X-ray Reflectivity Study on the Density and the Roughness of Silicon Oxide Thin Films under Various Fabrication Conditions

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CVD-SiO_2 films are technologically very important to semiconductor devices; however, one problem is that leak current of the films is higher than that of thermal oxidized SiO_2 films. We have found that radical oxidation of CVD-SiO_2 films reduce leak current of the films. In this study, we estimated the surface roughness and density of radical oxidized CVD-SiO_2 films using X-ray reflectivity study and investigated the relationship with the electric properties.

SiO_2 films of about 3 nm thick were grown on Si (100) wafers with CVD method. Some samples were then radical oxidized in Ar/O_2 plasma. The X-ray reflectivity measurements of the films were performed at BL16B2 using monochromatized X-rays of 0.124 nm.

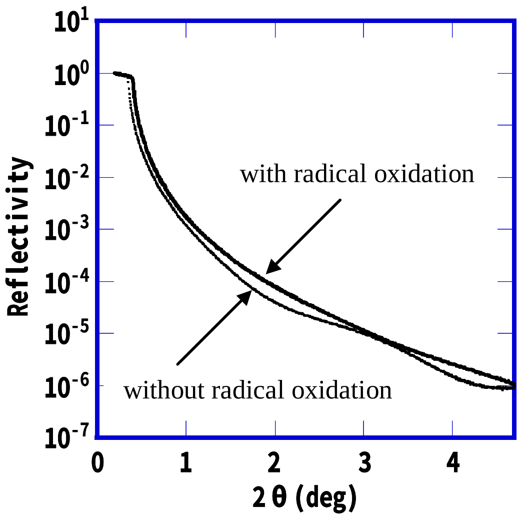

Fig. 1. X-ray reflection curves of CVD-SiO_2 films with and without radical oxidation.

Leak current of the radical oxidized CVD-SiO_2 film was lower than that of the as-deposited sample. Figure 1 shows x-ray reflection curves of the two samples. The oscillation amplitude of the radical oxidized sample was smaller than that of the as-deposited sample. The oscillation amplitude is related to the difference of density between the film and the substrate, and the results above indicate that the density of CVD-SiO_2 film is raised by radical oxidation. It is considered that the leak current would be reduced by the increase of the film density.

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1. Introduction

Merits of CVD silicon oxide

- low temperature process
- fabrication on any layer
- avoiding effect of substrate defect

Demerits of CVD silicon oxide

- high leak current ← The density would be low.

Advantages of O_2 radical treatment

- high oxidizability
- low temperature proces
- affecting only surface

  (no oxidation of under layer by small diffusion constant)

Conventional methods to estimate density

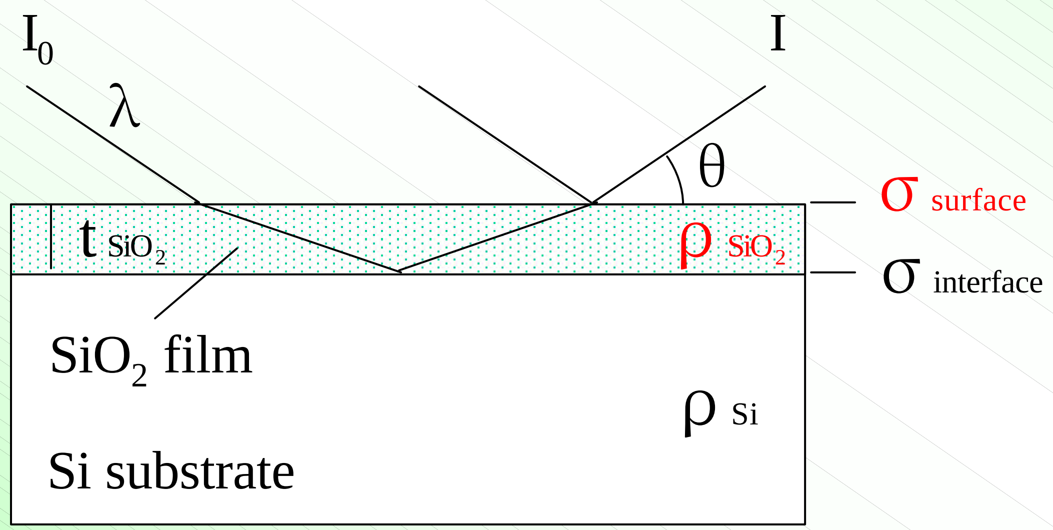
- XPS : variance of bonding angle
- TDS : H_2O desorption (adsorption)

← indirect and non quantitative

X-ray reflectivity mesurements were performed to the CVD silicon oxide film treated by O_2 radical, and density and roughness were investigated.

2. Theory

Case of a silicon oxide layer on a silicon substrate.



ρ_{SiO_2} : density of SiO_2 film

ρ_{Si} : density of Si substrate

$\sigma_{surface}$: roughness of SiO_2 surface

$\sigma_{interface}$: roughness of SiO_2/Si interface

t_{SiO_2} : thickness of SiO_2 film

λ : wavelength of X-ray

θ : incident angle

X-ray reflection curve is described as (L. G. Parratt, Phys. Rev. **95**, 359(1954).)

$$I / I_0 = R\,(\rho_{SiO_2},\ \rho_{Si},\ \sigma_{surface},\ \sigma_{interface},\ t_{SiO_2},\ \lambda,\ \theta)$$

density   surface roughness   interface roughness

These parameter is able to be obtained by curve fitting.

Density | Surface roughness | interface roughness

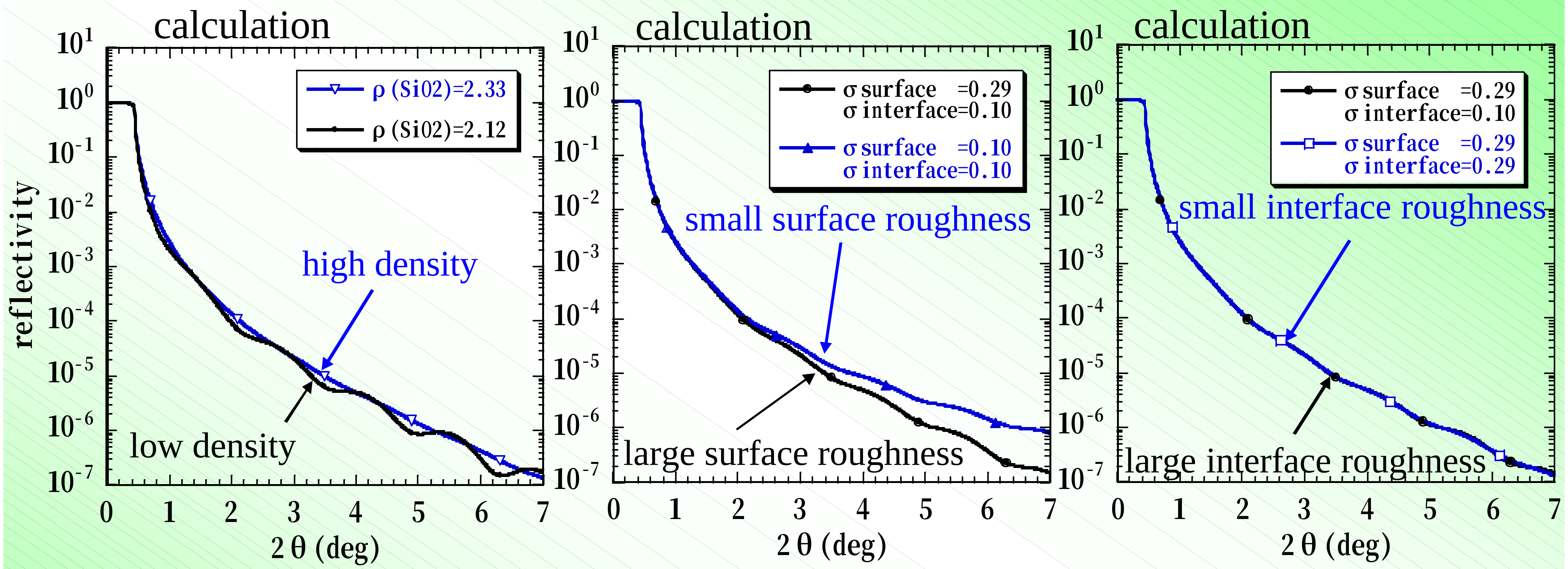


Fig. 1. The shape variation of X-ray reflection curves versus density (density difference between film and substrate), surface roughness and interface roughness.

Density is	high	→	Oscillation amplitude is	small
Surface roughness is	small	→	Decay is	small
Interface roughness is	small	→	Spectrum shape is	not change

3. Experiments

(1) Sample perparation

- HF etching
- CVD silicon oxide
- O_2 radical treatment

reference： pyrogenic, without O_2 radical treatment

(2) Meausrements

・X-ray reflectivity（denisty, surface roughness）

Beam line ： SPring-8 BL16B2

Energy ： 10 keV, Al filter

・I-V（leak currents）

・XPS（chemical bonding state, Si 2p, O 1s → density）

・TDS（H_2O desorption, m/e=18 (H_2O) → density）

・AFM（surface roughness）

4. Results

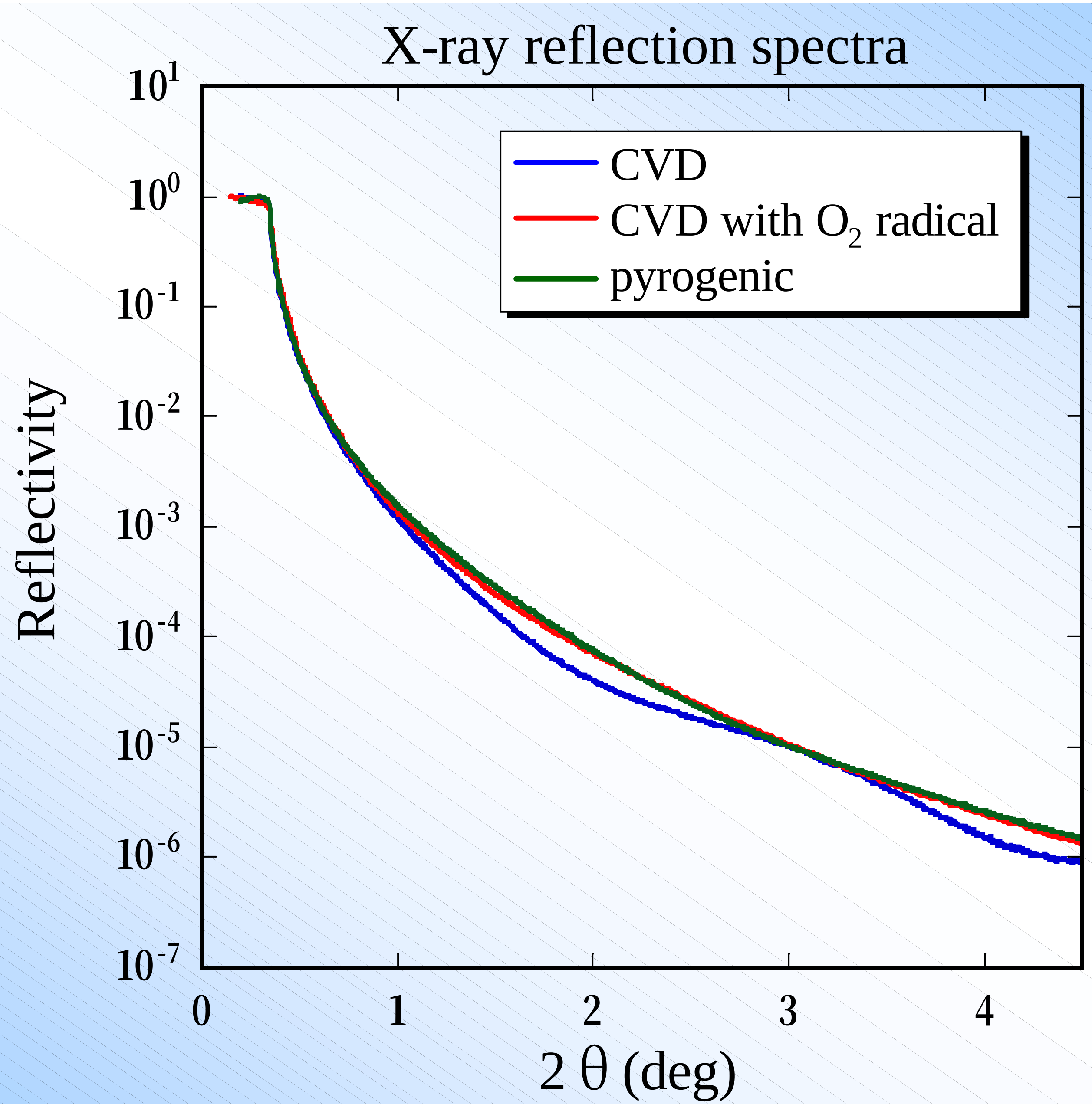
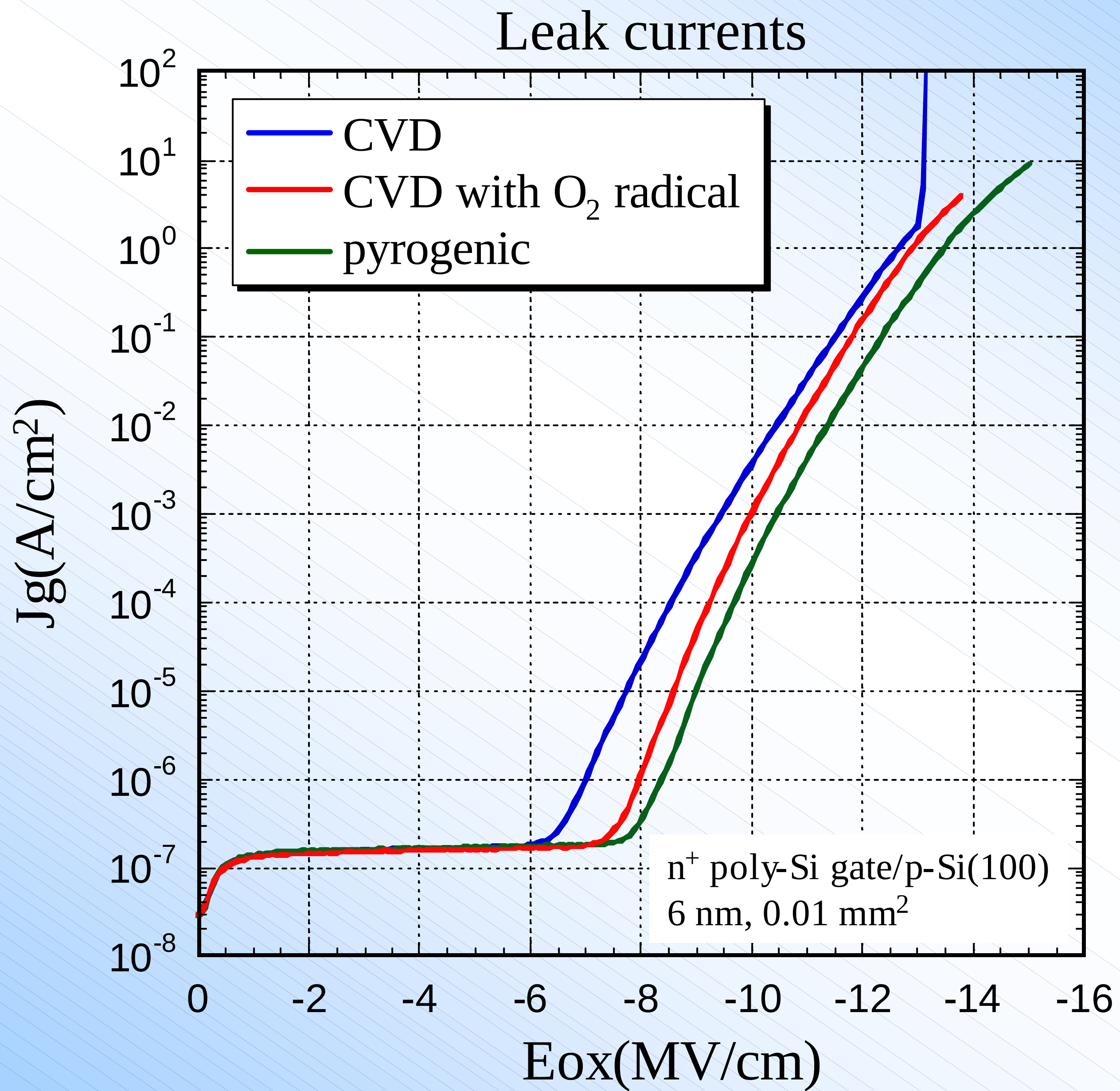


Fig. 2. X-ray reflection curves of various silicon oxide films.

The oscillation amplitude decreased with O_2 radical treatment.



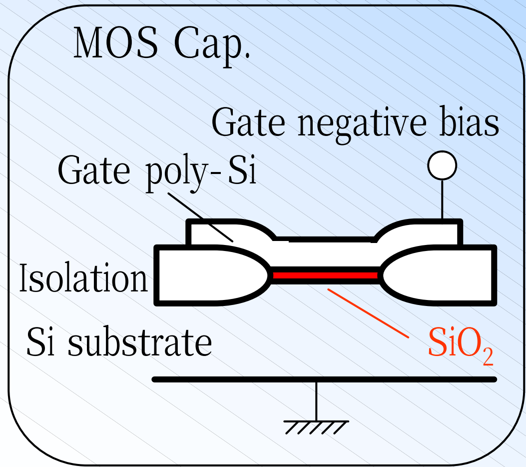


Fig. 3. I-V curves of silicon oxide gate insulator.

The leak current decreased with O_2 radical treatment.

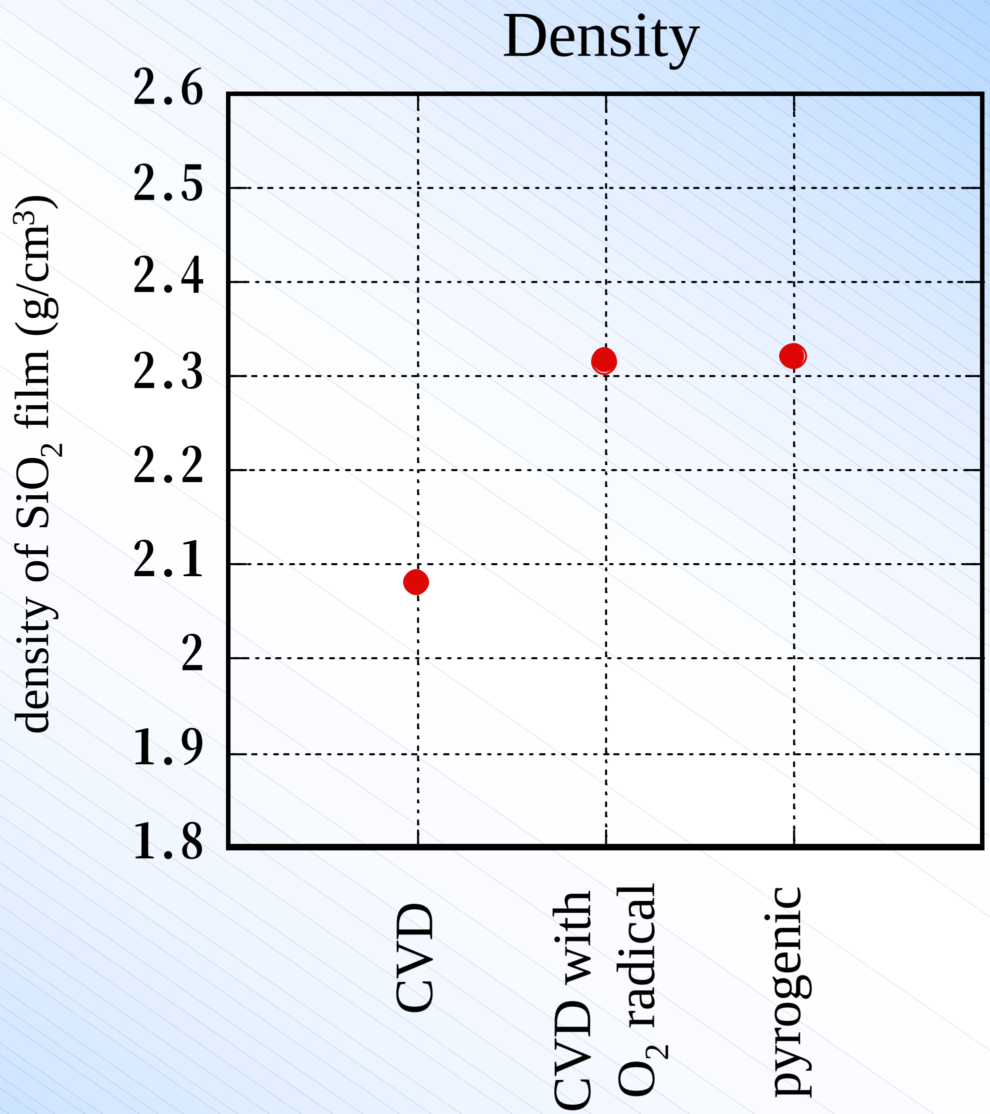


Fig. 4. The oxide film density obtained by X-ray reflection.

The silicon oxide film density increased with O_2 radical treatment.

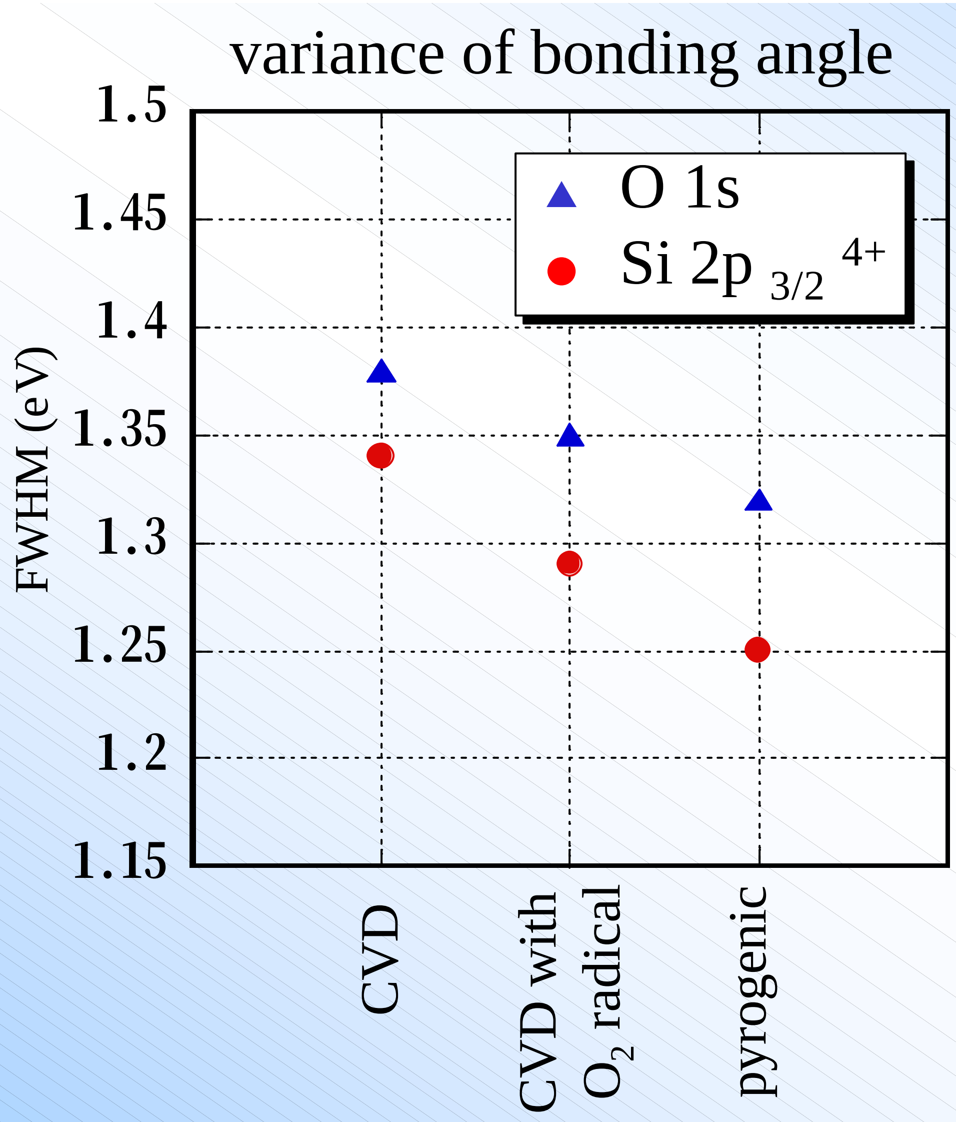


Fig. 5(a) FWHM of XPS peaks.

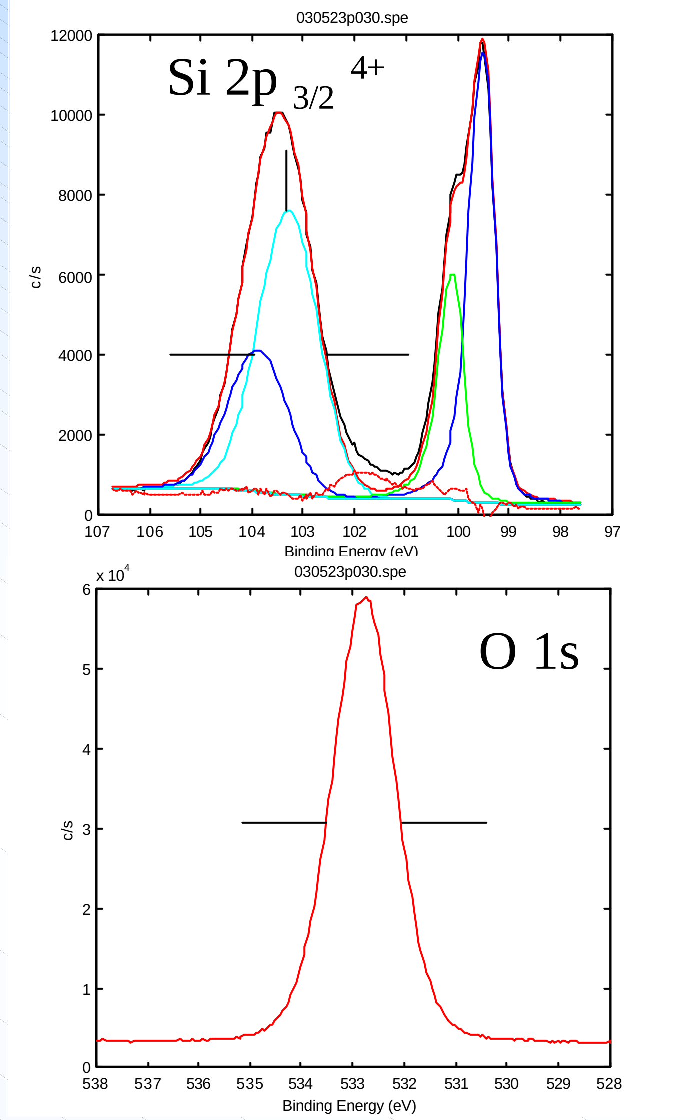


Fig. 5(b) XPS spectra.

The variance of bonding angle decreased. → The density would increase.

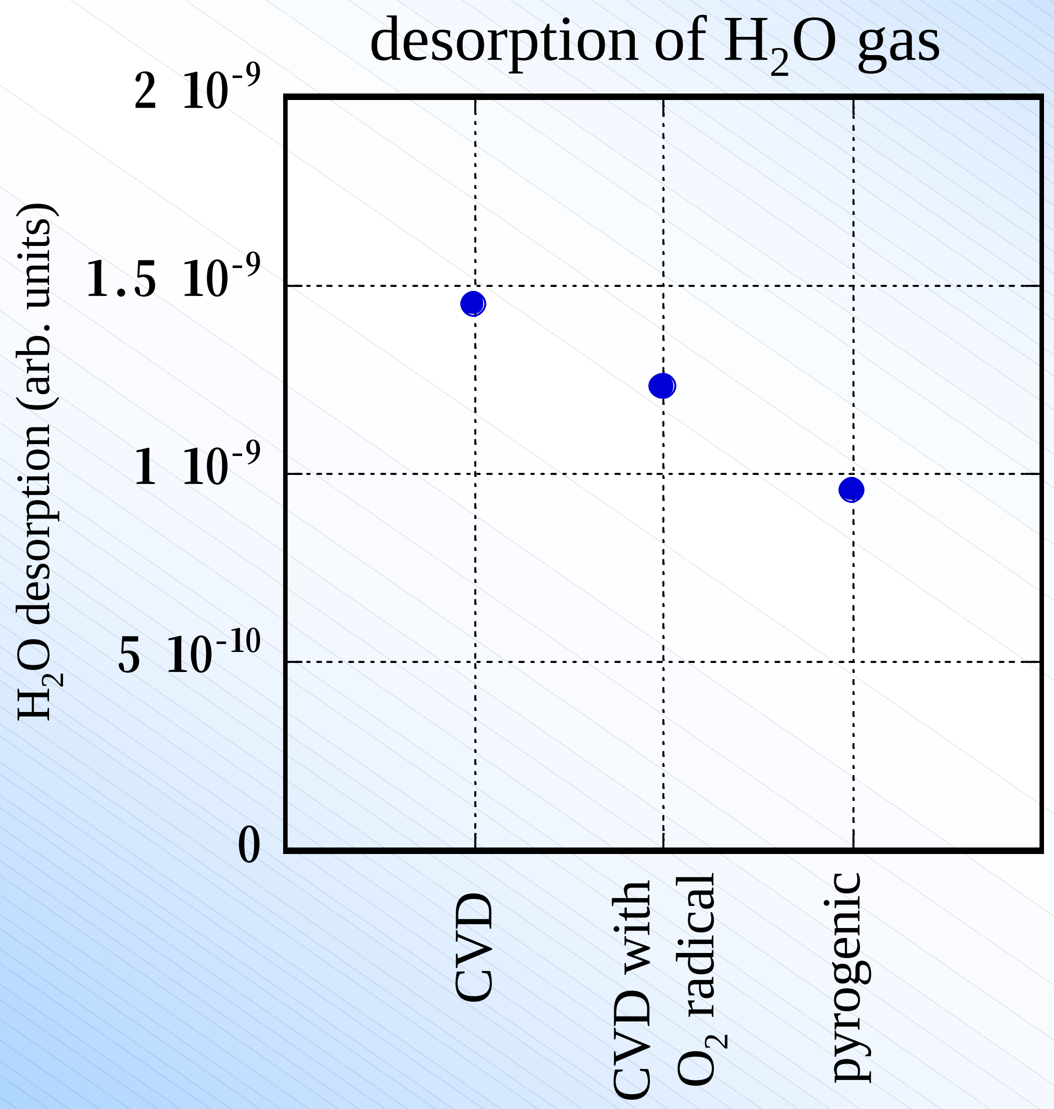


Fig. 6(a) The amounts of H_2O desorption with TDS.

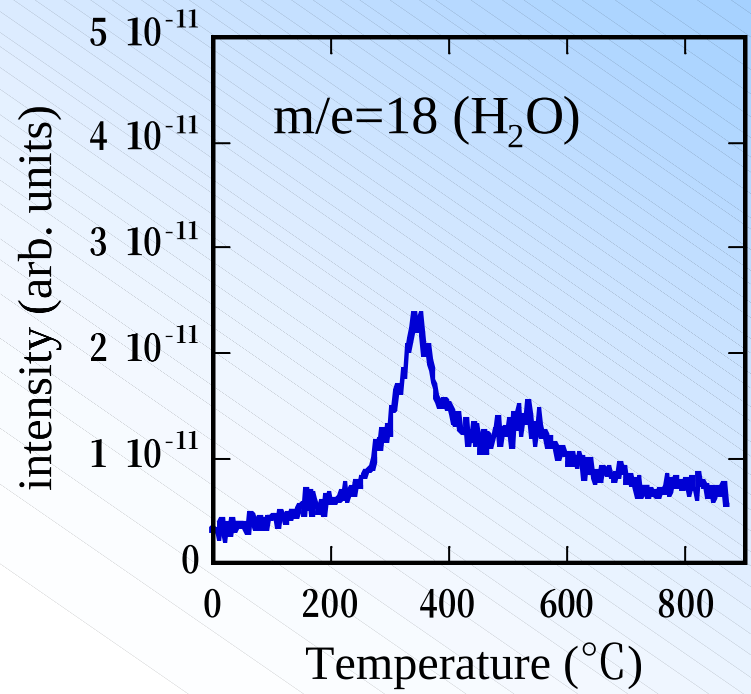


Fig. 6(b) TDS spectra.

The amounts of H_2O desorption decreased. → The density would increase.

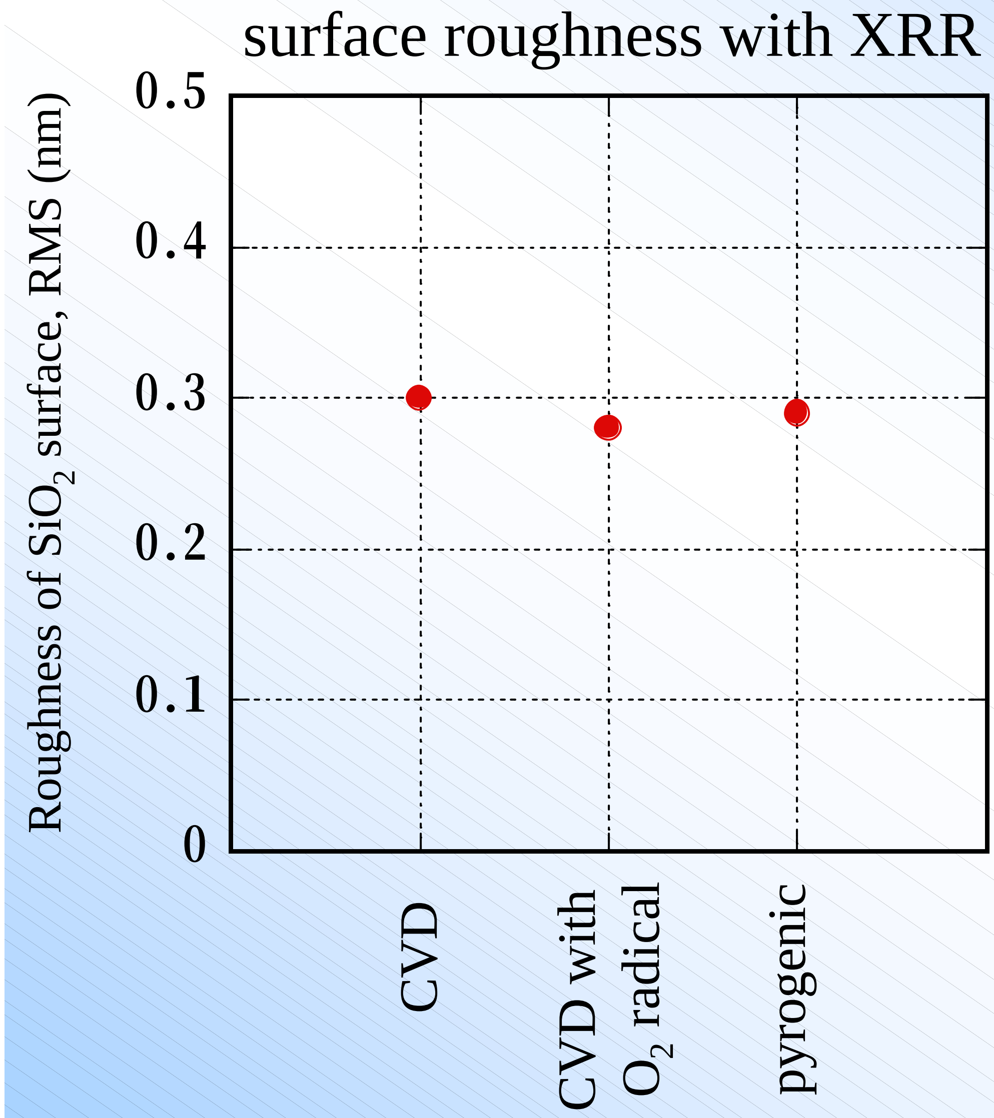


Fig. 7. The surface roughness of oxide film obtained by X-ray reflection.

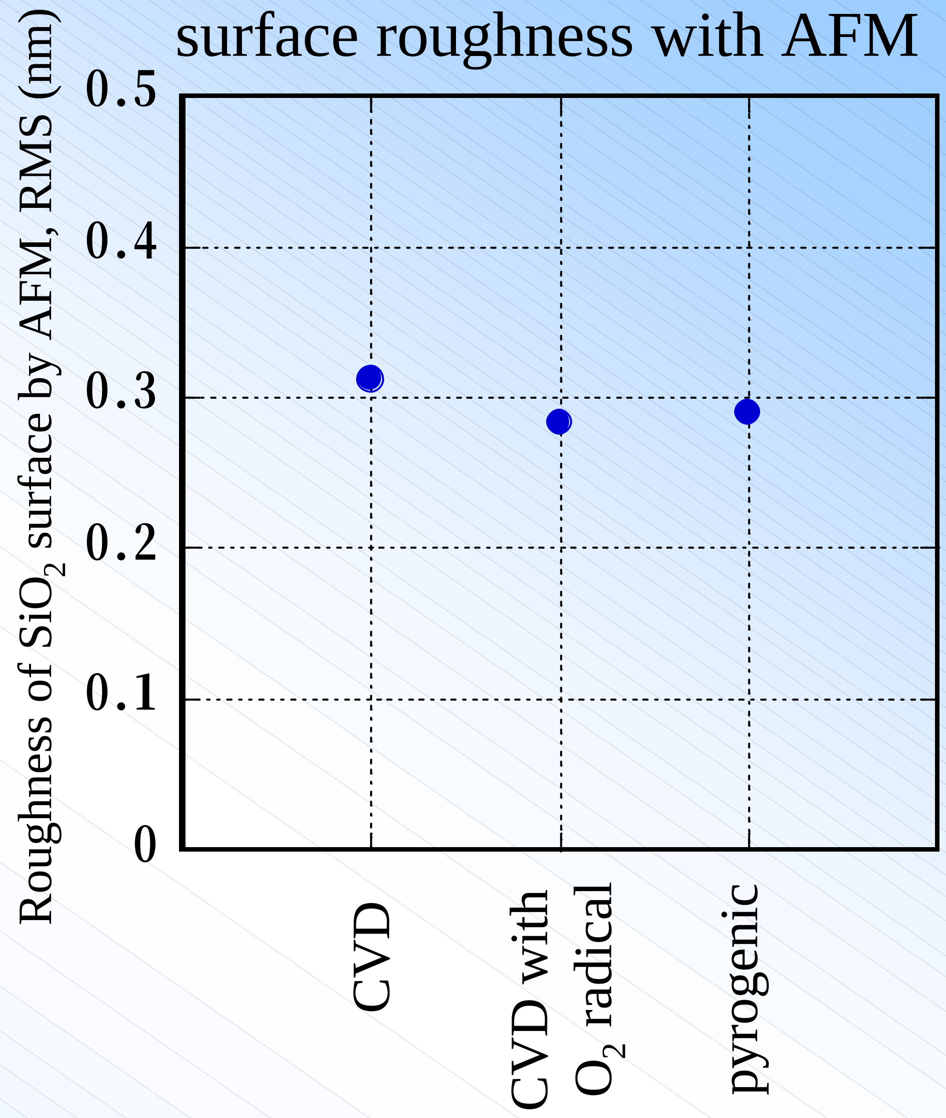


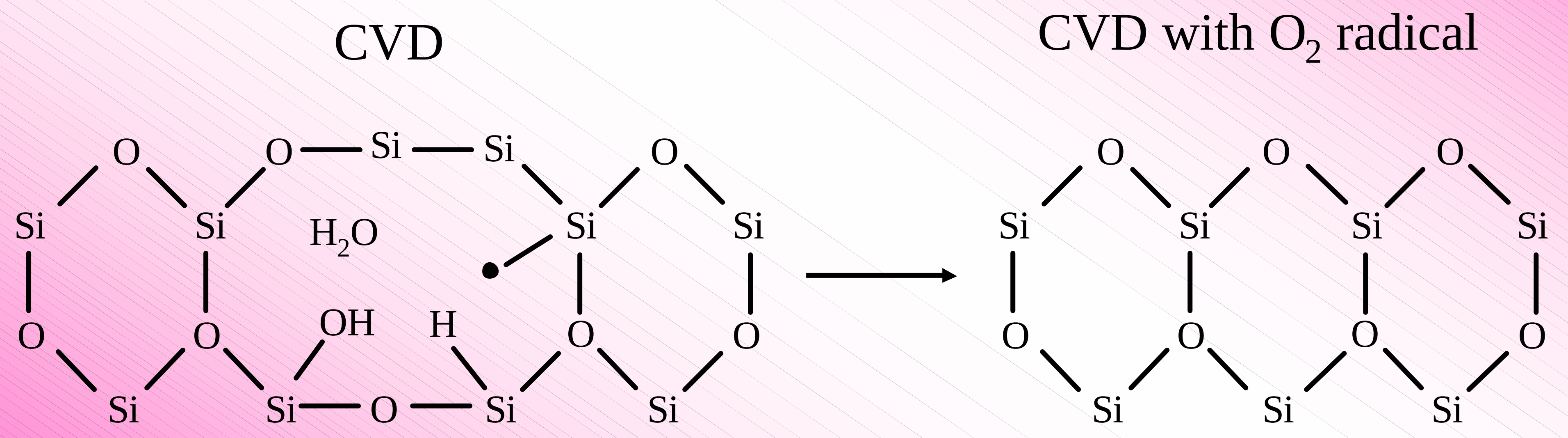
Fig. 8. The surface roughness of oxide film obtained by AFM.

The surface roughness was not change with O_2 radical treatment.

5. discussion

O_2 radical treatment cause CVD silicon oxide film that

density increases.
variance of bonding angle decreases.
amounts of H_2O desorption decreases.
leak current decreases.



The Si-O network would be compact and defects would be erased by O_2 radical treatments. Therefore leak current would be reduced.

6. conclusion

- X-ray reflectivity study is very powerful tool to estimate film density and surface roughness of silicon oxide thin films.

- O_2 radical treatment is very effective method to improve the insulation characteristics of CVD silicon oxide films.