Revealing Hidden Interfacial States in NO Passivated 4H-SiC/SiO_2 Structures using TEM-EELS and XPS*

Joshua Taillon,[1] Karen Gaskell,[2] Sarit Dahr,[3] Gang Liu,[4] Leonard Feldman,[4] Tsvetanka Zheleva,[5] Aivars Lelis,[5] and **Lourdes Salamanca-Riba**[1]

2016 March APS, X7.03
Friday March 18, 2016 – Room 303
8:24AM – Baltimore, MD

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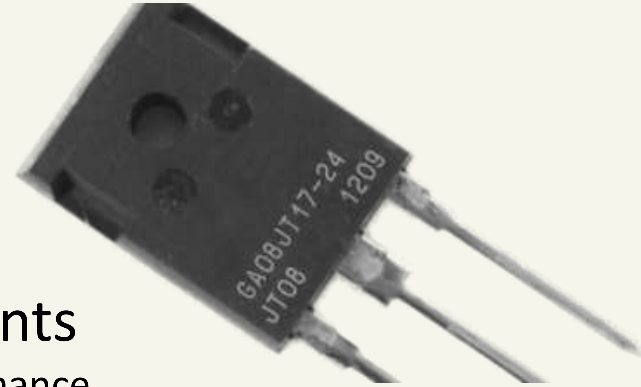
[1] Materials Science and Engineering, University of Maryland College Park
[2] Chemistry and Biochemistry, University of Maryland College Park
[3] Department of Physics, Auburn University
[4] Institute for Advanced Materials, Rutgers University
[5] U.S. Army Research Laboratory

Motivation and background



- SiC: Very promising for high temperature, high power, and high radiation environments
  - NO post oxidation anneal (POA) drastically improves performance

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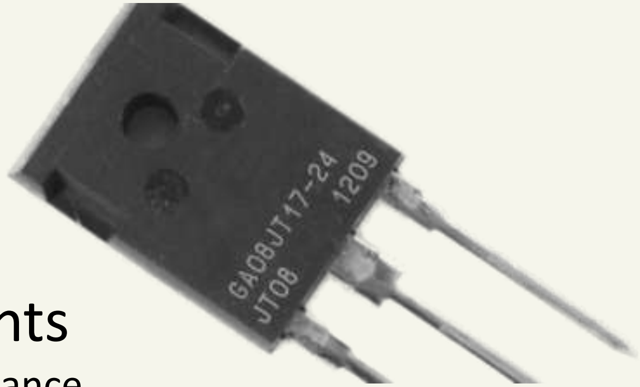

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  - Carrier mobility
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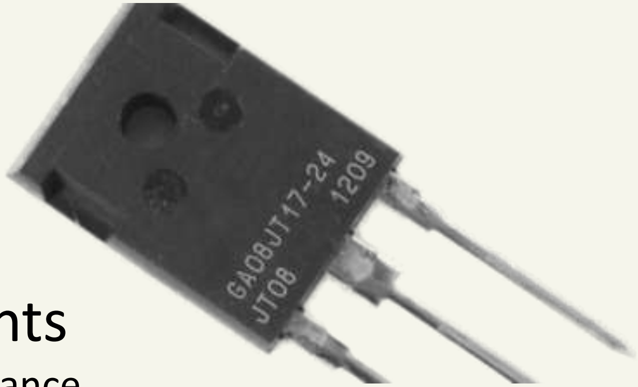

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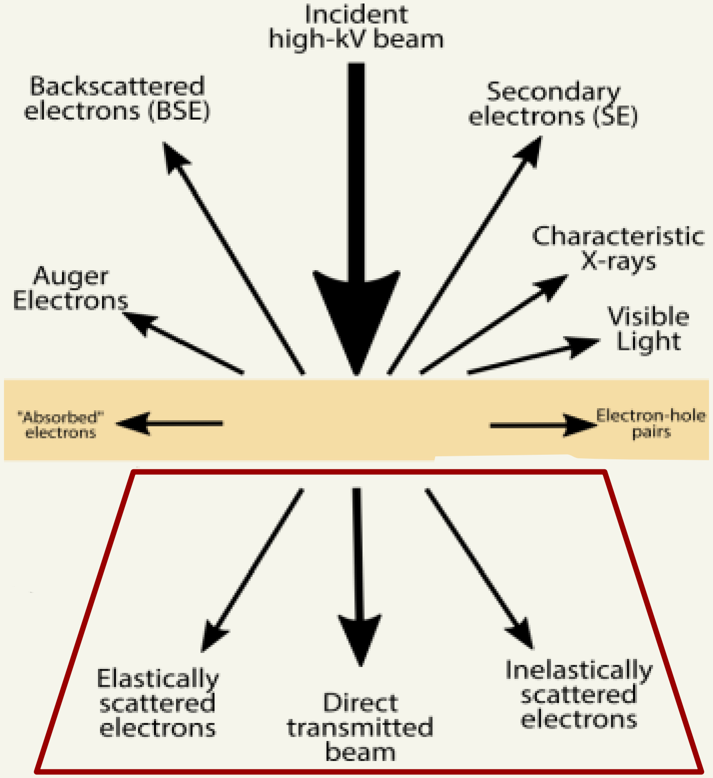
  - NO post oxidation anneal (POA) drastically improves performance
- Electrically active defects limit:
  - Carrier mobility
  - Reliability
  - Device stability
- What is the true nature of the interface, and how do our processing techniques really affect it?
  - Some work indicates a distinct transition region (EELS)[1, 2]
  - Others suggest abrupt transition (XPS, MEIS, etc.)[3 – 4]

[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

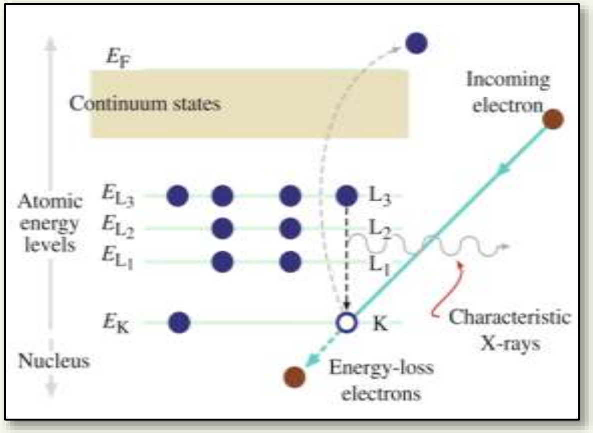
Electron Energy Loss Spectroscopy (EELS)

- E-beam has many interactions with specimen:

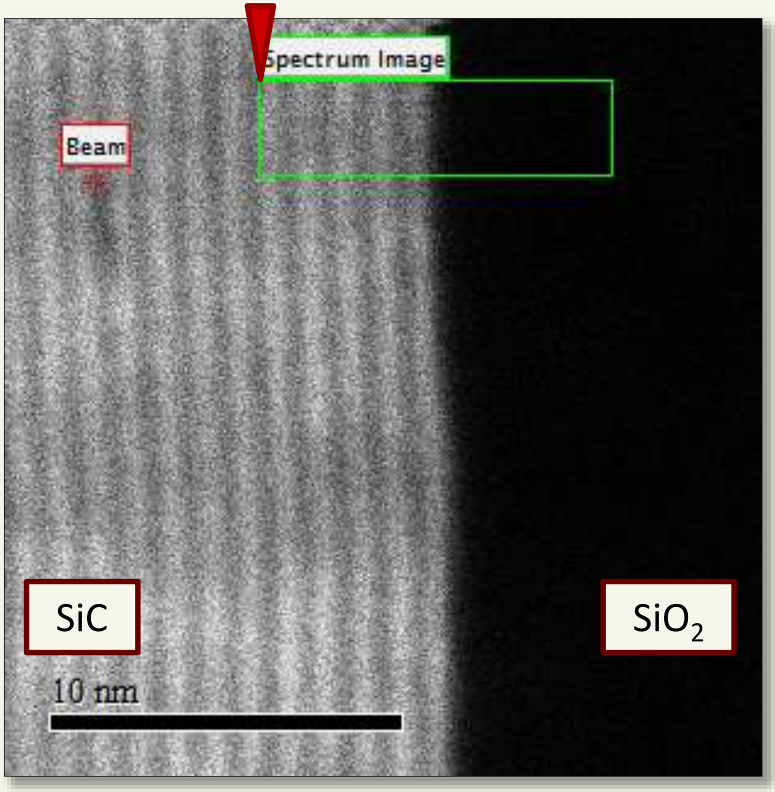


Adapted from Williams and Carter, *Transmission Electron Microscopy*, (2009).

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

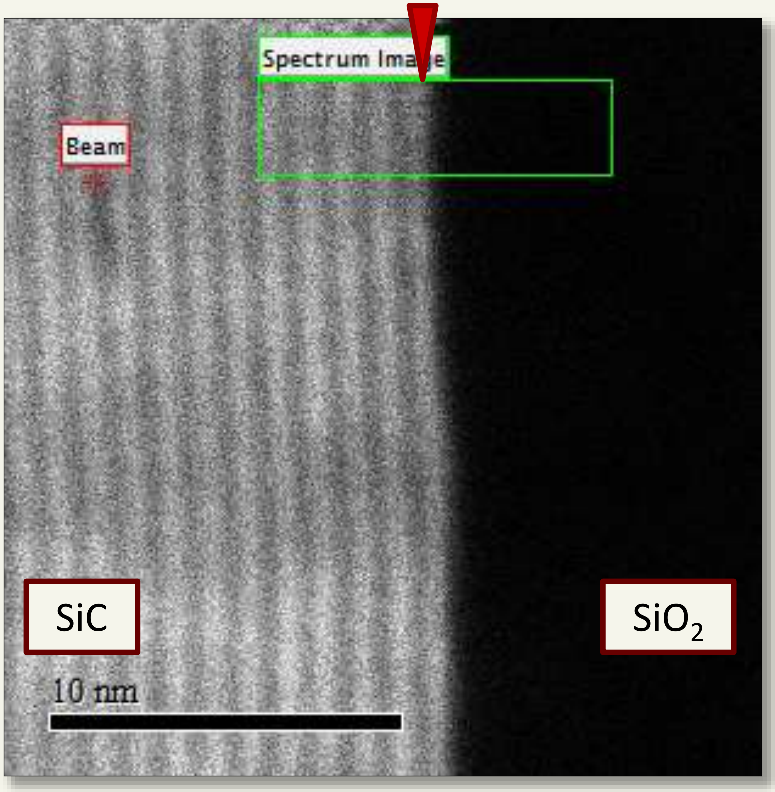


EELS Spectrum Imaging



HAADF Survey Image

EELS Spectrum Imaging



HAADF Survey Image

EELS Spectrum Image

Simultaneous HAADF Signal

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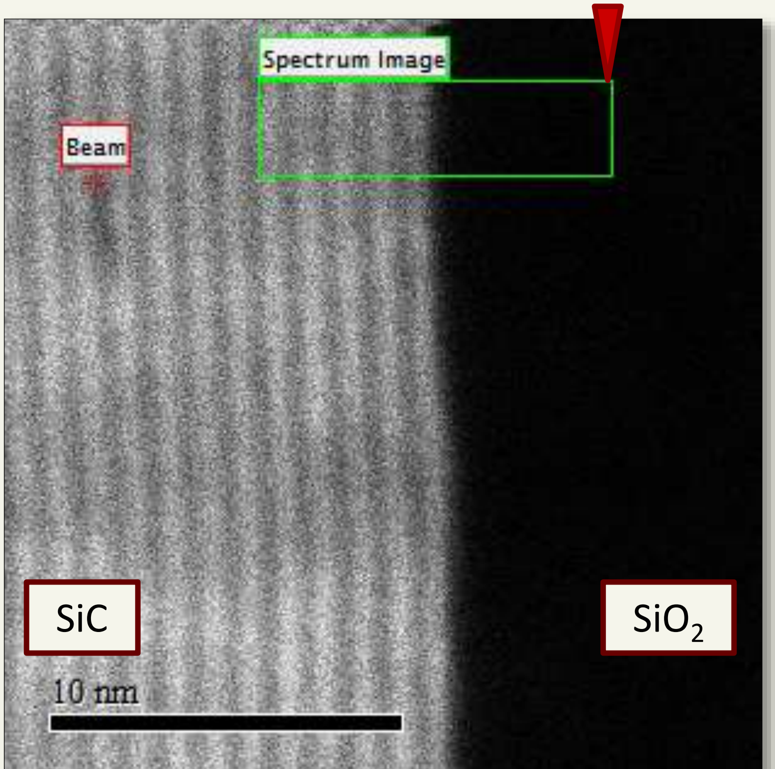

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SiC SiO_2

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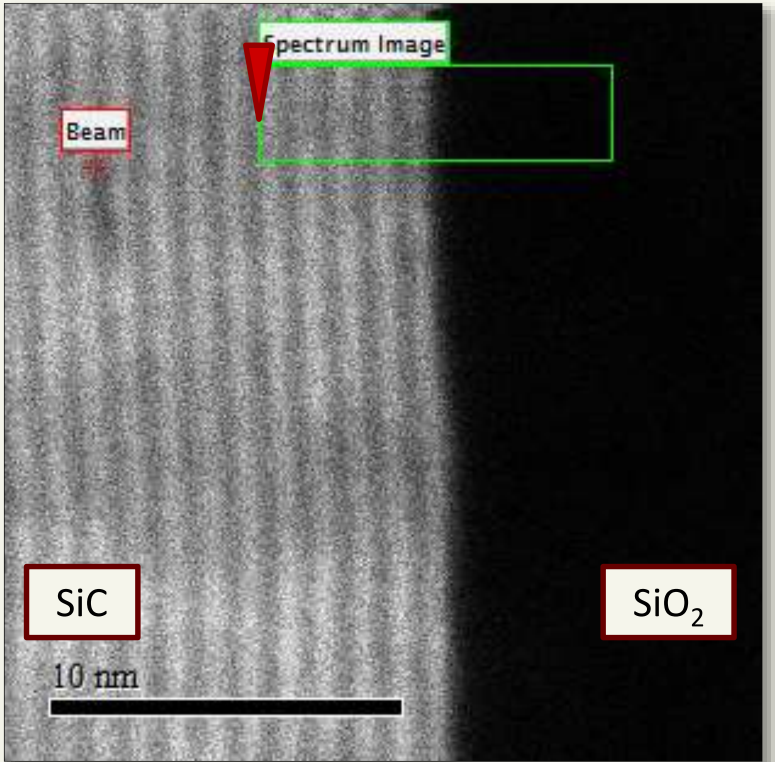

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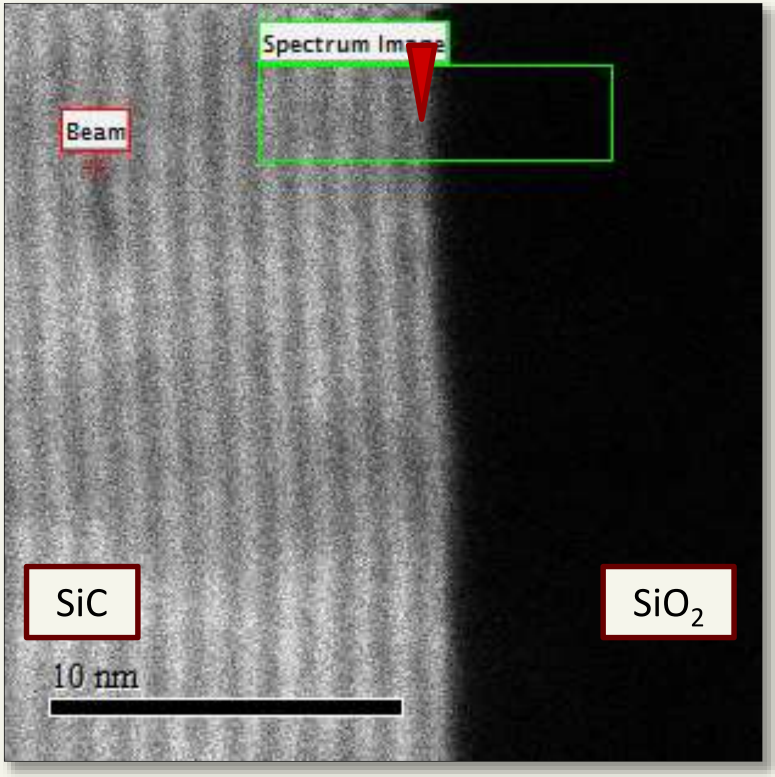

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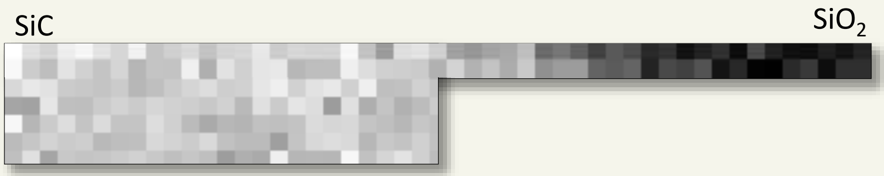

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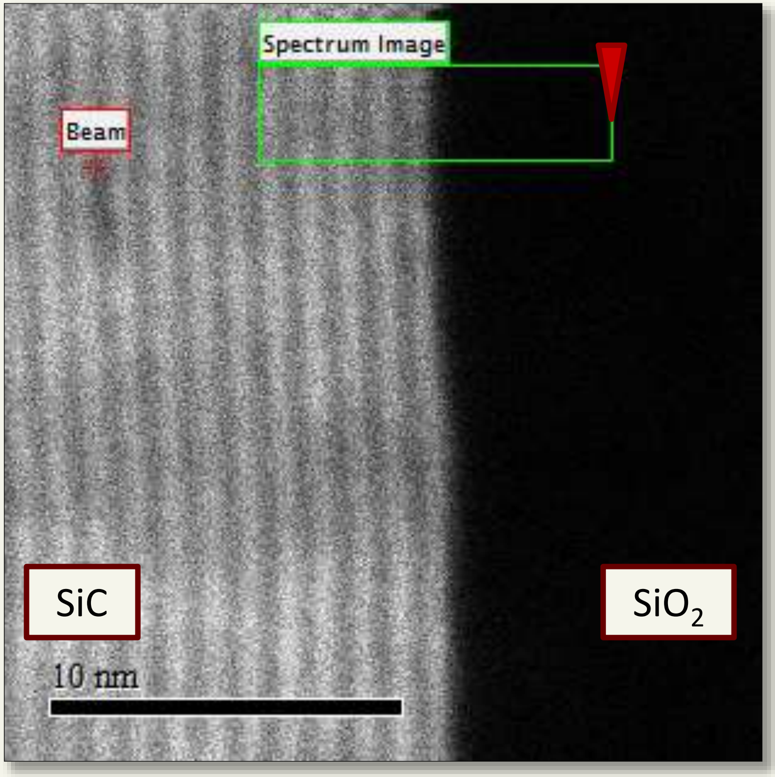

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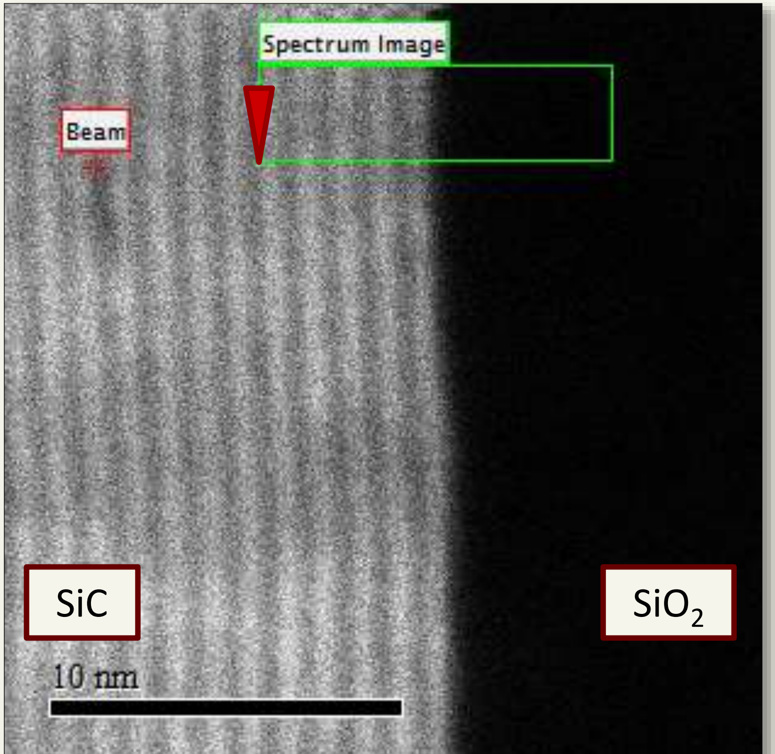

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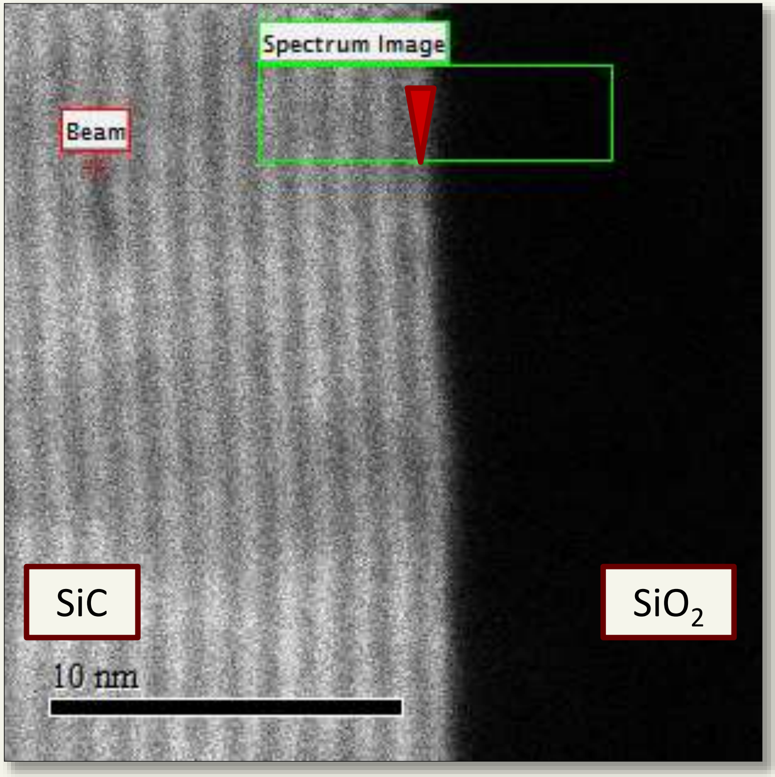

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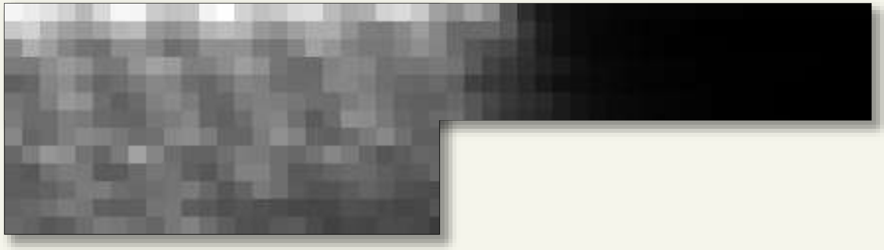
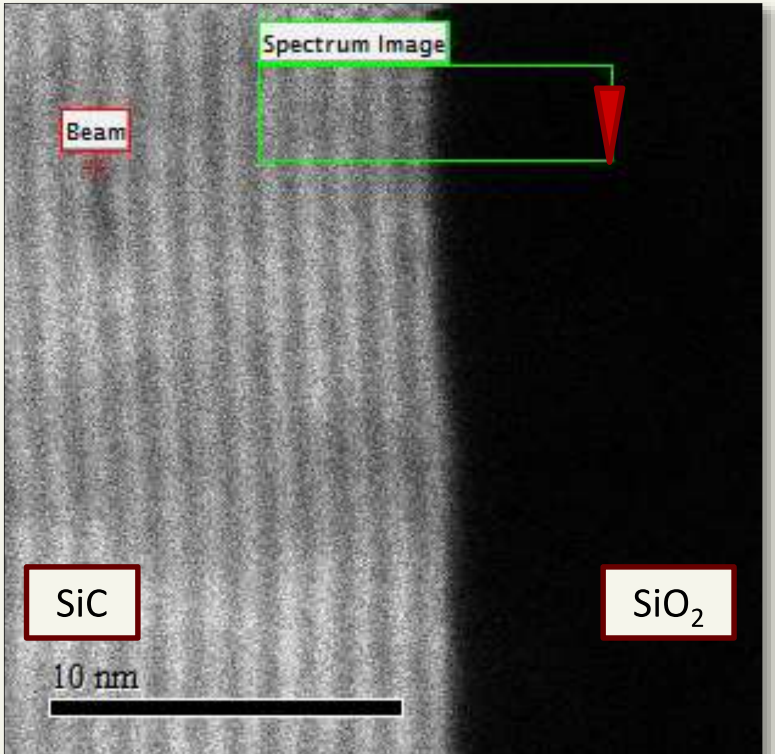
EELS Spectrum Image



Simultaneous HAADF Signal

EELS Spectrum Imaging



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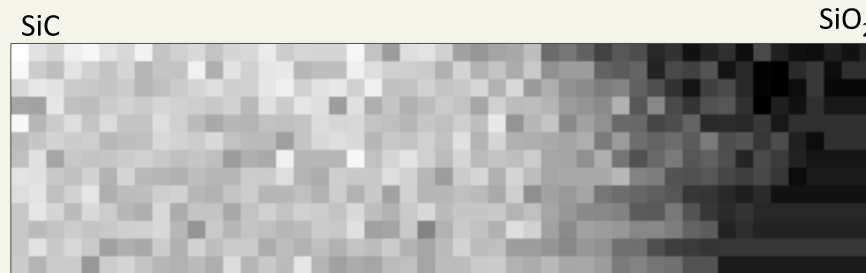

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Simultaneous HAADF Signal

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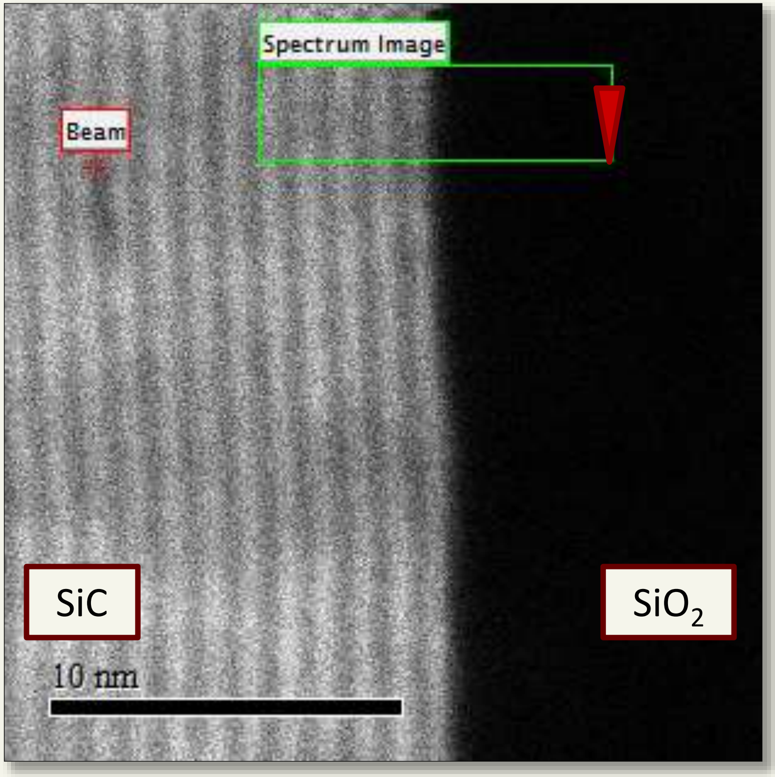
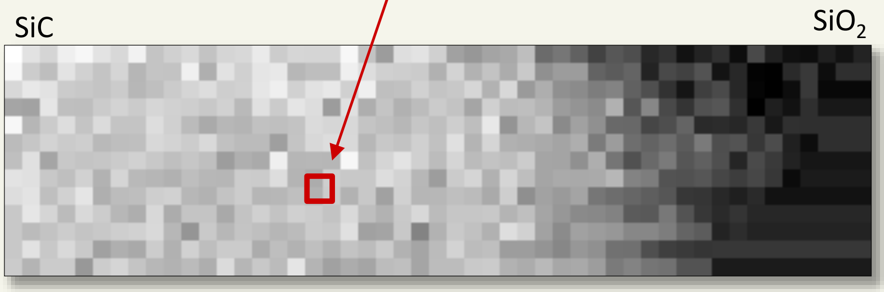


HAADF Survey Image

One spectrum per pixel

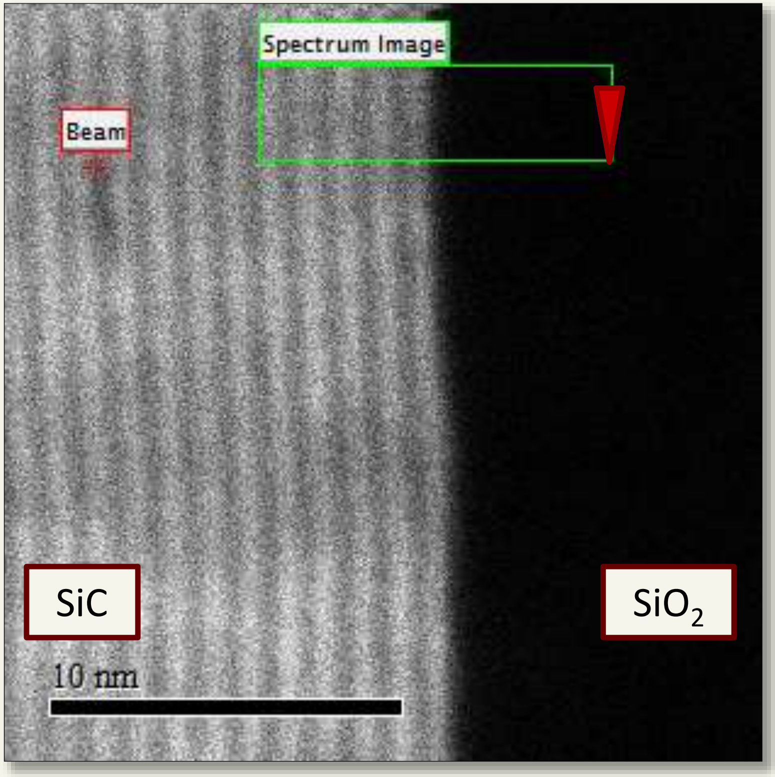


EELS Spectrum Image

Simultaneous HAADF Signal

A. JAMES CLARK
SCHOOL OF ENGINEERING

EELS Spectrum Imaging



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One spectrum per pixel

SiC

SiO_2

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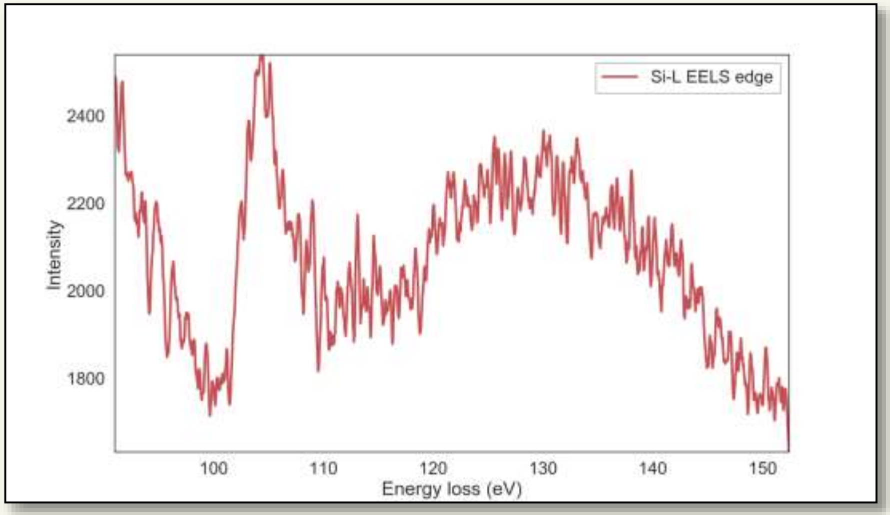
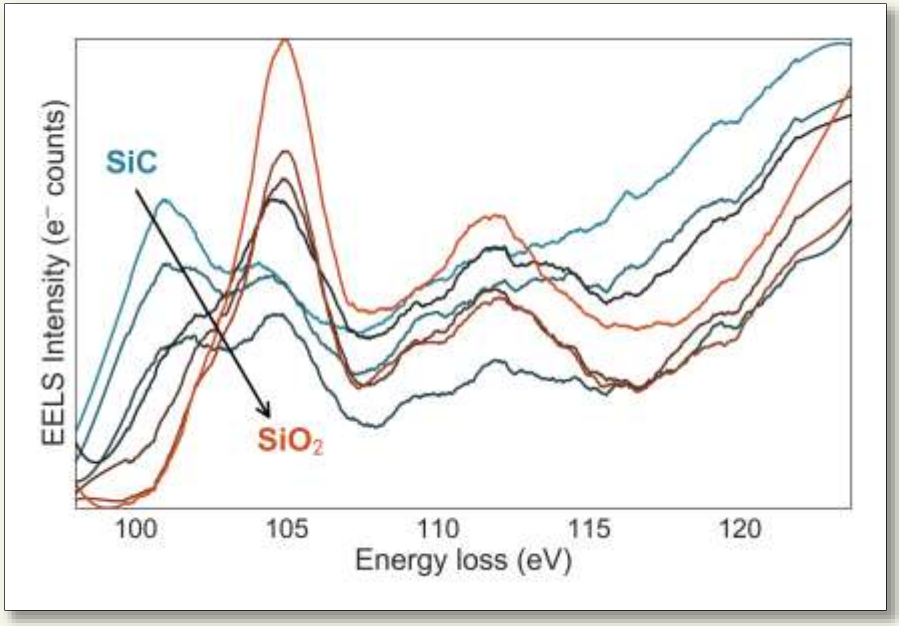
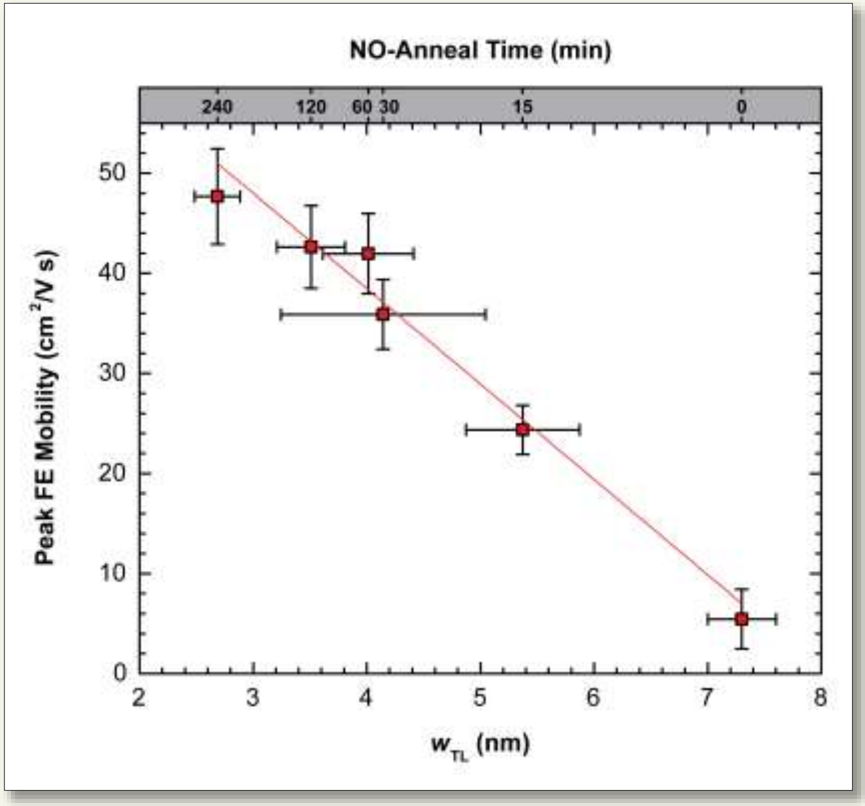


NO-anneal effects (previous results)



- Measured w_{TL} with chemical shift
- w_{TL} correlates inverse-linearly with μ_{FE}
- NO-anneal removes/passivates mobility-limiting defects
  - Compositionally and electronically



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

**Samples fabricated from 2010 SiC stock*

Newer work - Orientation effects

- Investigating effects of substrate orientation:
  - Newer SiC stock (2014)
  - $[0001]$ Si-face, with and without miscut
  - $[11\bar{2}0]$ a-face
  - Each with and without NO

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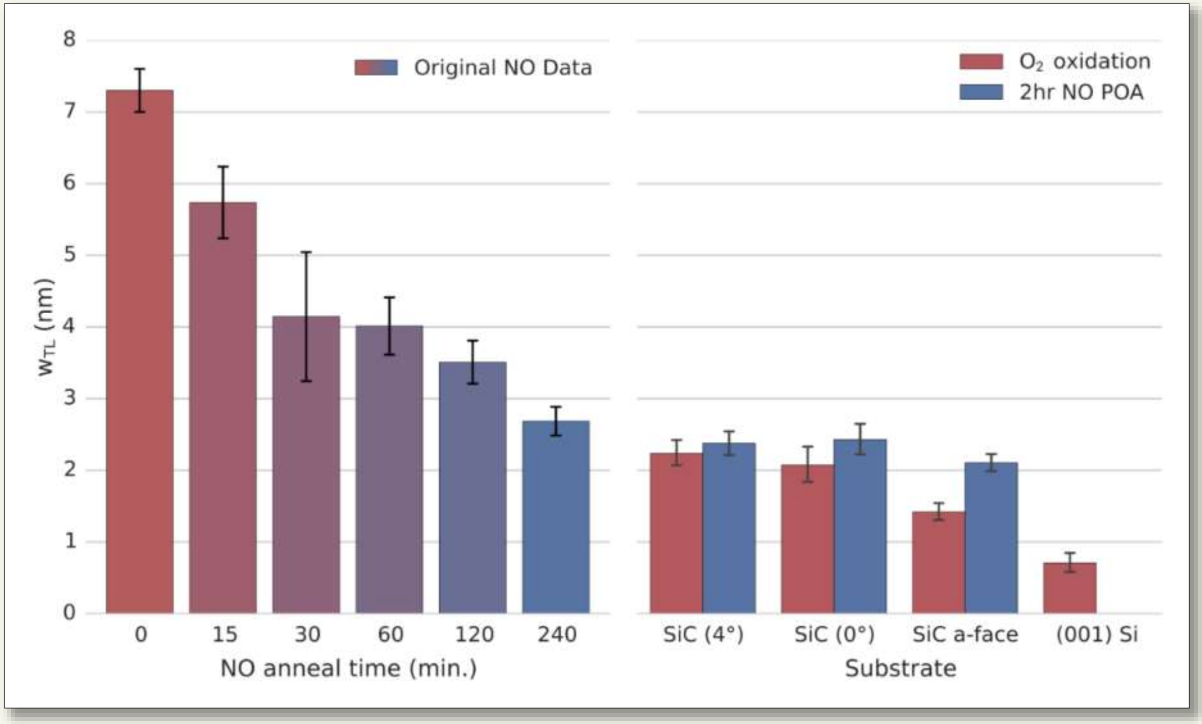

- Little to no effect of NO annealing
- *Contradicts expectations from previous result*

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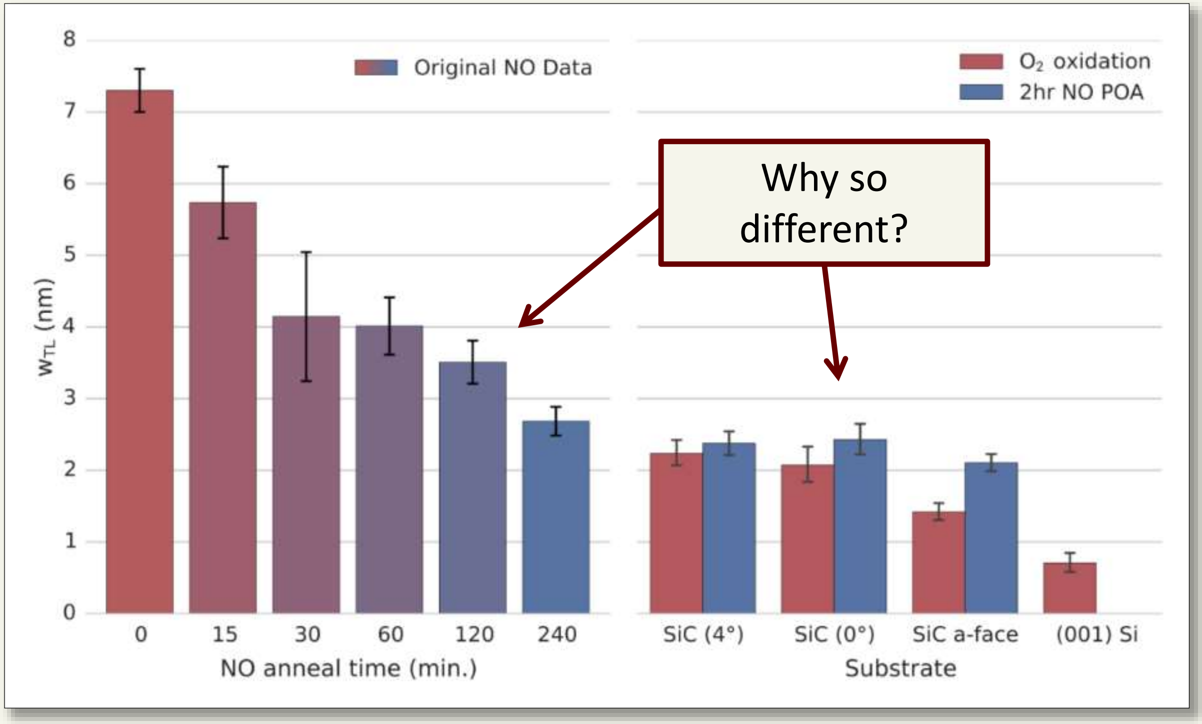

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NEW ANALYSIS TECHNIQUE

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
  - Unsupervised learning promotes bias-free analysis

Decomposition analysis

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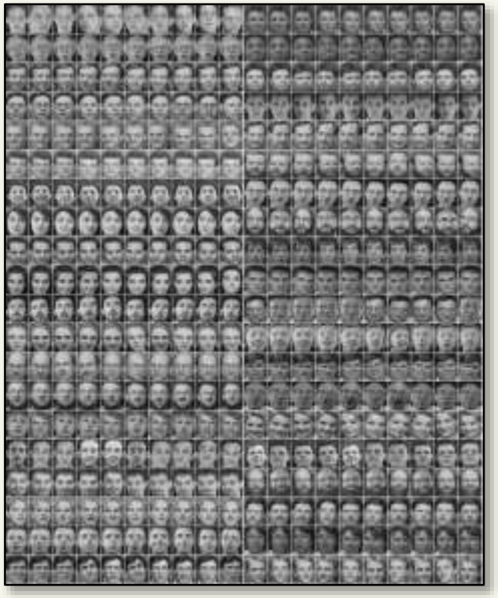
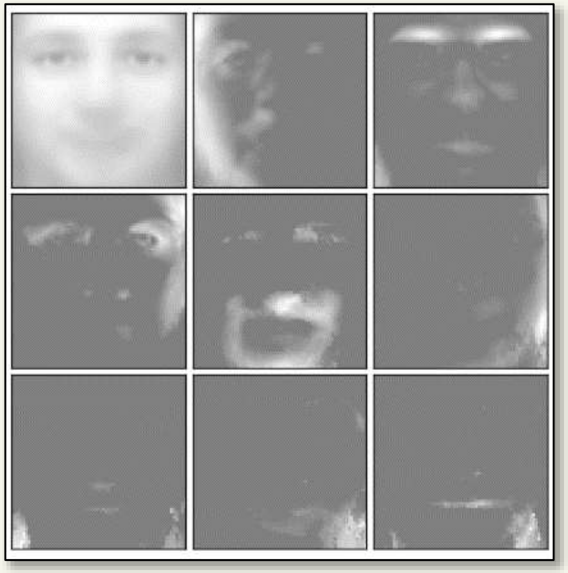
- Non-negative matrix factorization (NMF)



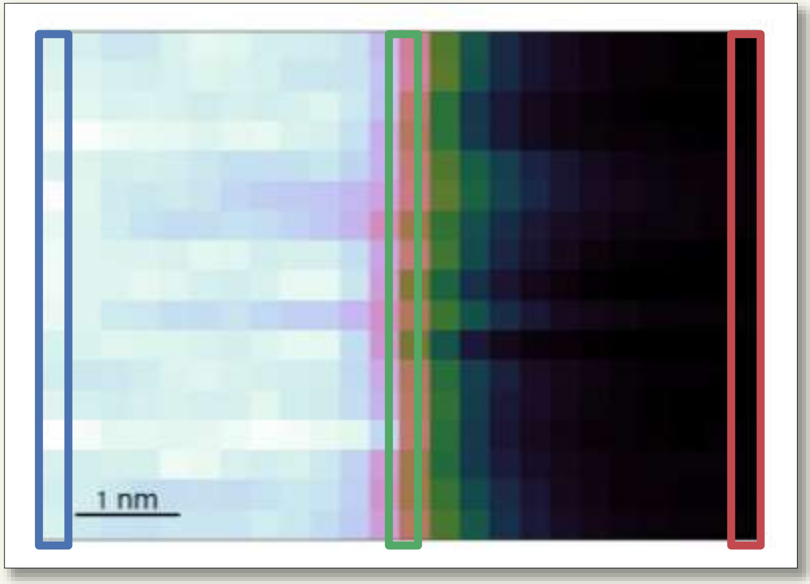
Olivetti Faces Dataset

NMF Components

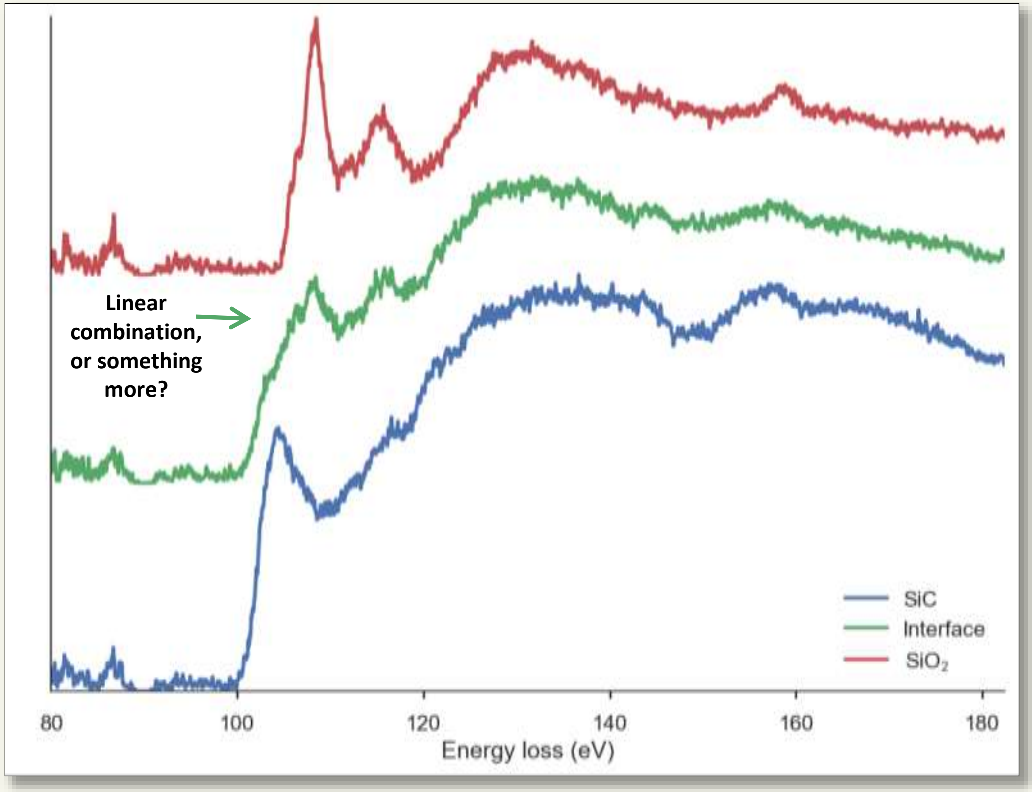


Interface components at NO-annealed interfaces

SiC/SiO_2 EELS Spectrum Image

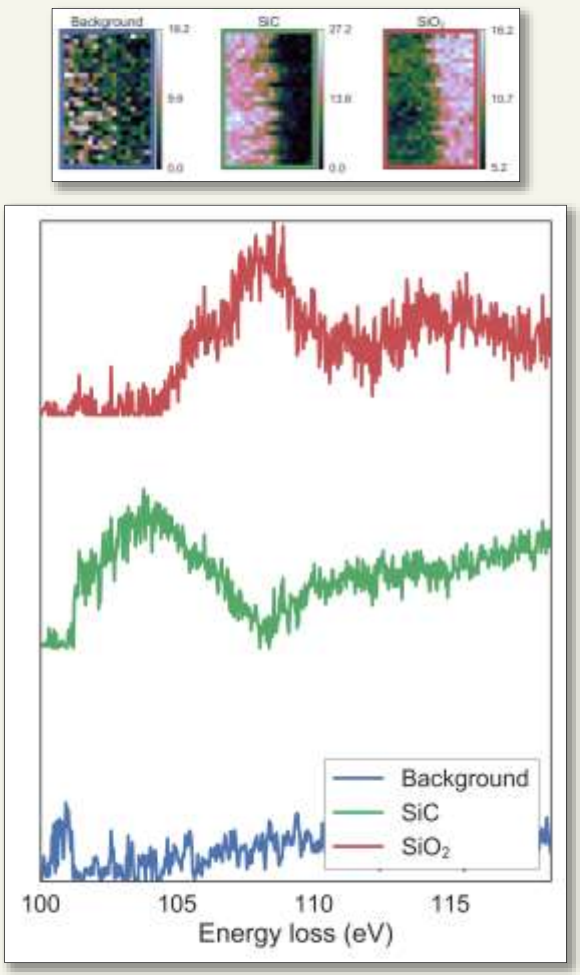


- Simple sum improves S/N, but cannot detect faint or overlapping signals

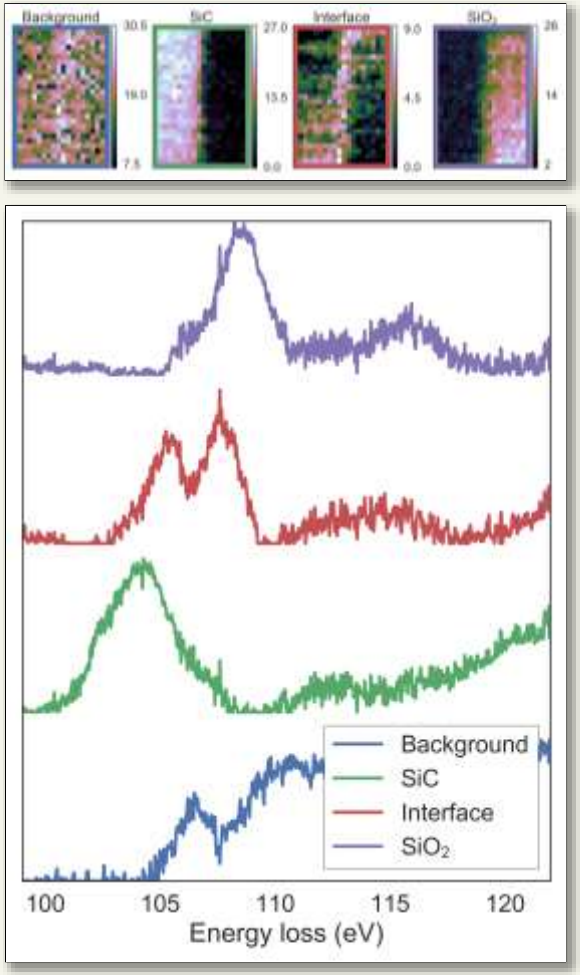


Decomposition analysis – Si $L_{2,3}$

a-face without NO



a-face with NO



- No significant variation between orientations
  - *a*-face data shown

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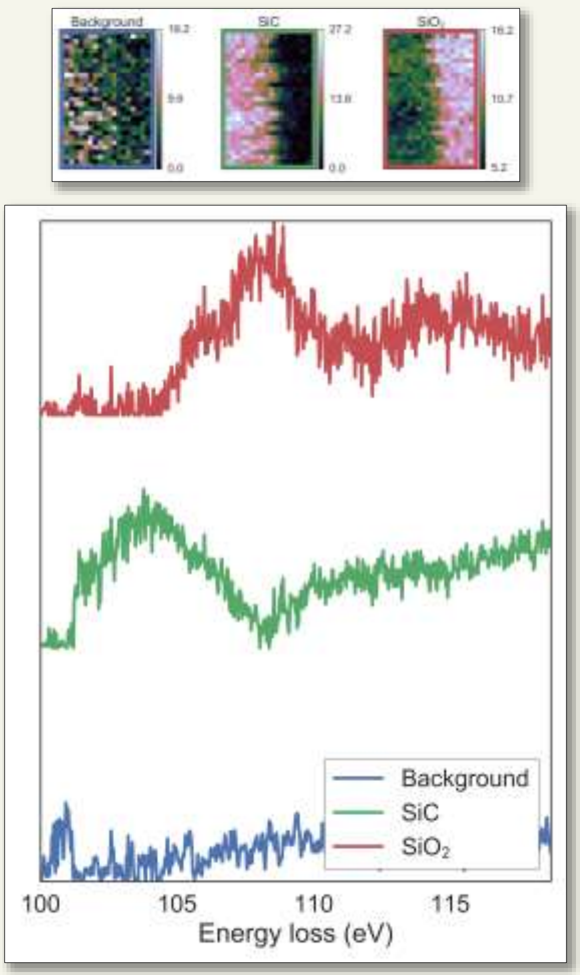
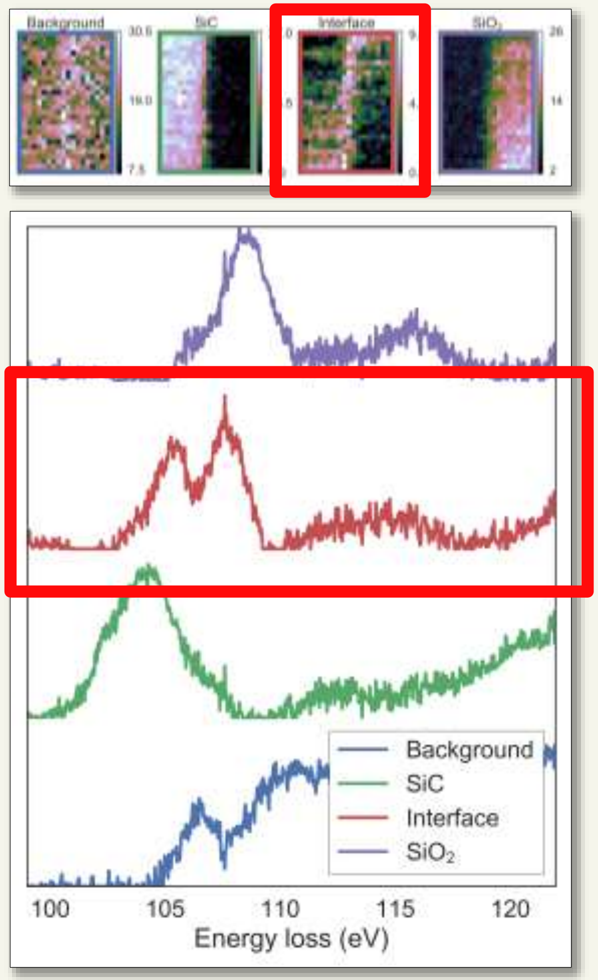


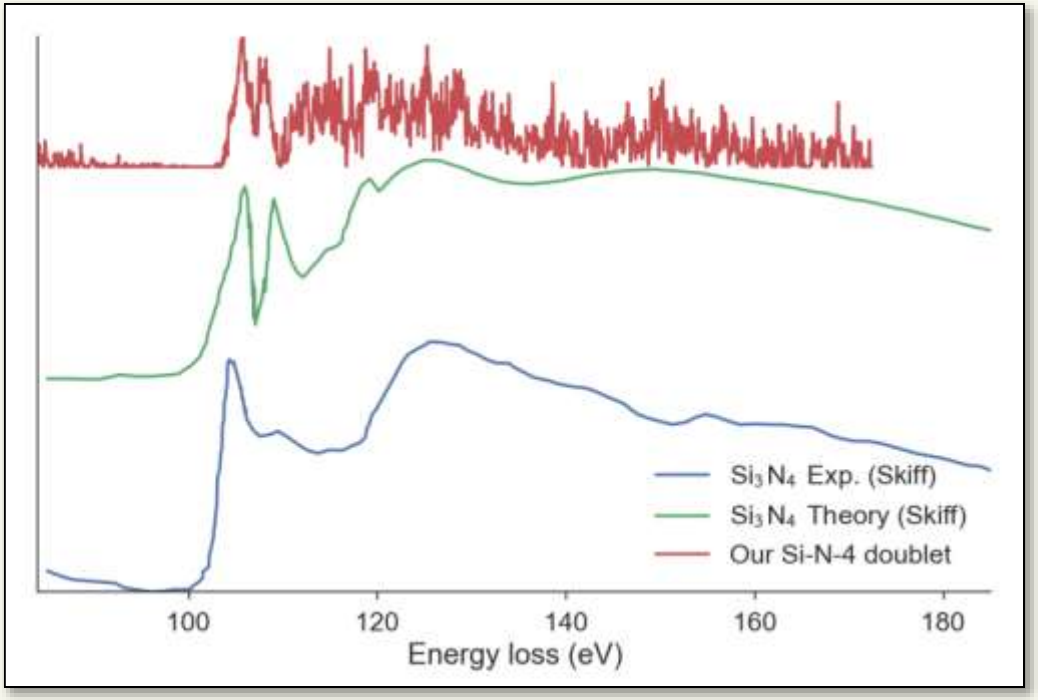
a-face with NO



- No significant variation between orientations
  - *a*-face data shown
- NO anneal gives rise to interfacial state with doublet peak

Si-$L_{2,3}$ Interface Component – N Bonding

- 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

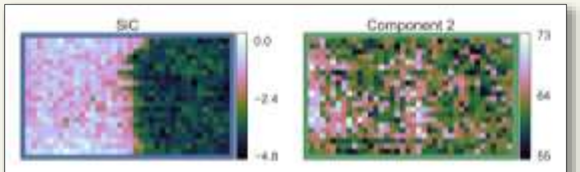


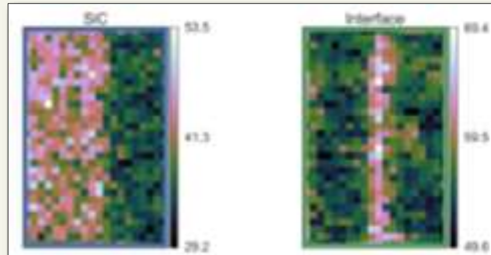
Skiff, W. M., *et al.*, *J. Appl. Phys.* **62,** 2439–2449 (1987).

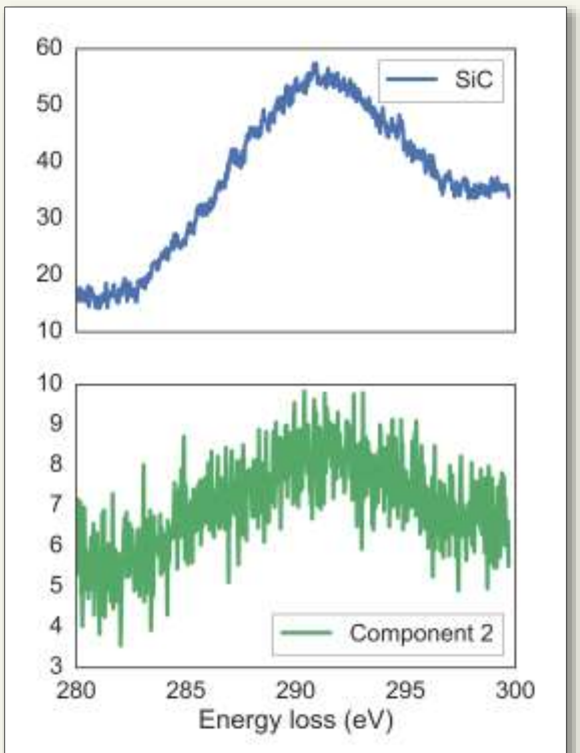
Decomposition analysis – C *K* edge

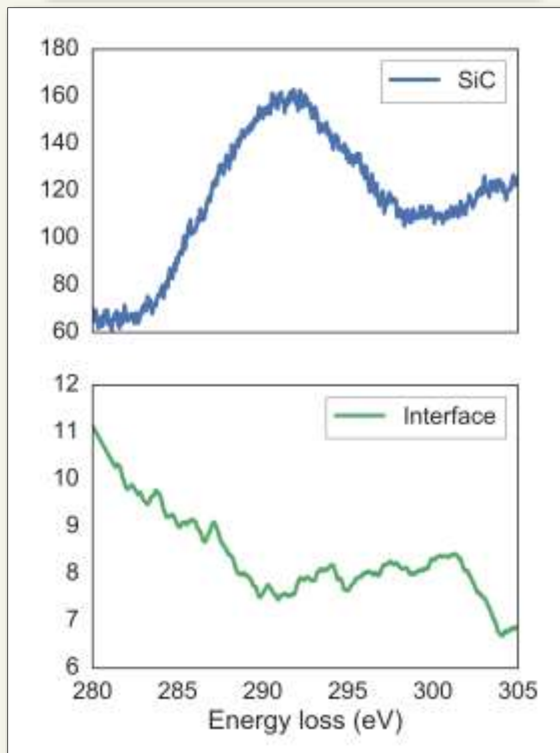
a-face without NO

a-face miscut with NO





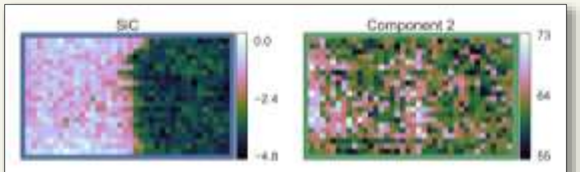


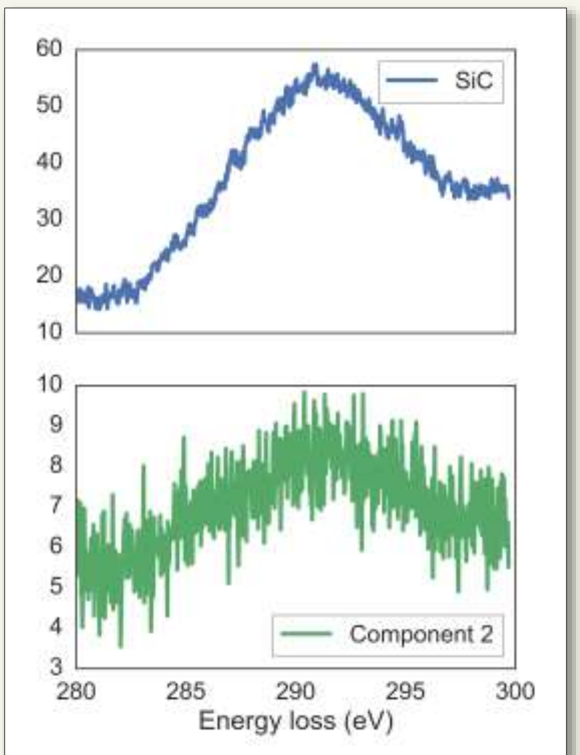


- No significant variation between orientations
    - *a*-face data shown

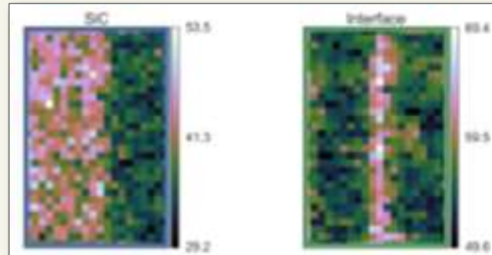
Decomposition analysis – C *K* edge

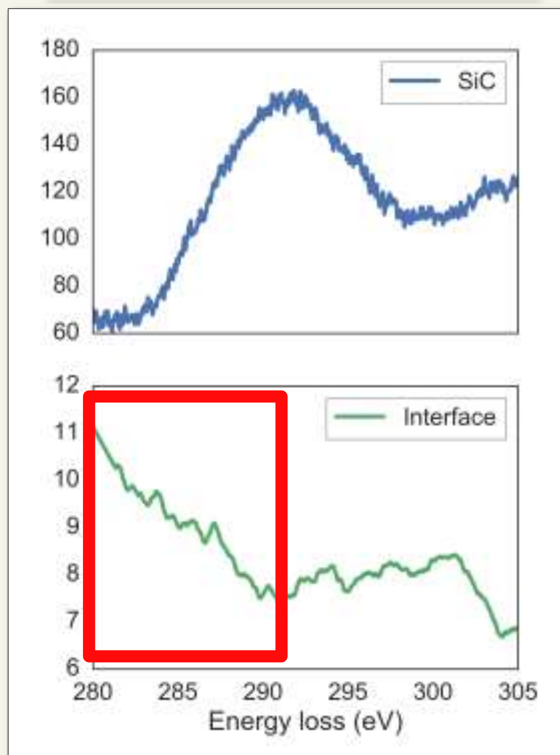
a-face without NO





a-face miscut with NO

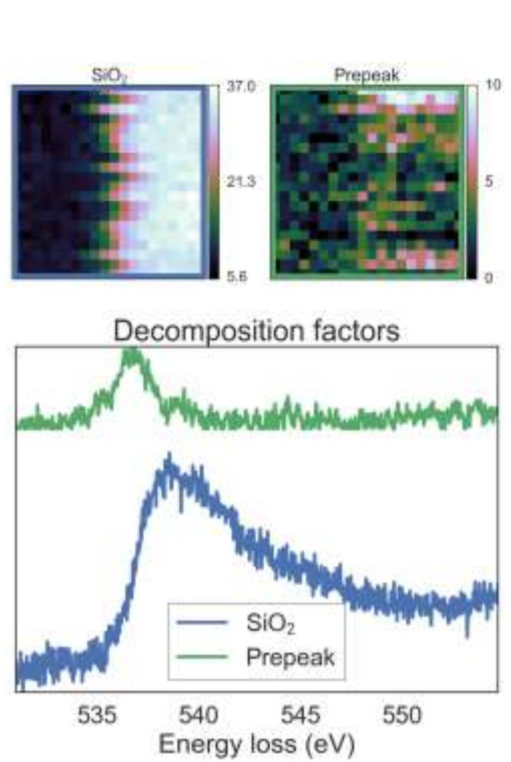
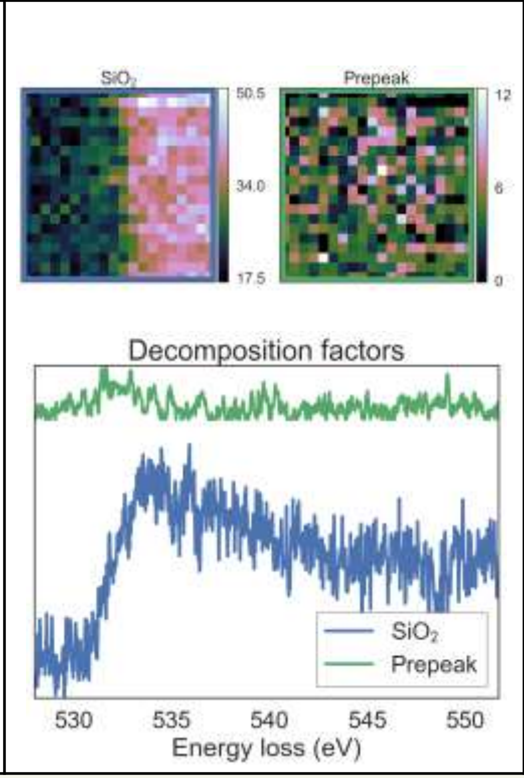
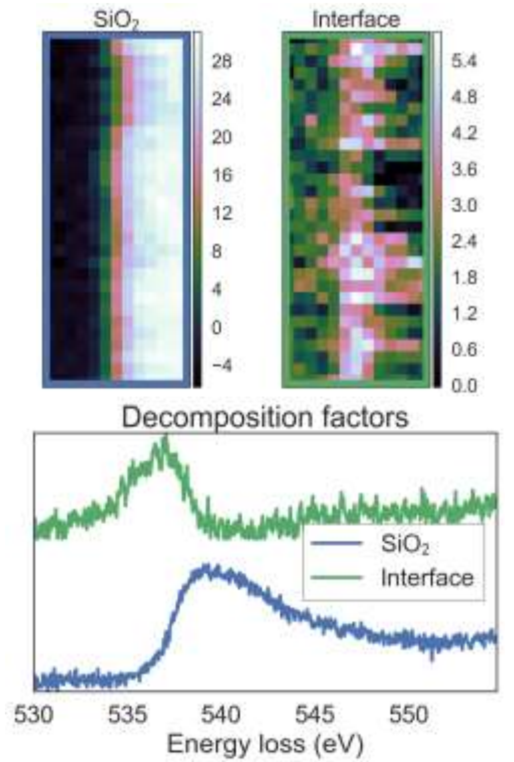




- No significant variation between orientations
  - *a*-face data shown

- NO anneal gives rise to interfacial state with pre-peak intensity attributable to *sp*^{2} from N-bonding*

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

O *K* edge analysis	Si-face 4° miscut (Si-NO-4)	Si-face no miscut (Si-NO-0)	a-face no miscut (a-NO-0)
Decomposition Results			

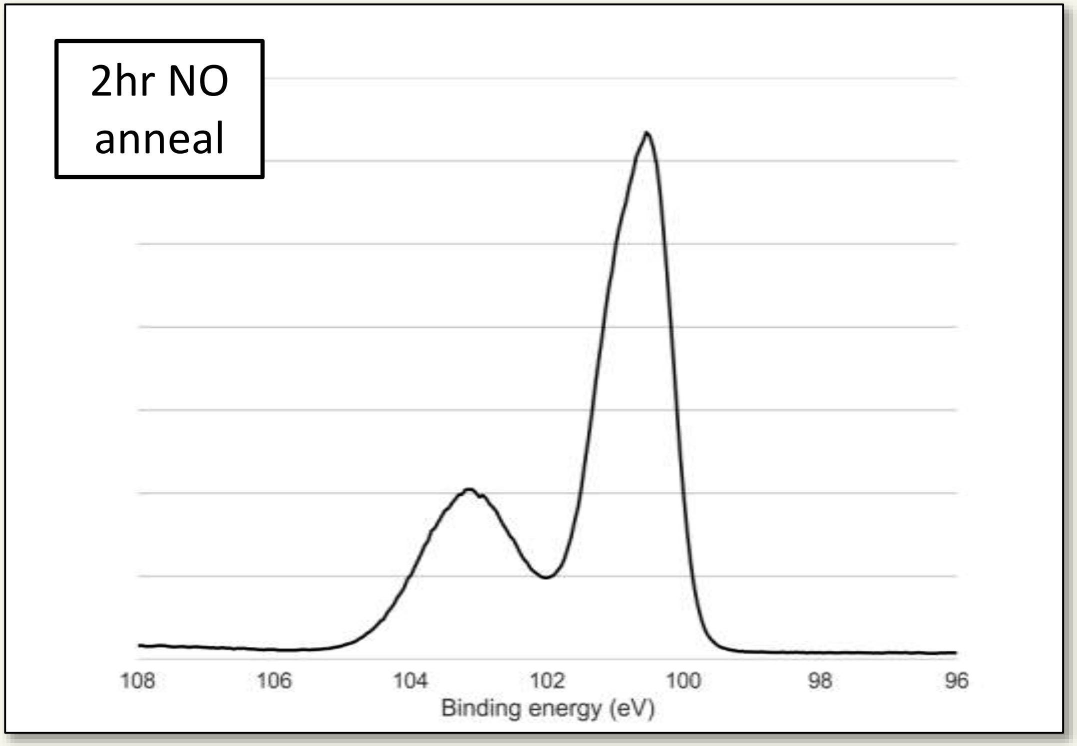
- All samples have an O-*K* pre-peak feature, but a-face is strongest and localized near the interface
- Intensity at lower energy indicates acceptor levels near edge of band gap, providing insight into the origins of improved mobility*

*Muller, D. A. et al. *Nature* 399, 758–761 (1999).

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*

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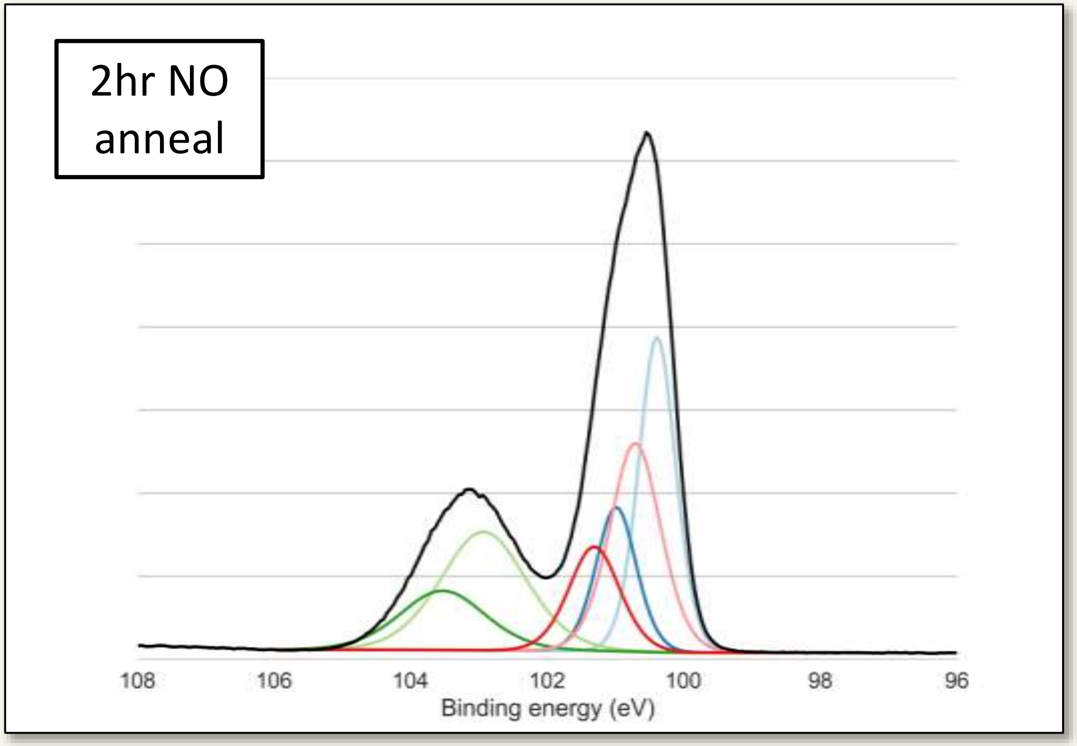

Peak-fitted Si-*2p*

XPS Interfacial analysis – Si-*2p*

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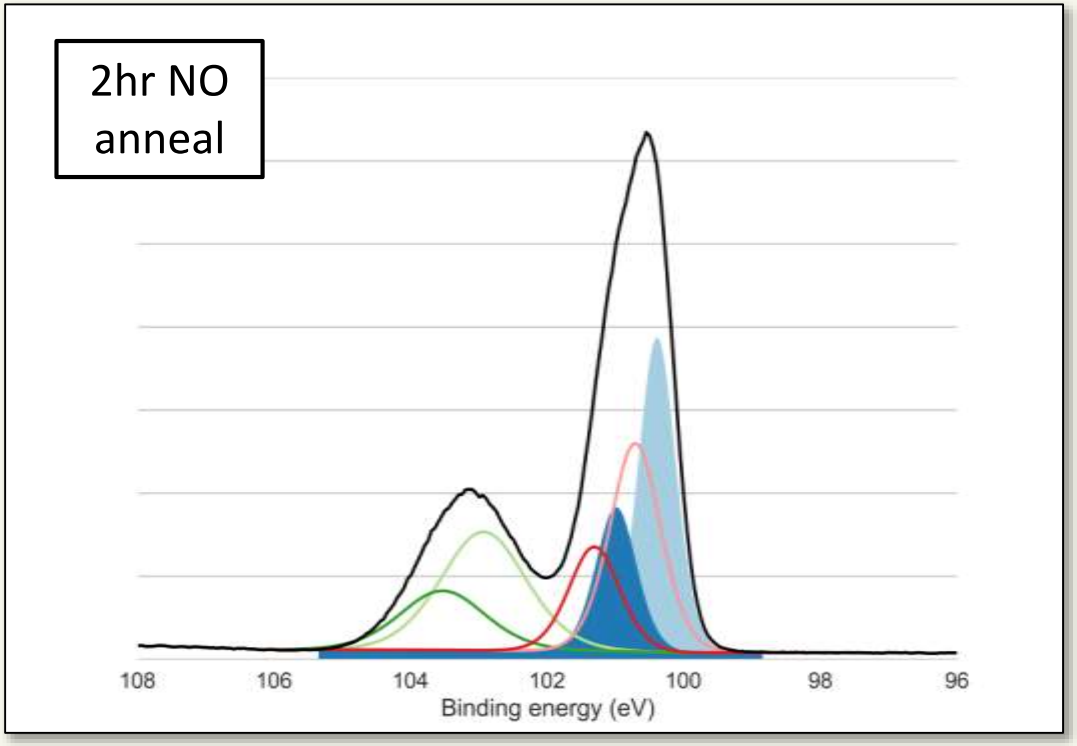

SiC signal

XPS Interfacial analysis – Si-*2p*

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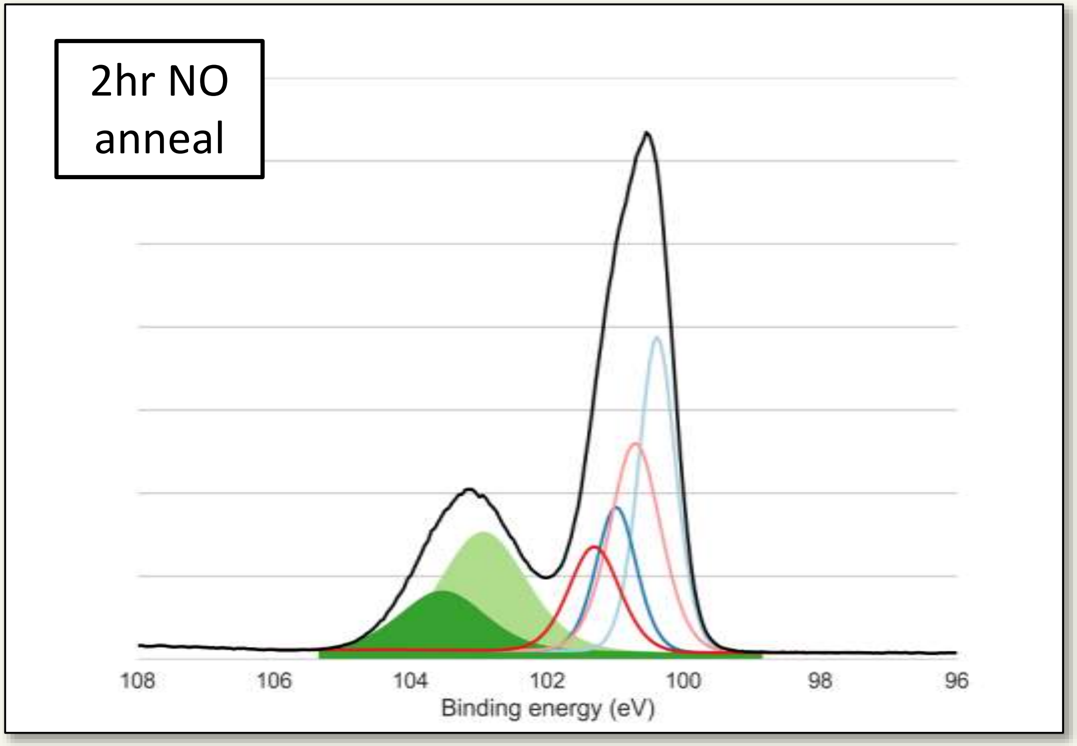

SiO_2 signal

XPS Interfacial analysis – Si-*2p*

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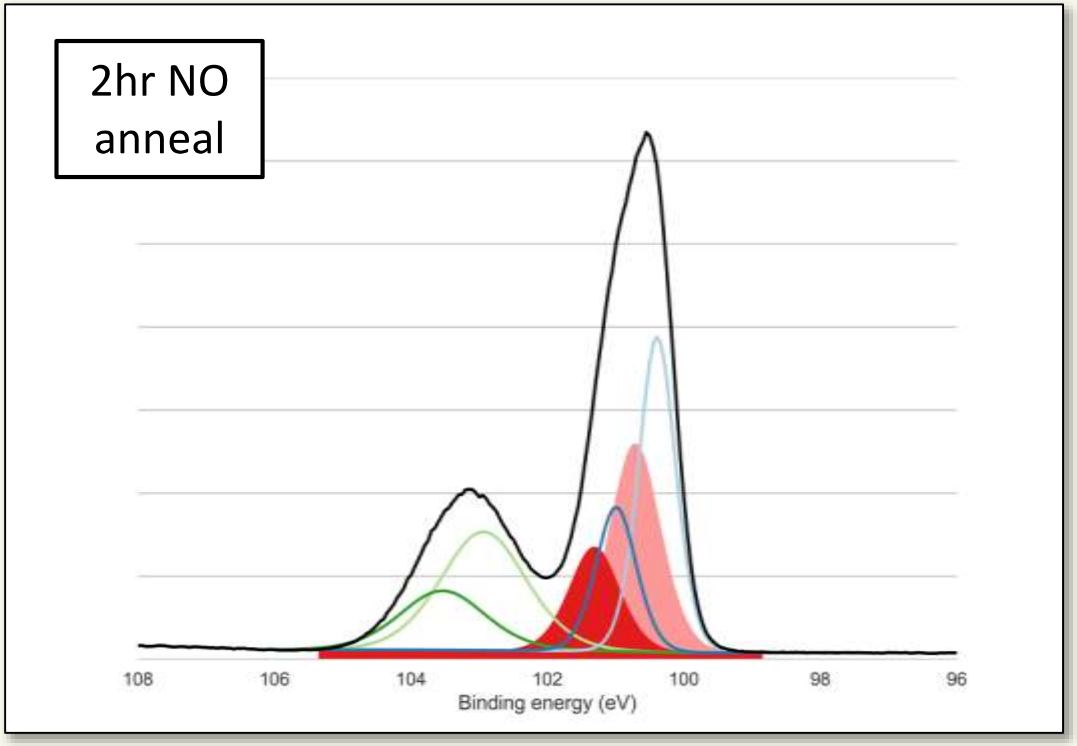
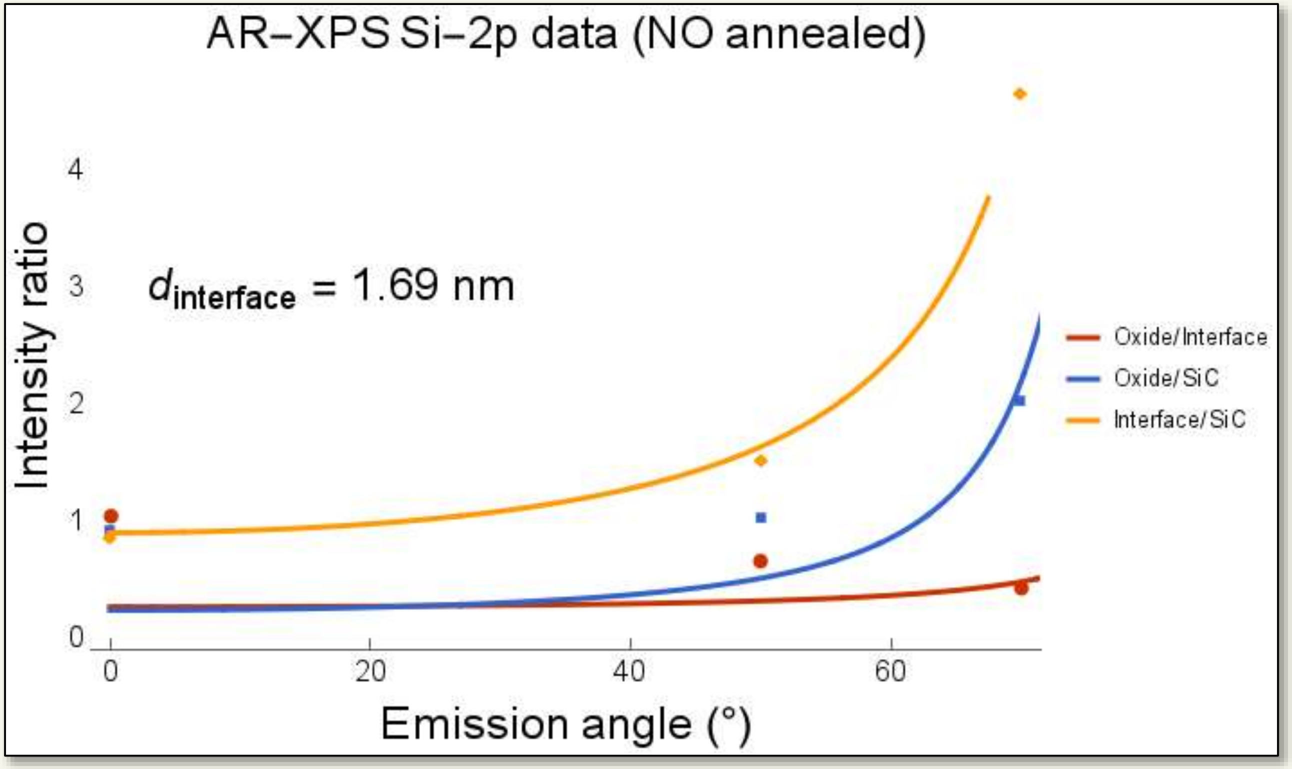


Interface signal

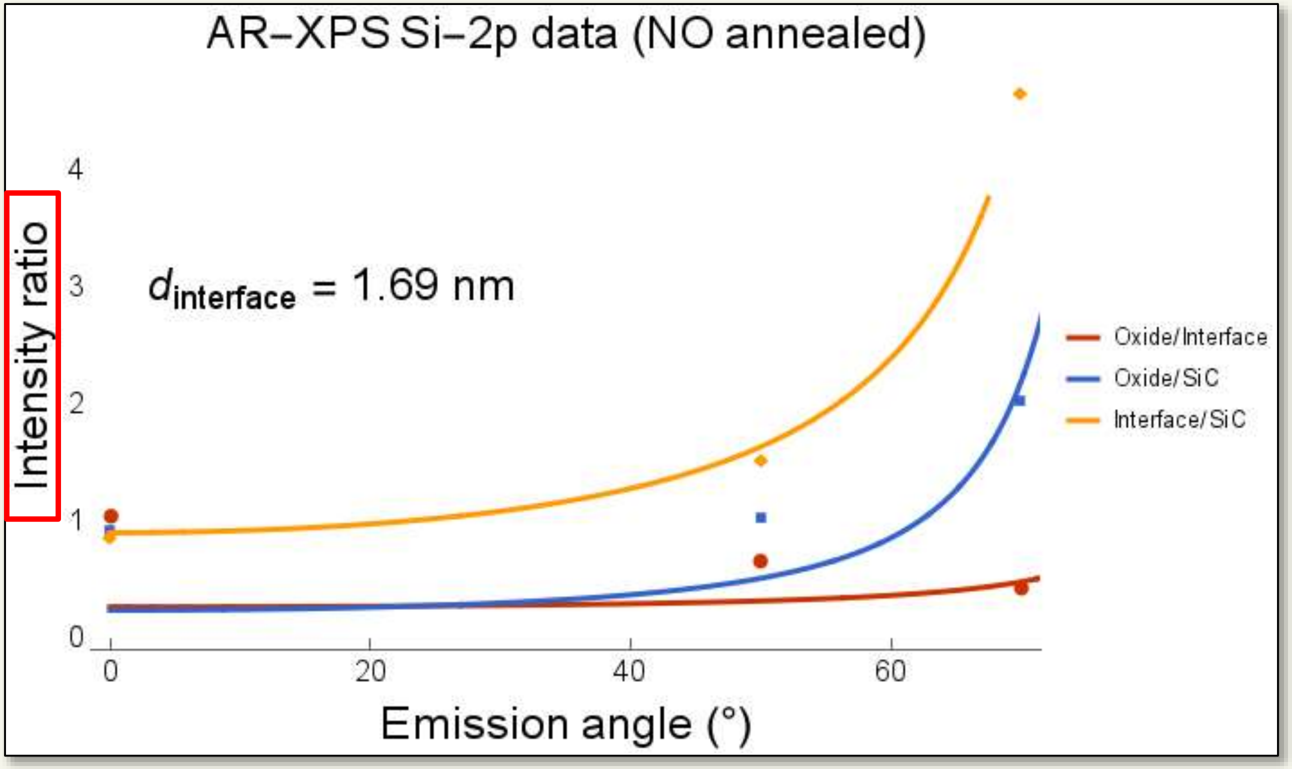
Angle resolved XPS - interface width



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

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

Angle resolved XPS - interface width



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

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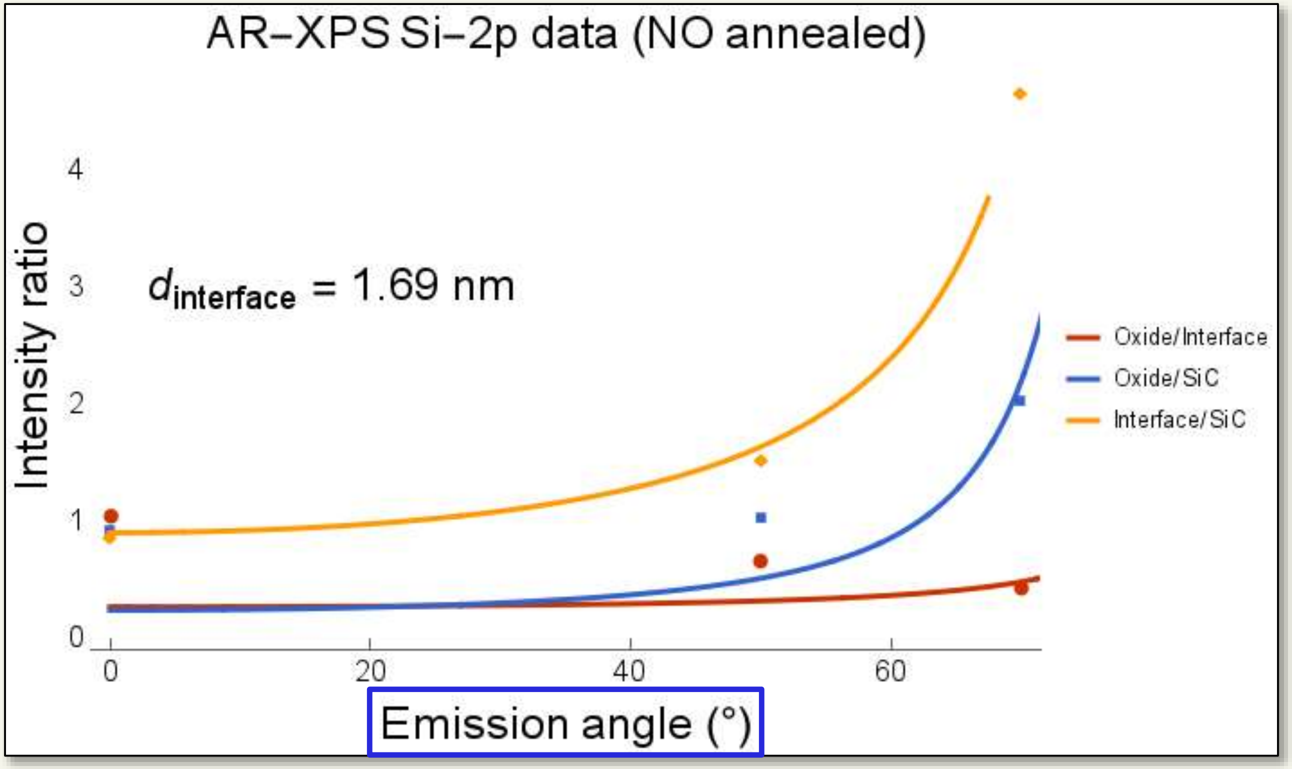
Angle resolved XPS - interface width



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

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

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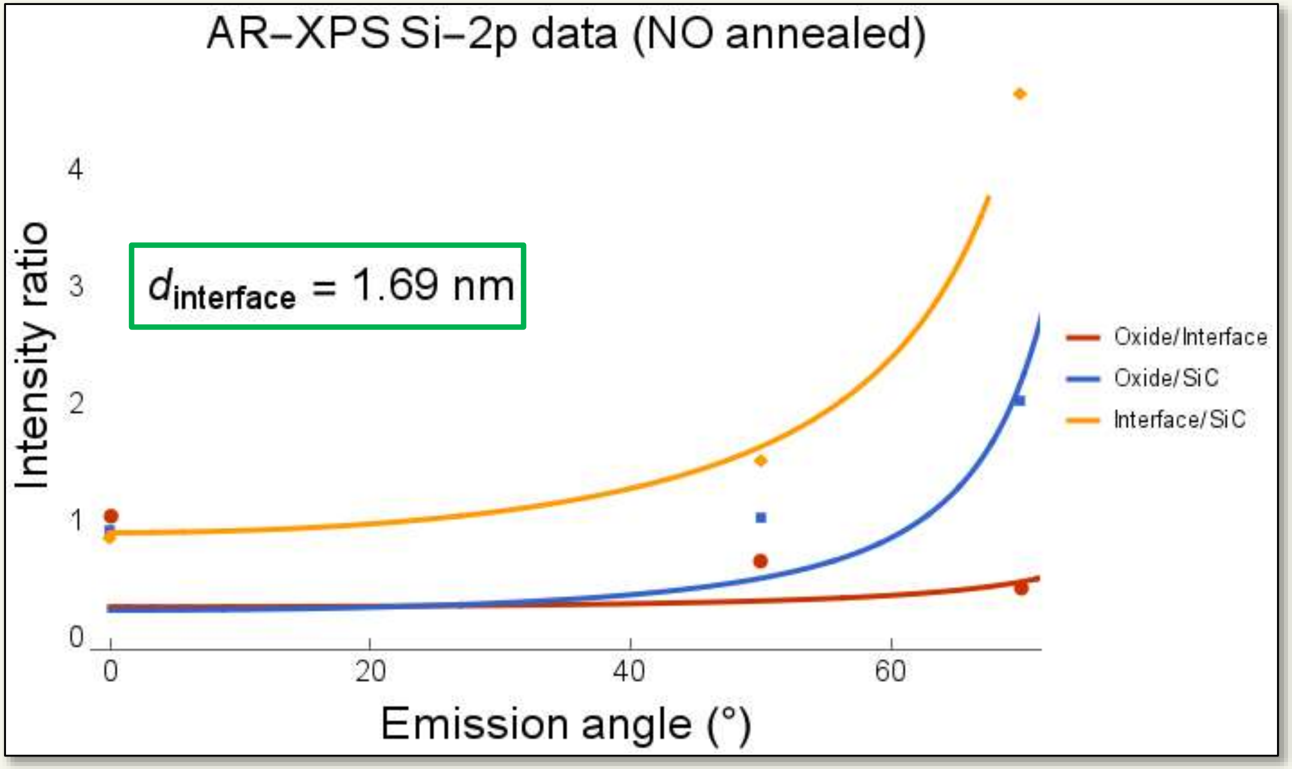

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Face / Treatment	$d_{\text{interface}}$
O_2 oxidation	2.97 nm
O_2 oxidation + NO Post-anneal	1.69 nm

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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, Geometric Phase Analysis)

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

Questions/comments/discussion?

Joshua Taillon: jtaillon@umd.edu

Lourdes Salamanca-Riba: riba@umd.edu