# 4. SR 光による光学薄膜の膜質評価

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ECR スパッタ法は成膜安定性に優れ基板へのダメージも通常のスパッタ法よりも小さいことから, 従来の蒸着法に代わり, 光学部品の反射膜などへの適用が進んでいるが, 膜質については十分な研究がなされていない。膜厚や膜密度は X 線反射率法で非破壊に調べることができるが, Si 基板上の SiOx 膜は基板との密度差が小さいことから, 実験室装置では困難である。我々は, 高輝度 SR 光を用いて, Si 基板上の ECR-SiOx 薄膜と蒸着 SiOx 膜で, 膜密度や表面粗さがどのように異なるかを調べた。ECR スパッタ法および蒸着法で 100nm 厚の SiOx 薄膜を Si 基板上に形成した。光学屈折率および膜組成比がほぼ同じ膜について, SPring-8・BL16B2 のθ-2θゴニオメータを用い, X 線エネルギー＝10keV で X 線反射率測定を行った。Fig.1 に 100nm 厚 ECR 膜(図中 S-)と蒸着膜(同 EB-)の X 線反射率曲線を示す。ECR 膜は蒸着膜よりも反射率曲線の振幅が明らかに小さい。フィッティング解析の結果, ECR 膜は蒸着膜よりも密度が約 10%大きく, 表面粗さは約 3/4 であることが分かった。ECR 膜は, 蒸着膜と比べて光学特性や組成比が同じでも, 高密度で表面平滑性に優れるといった膜質の違いがあることを, SR 利用 X 線反射率により明らかにできた。

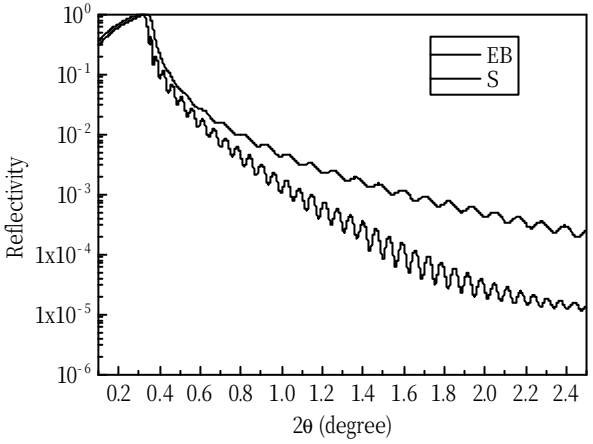


Fig.1.　X-ray reflectivity of EB- and S- $SiO_x$ / Si.

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Changes for the Better

# SR光による光学薄膜の膜質評価

## X-ray reflective studies of SiOx thin films for optical coating

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# - Background -

Optical coatings : important roles in optical devices, such as LD.

ECR sputtering method: attractive for optical coatings

- *less damage to substrates than conventional sputtering methods*
- *better controllability than evaporation methods*

However, the properties of the fabricated films have not been well studied.

XRD: not applicable because there are amorphous
XAFS: difficult due to the absorption energy of Si
XRR: applicable for the study of the density and the roughness!

Our goal: to know the difference of the density and the roughness in SiOx films by ECR sputtering and evaporation

# Samples and Experiments

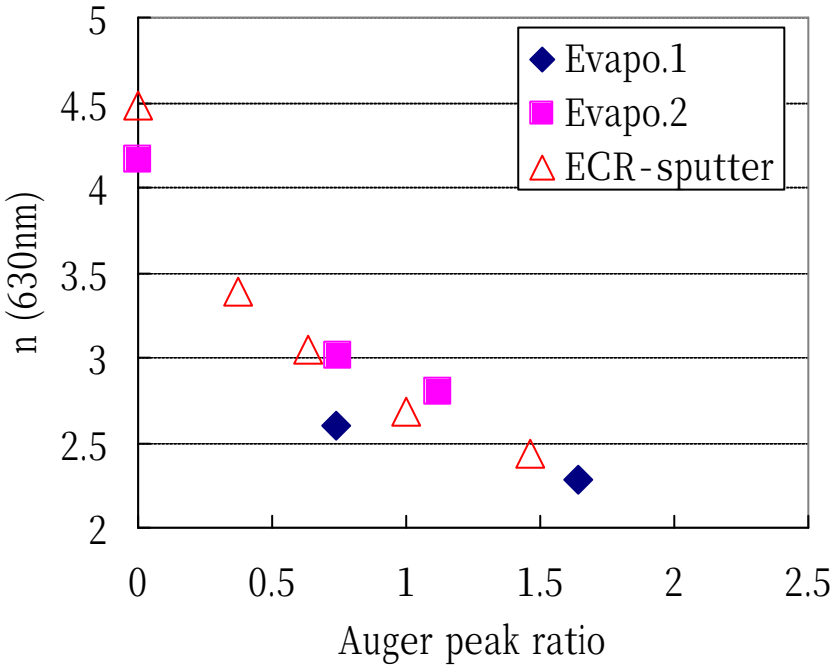
## Samples : SiOx/Si

| No. | Method | $O_2$ flow | Si/O ratio (Auger analysis) | Refractive index @630nm | Extinction index @630nm |
|---|---|---|---|---|---|
| 1 | Evaporation | 0.4 sccm | 1.1 | 2.6 | 0 |
| 2 | ECR | 0.4 sccm | 0.6 | 3.0 | 0.2 |
| 3 | ECR | 1 sccm | 1.0 | 2.6 | 0.13 |
| 4 | ECR | 6 sccm | 1.5 | 2.4 | 0.07 |

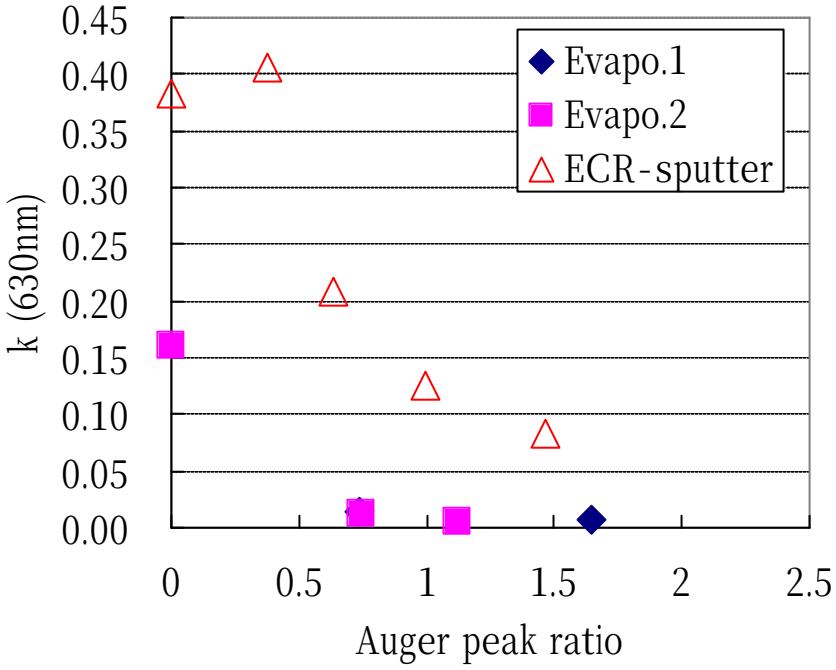
## X-ray Reflection measurements at SPring-8 BL16B2

- X-ray energy = 10 keV (0.12398 nm)
- Step measurements by a θ-2θ goniometer and a scintillation counter

# $O_2$ Contents vs. Optical Properties



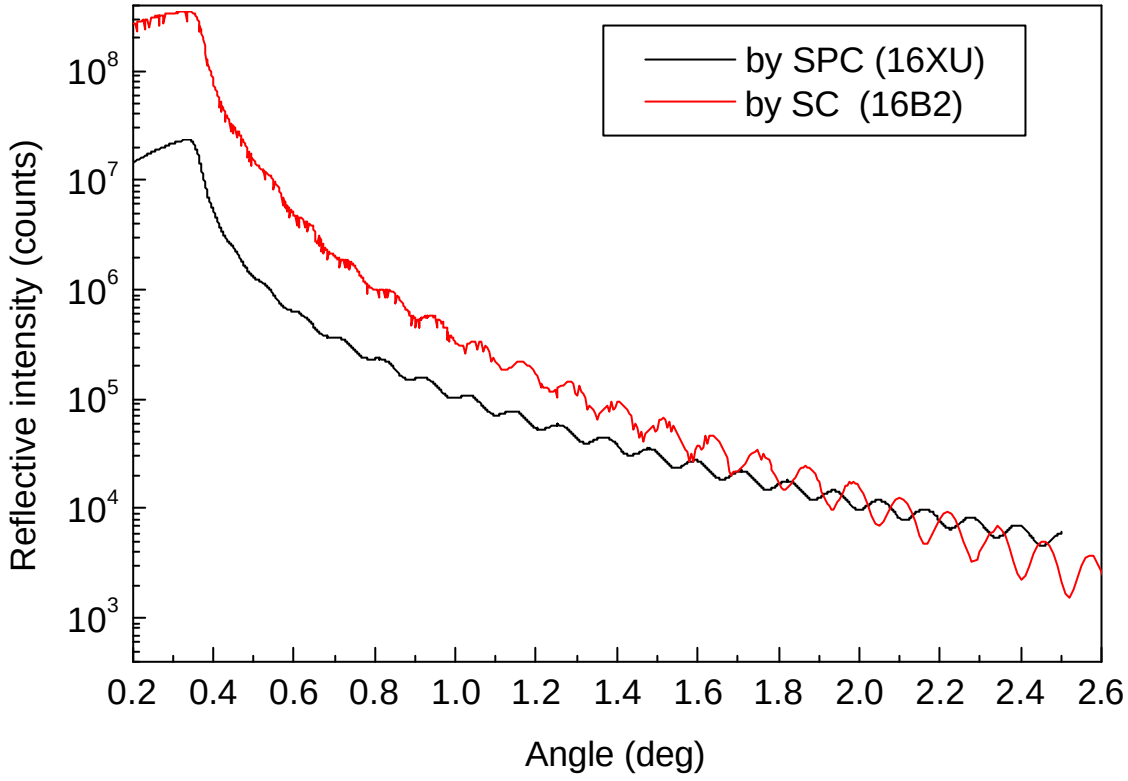

Si/O ratio vs. refractive index




Si/O ratio vs. extinction index

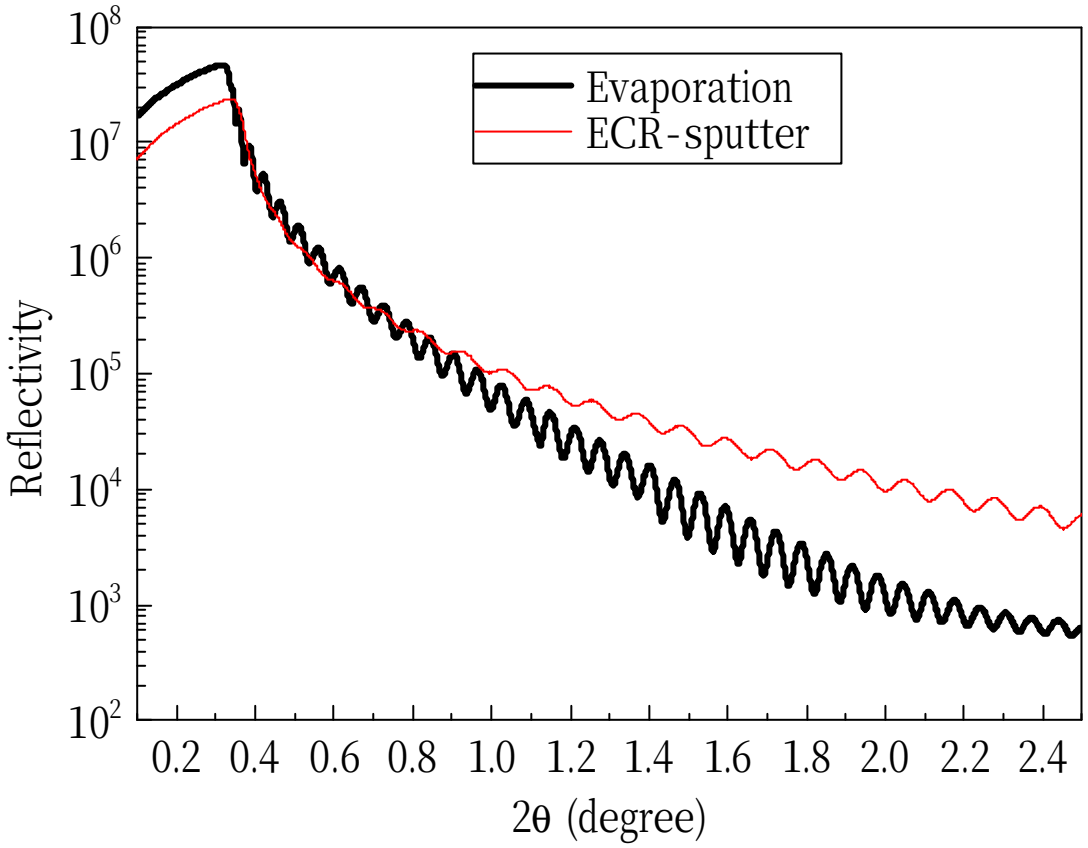
The refractive index can be predicted only from Si/O ratio, while the ECR-sputtered films shows significant values of extinction index.

# Example of failure measurements



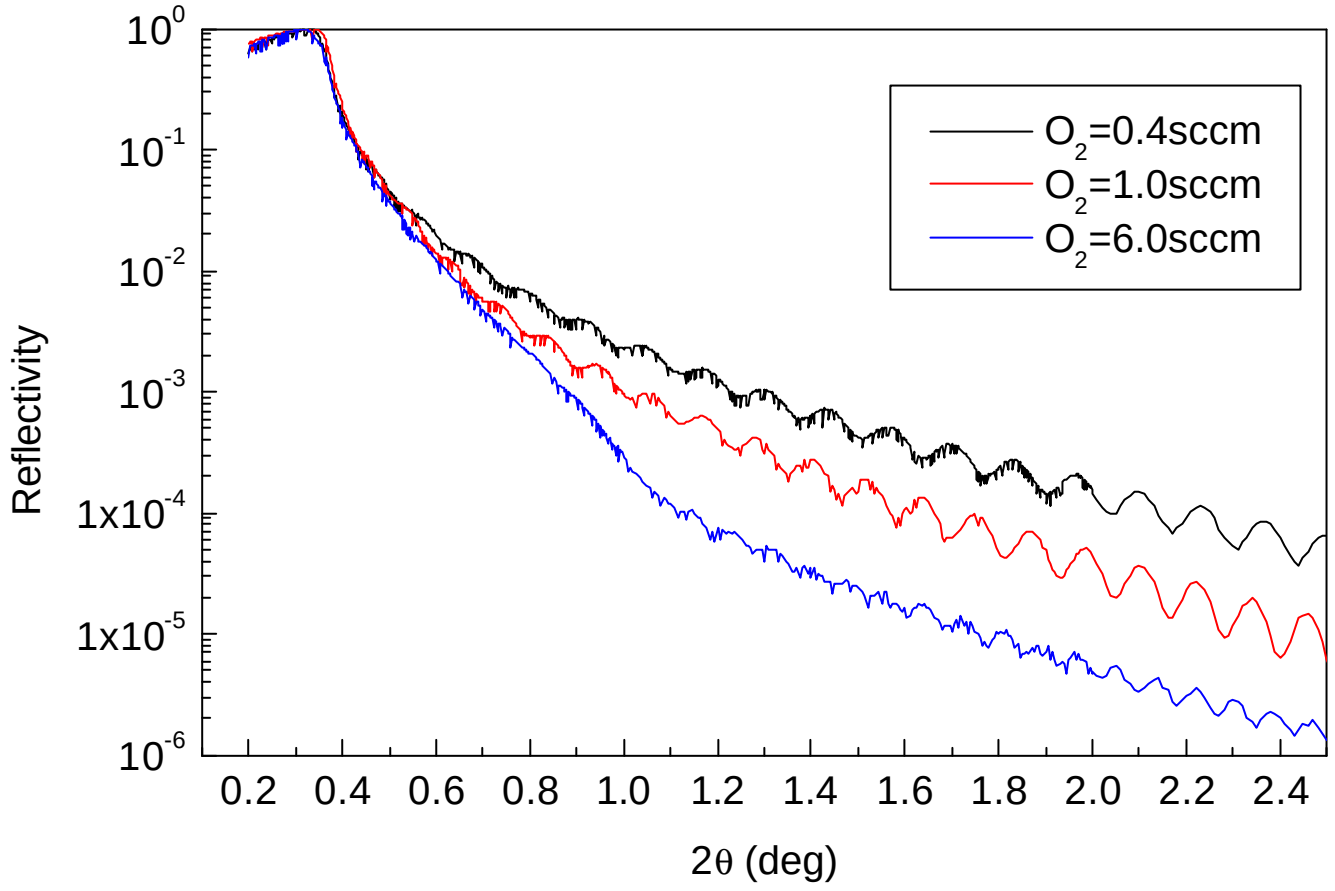

The linearity of the Sealed Proportional Counter / amplifier is not good enough for the measurements.

# X-ray Reflection curve of SiOx films
## (evaporation vs. ECR-sputter)




ECR film shows smaller amplitude and smaller dump.
→ ECR films should have higher density and smoother surface.

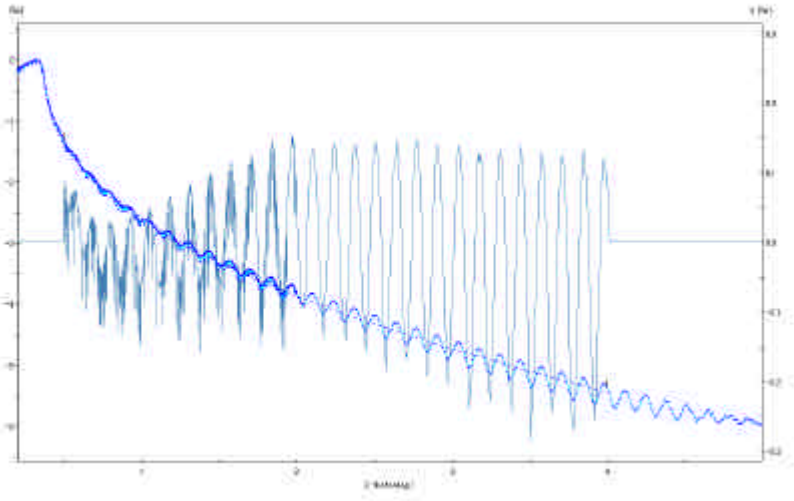
# X-ray Reflection curve of SiOx films
## (of ECR-sputterd with different $O_2$ flow rate)




The film by higher $O_2$ flow shows larger dumping curve.

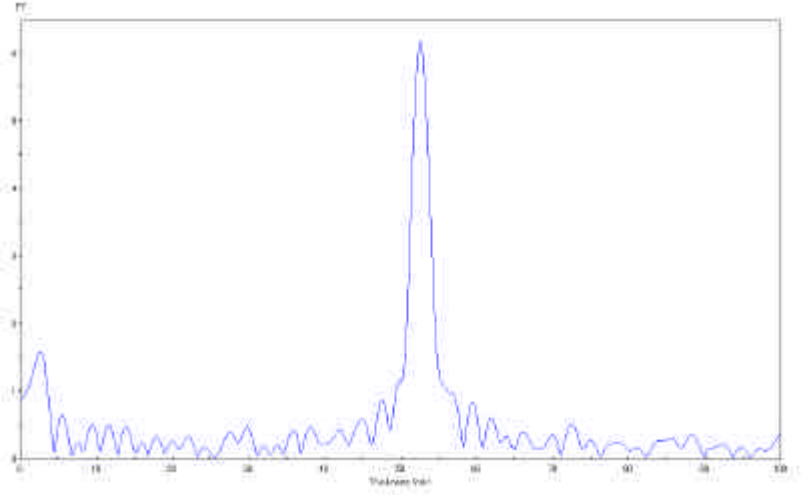
# Analysis of X-ray Reflection Curve

Extraction of the oscillation



Needs predicted value of density and roughness of the film when calculating the base line.

Fourier transform



The peak shows the thickness of the film.

More precise value of the thickness, the density and the roughness of the films can be obtained by curve fitting analysis.
The analysis was done the angle range of 1.0 – 3.5 degrees.

# Curve Fitting Results

| No. | Refractive index @630nm | Extinction index @630nm | thickness (nm) | density ($g/cm^3$) | roughness (nm) |
|---|---|---|---|---|---|
| 1 (evaporation) | 2.6 | 0 | 108.3 | 1.975 | 0.729 |
| 2 (ECR) | 3.0 | 0.2 | 52.4 | 2.125 | 0.258 |
| 3 (ECR) | 2.6 | 0.13 | 58.6 | 2.154 | 0.535 |
| 4 (ECR) | 2.4 | 0.07 | *80.7<br>4.2 | (1.905)<br>(2.229) | (0.522)<br>(2.263) |

*A single layer model did not fit the experimental curve.

The density and the roughness did not show any correlation with optical properties.

# Conclusions

- ECR-sputtered films have higher density and smoother surface than evaporation films.
- Density and/or roughness of the film have no correlation with optical refractive index.
- Films of large extinction index showed higher density, however, the correlation is not clear.