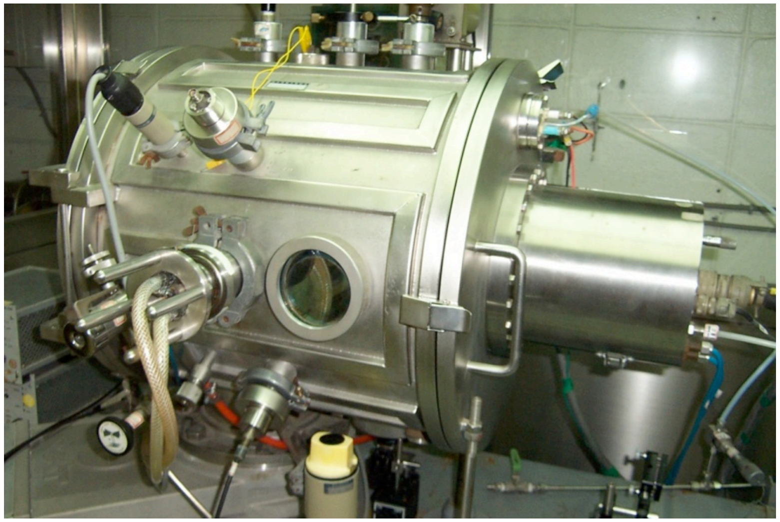
H: TIO_2, TA_2O_5 L: SIO_2

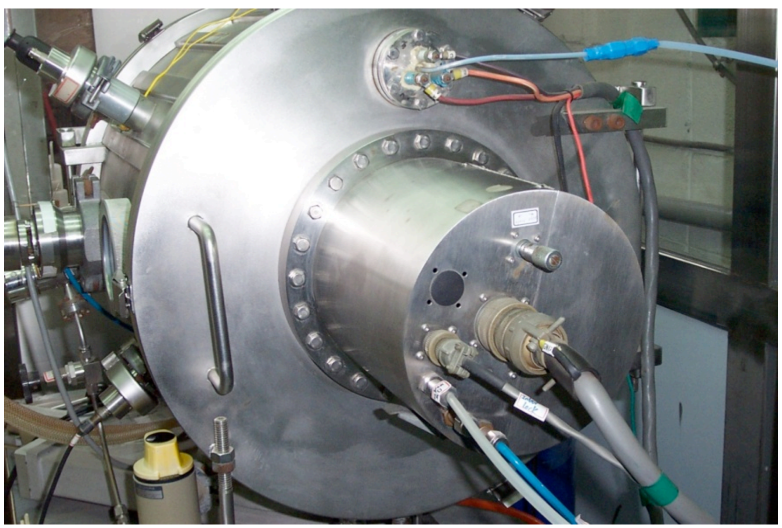
Coating Apparatus

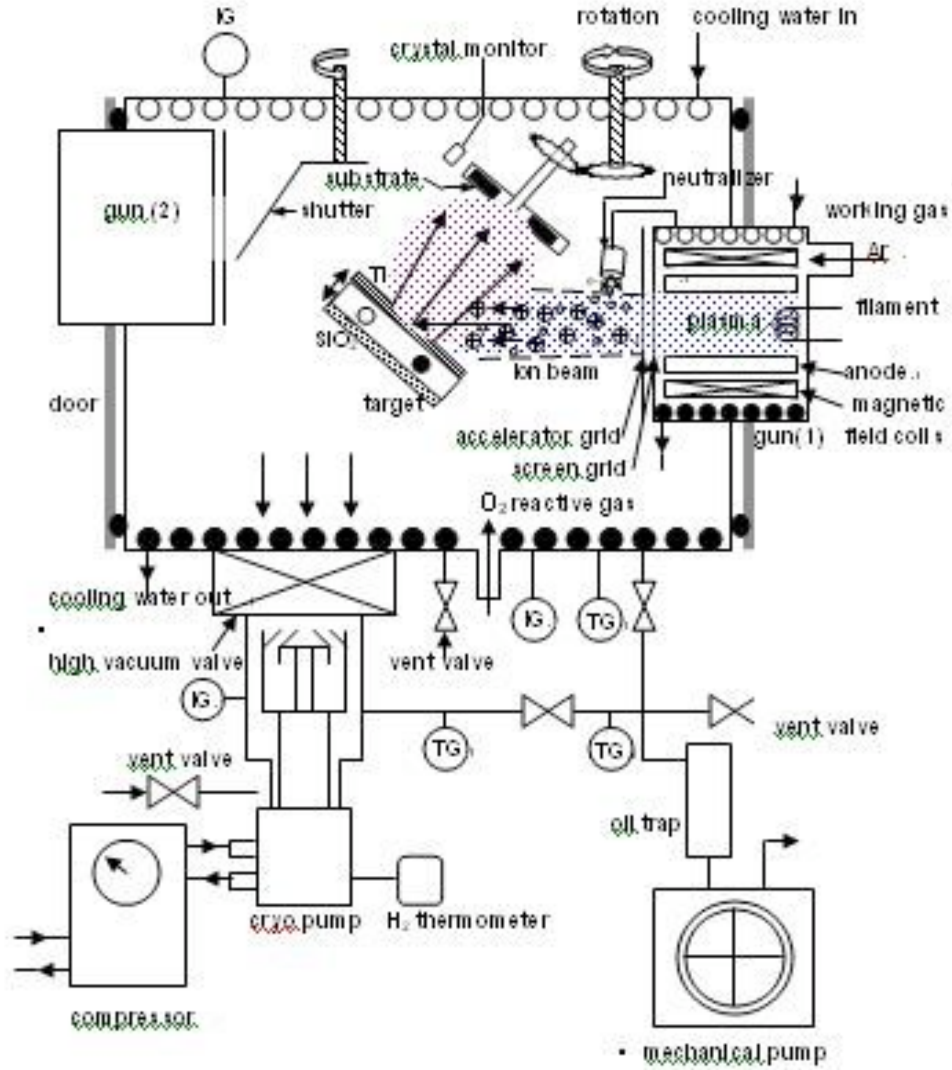
Ion Beam Sputtering

--- dense film

--- higher refractive index

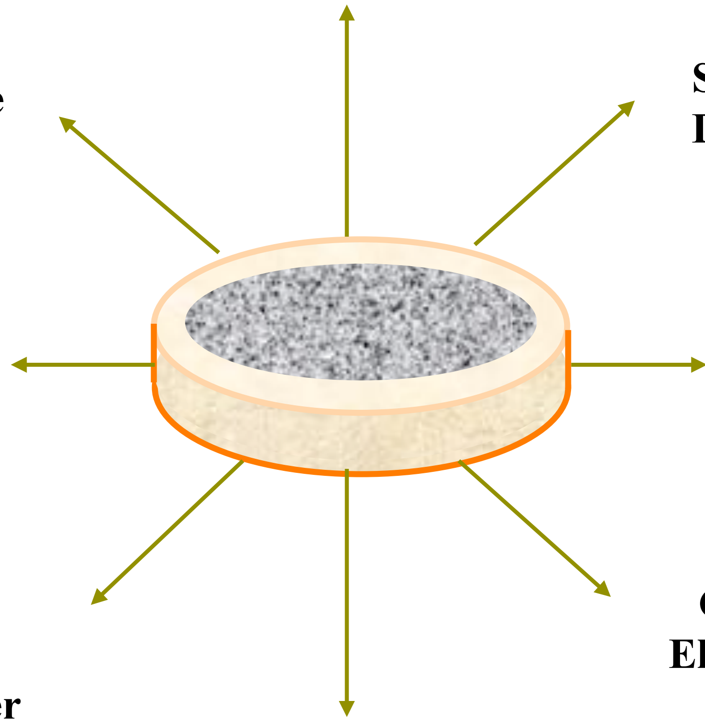






IG
rotation
cooling water in
crystal monitor
substrate
neutralizer
gun (2)
shutter
working gas
Ar
Ti
filament
SiO2
plasma
ion beam
anode
target
magnetic
field coils
door
gun(1)
accelerator grid
screen grid
O2 reactive gas
cooling water out
IG
TG
high vacuum valve
vent valve
IG
TG
TG
vent valve
vent valve
oil trap
cryo pump
H2 thermometer
compressor
mechanical pump

Instrumental analysis for mirror characterization



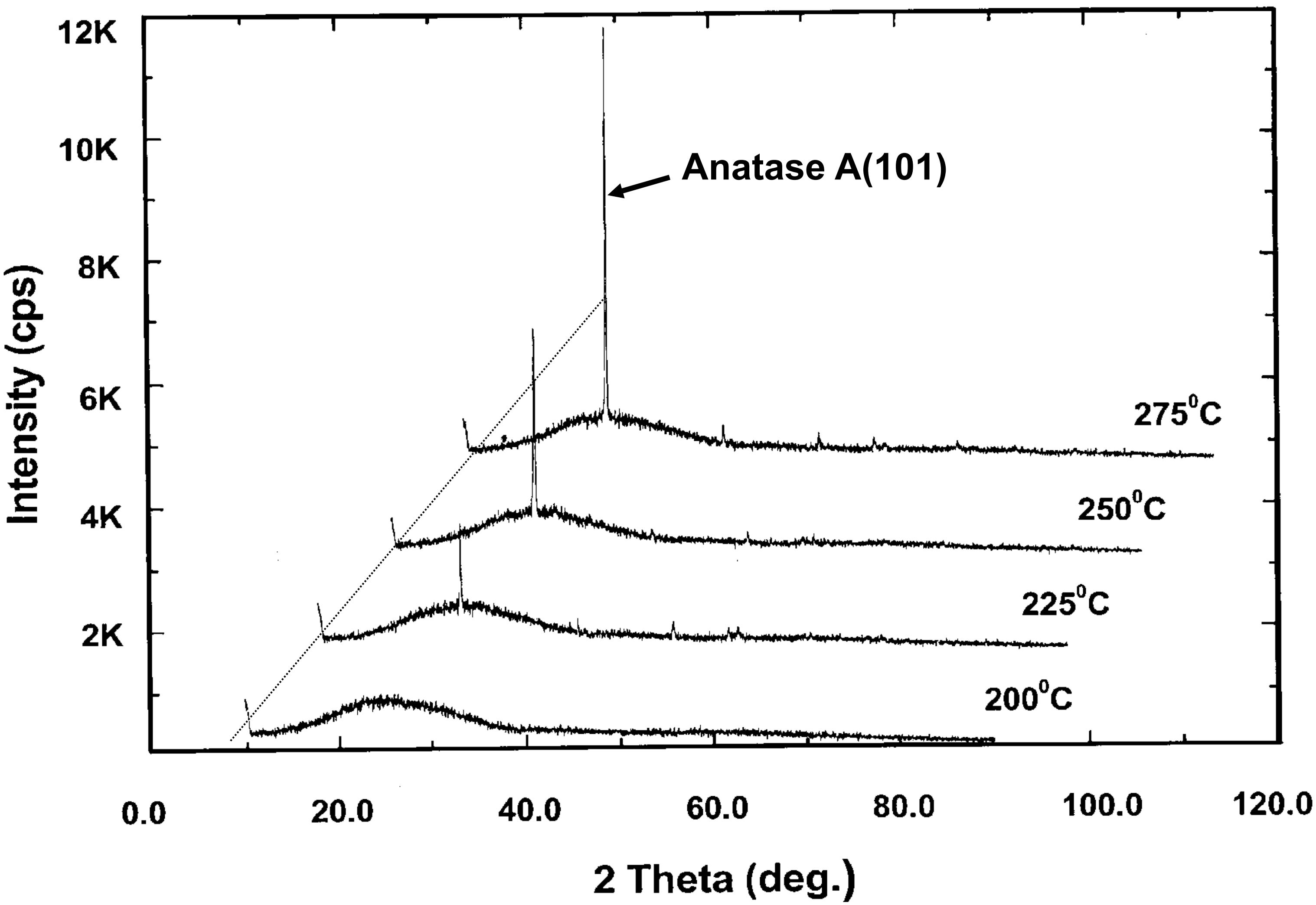
Single TiO_2 film

Annealing effect on ion-beam-sputtered titanium dioxide film

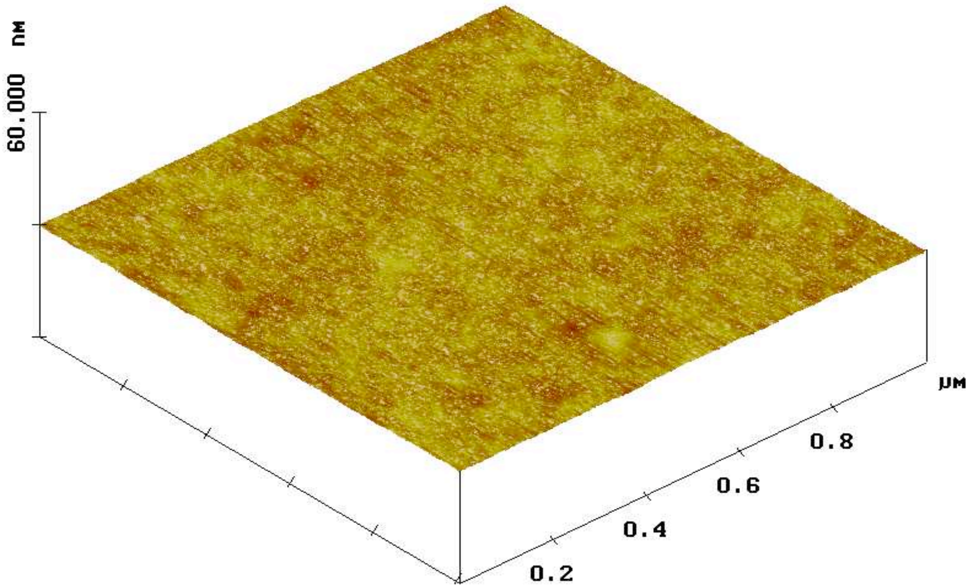
Wen-Hsiang Wang and Shiuh Chao

Department of Electrical Engineering, National Tsing Hua University, Hsin-Chu, Taiwan

September 15, 1998 / Vol. 23, No. 18 / OPTICS LETTERS 1417

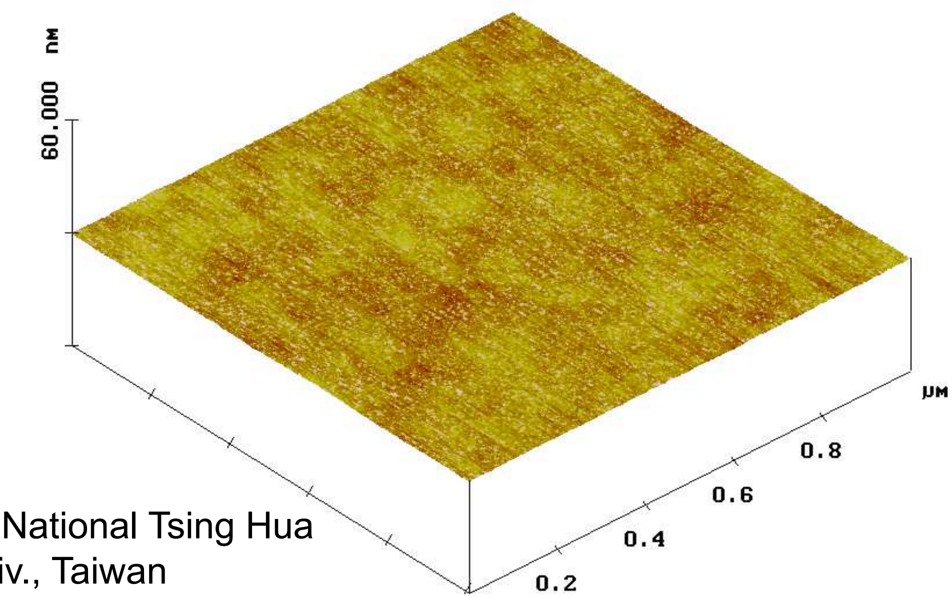


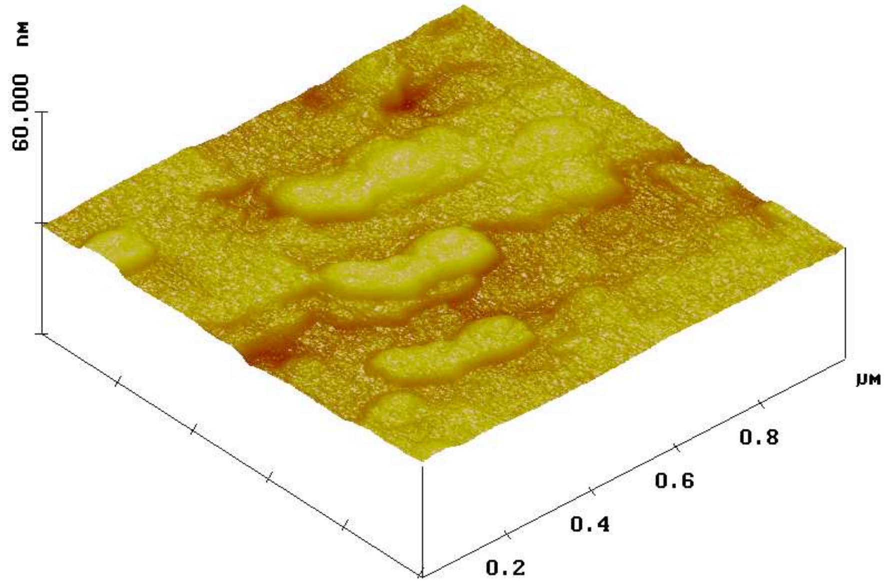
12K
10K
8K
6K
4K
2K
Intensity (cps)
Anatase A(101)
275⁰C
250⁰C
225⁰C
200⁰C
0.0
20.0
40.0
60.0
80.0
100.0
120.0
2 Theta (deg.)



(A) as-deposited

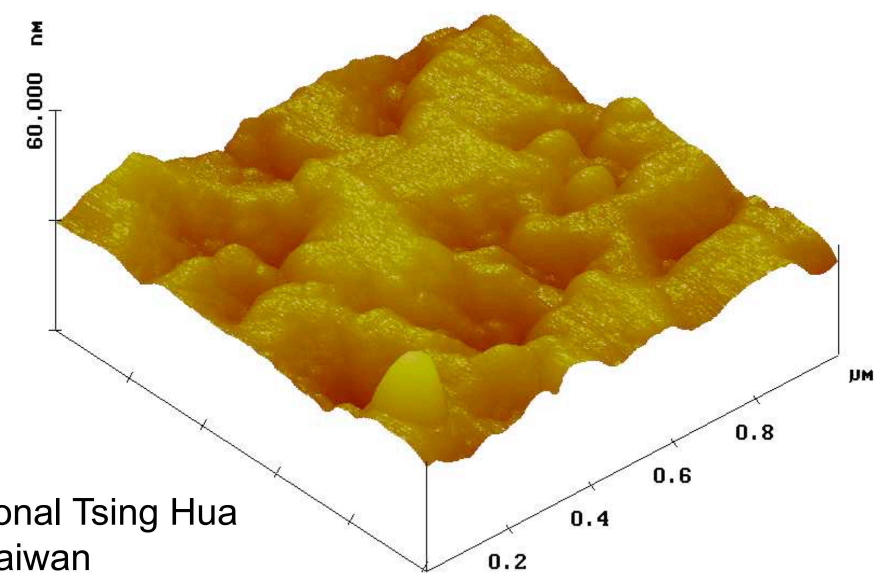
(B) annealed 200 ^oC





(C) annealed 225 °C

(D) annealed 300°C



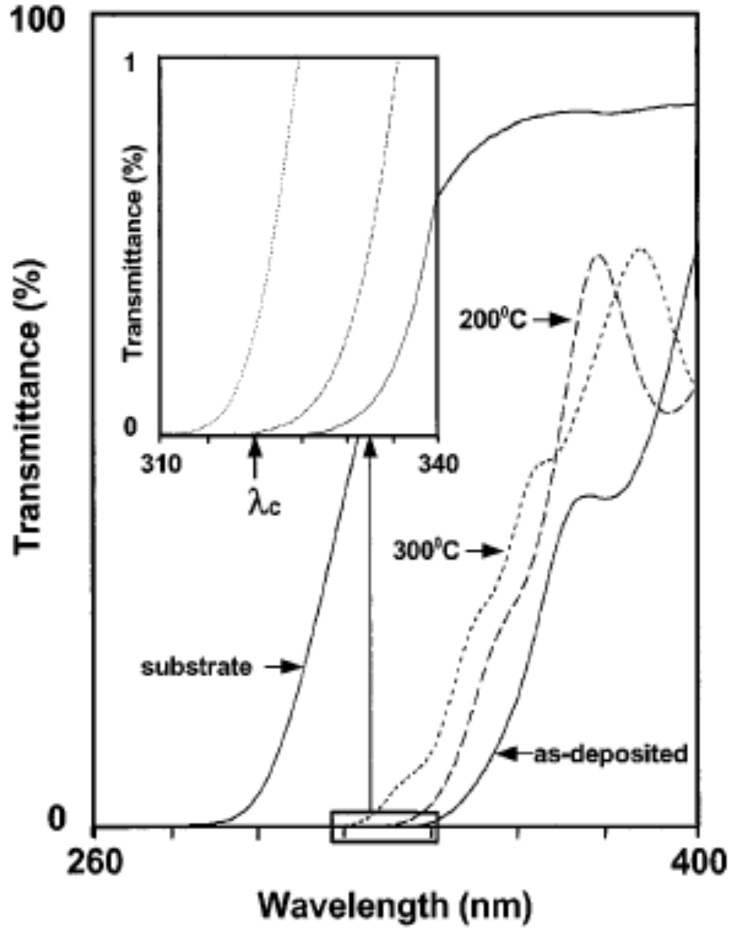
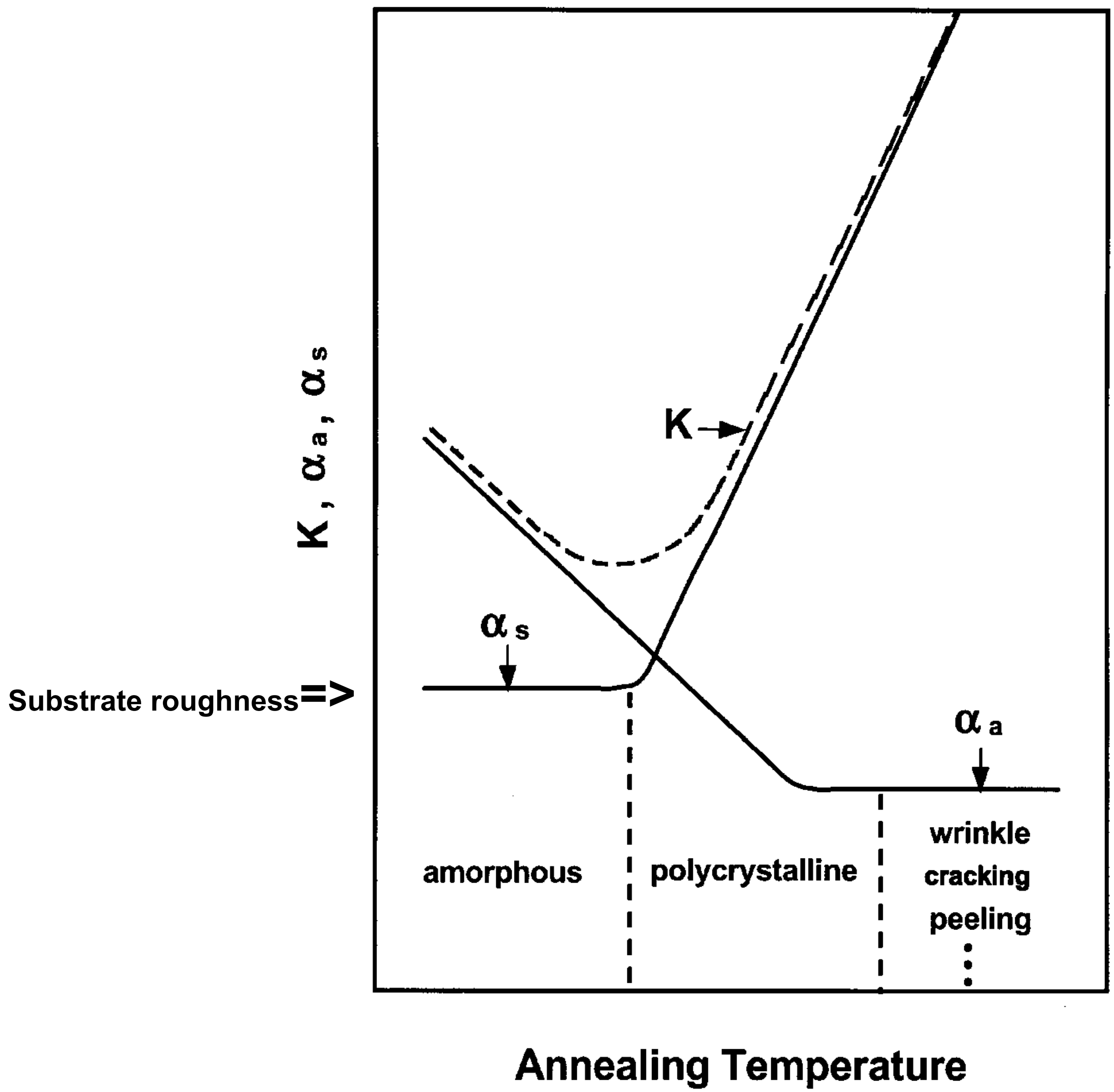


Fig. 2. Transmission spectra in the short-wavelength region for the as-deposited sample and the samples annealed at 200 and 300 °C.

annealing temp.(^{0}C)	632.8nm n	632.8nm k	550.0nm n	550.0nm k	450.0nm n	450.0nm k	I/I_0 (%)	λc (nm)	rms surface roughness(Å)
before annealing	2.50	0.0001	2.54	0.0003	2.66	0.0013	0	327.6	1.2
150	2.50	$<10^{-4}$	2.54	0.0002	2.66	0.0012	0	325.0	1.5
200	2.50	$<10^{-4}$	2.54	0.0001	2.66	0.0010	0	322.4	1.4
225	2.49	0.0001	2.53	0.0002	2.65	0.0011	20	319.2	8.4
250	2.48	0.0004	2.52	0.0007	2.64	0.0023	65	316.5	13.4
275	2.48	0.0007	2.52	0.0012	2.64	0.0040	100	313.8	17.2
300	2.48	0.0015	2.52	0.0025	2.64	0.0052	100	313.5	32.6
350	2.48	0.0018	2.52	0.0027	2.64	0.0062	100	313.5	33.0



(1) GOOD POLISHING=> REDUCE SUBSTRATE ROUGHNESS

(2) RAISE UP THE CRYSTALLIZATION TEMPERATURE OF THE FILM

Single TiO_2-SiO_2 mixed film

Characteristics of ion-beam-sputtered high refractive-index TiO2-SiO2 mixed films

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Vol. 16, No. 6/June 1999/J. Opt. Soc. Am. A 1477

5% SiO_2

annealing temp.(^{0}C)	632.8nm n	632.8nm k	550.0nm n	550.0nm k	450.0nm n	450.0nm k	I/I_0 (%)	λc (nm)	rms surface roughness(Å)
before annealing	2.47	$<10^{-4}$	2.52	0.0002	2.64	0.0012	0	325.0	1.6
150	2.47	$<10^{-4}$	2.52	0.0002	2.64	0.0011	0	321.8	1.5
200	2.47	$<10^{-4}$	2.52	0.0001	2.64	0.0010	0	319.8	1.4
250	2.47	$<10^{-4}$	2.52	0.0001	2.63	0.0010	0	315.3	1.5
275	2.46	0.0006	2.51	0.0011	2.63	0.0033	57	313.5	10.5
300	2.46	0.0009	2.51	0.0015	2.63	0.0038	100	313.2	16.8

表(二) 二氧化矽組成比例為5%的二氧化鈦光學混合膜在不同退火溫度下之折射係數(n)、消光係數(k)、銳鈦礦相對X光結晶強度(I/I_0)、截止波長(λc)及表面粗糙度均方根值

λc : cut-off wavelength

9% SiO_2

annealing temp.(^{0}C)	632.8nm		550.0nm		450.0nm		I/I_0 (%)	λc (nm)	rms surface roughness(Å)
	n	k	n	k	n	k			
before annealing	2.42	$<10^{-4}$	2.46	$<10^{-4}$	2.57	0.0010	0	319.7	1.3
200	2.42	$<10^{-4}$	2.45	$<10^{-4}$	2.56	0.0008	0	315.6	1.4
275	2.40	$<10^{-4}$	2.44	$<10^{-4}$	2.54	0.0006	0	312.5	1.5
300	2.38	0.0002	2.43	0.0004	2.53	0.0015	4	311.9	3.2
325	2.37	0.0009	2.43	0.0015	2.52	0.0025	45	310.9	8.5
350	2.36	0.0025	2.42	0.0029	2.51	0.0034	85	310.6	16.0
400	2.36	0.0032	2.41	0.0036	2.50	0.0044	100	310.2	24.4

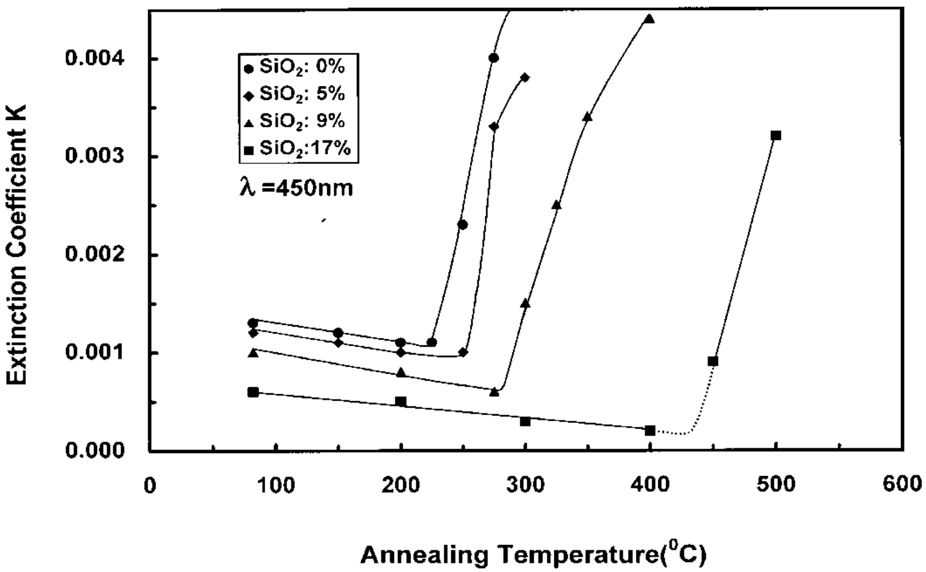
-99-

表(三) 二氧化矽組成比例為 9%的二氧化鈦光學混合膜在不同退火溫度下之折射係數(n)、消光係數(k)、銳鈦礦相對 X 光結晶強度(I/I_0)、截止波長(λc)及表面粗糙度均方根值

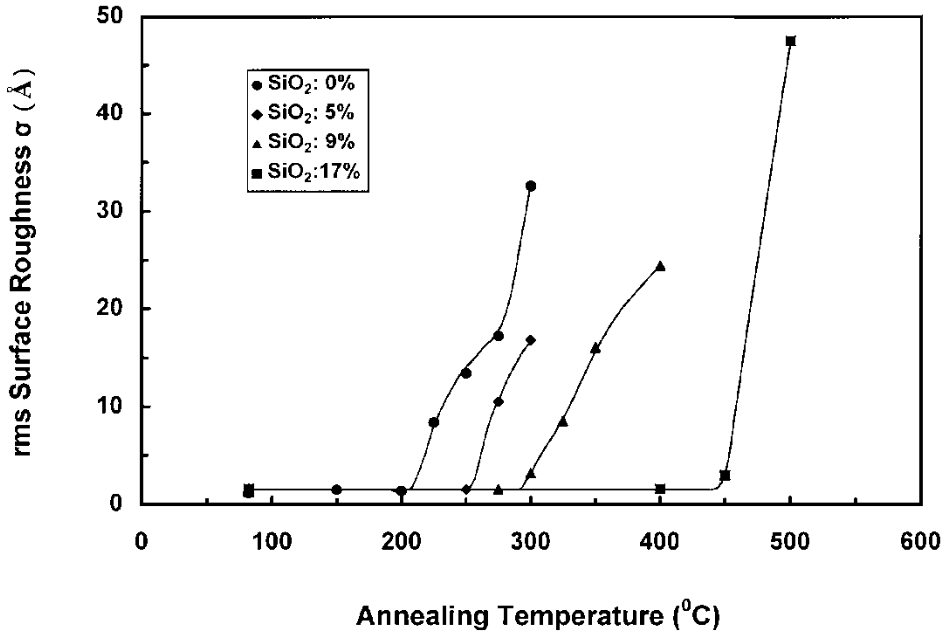
17% SiO_2

annealing temp.(^0C)	632.8nm		550.0nm		450.0nm		I/I_0 (%)	λc (nm)	rms surface roughness(Å)
	n	k	n	k	n	k			
before annealing	2.33	$<10^{-4}$	2.37	$<10^{-4}$	2.48	0.0006	0	314.1	1.6
200	2.32	$<10^{-4}$	2.36	$<10^{-4}$	2.47	0.0005	0	309.6	1.7
300	2.30	$<10^{-4}$	2.34	$<10^{-4}$	2.44	0.0003	0	307.6	1.7
400	2.28	$<10^{-4}$	2.32	$<10^{-4}$	2.41	0.0002	0	304.1	1.6
450	2.25	0.0004	2.29	0.0005	2.36	0.0009	0	303.4	3.0
500	2.21	0.0010	2.24	0.0012	2.32	0.0032	0	-	47.5
550	-	-	-	-	-	-	10	-	-

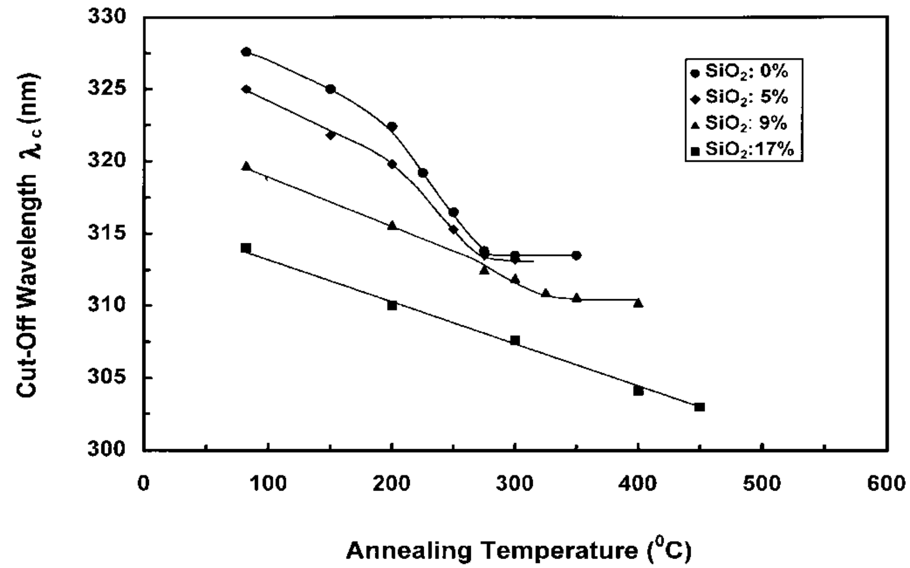
表(四) 二氧化矽組成比例為17%的二氧化鈦光學混合膜在不同退火溫度下之折射係數(n)、消光係數(k)、銳鈦礦相對X光結晶強度(I/I_0)、截止波長(λc)及表面粗糙度均方根值



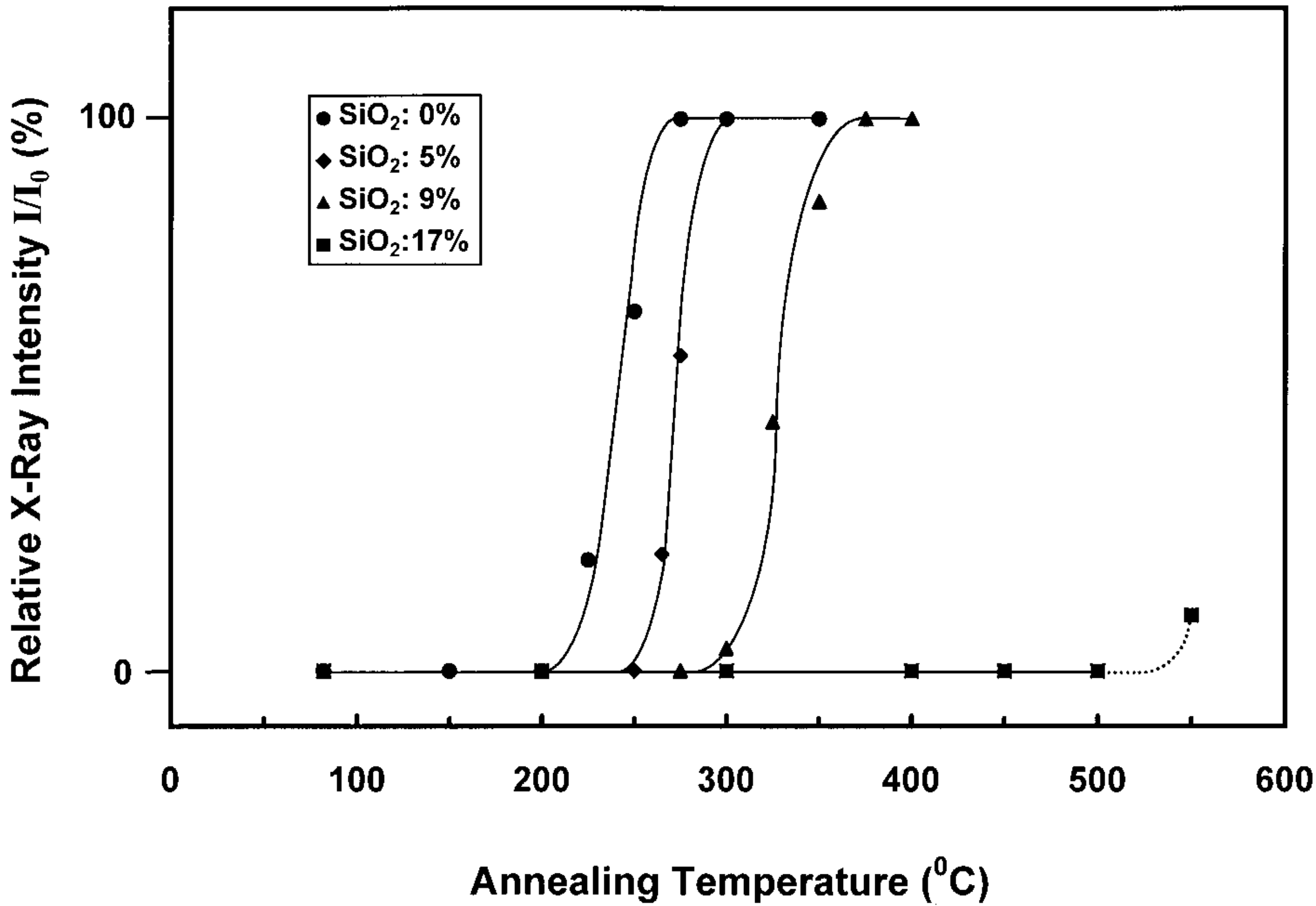
Extinction Coefficient K
0.004
0.003
0.002
0.001
0.000
SiO₂: 0%
SiO₂: 5%
SiO₂: 9%
SiO₂:17%
λ =450nm
0
100
200
300
400
500
600
Annealing Temperature(°C)



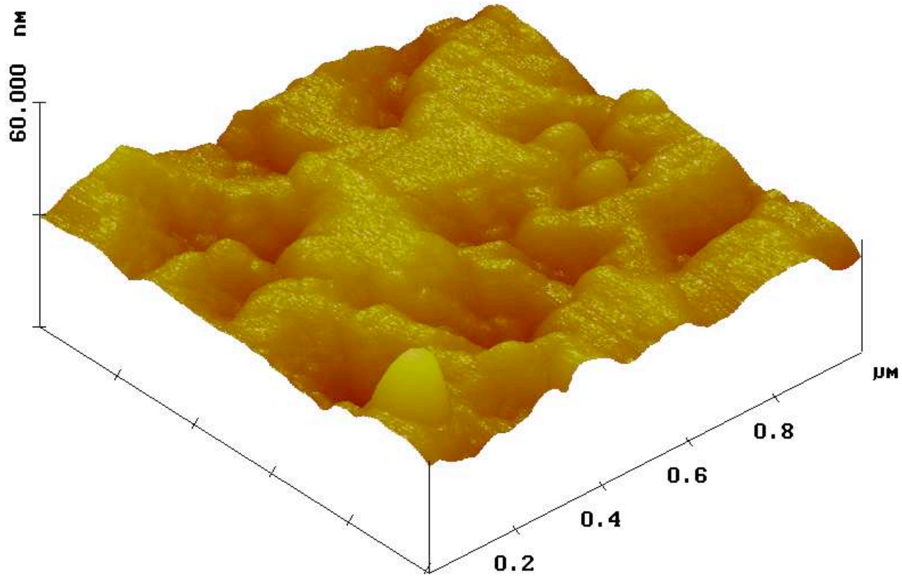
rms Surface Roughness σ (Å)
50
40
30
20
10
0
SiO₂: 0%
SiO₂: 5%
SiO₂: 9%
SiO₂:17%
0
100
200
300
400
500
600
Annealing Temperature (°C)



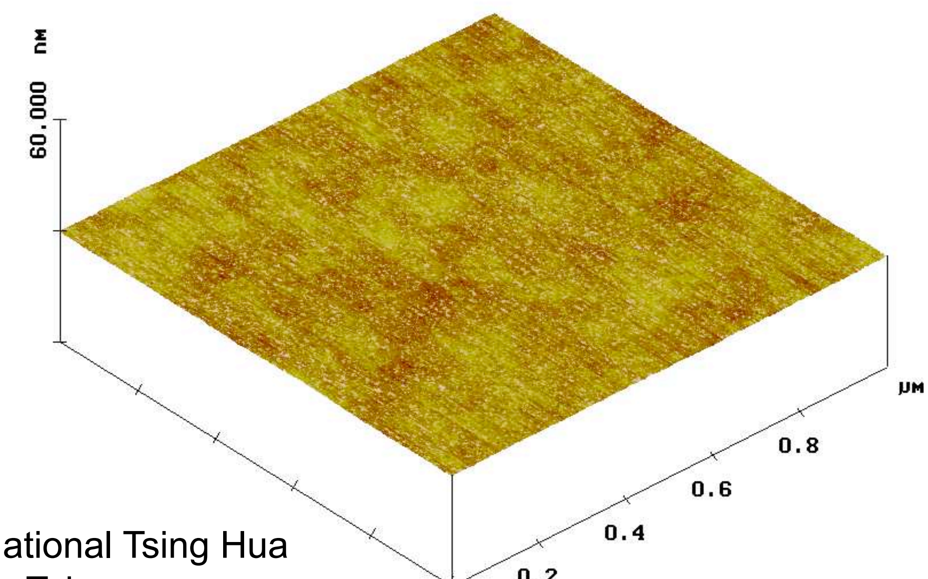
Cut-Off Wavelength λc (nm)
330
325
320
315
310
305
300
SiO₂: 0%
SiO₂: 5%
SiO₂: 9%
SiO₂:17%
0
100
200
300
400
500
600
Annealing Temperature (°C)



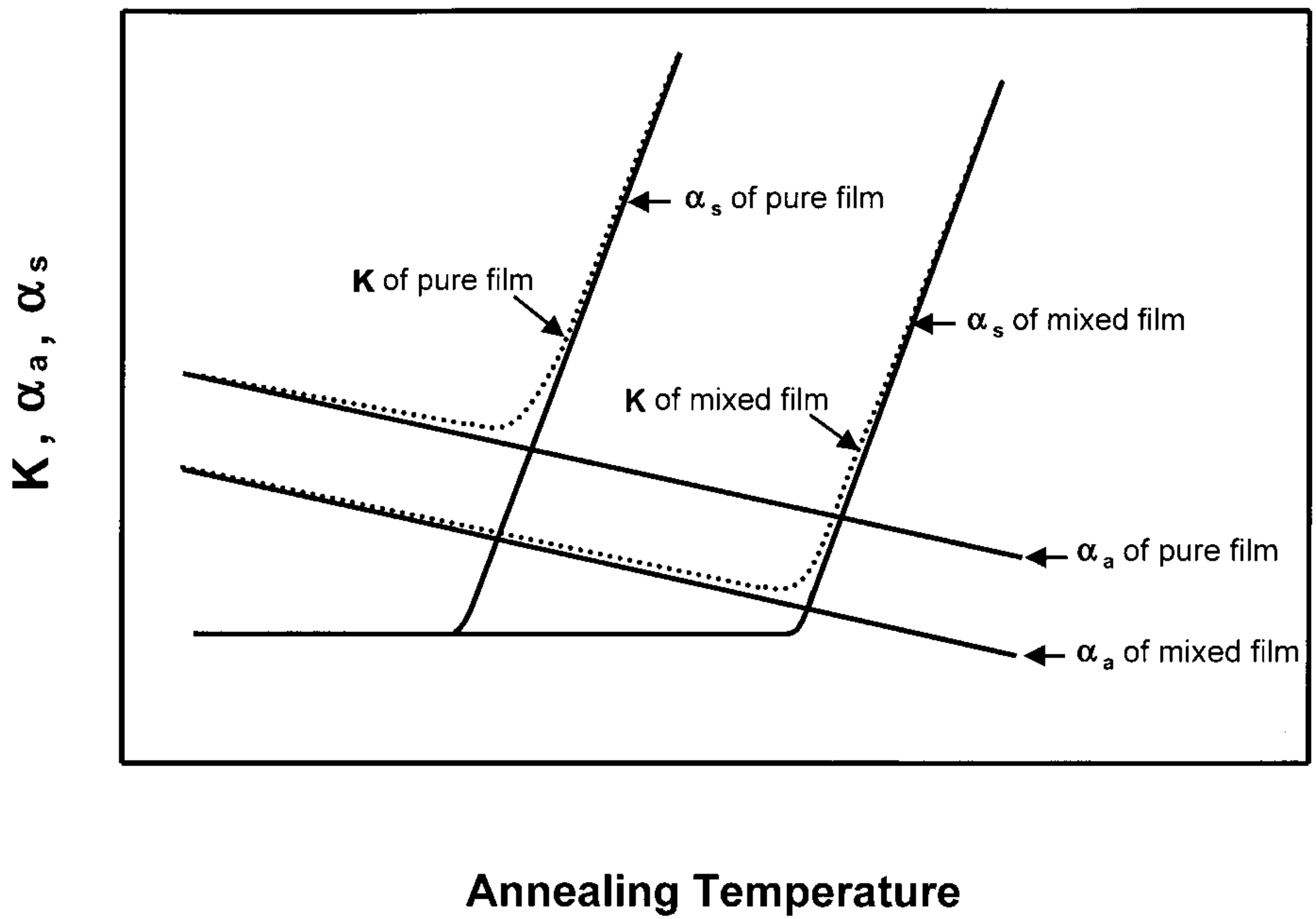
SiO2: 0%
SiO2: 5%
SiO2: 9%
SiO2:17%
Relative X-Ray Intensity I/I0 (%)
100
0
0
100
200
300
400
500
600
Annealing Temperature (0C)



(A) SiO_2: 0% annealed 300^{o}C

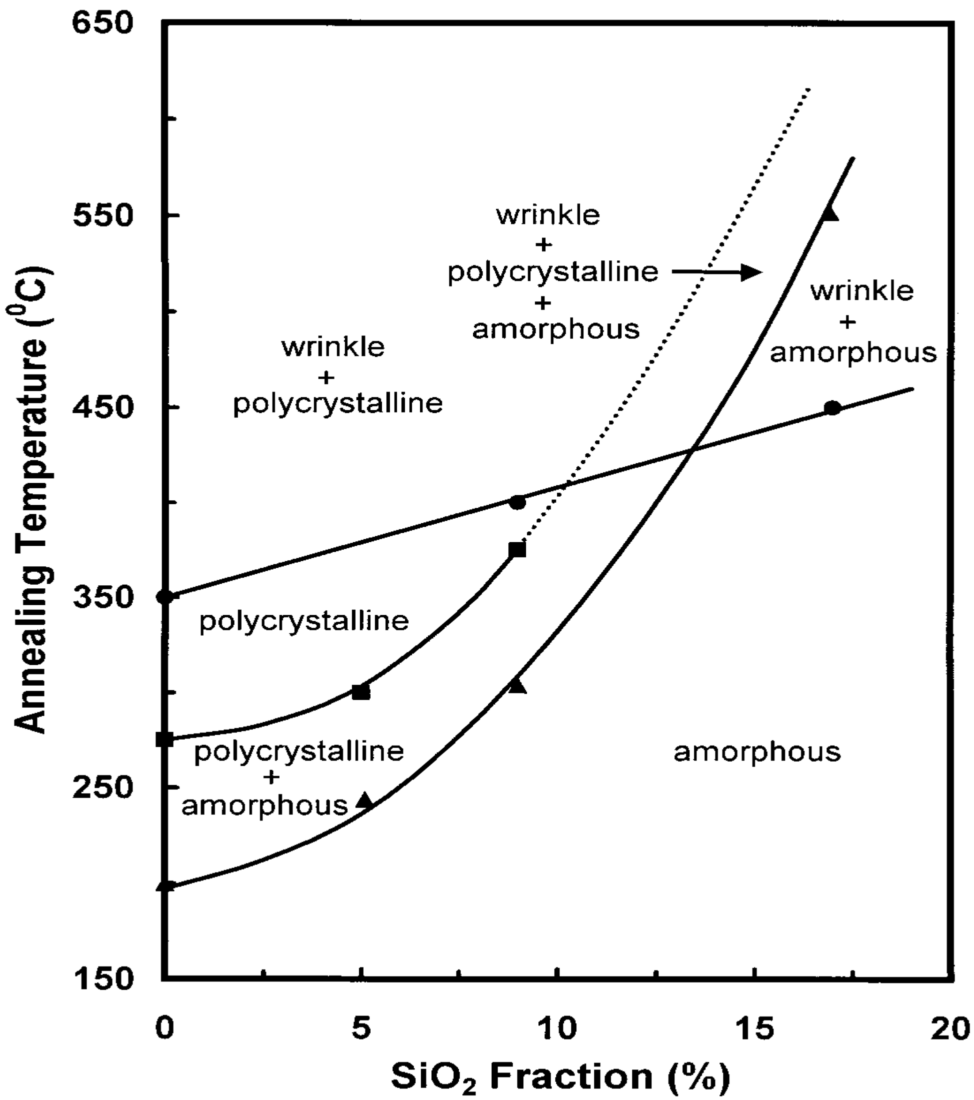


(B) SiO_2: 17% annealed 300^{o}C



K , αa , αs
αs of pure film
K of pure film
αs of mixed film
K of mixed film
αa of pure film
αa of mixed film
Annealing Temperature

“phase diagram” of the mixed film

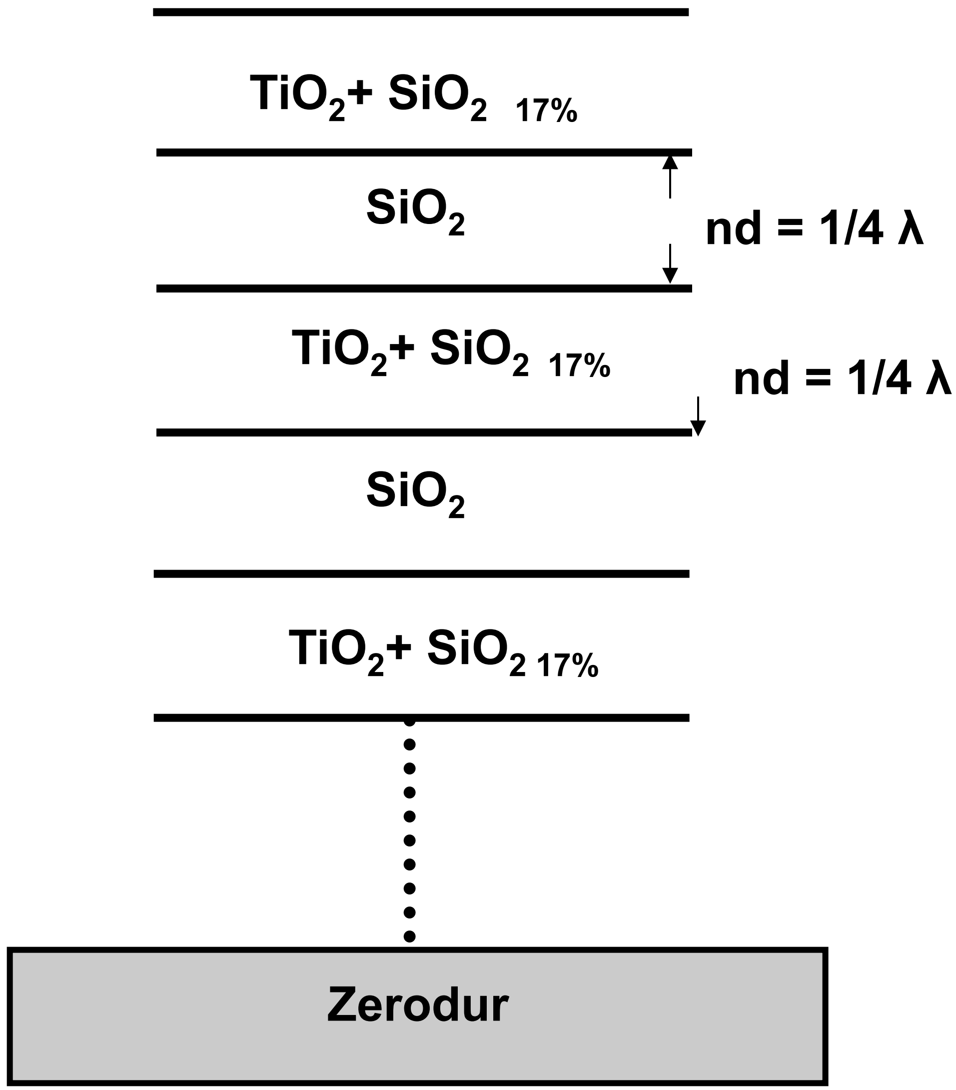


Multi-layer dielectric mirror with the mixed film

Low-loss dielectric mirror with
ion-beam-sputtered TiO_2–SiO_2 mixed films

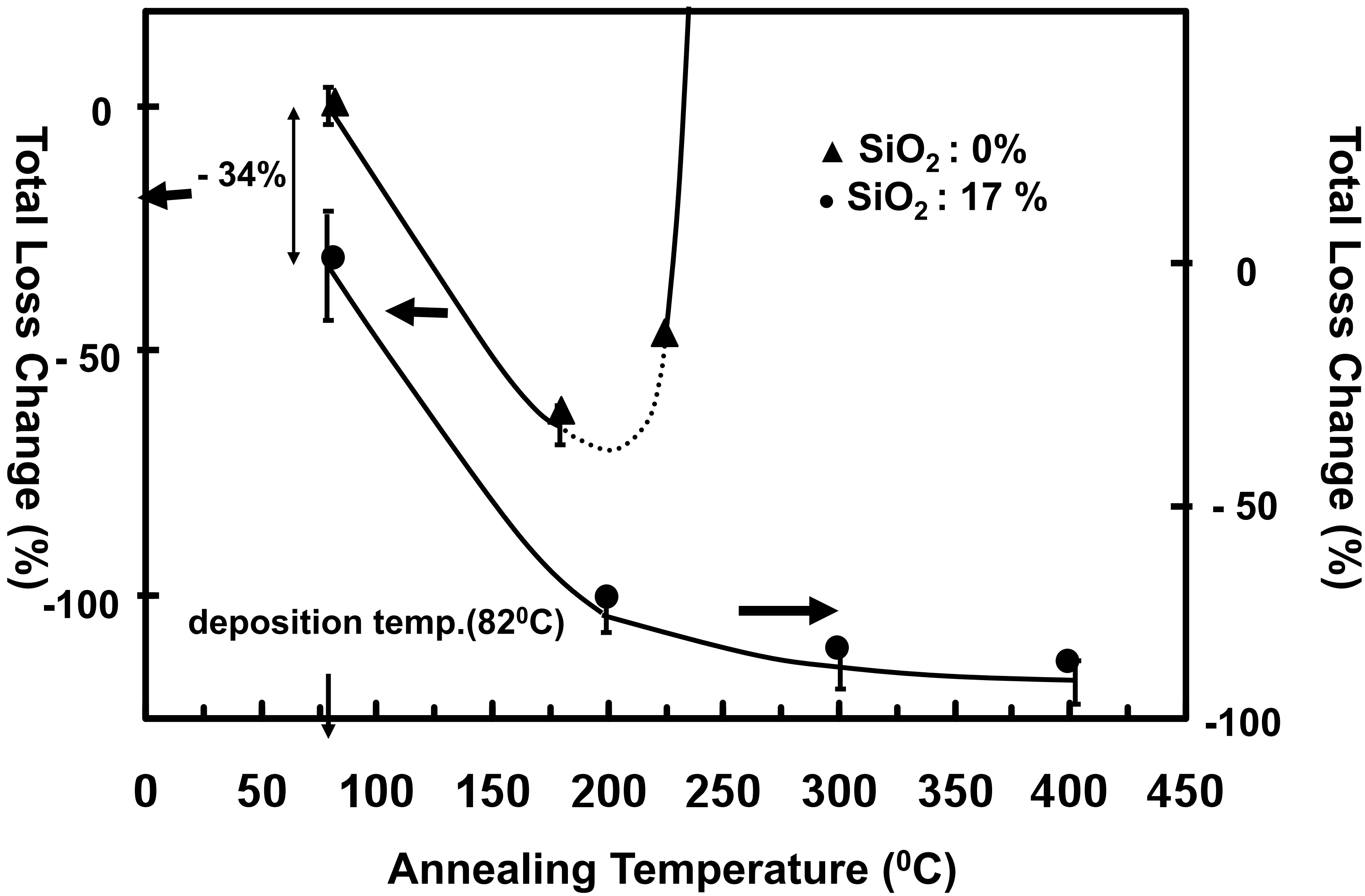
Shiuh Chao, Wen-Hsiang Wang, and Cheng-Chung Lee

1 May 2001 y Vol. 40, No. 13 y APPLIED OPTICS 2177



TiO2+ SiO2 17%
SiO2
nd = 1/4 λ
TiO2+ SiO2 17%
nd = 1/4 λ
SiO2
TiO2+ SiO2 17%
Zerodur

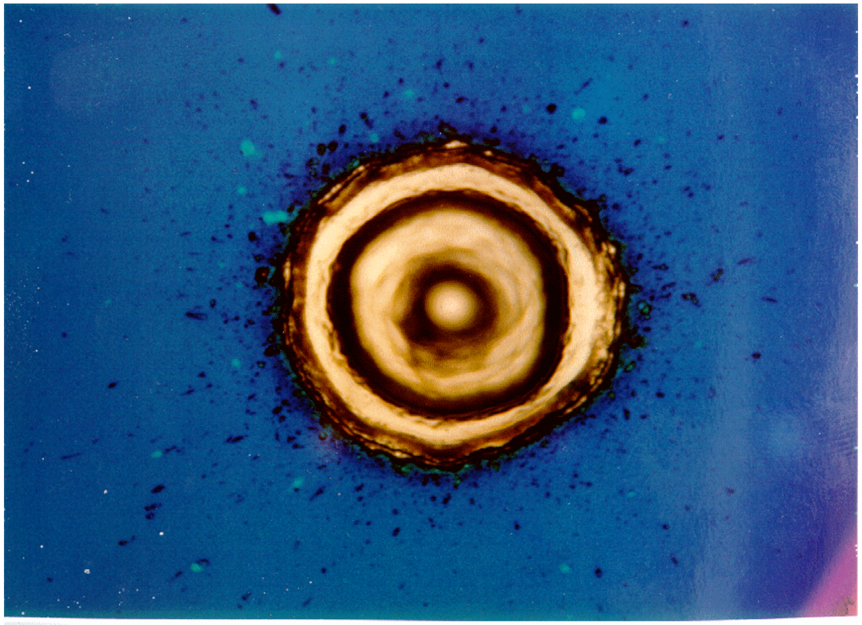
sample number	SiO_2(%) fraction	Annealing temp.(^0C)	Transmitt-ance (ppm)	Total loss (A+S)(ppm)	Total loss change(%)	Roughness rms(nm)	Damage(kJ/cm^2) threshold[c]
1	0	as-deposited	35	430±12	0	0.18	2.80
2	0	180	33	142±10	-67±2	0.19	6.12
3[a]	0	225	39	216[b]	-50	0.55[b]	immediate damage
4	17	as-deposited	90	282±36	0	0.16	4.27
5	17	200	94	72±7	-74±2	0.17	>6.37
6	17	300	96	41±12	-85±4	0.16	>6.37
7	17	400	99	34±11	-88±4	0.17	>6.37



Total Loss Change (%)
0
- 50
-100
- 34%
SiO2 : 0%
SiO2 : 17 %
deposition temp.(82⁰C)
Total Loss Change (%)
0
- 50
-100
0
50
100
150
200
250
300
350
400
450
Annealing Temperature (⁰C)

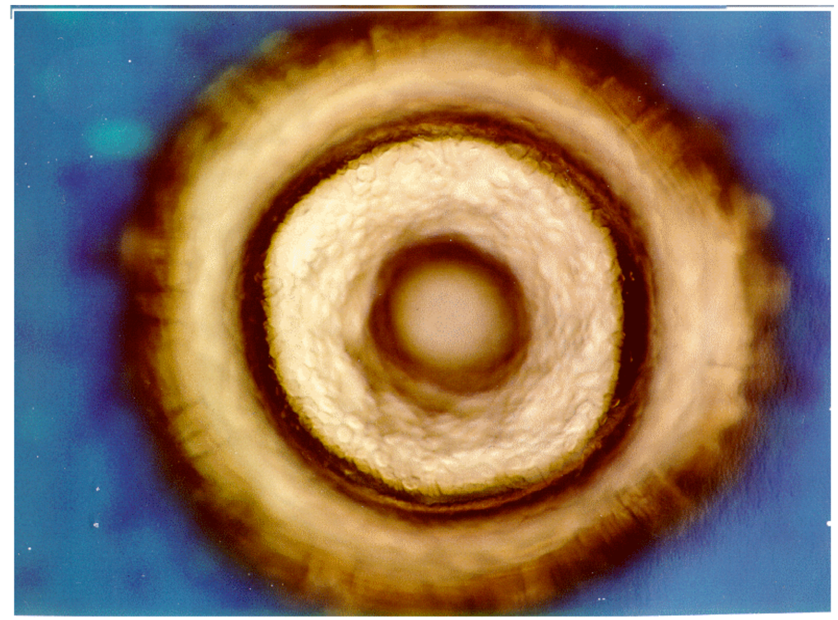
Laser Induced Damage

(A)



3 mm

(B)

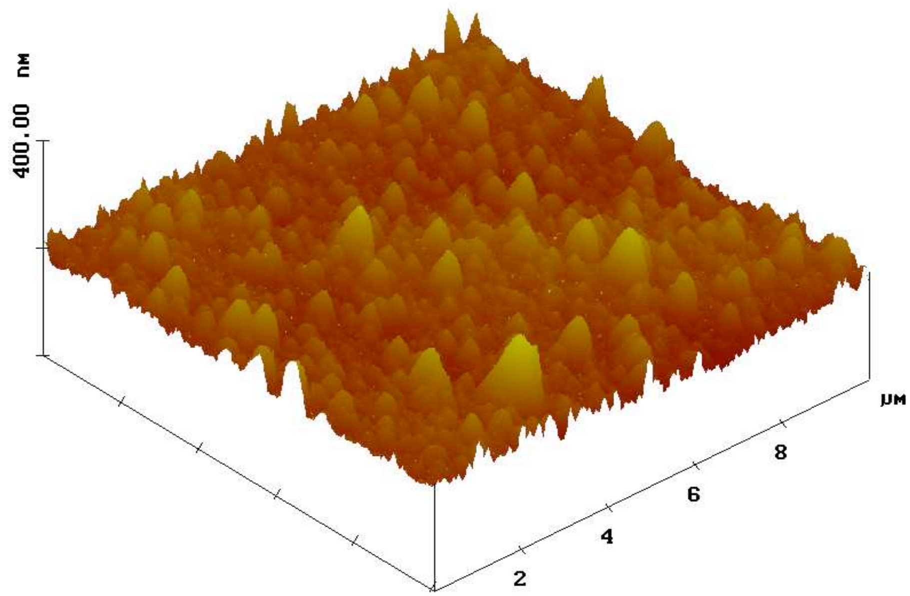


1.5 mm

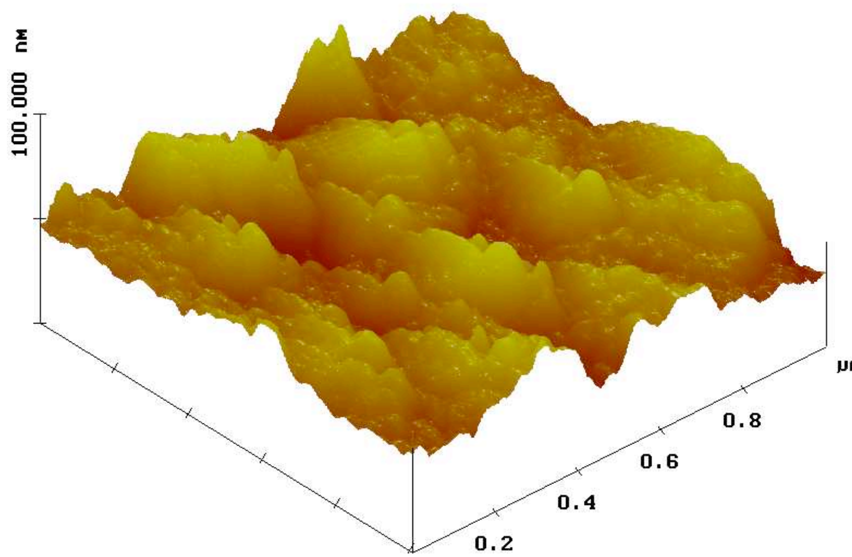
SiO_2:0% as-deposited
damage threshold: 2.8 KJ/cm^2

(A) (B): damaged spot under optical microscope

(C)



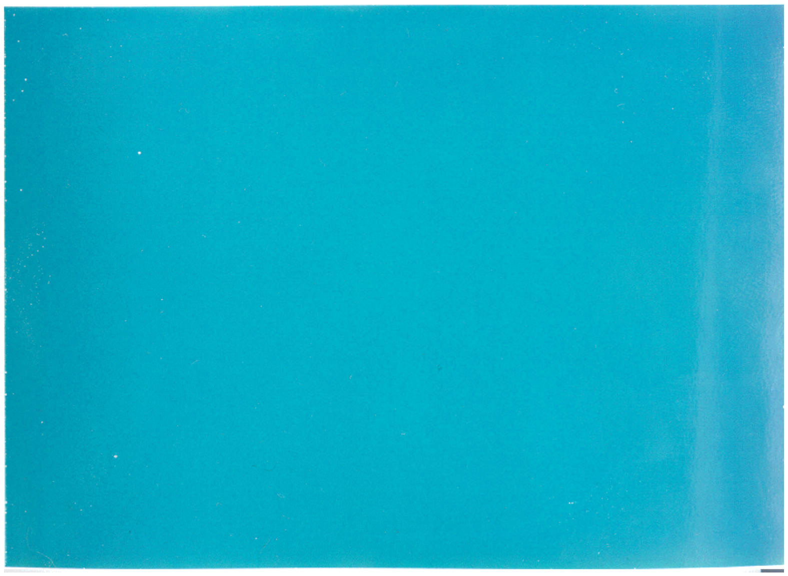
(D)



SiO_2:0% as-deposited
damage threshold: 2.8 KJ/cm^2

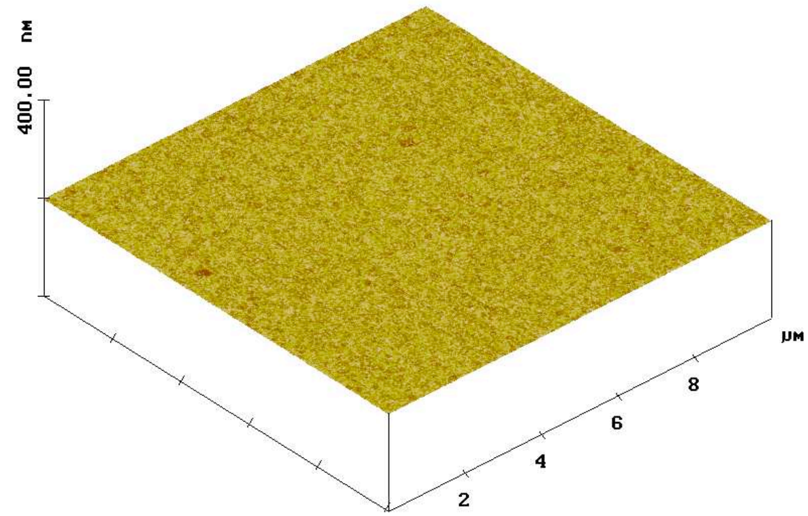
(C))(D) damaged spot under AFM

(A)



3 mm

(B)

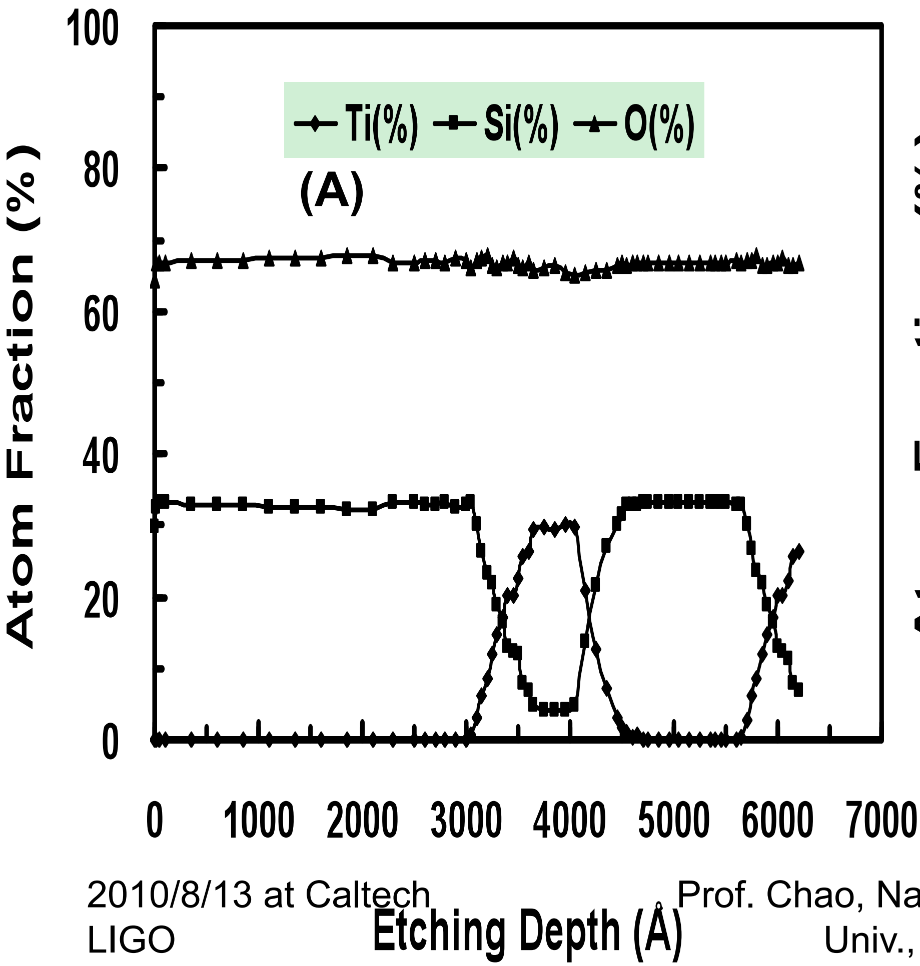


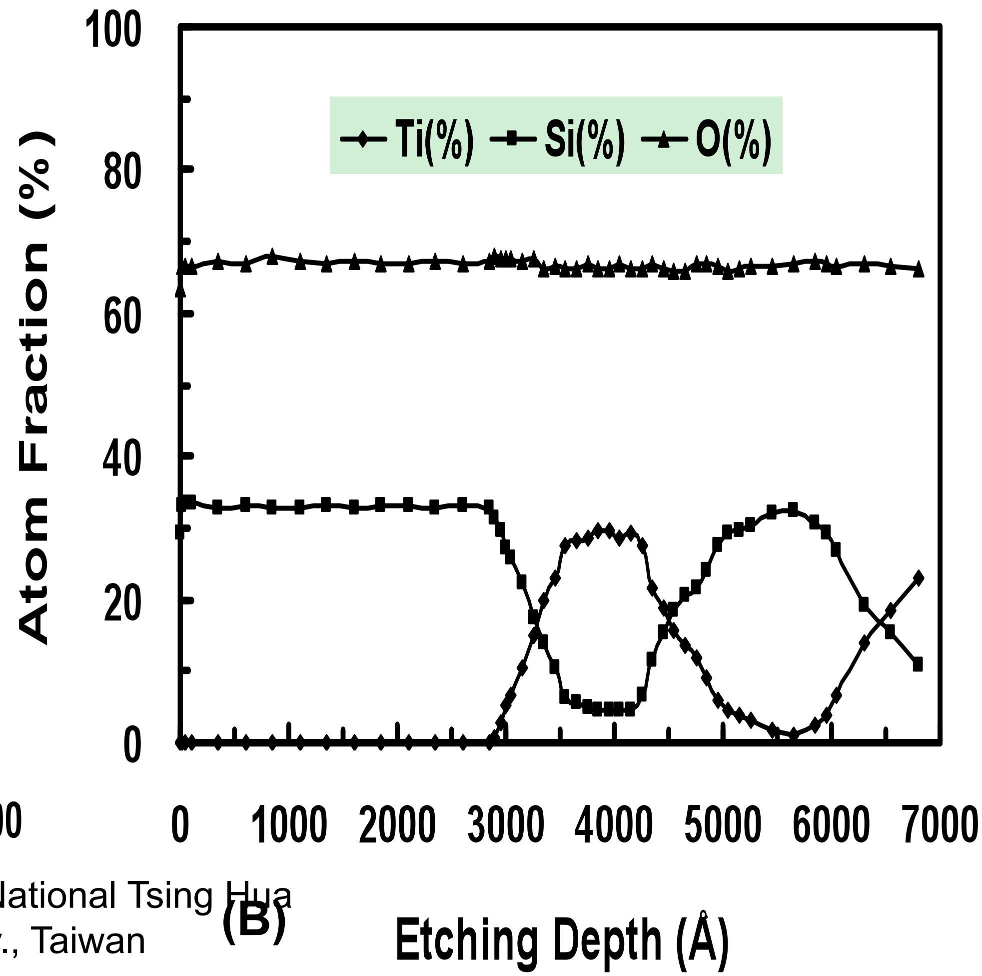
SiO_2: 17% annealed 200°C
Damage threshold: >6.37 KJ/cm^2

(A) Optical microscope (B) AFM of the exposed spot => no damage

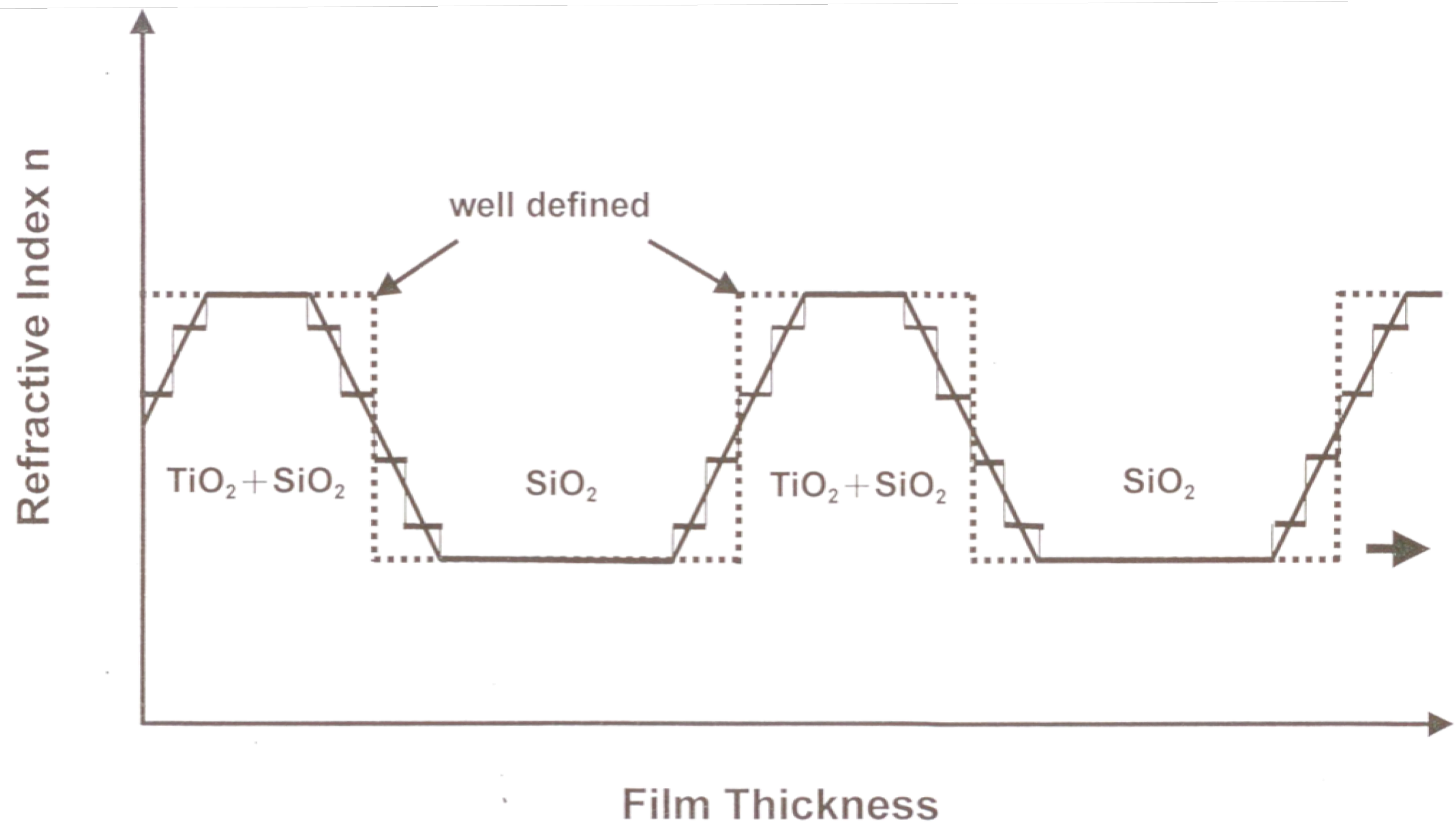
Depth profile for the mirrors (A) as-deposited(B) after 400^{o}C annealing (17% mixing)

(Tracing the Ti_{2p} Si_{2p} and O_{1s} by ESCA)

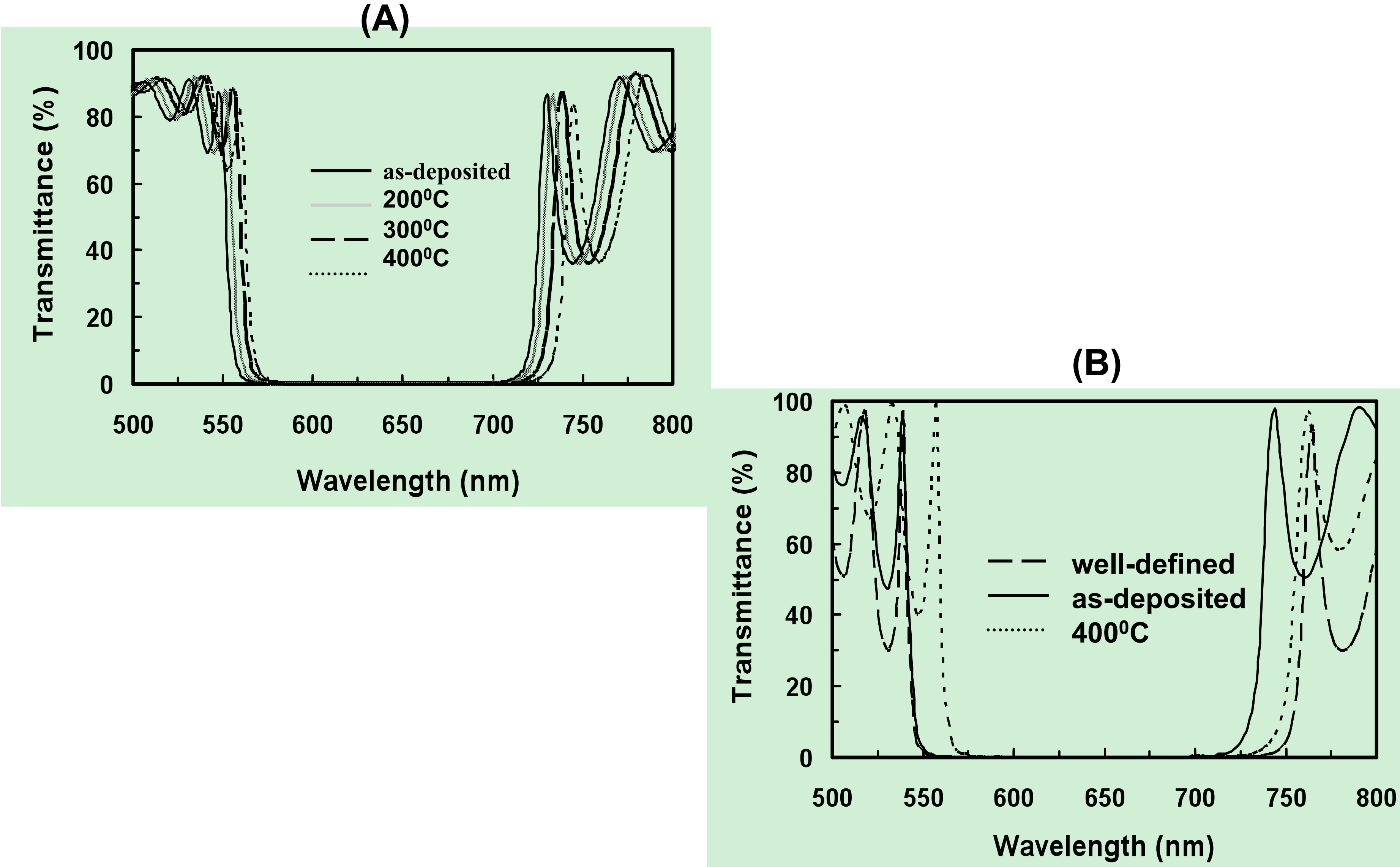




(B)



圖(三十九)以 TiO_2-SiO_2 混合膜為高折射係數層的雷射反射鏡無界面層存在的理想膜堆、界面折射率漸變膜堆與程式分割模擬漸變膜堆之折射係數分佈示意圖



圖(三十五)以TiO_2-SiO_2(17%)混合膜為高折射係數層的雷射反射鏡退火後　　的穿透光譜(A)為從光譜儀得到的量測值(B)為從ESCA對鏡片膜層原子分佈的偵測，以薄膜矩陣電腦模擬分析穿透率的結果

Other methods for depositing mixed films

TiO 2-S102 mixed films prepared by the **fast alternating sputter** **method**

Shiuh Chao, Cheng-Kuel Chang, and Jyh-Shin Chen

We introduced the fast alternating sputter method and its application on deposition of TiO2-SiO2 mixed films. By using fast alternating sputter, the TiO2 and SiO2 were completely mixed in the film, and no thinpair structure could be found by x-ray diffraction. The structure of the mixed films was amorphous in a wide composition range. The optical properties of the mixed films in the visible and near infrared changed from SiO2-dominant to TiO 2 -dominant as TiO2 content in the film increased.

1 August 1991 / Vol. 30, No. 22 / APPLIED OPTICS 3233

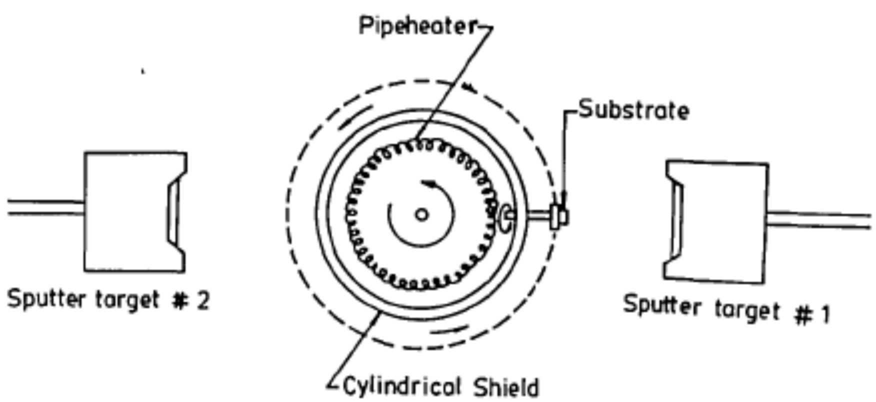


Fig. 1. Alternating sputter setup.

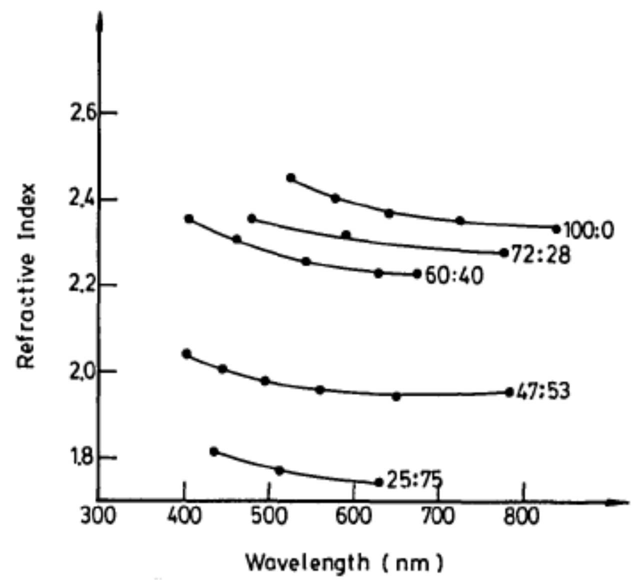


Fig. 6. Refractive index versus wavelength for the mixed films in Figs. 4 and 5.

Slow alternating sputter for "nano-film" ?

Mixed films of TiO_2–SiO_2 deposited by double electron-beam coevaporation

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We used double electron-beam coevaporation to fabricate TiO2–SiO2 mixed films. The deposition process included oxygen partial pressure, substrate temperature, and deposition rate, all of whichwere real-time computer controlled. The optical properties of the mixed films varied from pure SiO2 to pure TiO2 as the composition of the films varied accordingly. X-ray diffraction showed that the mixed films all have amorphous structure with a SiO2 content of as low as 11%. Atomic force microscopy showed that the mixed film has a smoother surface than pure TiO2 film because of its amorphous structure. Linear and Bruggeman's effective medium approximation models fit the experimental data better than other models.

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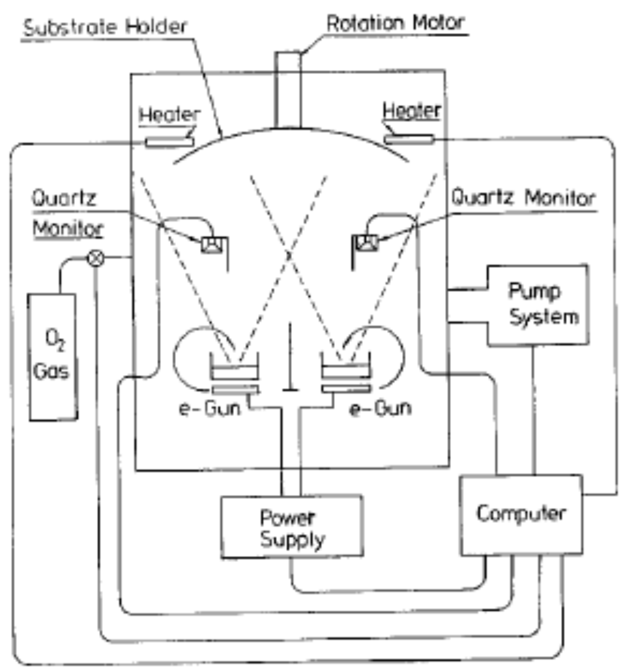

Fig. 1. Configuration of the Balzers BAP800 coating chamber.

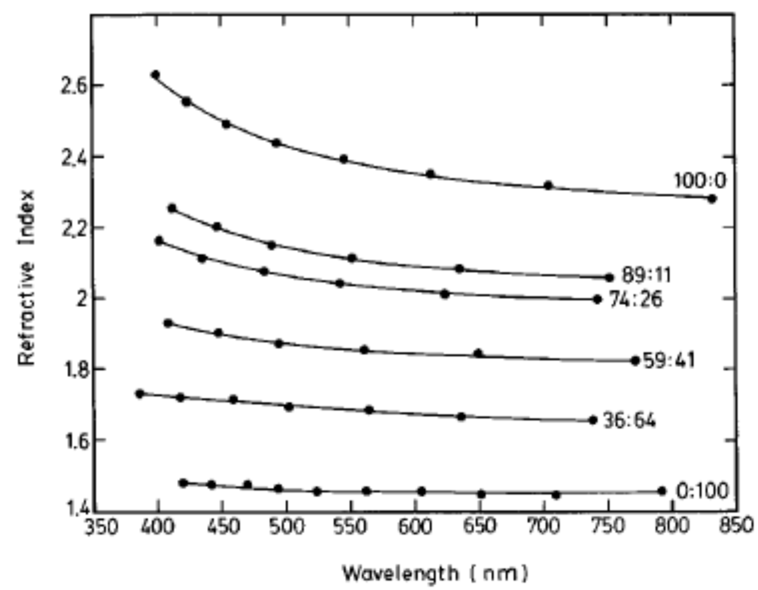

Fig. 7. Refractive index versus wavelength for the mixed films. The ratios are the TiO_2:SiO_2 mole fractions.

Mixed film for LIGO application

- One more factor ---- mechanical loss ---- should be added for investigation
- Near future: systematically characterizing the mechanical loss of the films
- Current effort: recovering the old coating conditions for the films