Probing the Nature of Interfacial States in NO Passivated 4H-SiC/SiO_2 Structures using TEM-EELS and XPS*

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[5] U.S. Army Research Laboratory

THE DEPARTMENT *of*
MATERIALS SCIENCE AND ENGINEERING

Outline

- Motivation behind analytical microscopy of SiC microelectronics
  - Impacts of NO post-annealing

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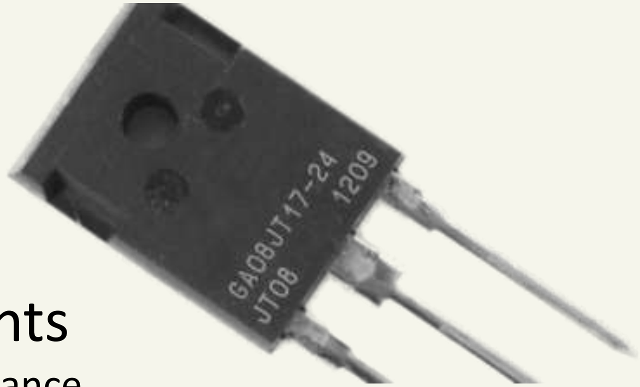
  - Hyperspectral imaging, machine learning techniques for signal deconvolution
  - Significant changes in interface character after NO-anneal
- Correlation with XPS results
  - What differences are observed with an NO-anneal?

Motivation and background



- SiC: Very promising for high temperature, high power, and high radiation environments
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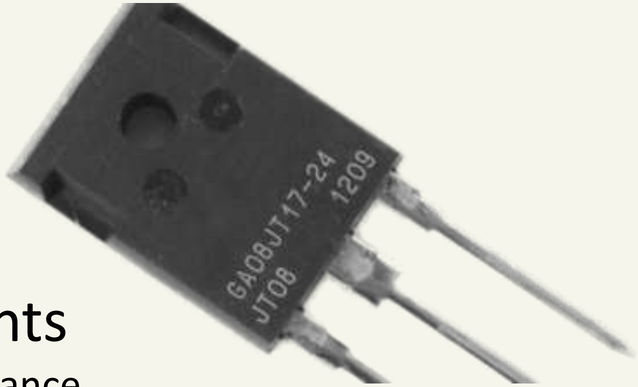

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  - Carrier mobility
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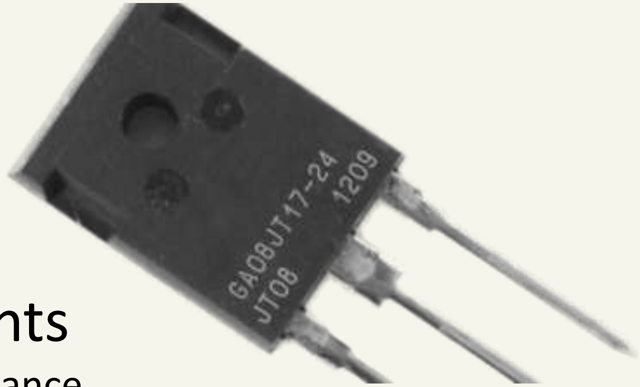


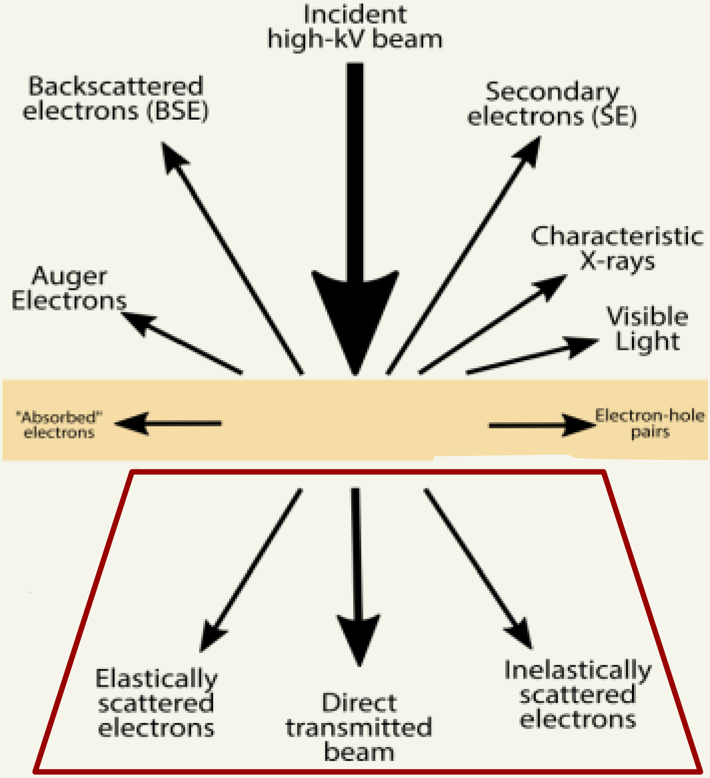
- SiC: Very promising for high temperature, high power, and high radiation environments
  - NO post oxidation anneal (POA) drastically improves performance
- Electrically active defects limit:
  - Carrier mobility
  - Reliability
  - Device stability
- What is the true nature of the interface, and how do our processing techniques really affect it?
  - Distinct transition region (EELS)[1, 2]
  - Abrupt transition (XPS, MEIS, etc.)[3 – 4]
  - What is NO post oxidation annealing really changing about the interface structurally and chemically?

[1] J. Taillon, L. Salamanca-Riba, *et al.*, J. Appl. Phys. 113, 044517 (2013).
[2] Chang, K. C. *et al*. J. Appl. Phys. 97, 104920 (2005).
[3] H. Watanabe, *et al.*, Appl. Phys. Lett., 99(2), 021907 (2011).
[4] X. Zhu, *et al.*, Appl. Phys. Lett., 97(7), 071908 (2010).

TEM-EELS EXPERIMENTS

Electron Energy Loss Spectroscopy (EELS)

- E-beam has many interactions with specimen:



Adapted from Williams and Carter, *Transmission Electron Microscopy*, (2009).

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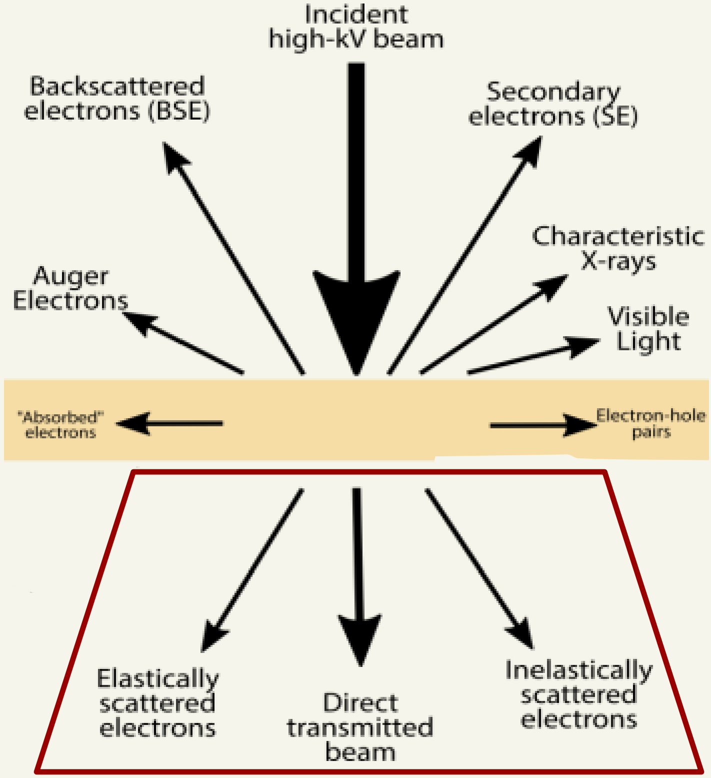
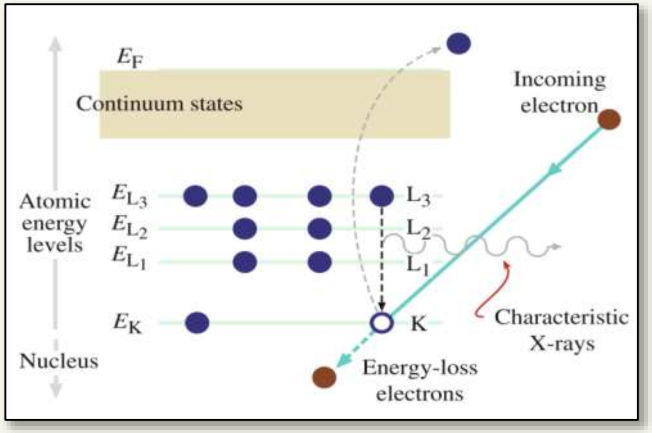
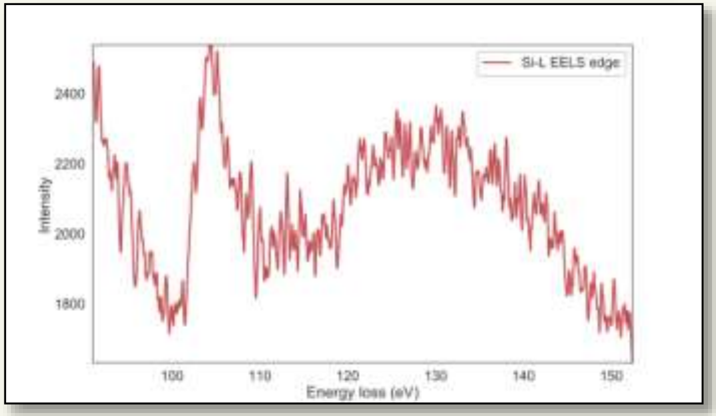


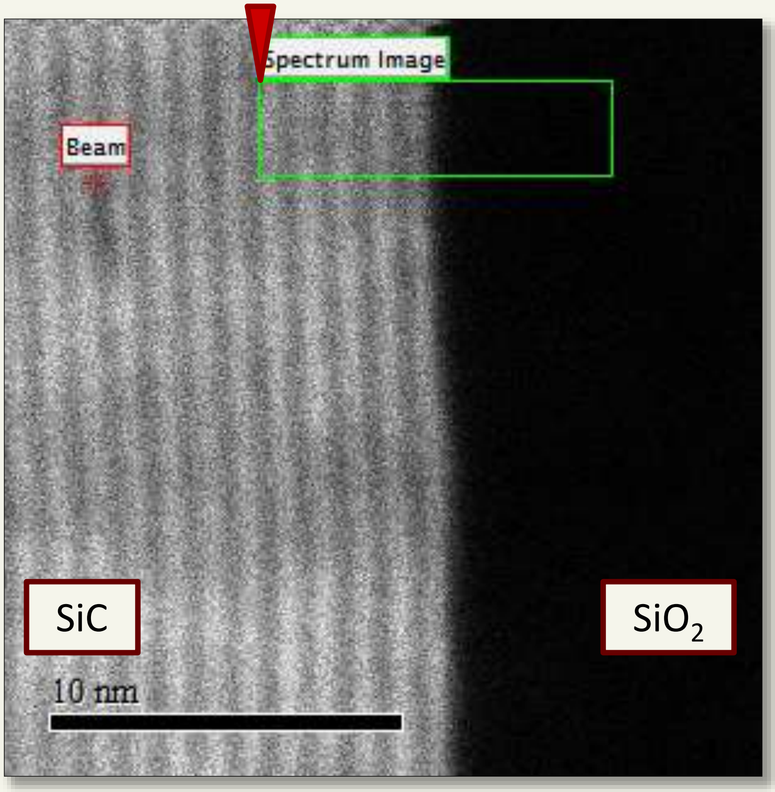
Adapted from Williams and Carter, *Transmission Electron Microscopy*, (2009).

- EELS is measurement of the energy lost when an electron interacts with the sample:



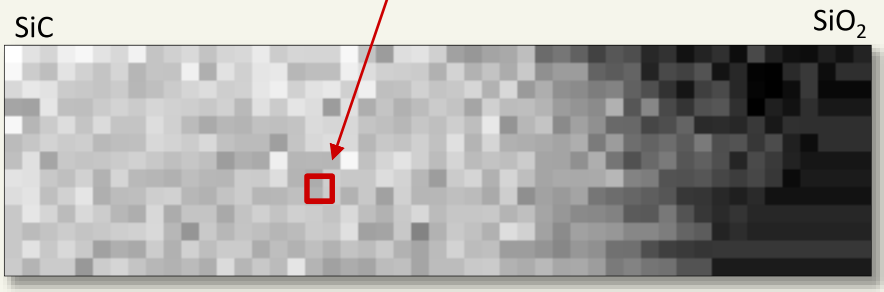


EELS Spectrum Imaging



HAADF Survey Image

One spectrum per pixel

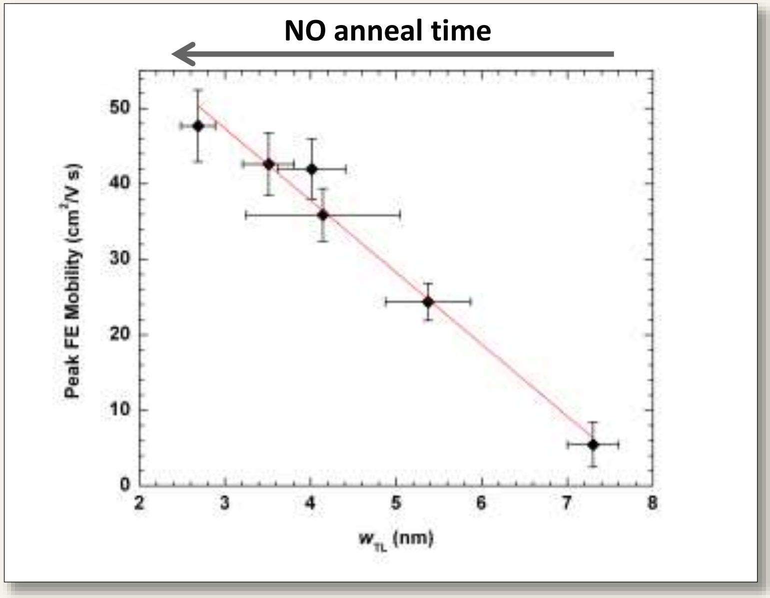


EELS Spectrum Image

Simultaneous HAADF Signal

NO-anneal effects (previous results)

- w_{TL} correlates inverse-linearly μ_{FE}
  - Also correlates with decreased trap density: John Rozen, *et al*. IEEE Trans. Elec. Dev. (2011).
- NO-anneal removes/passivates mobility-limiting defects
  - Compositionally and electronically
- **Conclusions:**
  - w_{TL} decreases with increasing NO anneal time
    - New chemical shift of Si-$L_{2,3}$ edge onset was most reliable method
    - No excess C on either side of interface

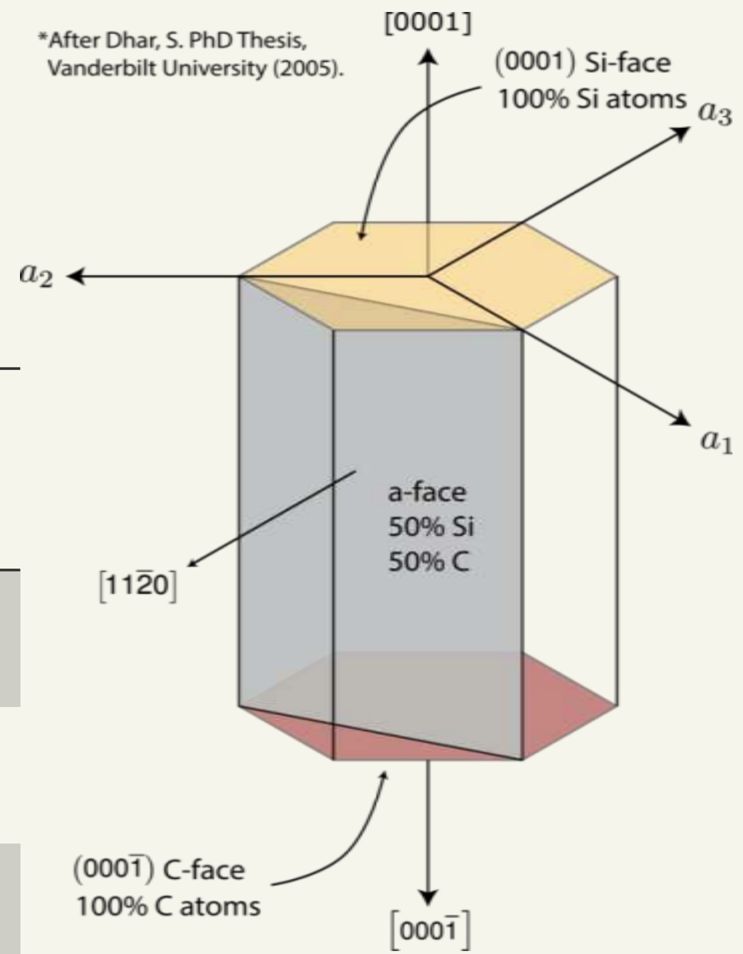


J. Taillon, L. Salamanca-Riba, *et al.*, *J. Appl. Phys.* **113**, 044517 (2013).

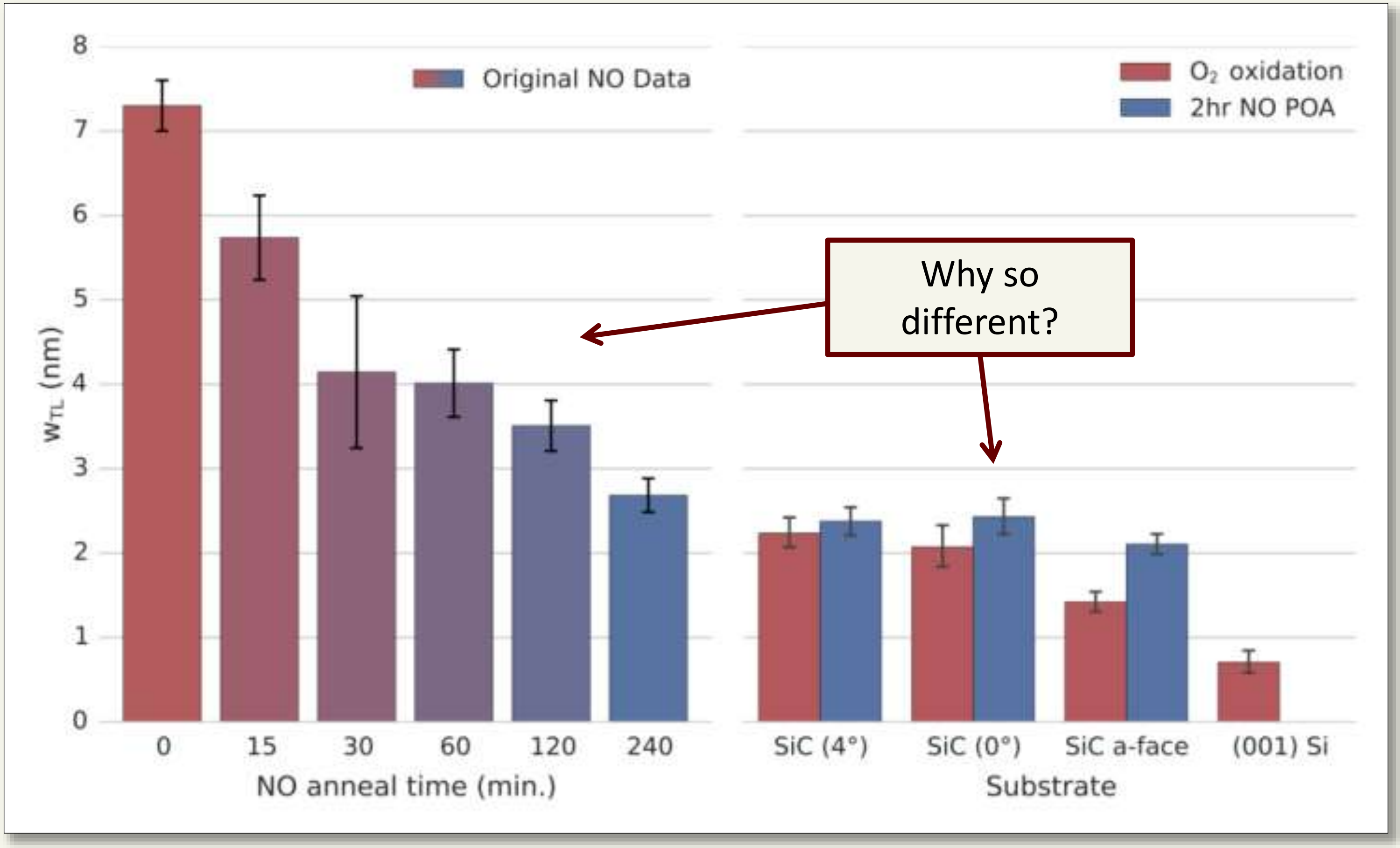
Orientation effects

- 2 x 3 matrix aimed at comparing substrate orientation (and miscut) with processing conditions:
  - NO POA is for 2hr, all SiC substrates are n-type, SiO_2 ~60 nm thick

Samples:	**Oxidation**	**NO POA**
Si-face on-axis	Si-O_2-0	Si-N-0
Si-face miscut (4°)	Si-O_2-4	Si-N-4
a-face on-axis	a-O_2-0	a-N-0



w_{TL} measurements



- Little to no effect of NO annealing
- *Contradicts expectations from previous result*

NEW ANALYSIS TECHNIQUE

Hyperspectral signal decomposition – machine learning

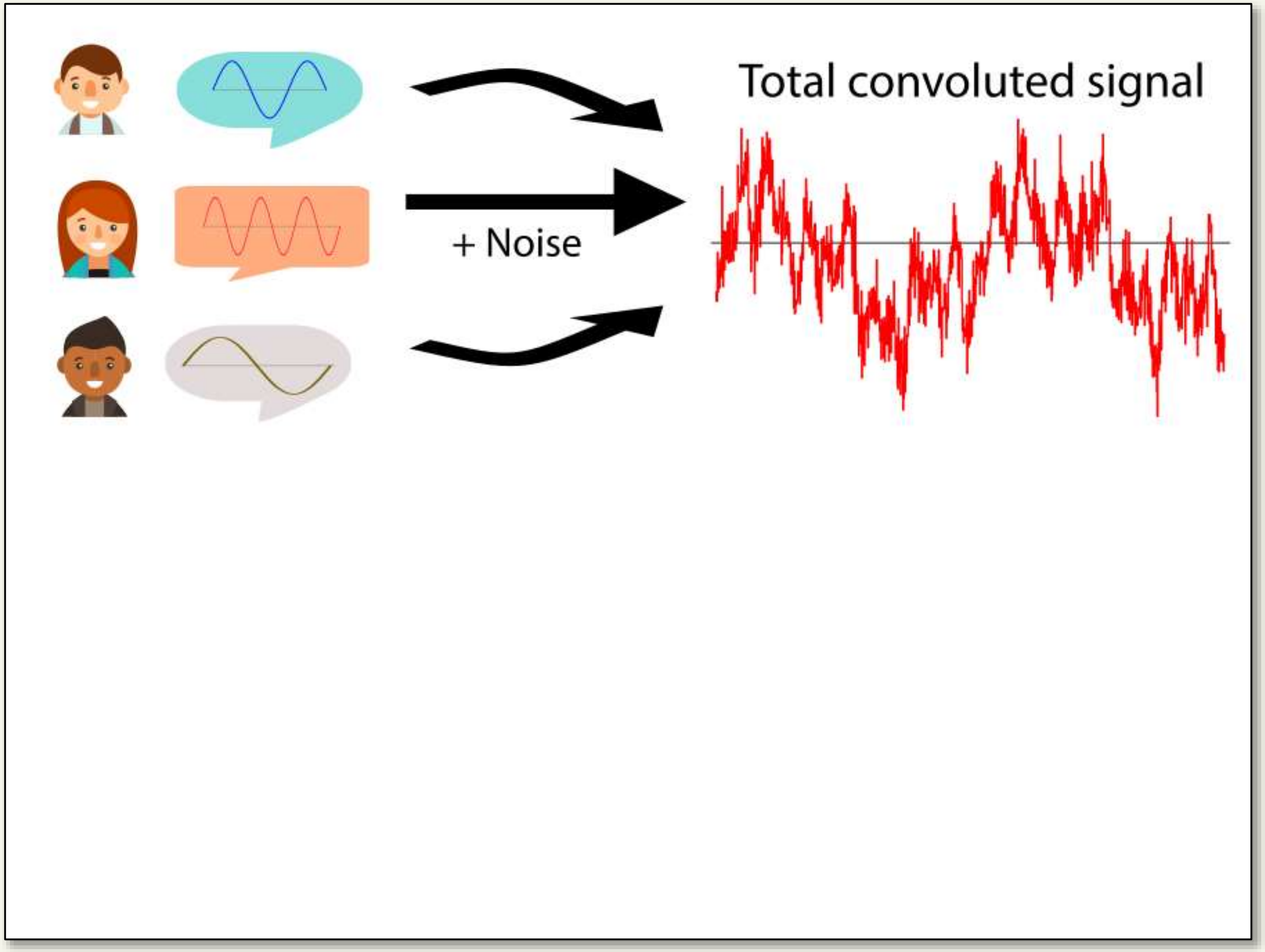
- Si-$L_{2,3}$
- C-K
- O-K

Decomposition analysis

- EELS spectrum imaging as "big data"
  - Machine learning improves sensitivity; highlights most important features

- Non-negative matrix factorization and Blind source separation:
  1) Identify number of important sources
  2) Maximize independence between components
  3) Describe data with as few components as necessary

Decomposition analysis – audio signal example



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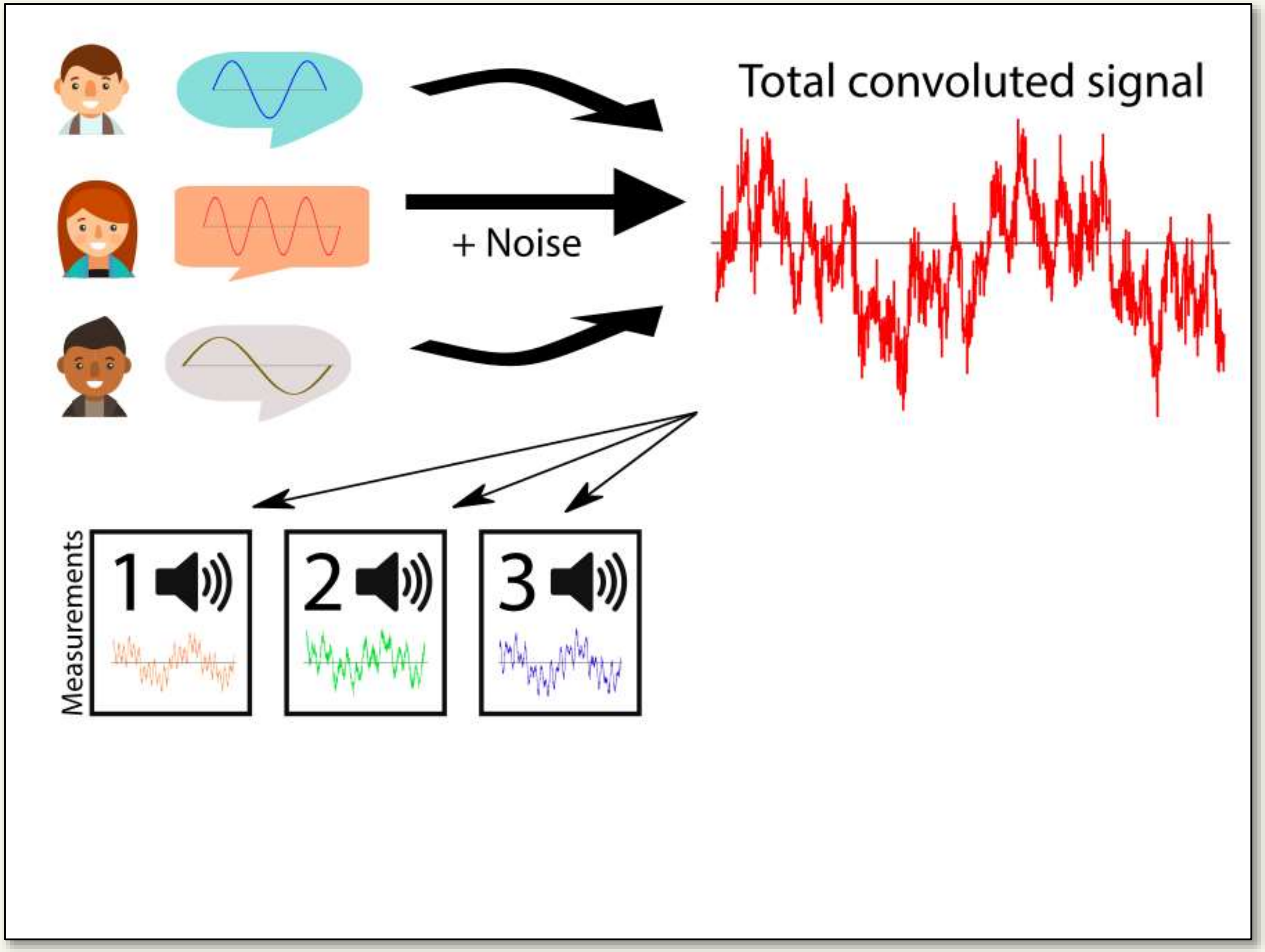

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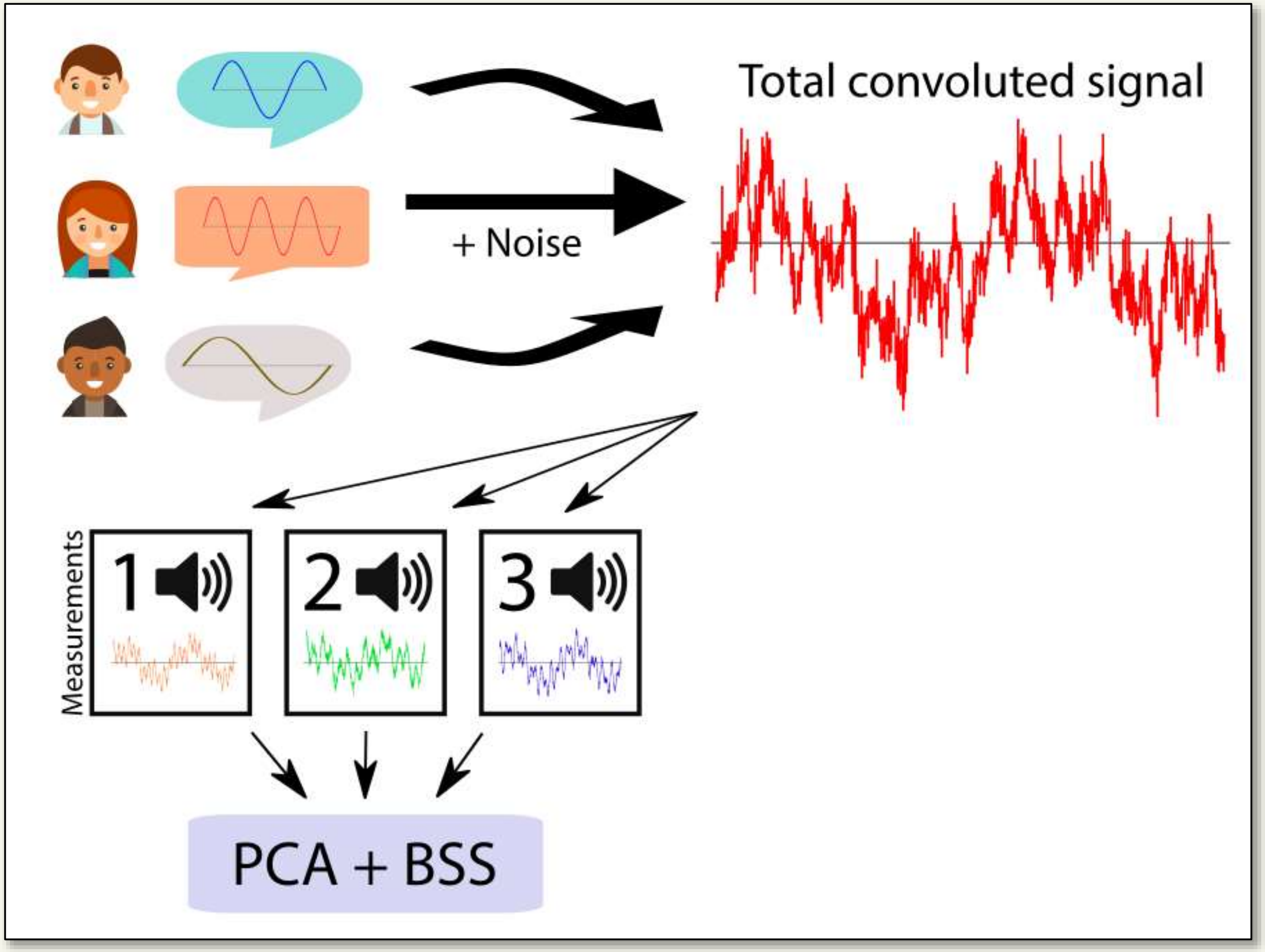

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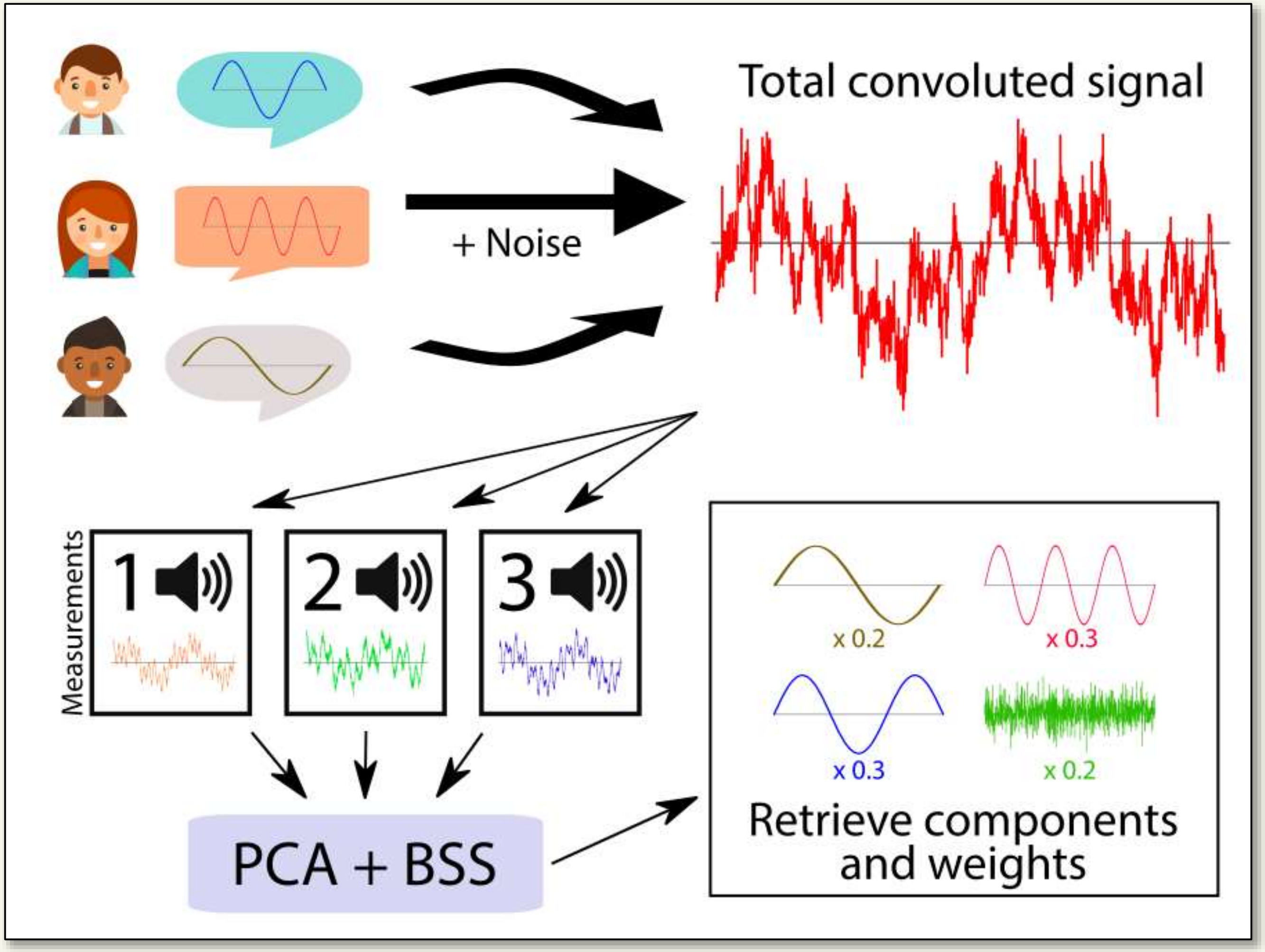
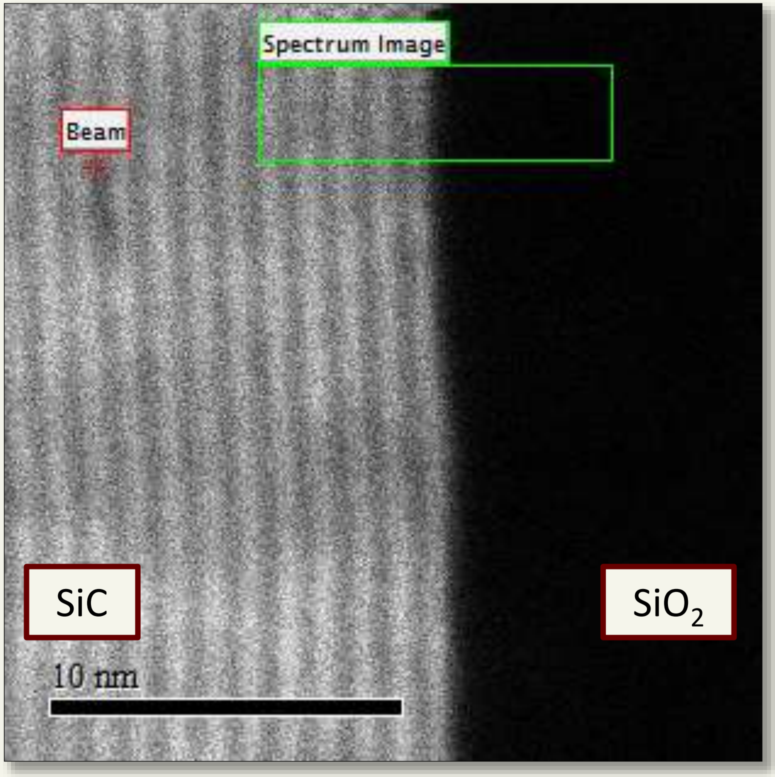


Decomposition analysis in spectrum imaging

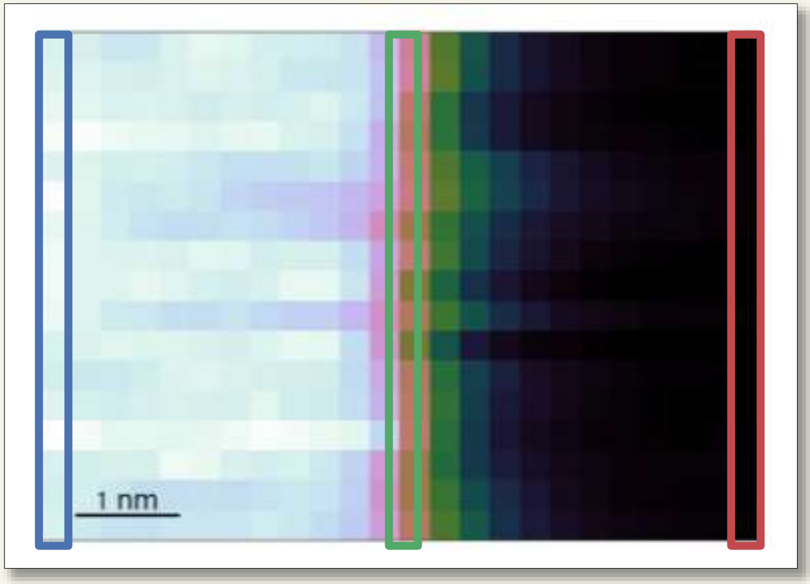


HAADF Survey Image

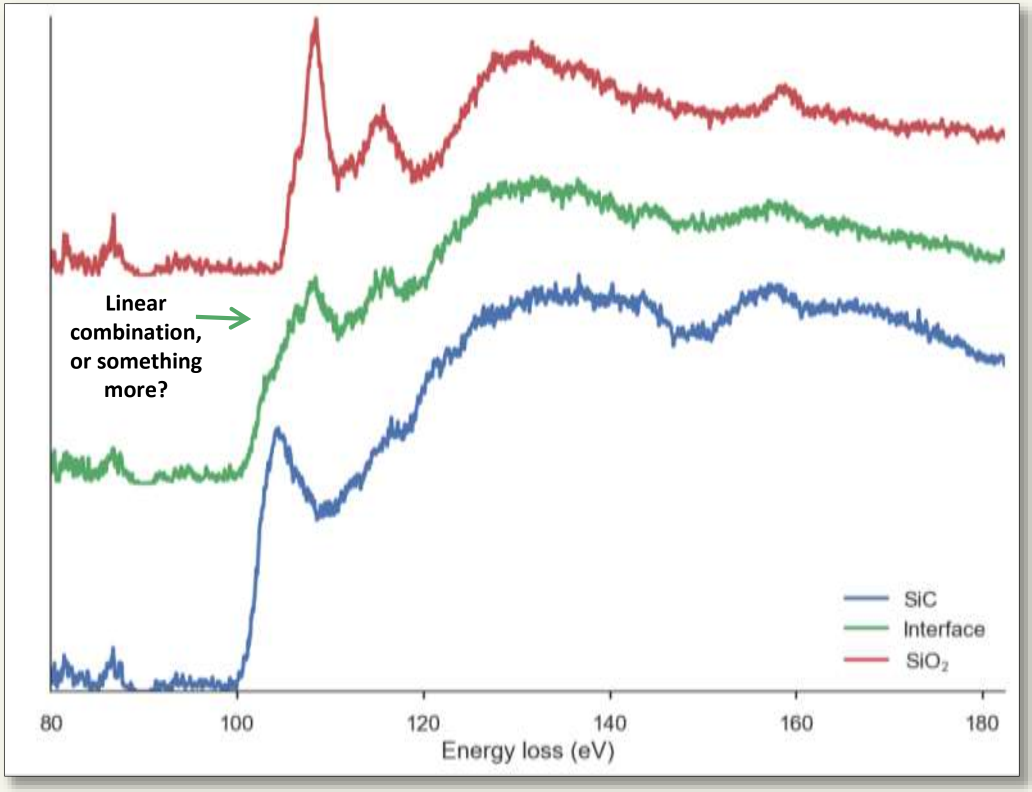
EELS Spectrum Image

- Each pixel of the spectrum image becomes a discrete observation
- More pixels = more sensitivity

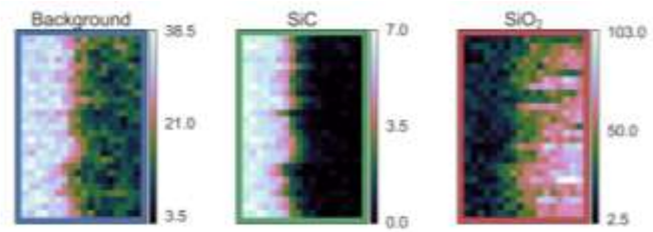
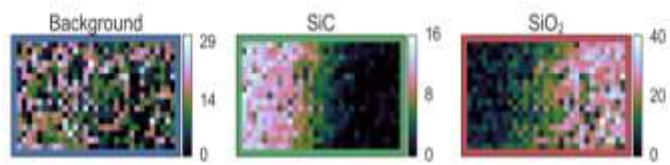
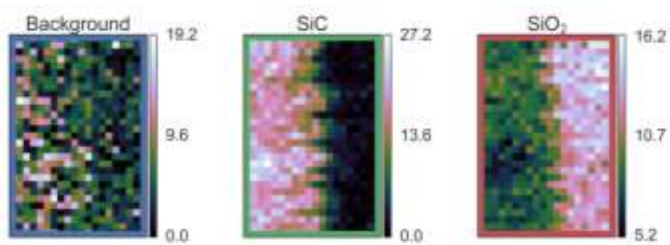
Interface components at NO-annealed interfaces



- Simple sum improves S/N, but cannot detect faint or overlapping signals



Decomposition of Si-$L_{2,3}$ EELS

Face / Treatment	O_2 oxidation	O_2 oxidation + NO POA
Si-face 4° miscut (Si-X-4)		
Si-face no miscut (Si-X-0)		
a-face no miscut (a-X-0)		

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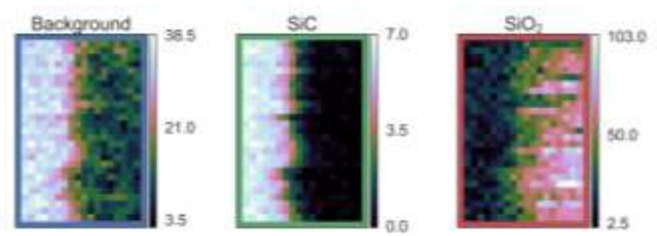
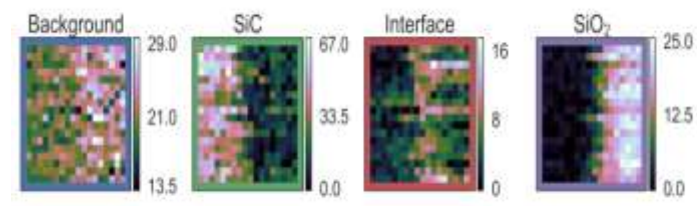
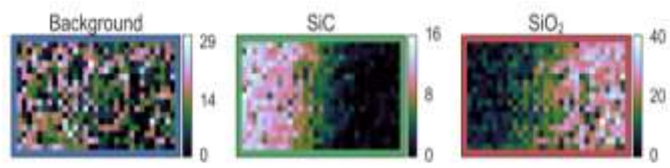
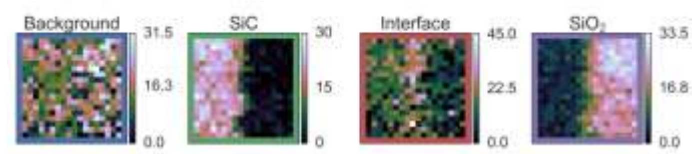
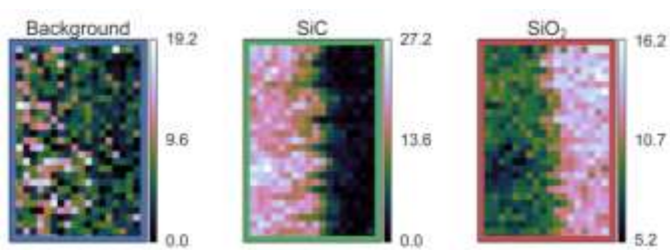
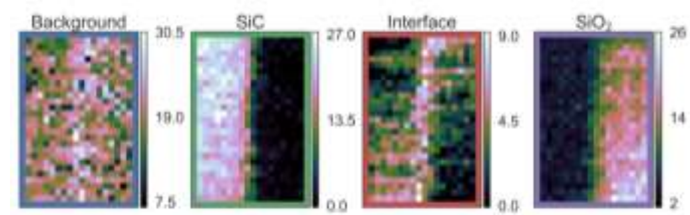
Si-face 4° miscut (Si-X-4)		
Si-face no miscut (Si-X-0)		
a-face no miscut (a-X-0)		

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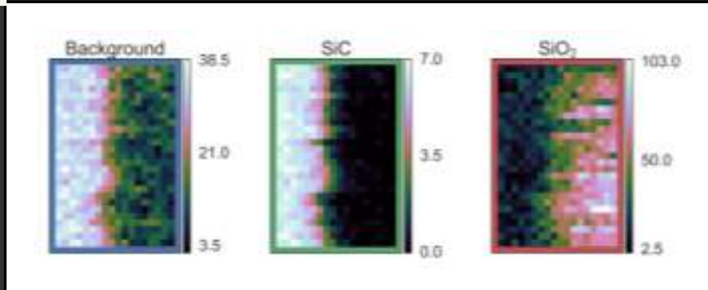
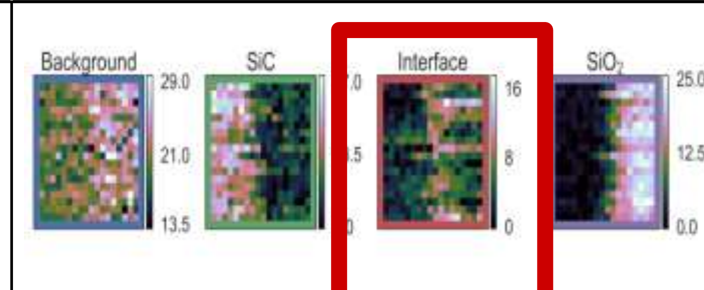
(Si-X-4)</td><td>
</td><td>
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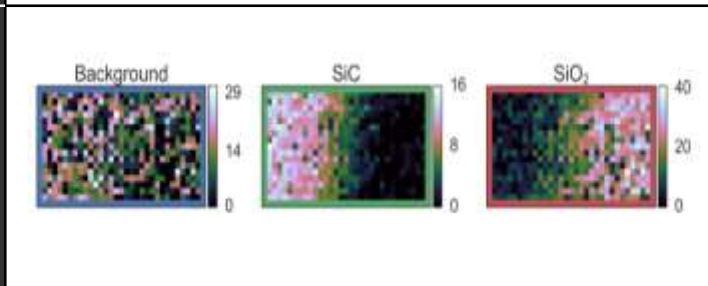
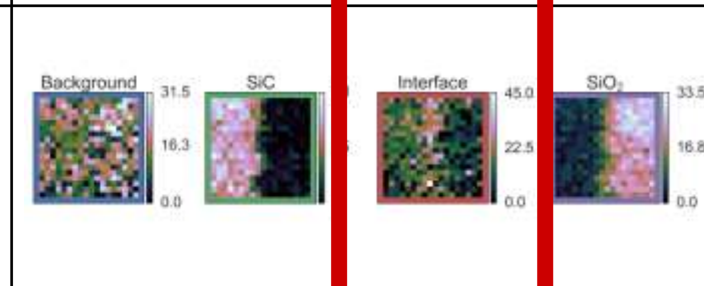
(Si-X-0)</td><td>
</td><td>
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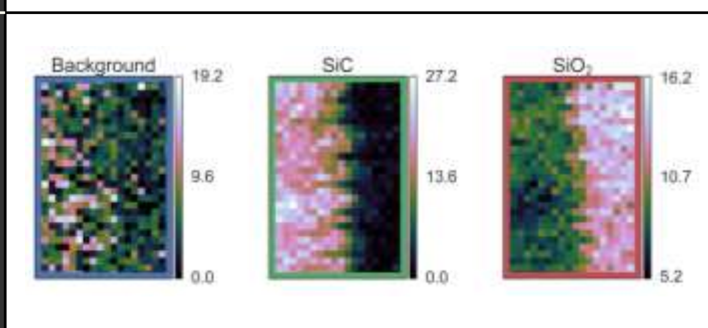
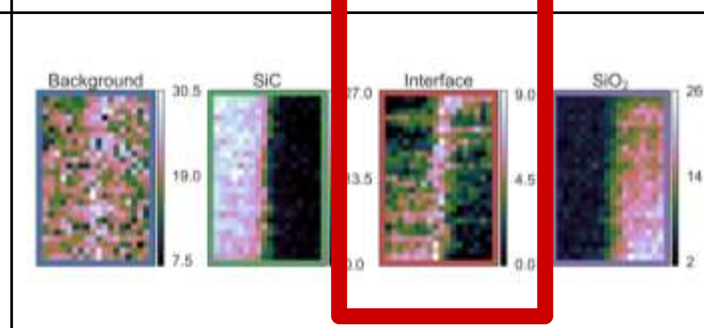
(a-X-0)</td><td>
</td><td>
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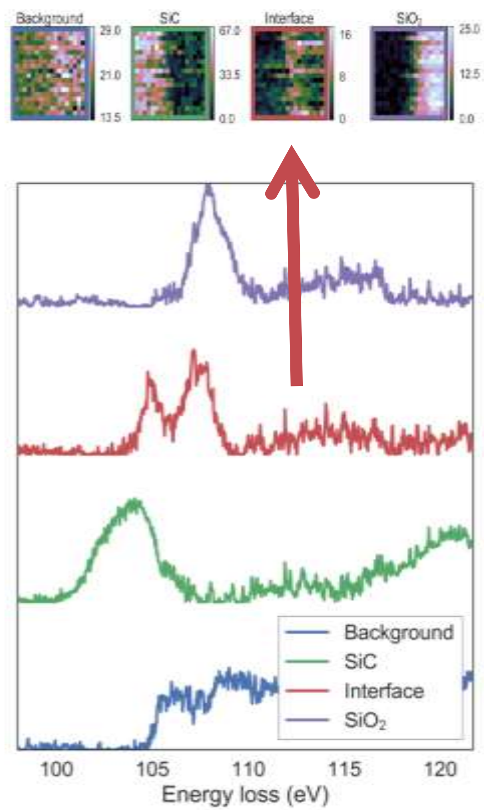
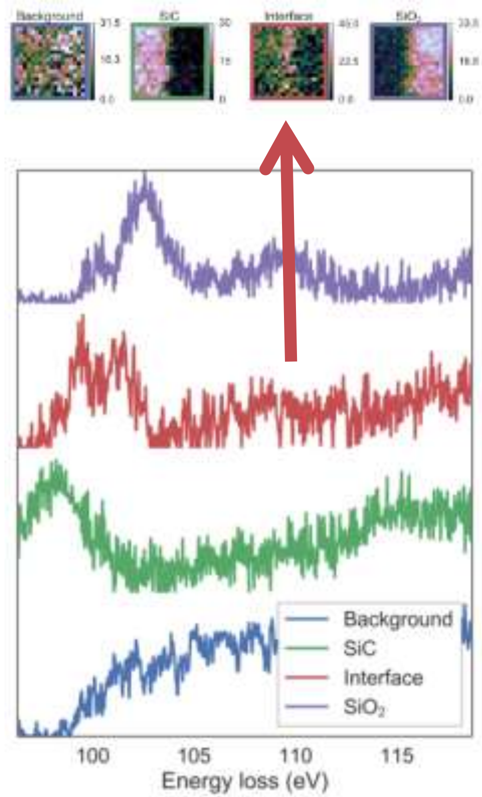
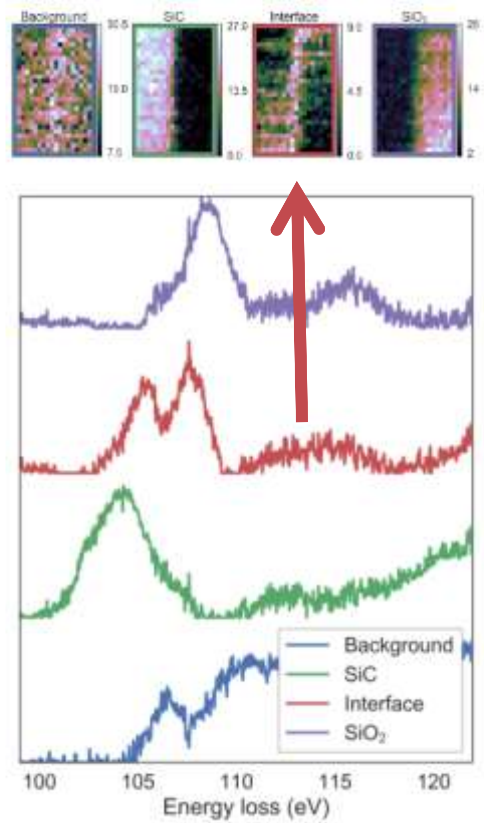
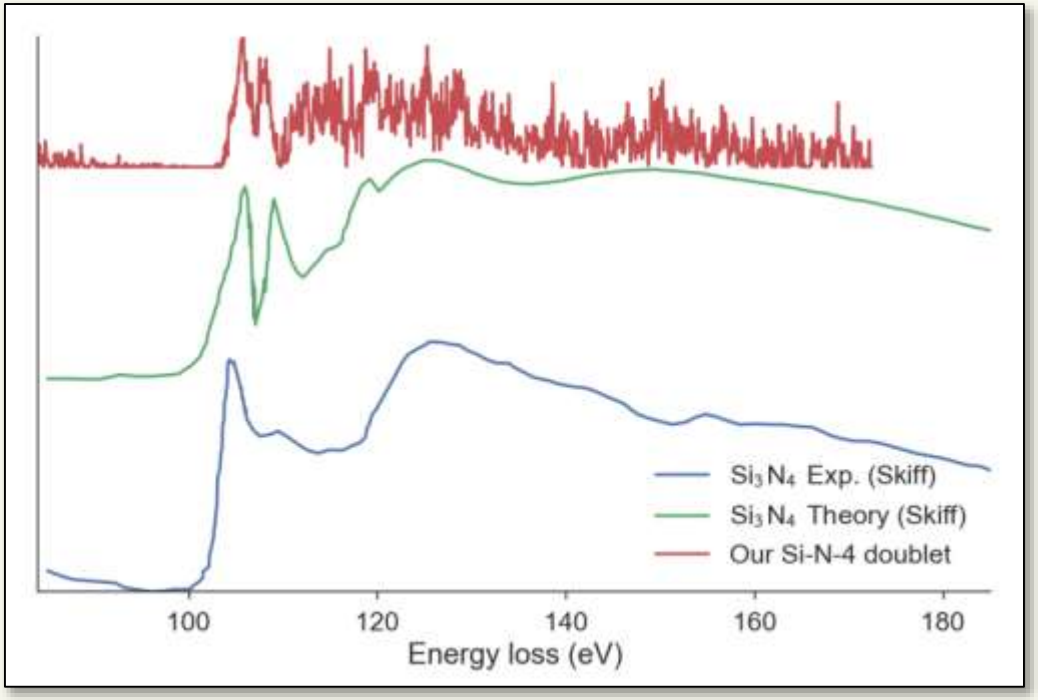
Face / Treatment	Si-face 4° miscut (Si-NO-4)	Si-face no miscut (Si-NO-0)	a-face no miscut (a-NO-0)
Decomposition components			

What could it be?

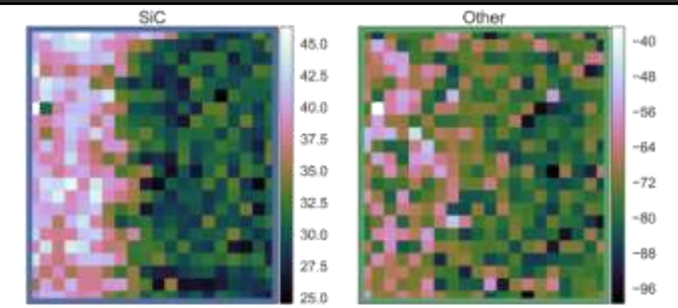
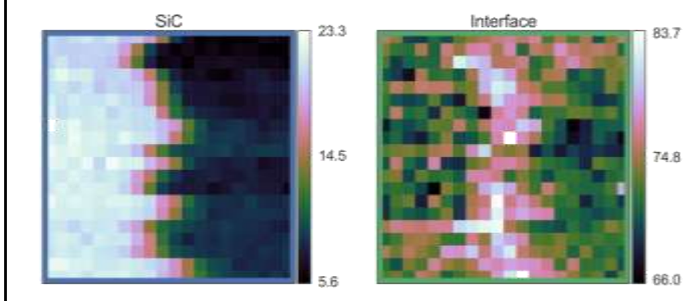
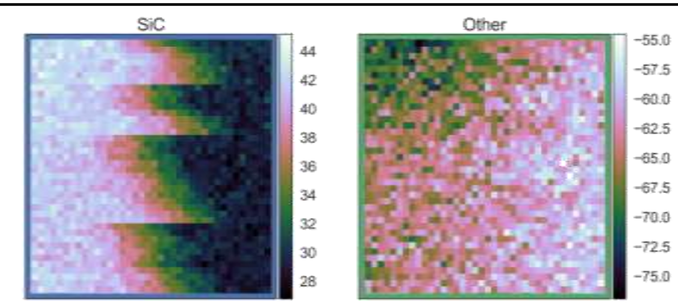
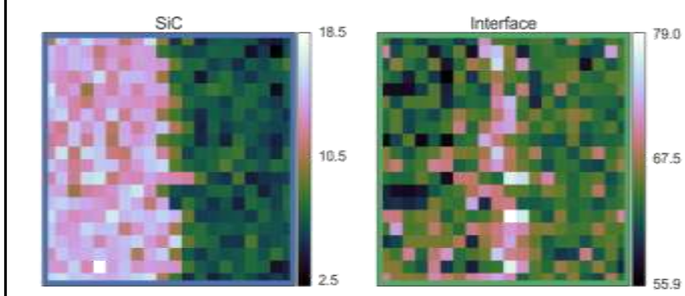
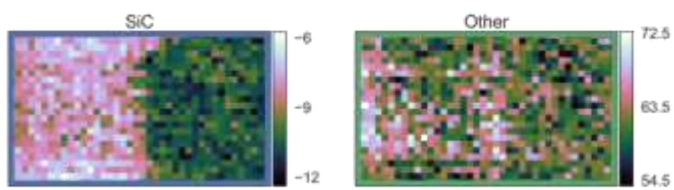
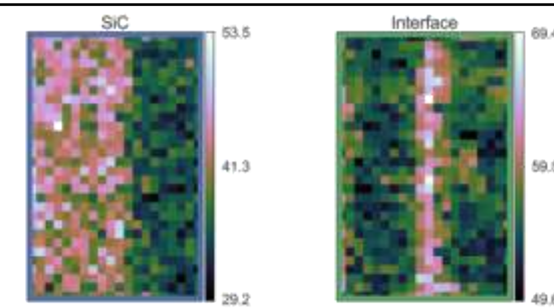
- Si_3N_4 theory and experiment (Skiff et al.)
  - Doublet peak at same energy as our peak
- Effect of N-bonding
  - Si-C-N-O bonding configurations?
  - Evidence of N-bonding at interface
  - DFT modeling will reveal further details

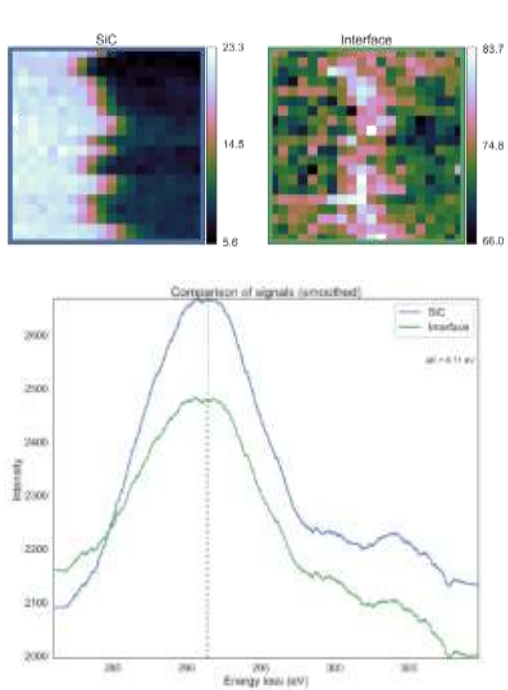
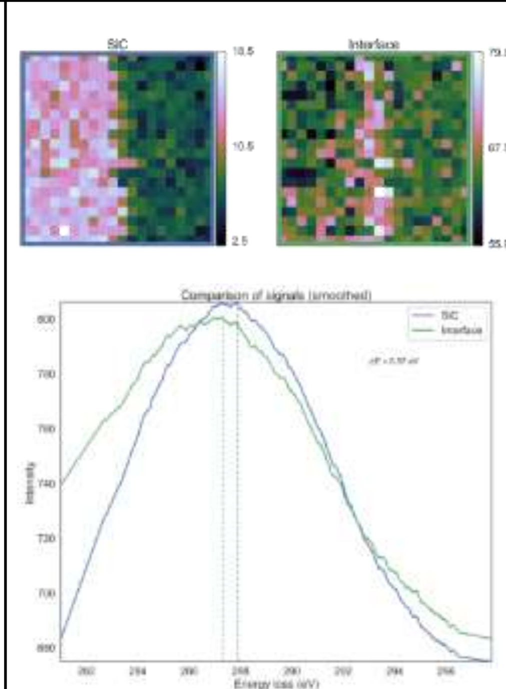
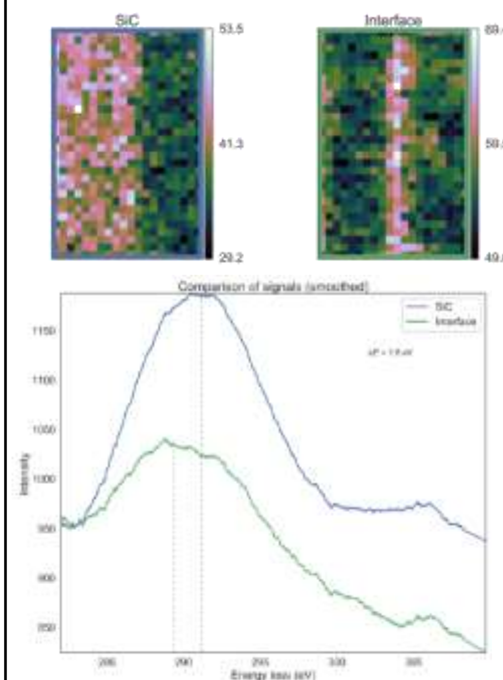


Skiff, W. M., *et al.*, *J. Appl. Phys.* **62,** 2439–2449 (1987).

Decomposition of C-*K* EELS

A. JAMES CLARK SCHOOL OF ENGINEERING

Face / Treatment	O_2 oxidation	O_2 oxidation + NO POA
Si-face 4° miscut (Si-X-4)		
Si-face no miscut (Si-X-0)		
a-face no miscut (a-X-0)		

Face / Treatment	Si-face 4° miscut (Si-NO-4)	Si-face no miscut (Si-NO-0)	a-face no miscut (a-NO-0)
Decomposition components			

- C signal Interface components at slightly lower primary peak energies attributable to possible sp^2 hybridization from N-bonding*

*J. Hu, *et al.*, *Phys. Rev. B* **57**, R3185 (1998).

Decomposition of O-*K* EELS

<table>
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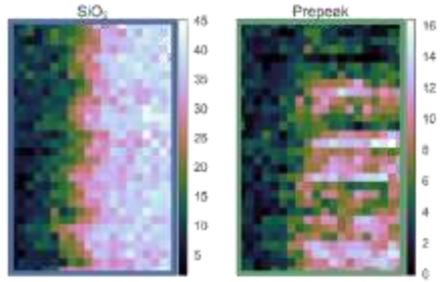
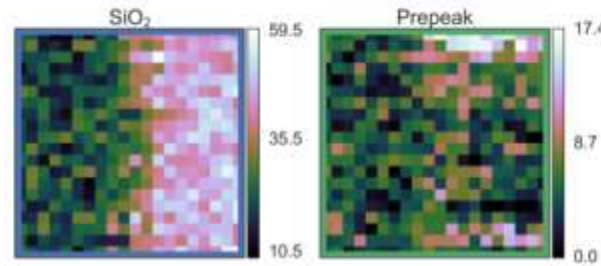
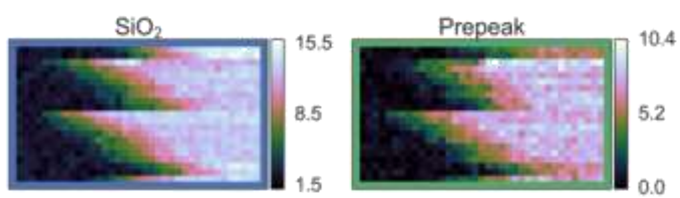
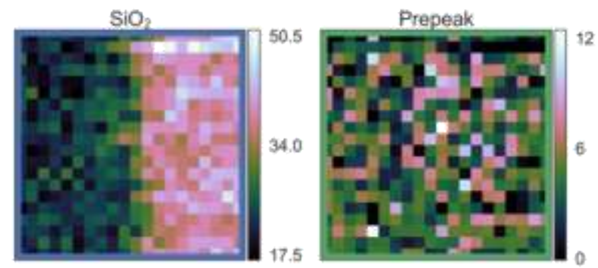
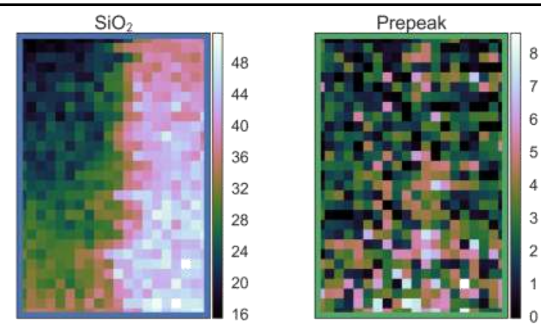
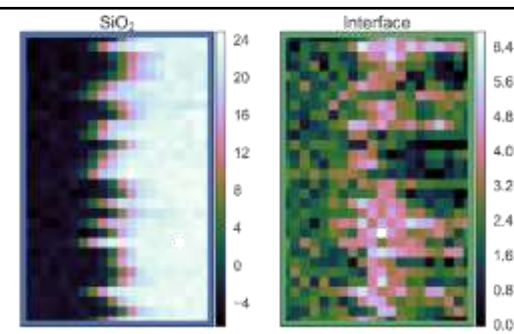
<tr><td>Si-face 4° miscut (Si-X-4)</td><td></td><td></td></tr>
<tr><td>Si-face no miscut (Si-X-0)</td><td></td><td></td></tr>
<tr><td>a-face no miscut (a-X-0)</td><td></td><td></td></tr>
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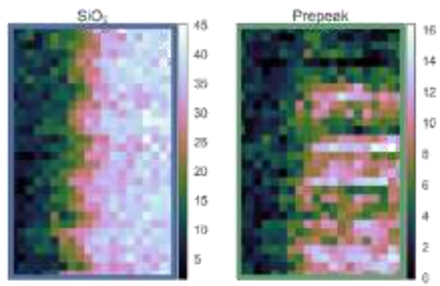
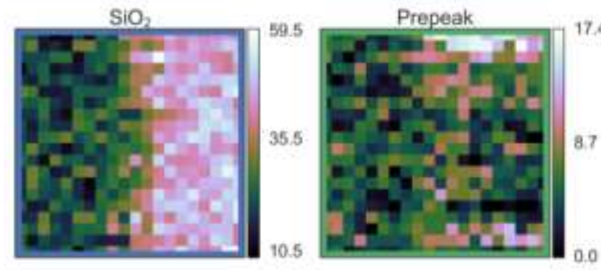
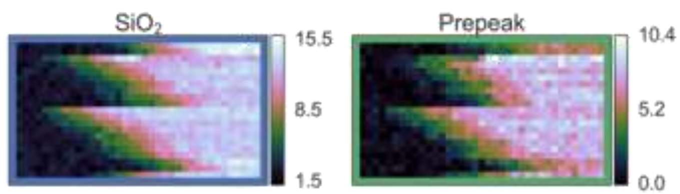
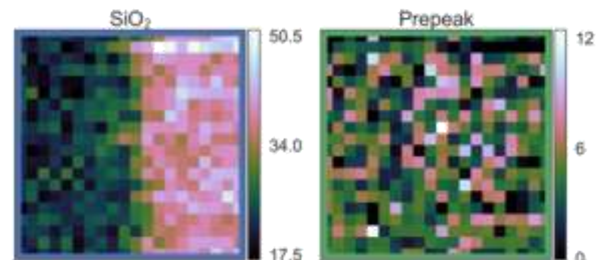
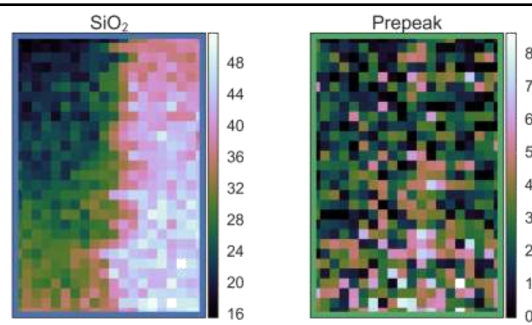
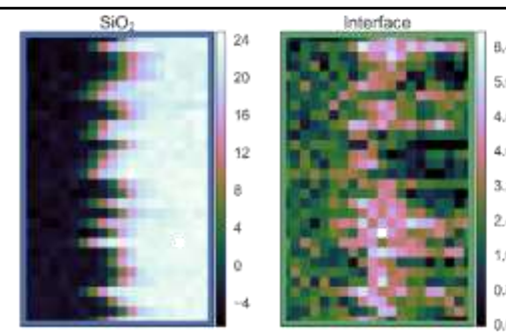
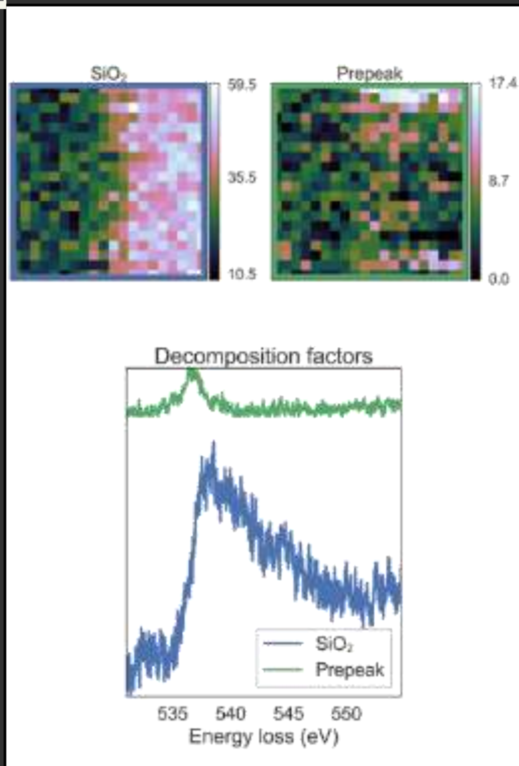
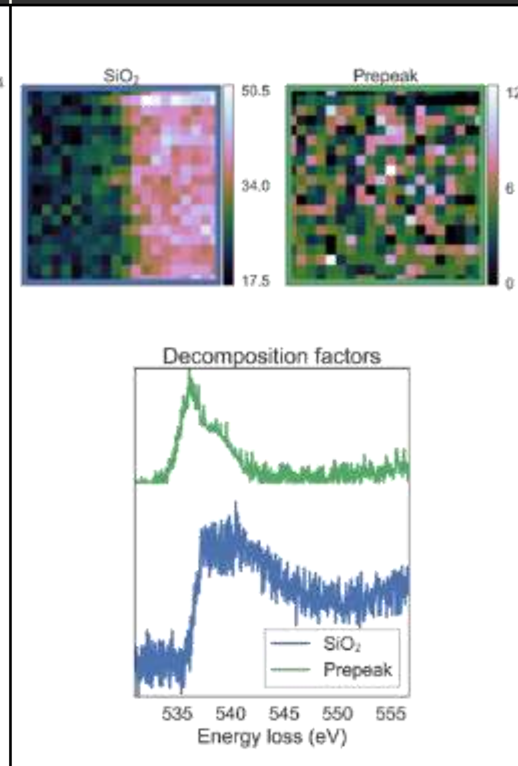
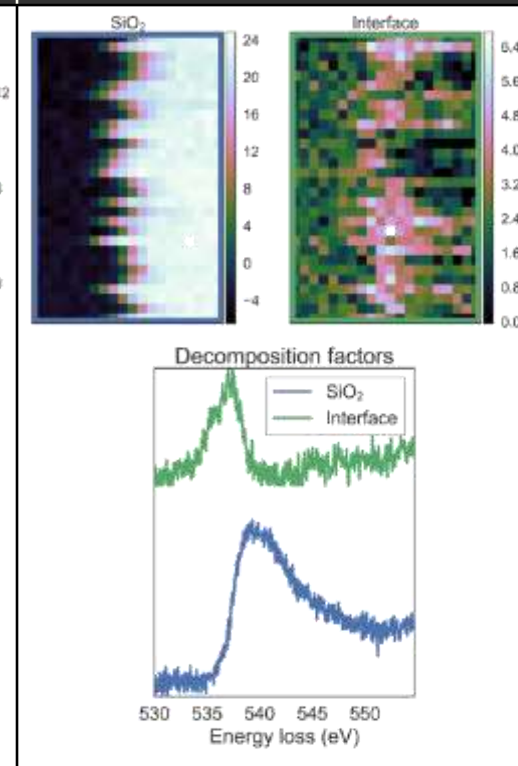
Decomposed O-K edges

Face / Treatment	O_2 oxidation	O_2 oxidation + NO POA
Si-face 4° miscut (Si-X-4)		
Si-face no miscut (Si-X-0)		
a-face no miscut (a-X-0)		

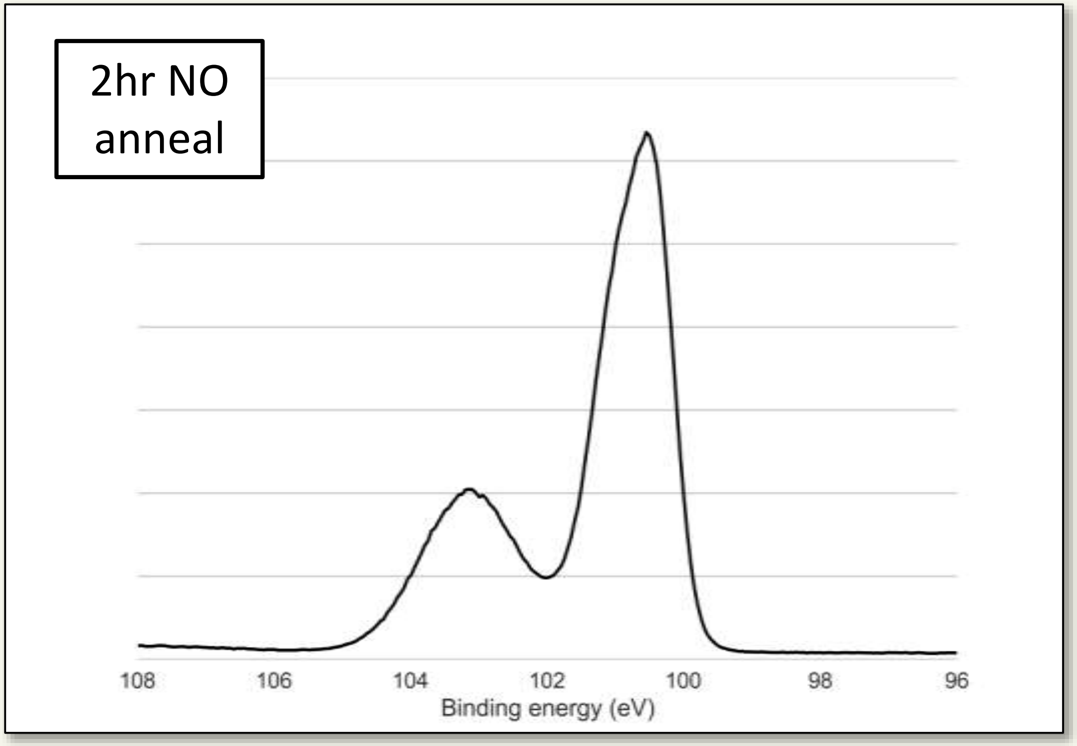
Face / Treatment	Si-face 4° miscut (Si-NO-4)	Si-face no miscut (Si-NO-0)	a-face no miscut (a-NO-0)
Decomposition components			

- All samples have an O-*K* pre-peak feature, but only a-face is localized near the interface
- Indicates defect states near edge of band gap, improving conductivity

XPS DEPTH PROFILING

XPS Interfacial analysis – Si-*2p*

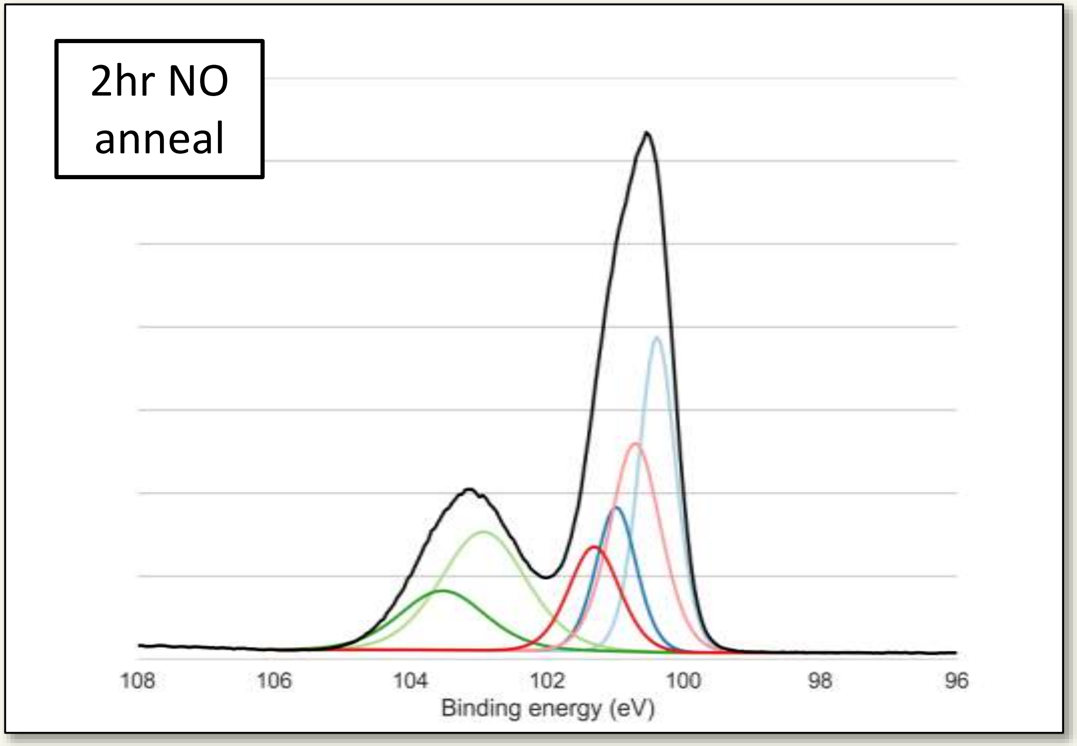
- Can we correlate the EELS?
- XPS is surface-sensitive binding energy measurement
- Measured oxidized and NO-POA samples etched near to the interface



"Raw" Si-*2p*

XPS Interfacial analysis – Si-*2p*

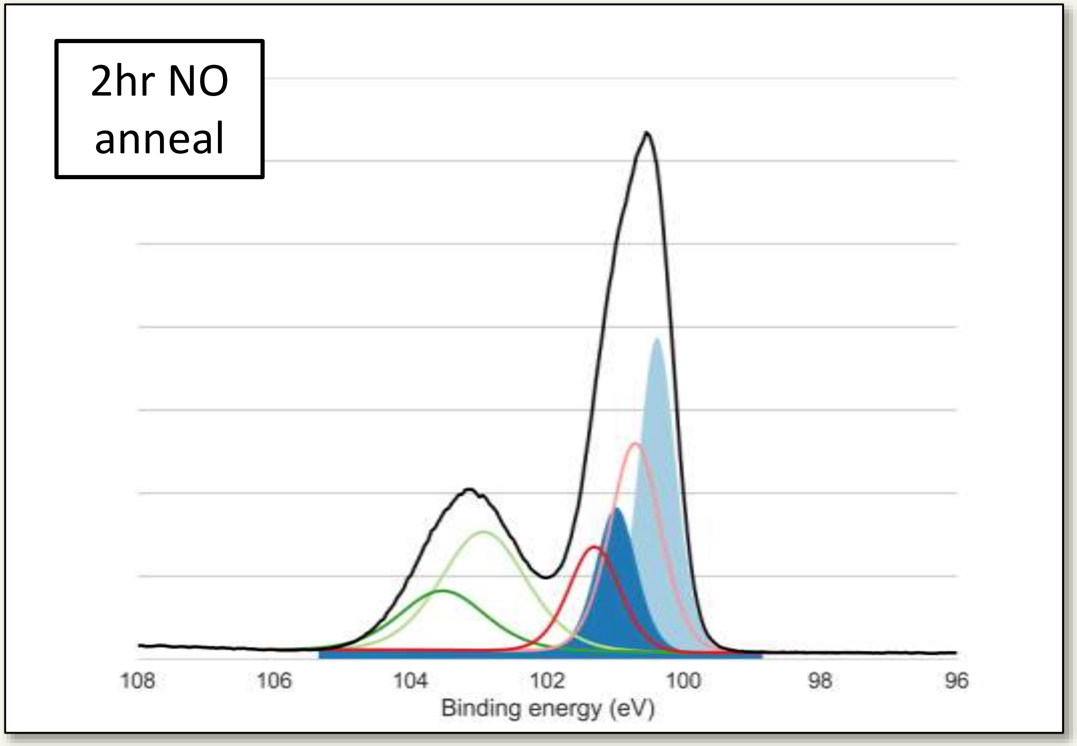
- Can we correlate the EELS?
- XPS is surface-sensitive binding energy measurement
- Measured oxidized and NO-POA samples etched near to the interface



Peak-fitted Si-*2p*

XPS Interfacial analysis – Si-*2p*

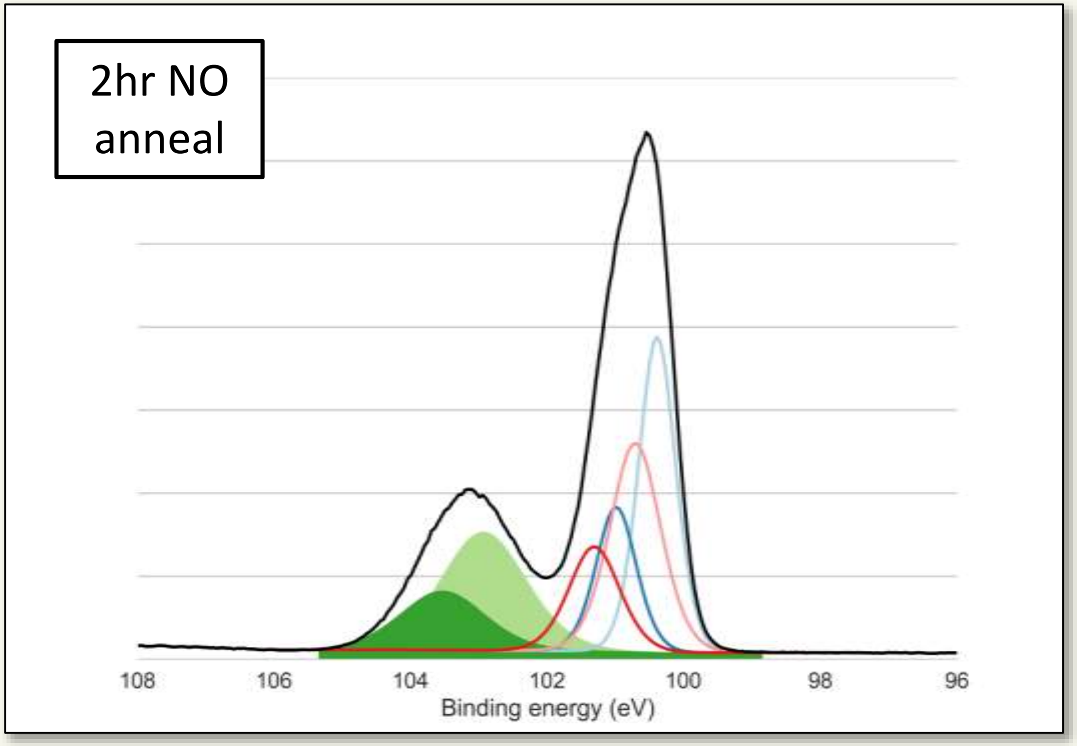
- Can we correlate the EELS?
- XPS is surface-sensitive binding energy measurement
- Measured oxidized and NO-POA samples etched near to the interface



SiC signal

XPS Interfacial analysis – Si-*2p*

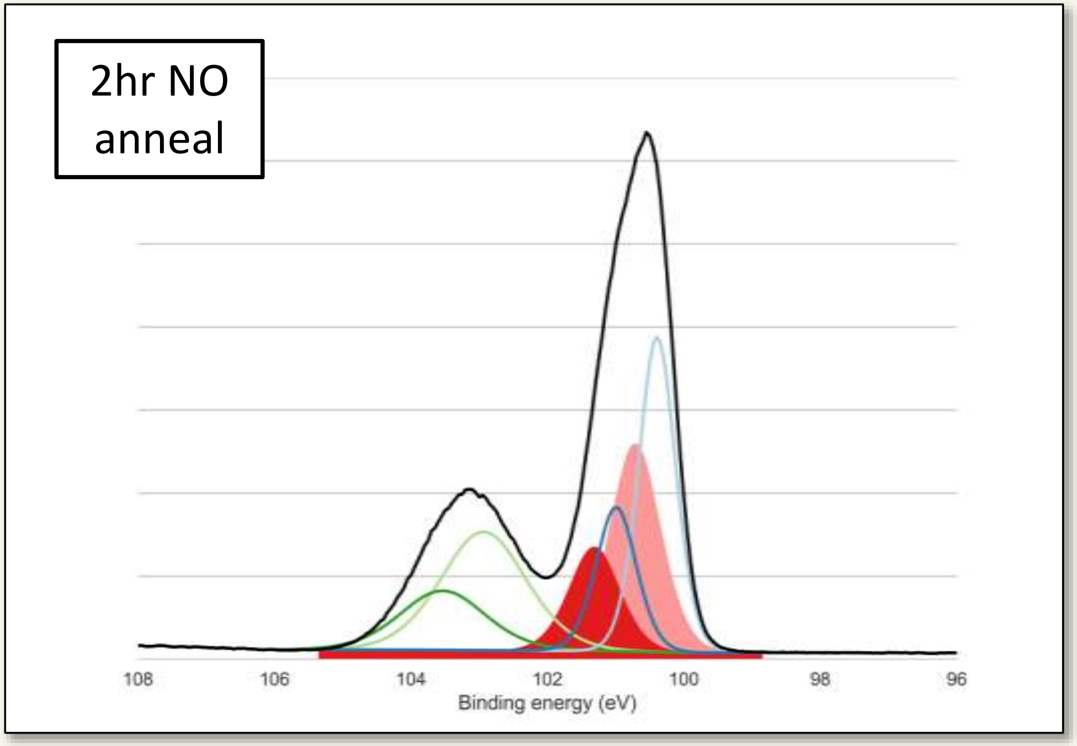
- Can we correlate the EELS?
- XPS is surface-sensitive binding energy measurement
- Measured oxidized and NO-POA samples etched near to the interface



SiO_2 signal

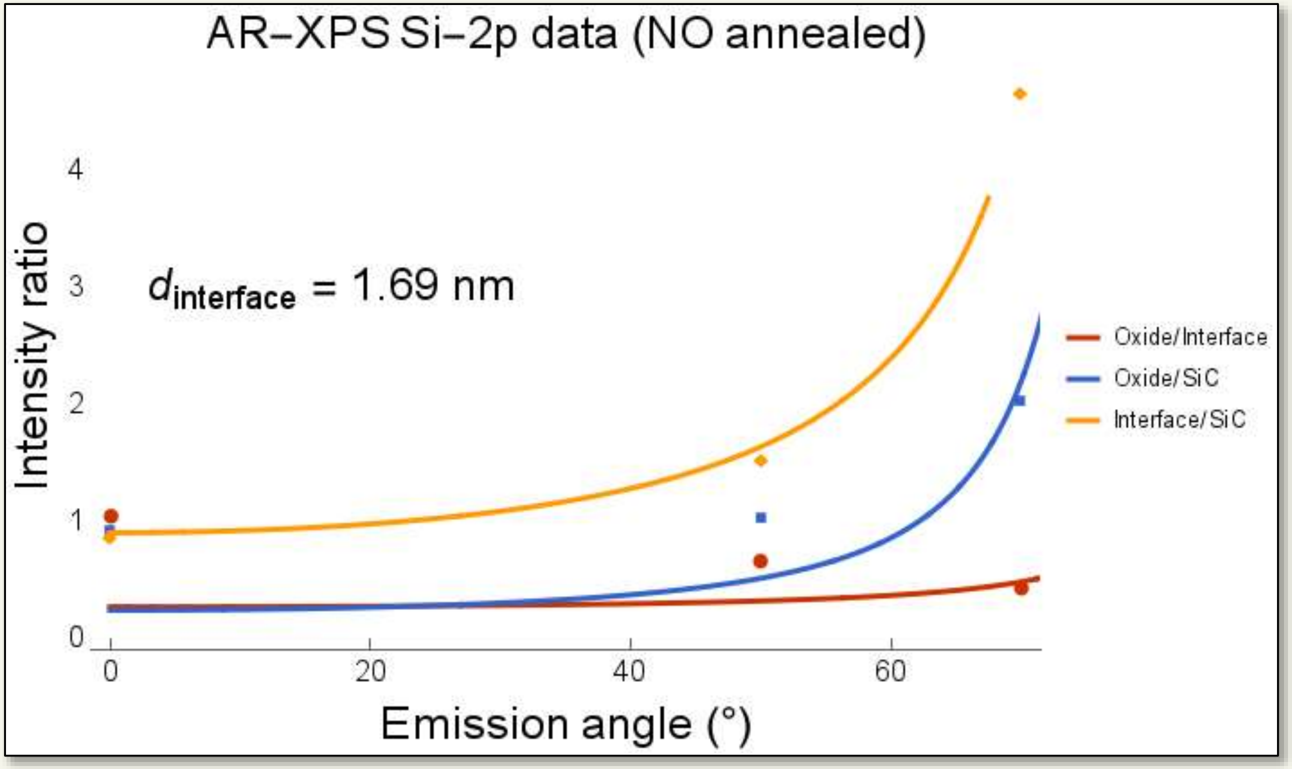
XPS Interfacial analysis – Si-*2p*

- Can we correlate the EELS?
- XPS is surface-sensitive binding energy measurement
- Measured oxidized and NO-POA samples etched near to the interface



Interface signal

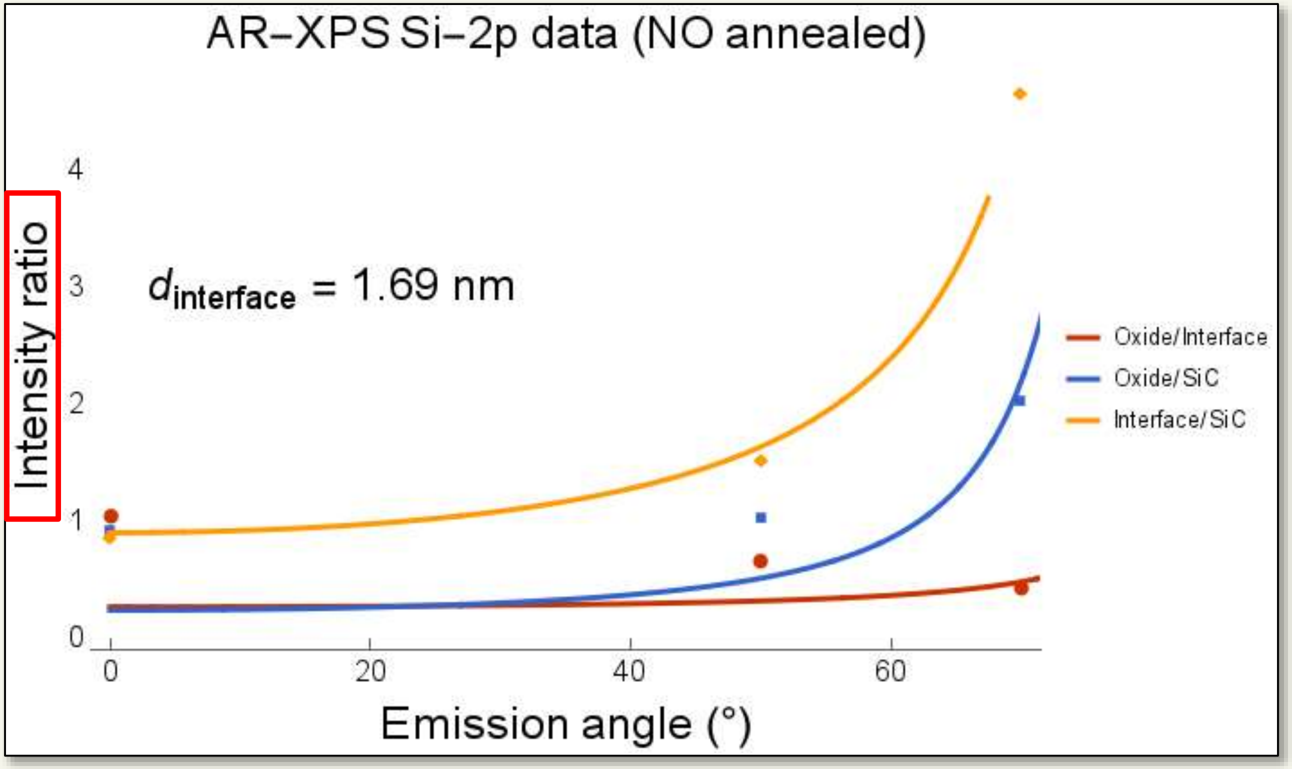
Angle resolved XPS - interface width



$$\frac{I_{int}}{I_{bulk}} = \frac{c_{int}}{c_{bulk}} \left[\exp\left(\frac{d_{int}}{\lambda \cos\theta} \right) - 1 \right]^{*}$$

*L. I. Johansson, *et al*. *Surface Science*, **529**, 515 (2003).

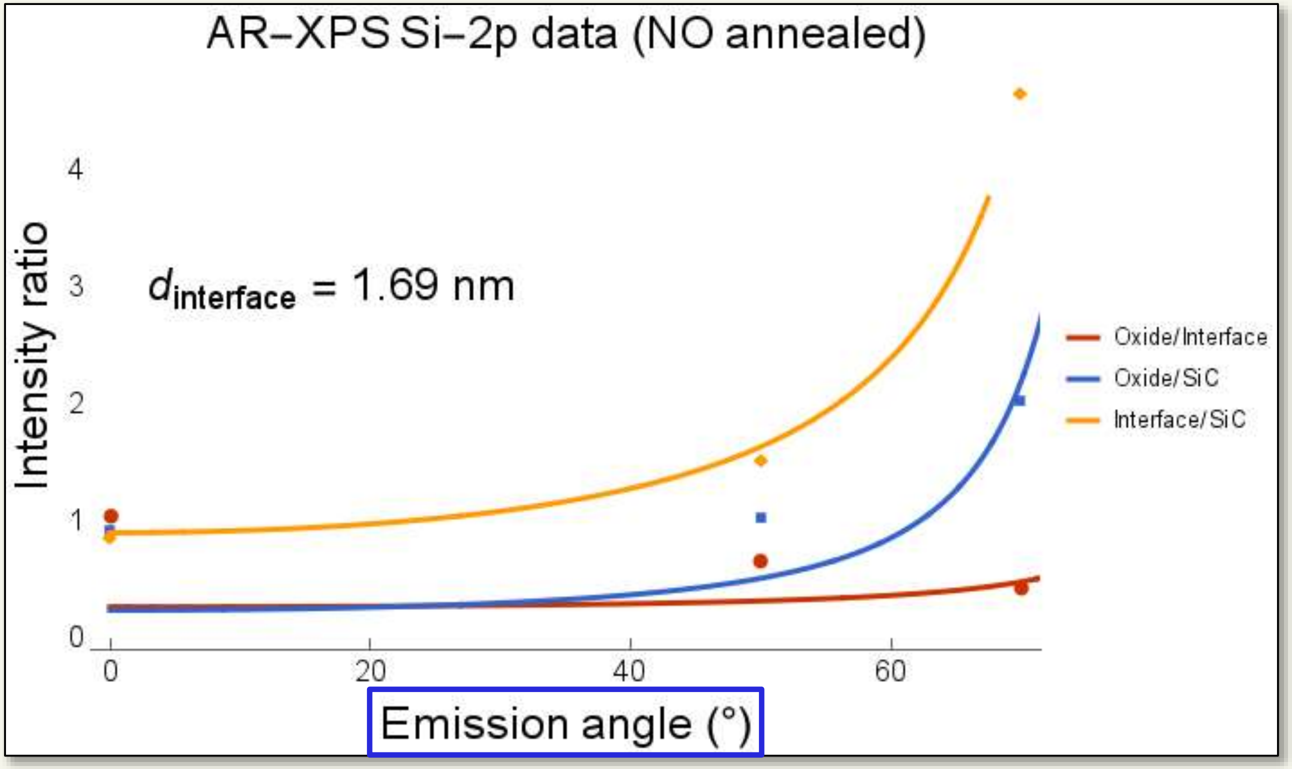
Angle resolved XPS - interface width



$$\boxed{\frac{I_{int}}{I_{bulk}}} = \frac{c_{int}}{c_{bulk}}\left[\exp\left(\frac{d_{int}}{\lambda\cos\theta}\right) - 1\right]^{*}$$

*L. I. Johansson, *et al*. *Surface Science*, **529**, 515 (2003).

Angle resolved XPS - interface width



$$\frac{I_{int}}{I_{bulk}} = \frac{c_{int}}{c_{bulk}} \left[\exp\left(\frac{d_{int}}{\lambda \cos \boxed{\theta}} \right) - 1 \right]^{*}$$

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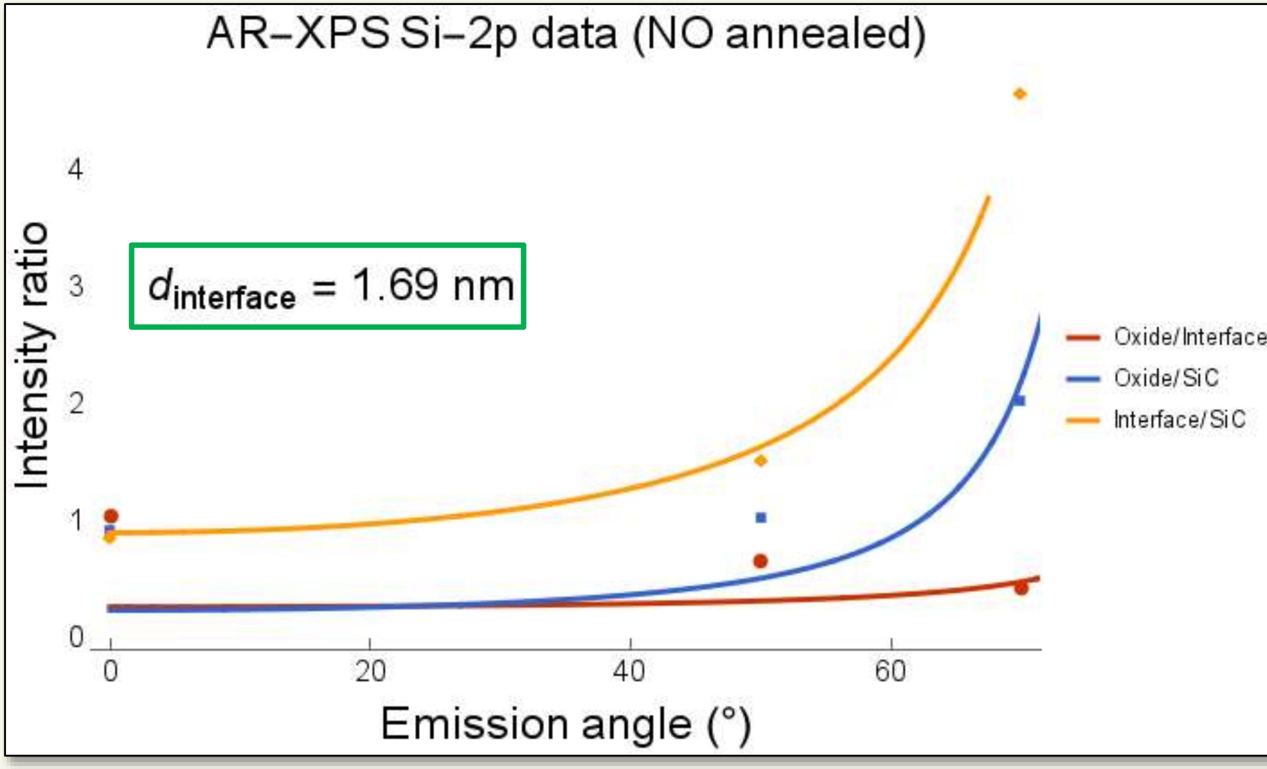
Angle resolved XPS - interface width



$$\frac{I_{int}}{I_{bulk}} = \frac{c_{int}}{c_{bulk}}\left[\exp\left(\frac{\boxed{d_{int}}}{\lambda\cos\theta}\right) - 1\right]^{*}$$

*L. I. Johansson, *et al*. *Surface Science*, **529**, 515 (2003).

Angle resolved XPS - interface width

Face / Treatment	$d_{\text{interface}}$
O_2 oxidation	2.97 nm
O_2 oxidation + NO Post-anneal	1.69 nm

Angle resolved XPS - interface width

Face / Treatment	$d_{interface}$
O_2 oxidation	2.97 nm
O_2 oxidation + NO Post-anneal	1.69 nm

- Similar values to EELS measurements
- Definitely not “abrupt”

Summary

- The shift of the Si-$L_{2,3}$ edge is a good indicator of the width of the transition region in 4H SiC/SiO_2.
  - Newer devices do not follow previously observed trend
  - Measuring interface width does not reveal what is happening inside
- Decomposition of EELS signals reveal a chemically/electrically distinct interface state in all NO-annealed samples
  - Likely significant impacts on mobility and performance
  - Spatial distribution matches measurements of w_{TL}
- XPS corroborates EELS findings of Si_3N_4-like N bonding at the interface, with similar spatial extent
  - **Transition region with approximately 1.5-2.0 nm**

Future work

- Theoretical modeling of DOS for explanation
- Exploration of lattice strain in different substrate orientations (CBED, Geo. Phase Analysis)

Acknowledgements

- ARL Contracts W911NF-11-2-0044 and W911NF-07-2-0046.
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HyperSpy
hyperspectral data analysis
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THANK YOU

Questions/comments/discussion?