Laboratory Scale Hard X-ray Photoelectron Spectroscopy in Comparison with SPring-8

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Motivation

[1] S. Tanuma et al, Surf. Interface Anal., **43** 689 (2011).
[2] M. B. Trzhaskovskaya et al, At. Data Nucl. Data Tables, **77**, 97 (2001).

HAXPES Characteristics

Advantages

1. Non-destructive evaluation
2. Deep detection depth[1]
3. No surface preparation
4. Variable detection depth (TOA)
5. Core level evaluation

Disadvantages

1. Synchrotron radiation facilities required
2. Cross section reduction[2]
3. Development of high voltage analyzer

In recent years,

A laboratory HAXPES equipment with 9.25 keV X-ray source was developed.

Accessibility is greatly improved.

In this study…

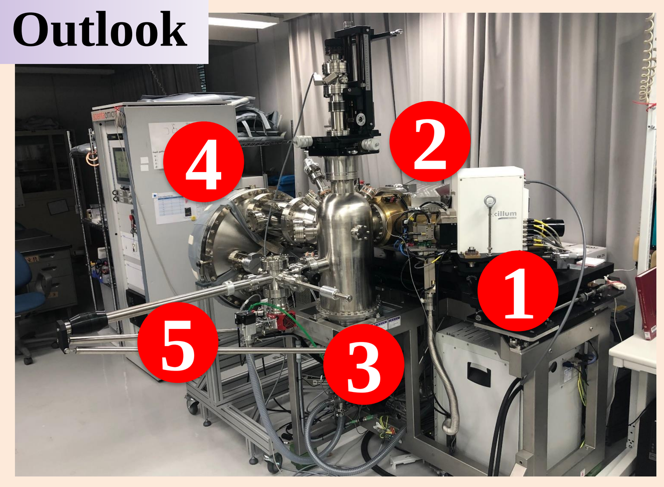
Present the feasibility of the laboratory HAXPES

i. **Clarify basic characteristics**
ii. **Evaluate samples in various fields**

Experiment

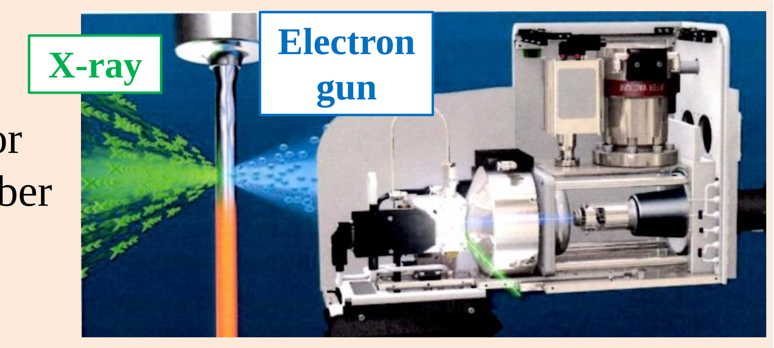
Laboratory HAXPES

Outlook



Components

1. X-ray Source
2. Monochromator
3. Analysis chamber
4. Detector
5. Load Lock



Vacuum Level

Analysis chamber: 1.0 x 10^{-10} mbar
Load Lock: 1.0 x 10^{-7} mbar

X-Ray Conditions [3]

- X-Ray Source : Liquid Gallium **9.25 keV**
- Photon Counts : 6.8 x 10^{8} photon/s (at sample)
- X-ray Spot Size : 30(V) x 45(H) μm^2 (at sample)
- Electron Gun
  Power : 20 – 250 W
  Spot Size : 20 (V) x 80(H) μm^2 (at Liquid Ga)

SPring-8 BL46XU

- X-Ray Energy : **7939 eV**
- Photon Counts : 1.0 x 10^{13} photon/s (at sample)
- X-ray Spot Size : ~ 30 μm^2 (at sample)
- Take-off Angle (TOA) : 80

XPS (AlKα)

Thermo Scientific K-ALPHA

- X-Ray Energy : AlKα **1486.6 eV**
- X-ray Spot Size : 30-400 μm^2 (at sample)
- Take-off Angle (TOA) : 90

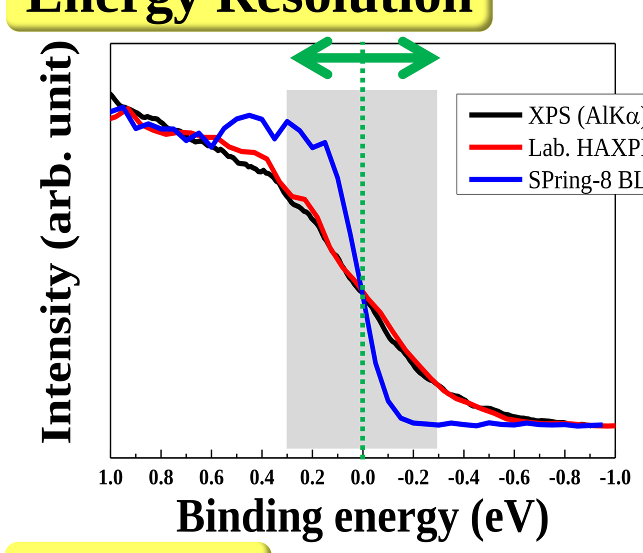
[3] A. Regoutz et al, Rev. Sci. Instrum., **89** 073105 (2018).

Results and Discussion

[4] M. P. Seah et al., Surf. Interface Anal. 35,514 (2003).
[5] K. Usuda et al., IEEE 2nd EDTM Conf., 2018, p. 19.
[6] S. Dahle et al., Plasma Chem. Plasma Process., **32**, 1109 (2012).

Basic Characteristics

Energy Resolution

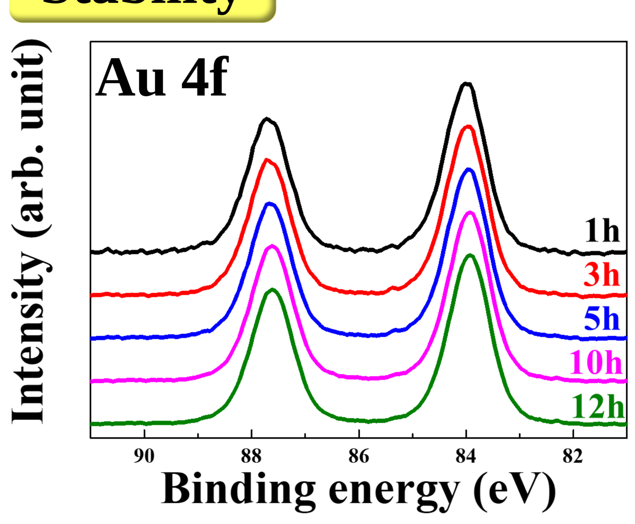


	Energy Resolution (eV)	Measurement Time (min)
XPS (AlKα)	0.63	50
Lab. HAXPES	0.62	130
SPring-8 BL46XU	0.24	10

Pass energy XPS: 50 eV, HAXPES: 200 eV

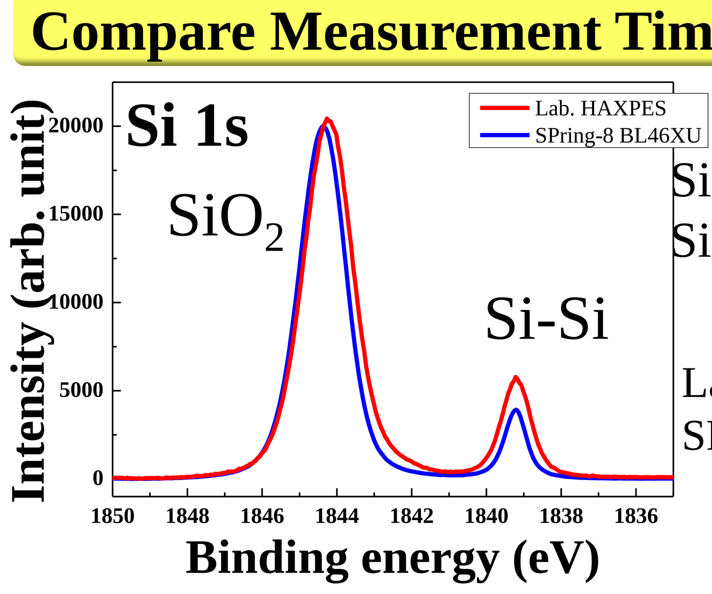
Energy Resolutions
Lab. HAXPES = XPS (AlKα) > SPring-8 BL46XU

Stability



✓ High stability
✓ Maintenance free for more than six months

Compare Measurement Time



Pass energy : 200 eV
SiO_2/c-Si
SiO_2 thickness: 30.0 nm

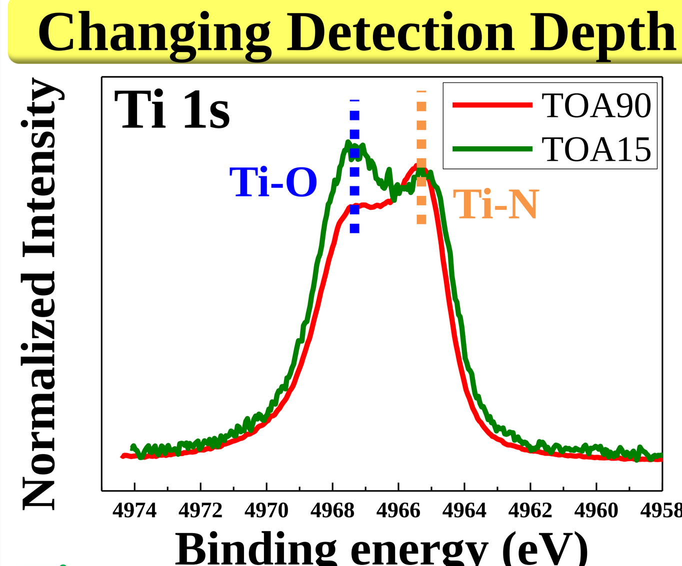
Measurement Time
Lab. HAXPES: 230 min
SPring-8 BL46XU: 20 min

Comparable spectrum to synchrotron is obtained with reasonable time.

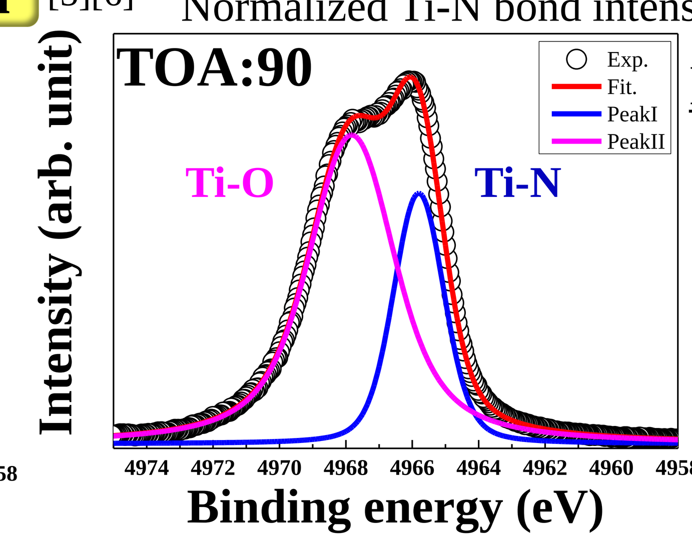
TiN/Si Sample

Sample TiN/c-Si TOA90: ~39 nm, TOA15: ~10 nm
TiN film thickness: 30 nm deposited by sputter

Changing Detection Depth [5][6]



Normalized Ti-N bond intensity

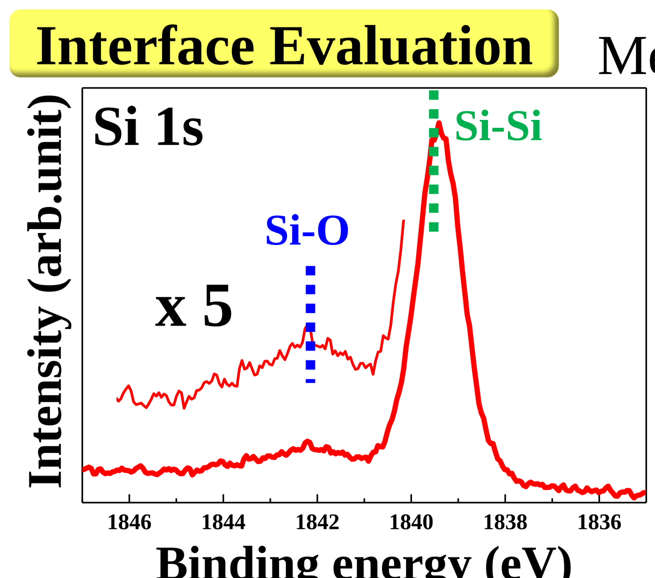


Measurement Time
TOA90: 20 min
TOA15: 140 min

Ti 1s spectrum

✓ TiN film mainly consist of Ti-N and Ti-O bond.
✓ Ti-O bonds exist on the sample surface.

Interface Evaluation Measurement Time: 230 min



Si-Si bond (bulk component) + Si-O bond

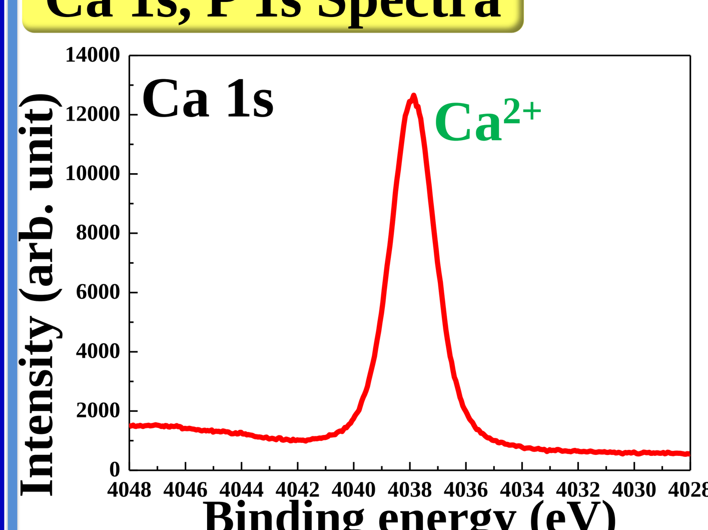
Si-O bond is between TiN film and Si substrate.

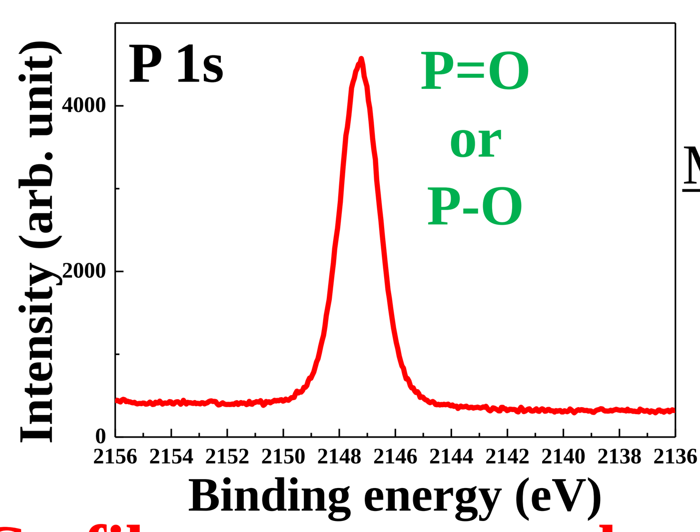
Non-destructive buried interface and film evaluation

Organic Material

Sample Hydroxyapatite: $Ca_{10}(PO_4)_6(OH)_2$
one of the main components of bone
Cu film deposition

Ca 1s, P 1s Spectra





Measurement Time
Ca 1s: 20 min
P 1s: 20 min

Spectra through thin Cu film to suppress charge-up effect

Future Plan

✓ Creation of a core level database for chemical bonding states evaluation.
✓ Examination of parameters such as relative sensitivity factor in GaKα X-ray source for quantitative evaluation.
✓ Evaluation on interface chemical bonding states by *in-situ* observation.

Conclusions

Present the feasibility of the laboratory HAXPES

- Sufficient resolution with reasonable measurement time.
- Non-destructive evaluation for buried interface and thin film in semiconductor material without synchrotron facility.
- Adoptable for insulator material with suppressing charge-up effect.

We can easily evaluate buried interface a wide variety of material

Quick feedback accelerate the research.

Acknowledgments

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