A. JAMES CLARK SCHOOL OF ENGINEERING

Systematic Characterization of the SiC/SiO_2 Transition Layer in NO-Annealed MOSFETs

Joshua Taillon,[1] Joon Hyuk Yang,[1] Claude Ahyi,[2] John Rozen,[3] John Williams,[2] Leonard Feldman,[3] Tsvetanka Zheleva,[4] Aivars Lelis,[4] and **Lourdes Salamanca-Riba**[1]

Thursday, August 16, 2012

[1] Materials Science and Engineering, University of Maryland College Park

[2] Physics, Auburn University

[3] Physics and Astronomy, Vanderbilt University

[4] Army Research Laboratory

THE DEPARTMENT *of* MATERIALS SCIENCE AND ENGINEERING

Outline

- Introduction, Motivation, Background, Goals
- Experimental Methods
  - EELS, Spectrum Imaging, w_{TL} determination
- Transition layer width results
  - Composition ratios
  - Interdiffusion
  - HAADF-STEM
  - Chemical shift
- Correlation with electronic measurements
- Conclusions, Remaining questions, etc.

Motivation and Background

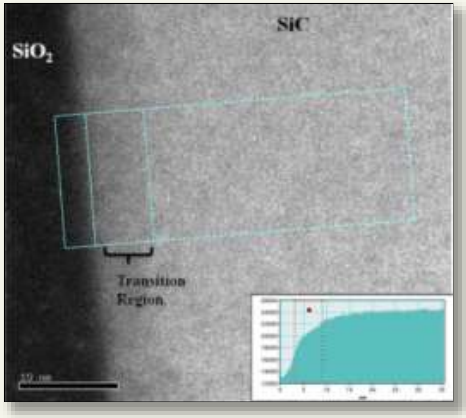
- SiC: Very promising for high temperature, high power, and high frequency devices
  - 4H polytype (bulk):[1] $\boldsymbol{E_g} = 3.23\ \text{eV}$, $\boldsymbol{\mu_e} \approx 850\ \frac{\text{cm}^2}{\text{V}\cdot\text{s}}$, $\boldsymbol{\varepsilon} = 10$, $\boldsymbol{\kappa} = 3.7\ \frac{\text{W}}{\text{cm}\cdot{}^\circ\text{C}}$
  - MOSFET devices limited by poor channel carrier mobility and reliability
  - Electrically active defects at the SiC/SiO_2 interface inhibit devices during channel inversion
- Possible nature of these defects?

Excess carbon at the interface (maybe?)	K. Chang, *et al*. J. Electron. Mater. **32**, 464 (2003). X. Shen, *et al*. J. Appl. Phys. **108**, 123705 (2010). Hatakeyama, *et al.* Mater. Sci. Forum **679**, 330 (2010).
3-fold Si and C coordination and C_i	M. Di Ventra, *et al.* Phys. Rev. Lett. **83**, 1624 (1999). S. Pantelides, *et al.* Mater. Sci. Forum **527**, 935 (2006).
V_{Si} and V_o at interface	C. Cochrane, *et al*. Appl. Phys. Lett. **100**, 23509 (2012). J. Rozen, *et al*. J. Appl. Phys. **105**, 124506 (2009).

[1] Semiconductor database: *http://www.ioffe.ru/SVA/NSM/Semicond/SiC/index.html*

Previous Work

- Transition region around SiC/SiO_2 interface
  - EELS evidence of enhanced C concentration in SiC at interface
    - T. Zheleva, *et al*. Appl. Phys. Lett. **93**, 022108 (2008).
  - Transition layer width (w_{TL}) lowered by NO post-anneal; measured with HAADF-STEM intensity profiles
    - Inverse linear correlation between w_{TL} and mobility
    - T. Biggerstaff, *et al*. Appl. Phys. Lett. **95**, 032108 (2009). →



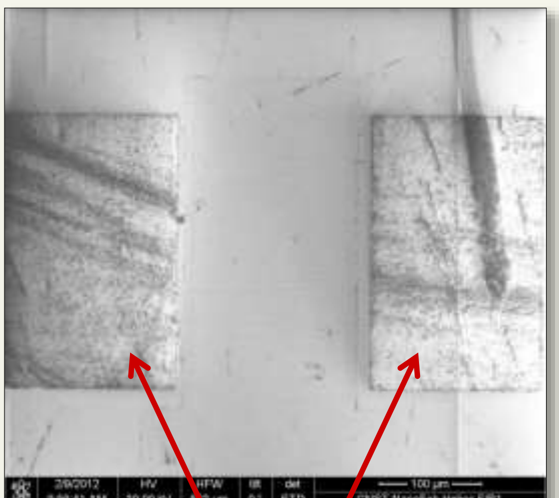
- Our previous results (2010 -)
  - Not annealed, 240 min NO anneal, 20 hr N_2 plasma anneal, 4 and 6 hr O_2 plasma oxidation
  - Annealing in all cases yielded sharper interfaces than non-annealed
    - N_2 plasma most effective, but no systematic results

Goals

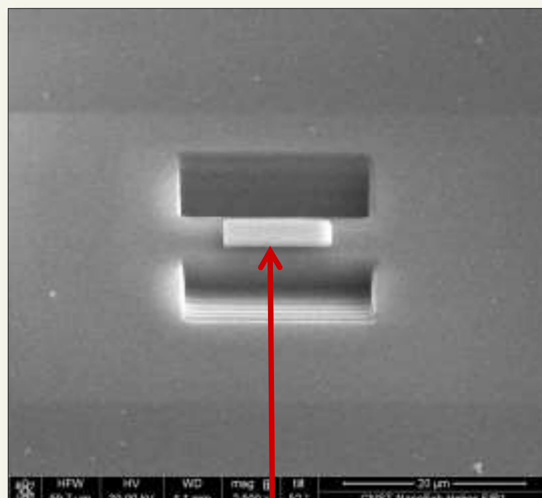
- Physically and chemically characterize transition layer as a function of NO post-annealing time
  - Systematic set of SiC MOSFETS that received 0, 15, 30, 60, 120, and 240 minute post-oxidation anneals at 1175°C
  - Using HRTEM, HAADF-STEM, and EELS
  - Correlate with measured device properties
  - Investigate conflicting claims of excess C at interface
- Develop reliable, objective, and reproducible methods by which to determine w_{TL}
  - For comparison to previous works and future sample sets

TEM Specimen Preparation

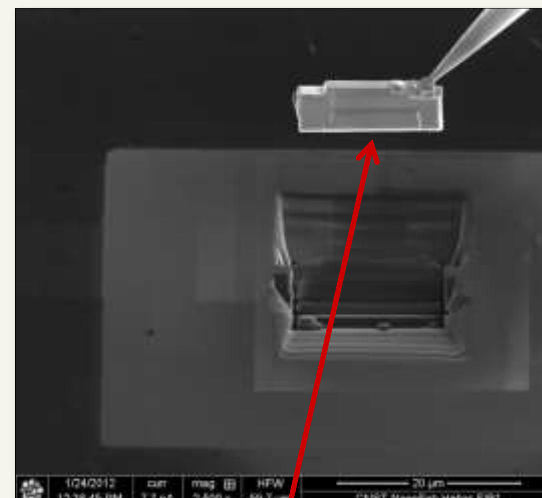
- Cross-sectional TEM specimen prepared with FIB
  - Mo gate metallization removed from devices with H_2O_2 etch
  - Two protective Pt layers (e^- beam and ion beam deposited) prevent Ga^+ ion implantation and oxide layer damage
  - Previous work used C protective layer, complicating analysis



S/D contacts



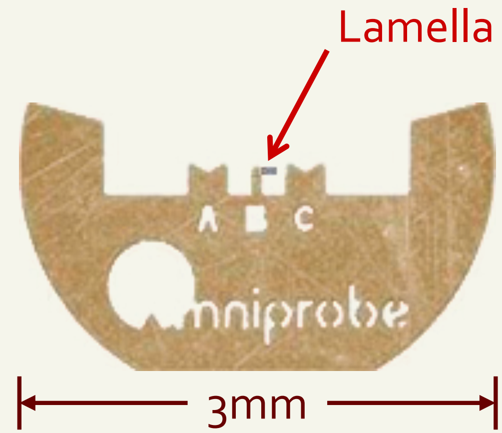
Protective Pt

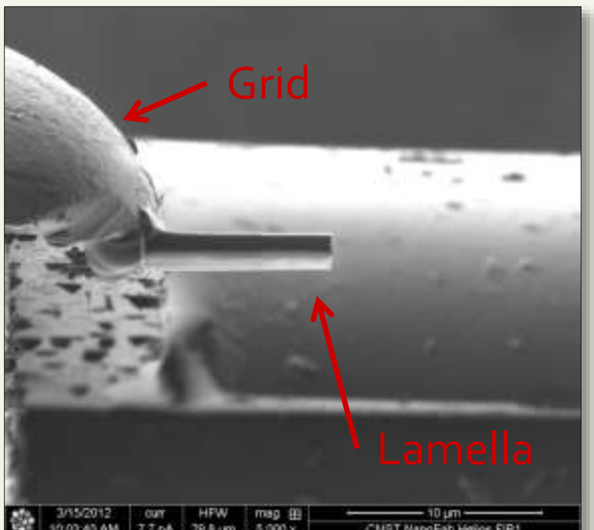


Lamella removed with tungsten probe

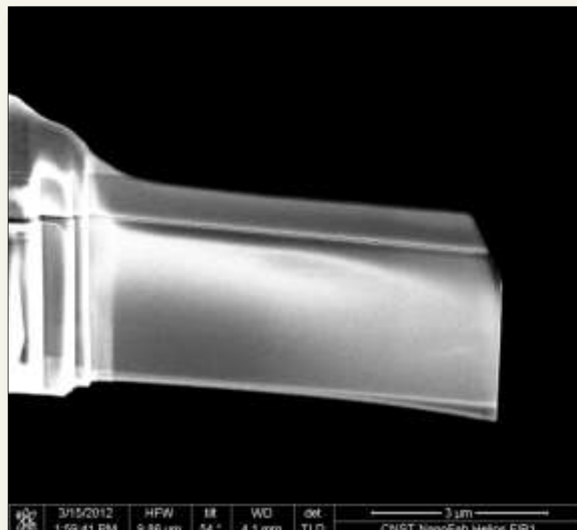
TEM Specimen Preparation

- Typical specimen dimensions
  - 6μm × 3μm × ≈ 80nm
  - Flat "flag" specimen attached to grid with Pt

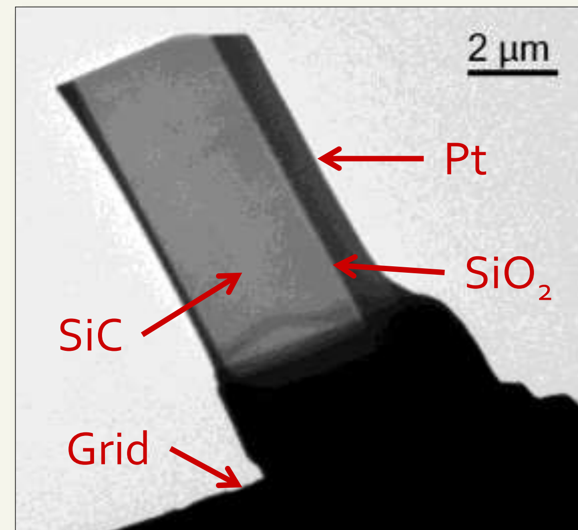




FIB image before thinning

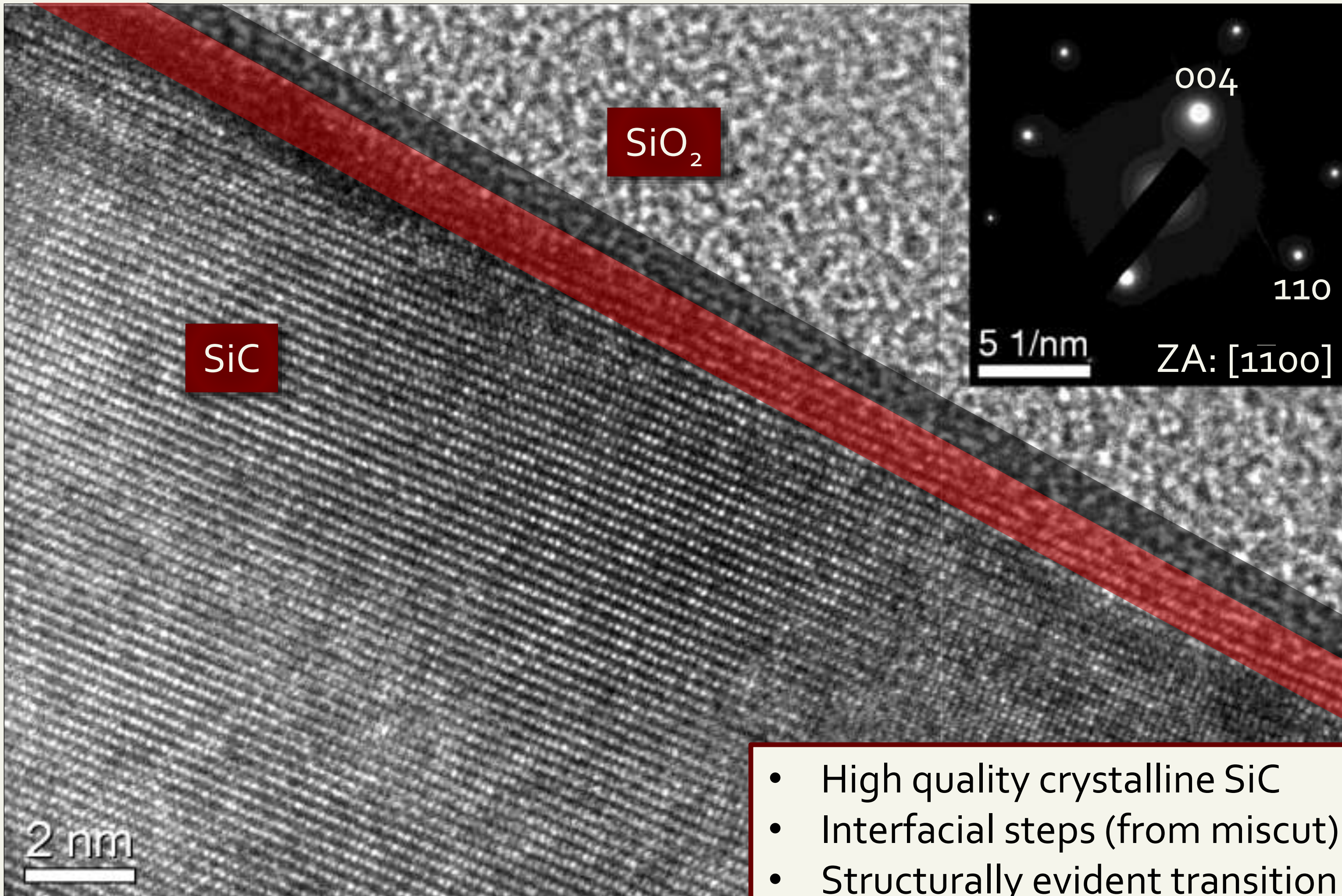


SEM image after thinning



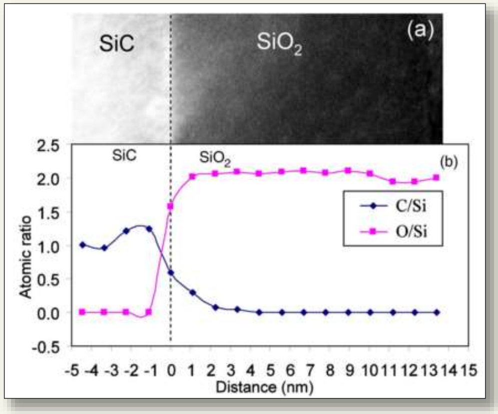
Low-mag TEM image

HRTEM of Transition Layer



- High quality crystalline SiC
- Interfacial steps (from miscut)
- Structurally evident transition layer

Transition Layer Thickness Measures



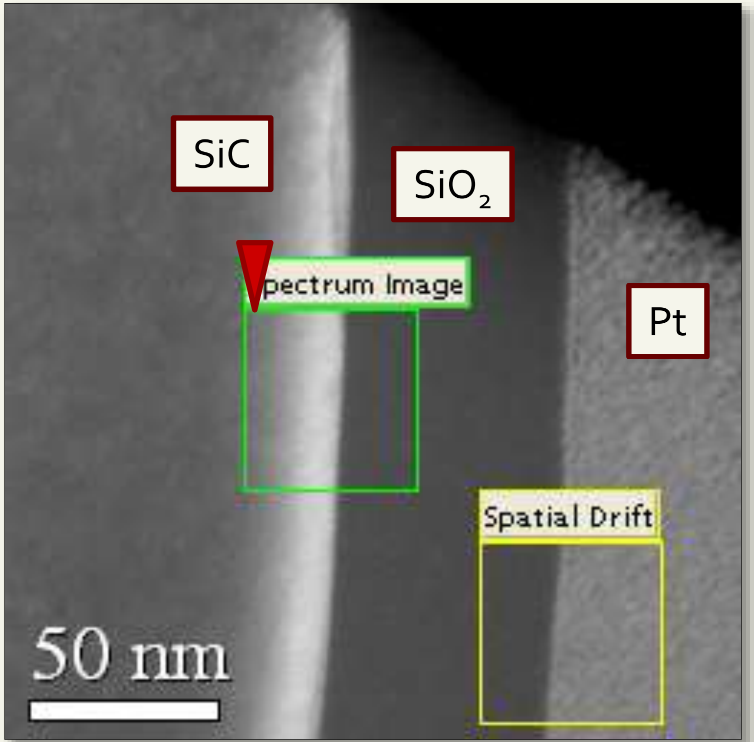
- EELS composition ratios ($^{C}/_{Si}$ and $^{O}/_{Si}$) →
  - Zheleva, *et al.* Appl. Phys. Lett. **93**, 022108 (2008).
  - Eliminates many sources of systematic error[1]
- Relative "interdiffusion" of C and O (EELS)
  - Gives idea of transition layer on each side of interface
  - C into SiO_2 and O into SiC
- HAADF-STEM image intensity profiles
  - HAADF reveals Z-contrast from variations in atomic composition
- Chemical shift of Si-$L_{2,3}$ EELS edge
  - Well-documented shift in edge onset energy (SiC: 100 eV; SiO_2: 104 eV)
    - G. Auchterlonie, *et al.* Ultramicroscopy, **31**, 217 (1989).
  - Reveals information about local Si bonding

[1] R. Brydson and R.M.S. (UK), *Electron Energy Loss Spectroscopy*, Microscopy Handbooks (Bios, 2001).

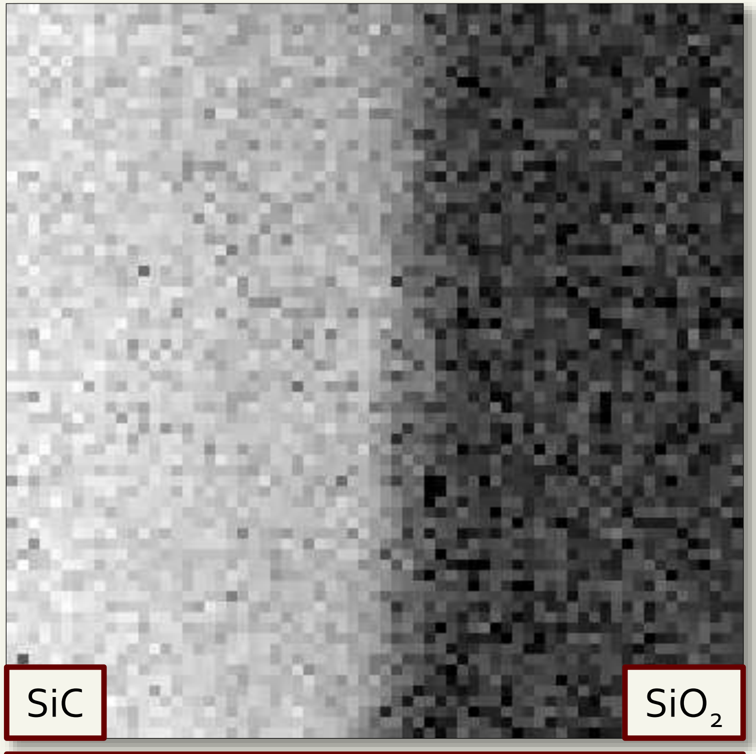
EELS Experimental Methods

- Gatan Tridiem post-column imaging filter
  - Simultaneous HAADF-STEM imaging and EELS collection
- Spectrum imaging
  - Raster beam across sample, collecting a full EELS spectrum at each pixel
  - Spectrum image (SI) represents a map of spectra
  - Allows for short collection time, limited beam damage
  - Sum parallel to interface to improve $^{S}/_{N}$ ratio
- Collection parameters:
  - Linear or areal SI across interface
  - Dwell time < 0.01 s
  - Spot size (~ spatial resolution) = 0.5 nm
  - Energy dispersion = 0.3 eV/channel

Spectrum Imaging

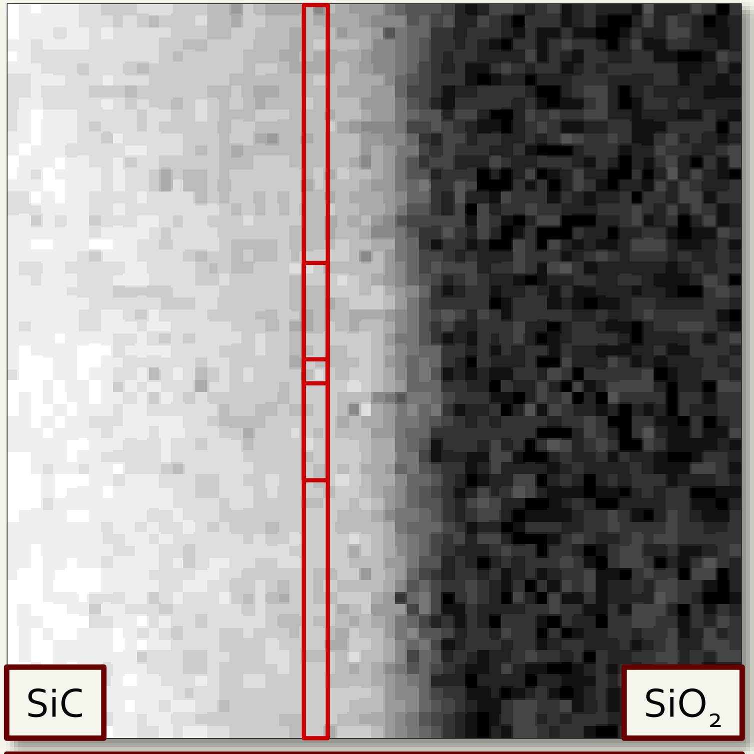


HAADF Image
(60 minute anneal)

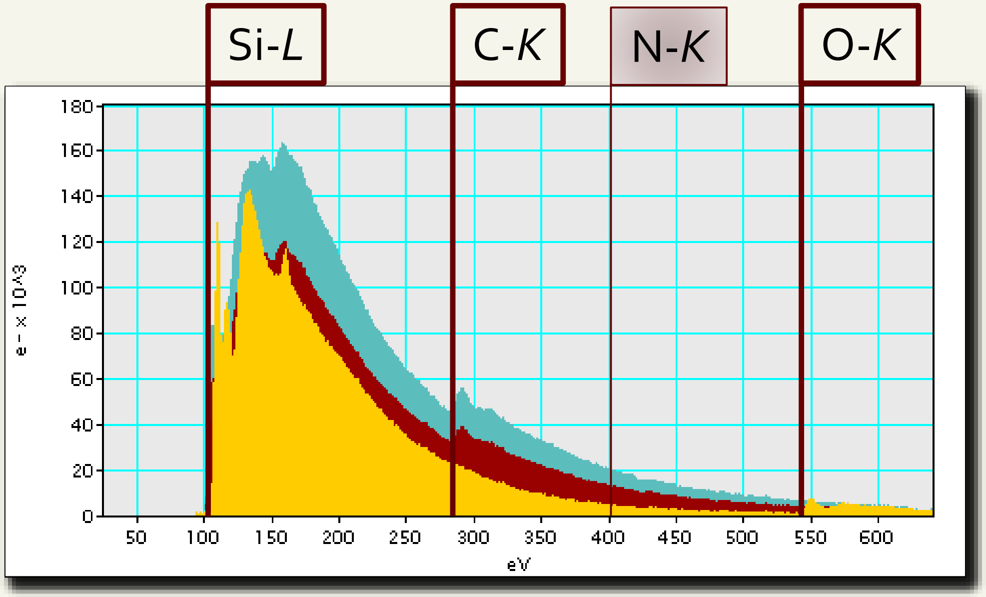


Spectrum Image
(60 minute anneal)

Spectrum Imaging



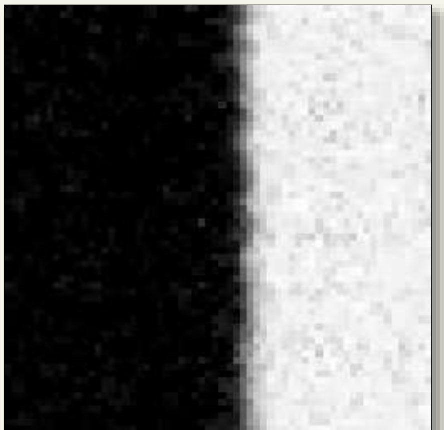
Spectrum Image
(60 minute anneal)



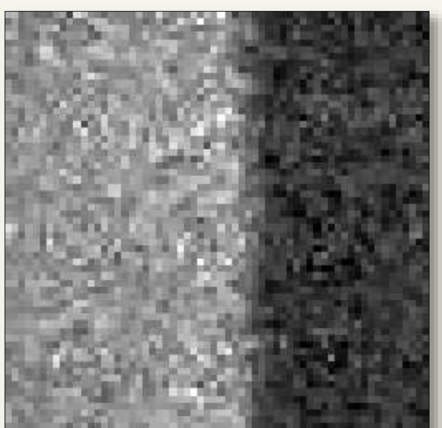
Background-subtracted spectrum
(60 minute anneal)

Elemental Composition Ratio

O

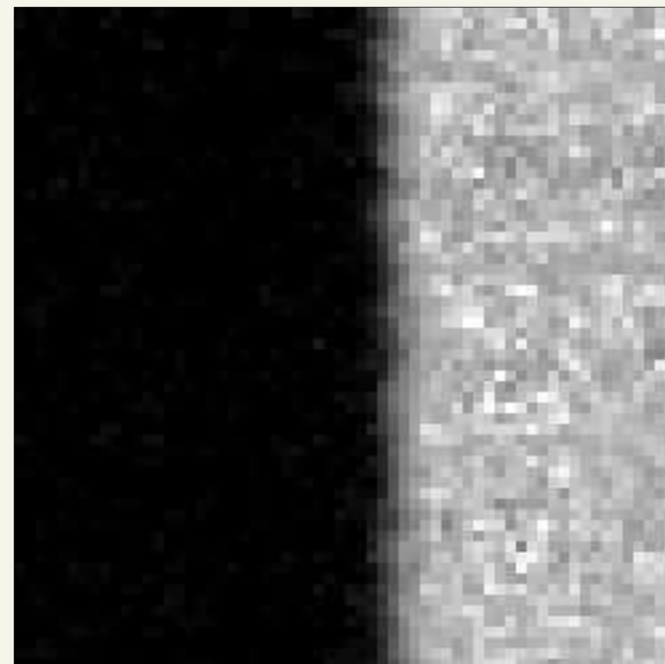


Si



Relative composition maps
(60 minute anneal)

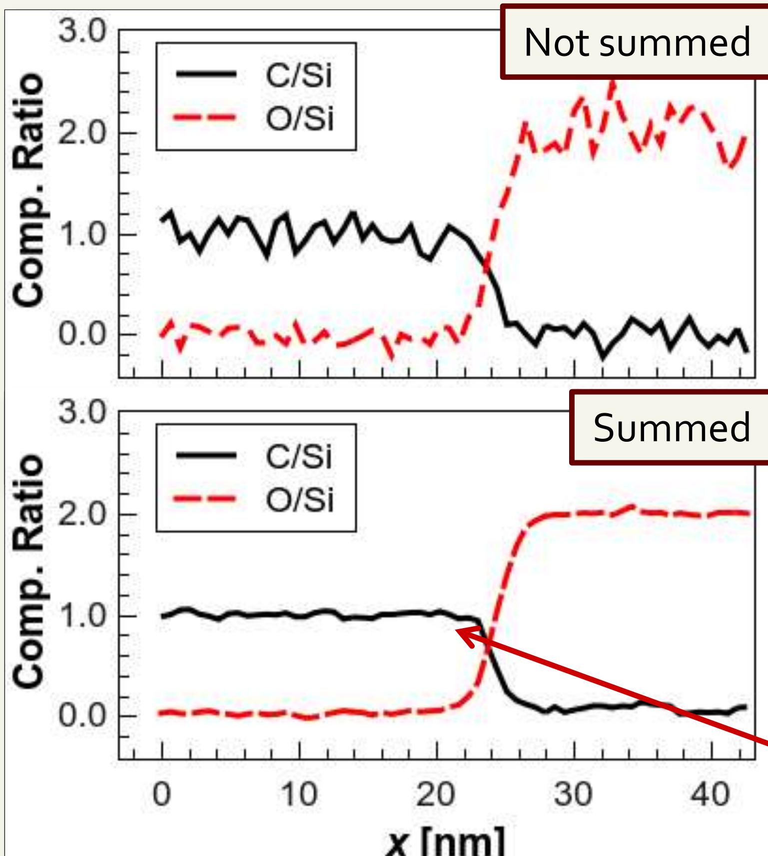
=



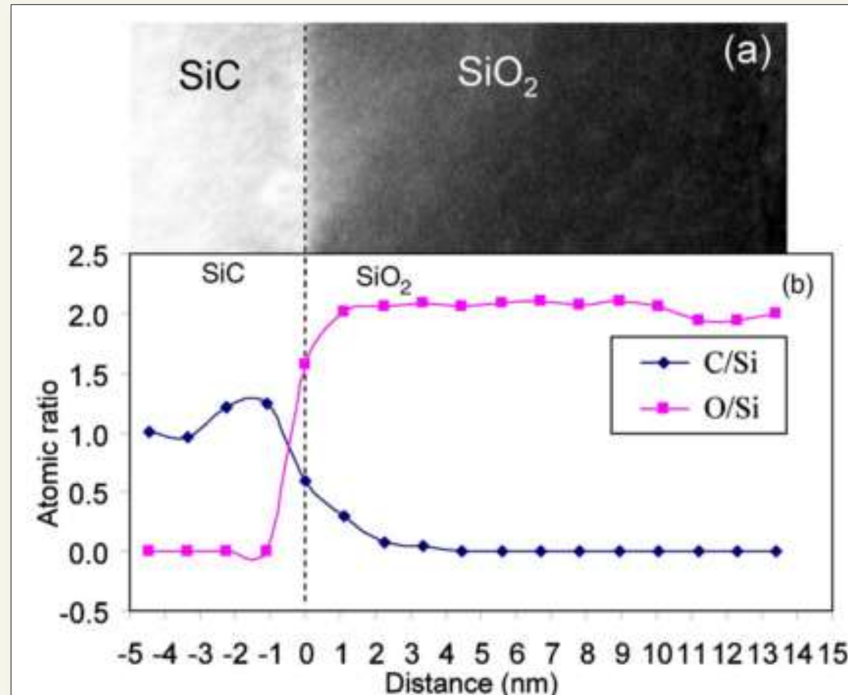
Composition Ratio Map
(60 minute anneal)

w_{TL} from Composition Ratios

- Profile of atomic ratio maps:



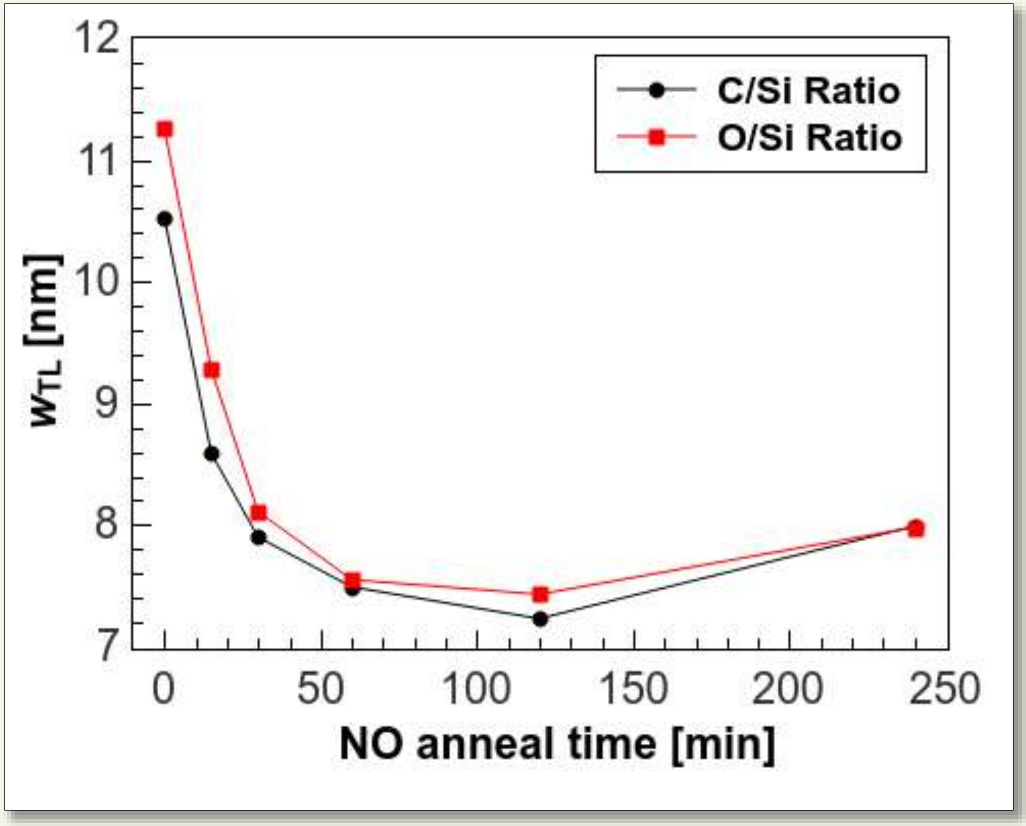
- Use $\frac{d}{dx}$ of profile to find w_{TL}:



No excess C at interface

Results (composition ratios)

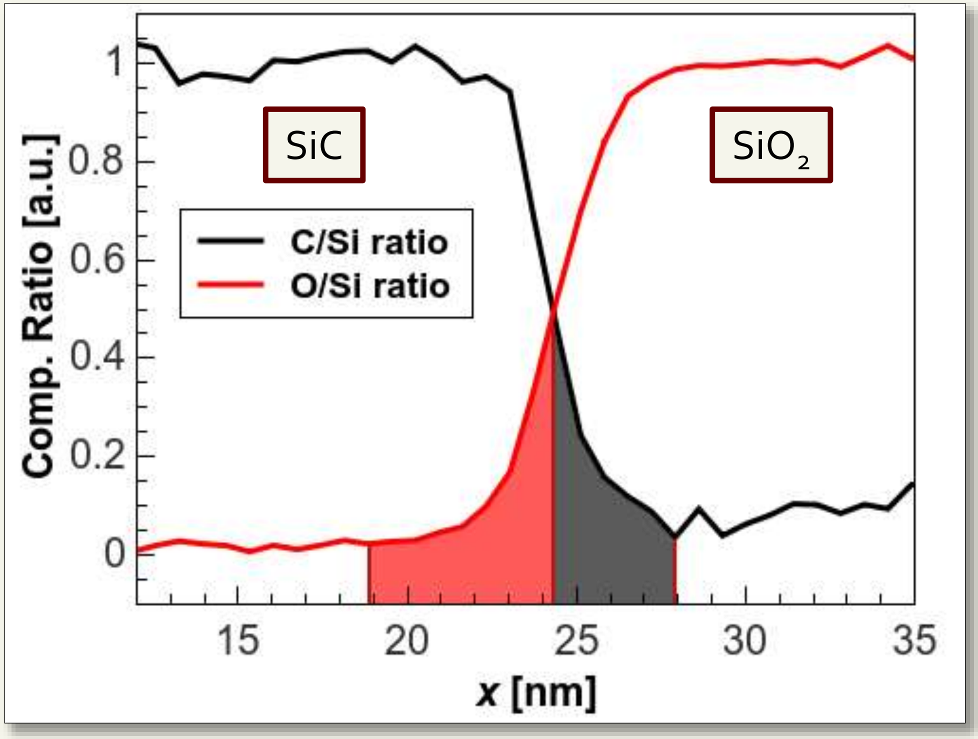
- w_{TL} decreases as anneal time increases
  - EELS signal from 240 min anneal sample very noisy (C implantation)
- NO anneal results in significant improvement!
  - Removal of defects
  - Smoothing of interface[1]
- $\frac{O}{Si}$ slightly larger than $\frac{C}{Si}$
  - Significant difference?



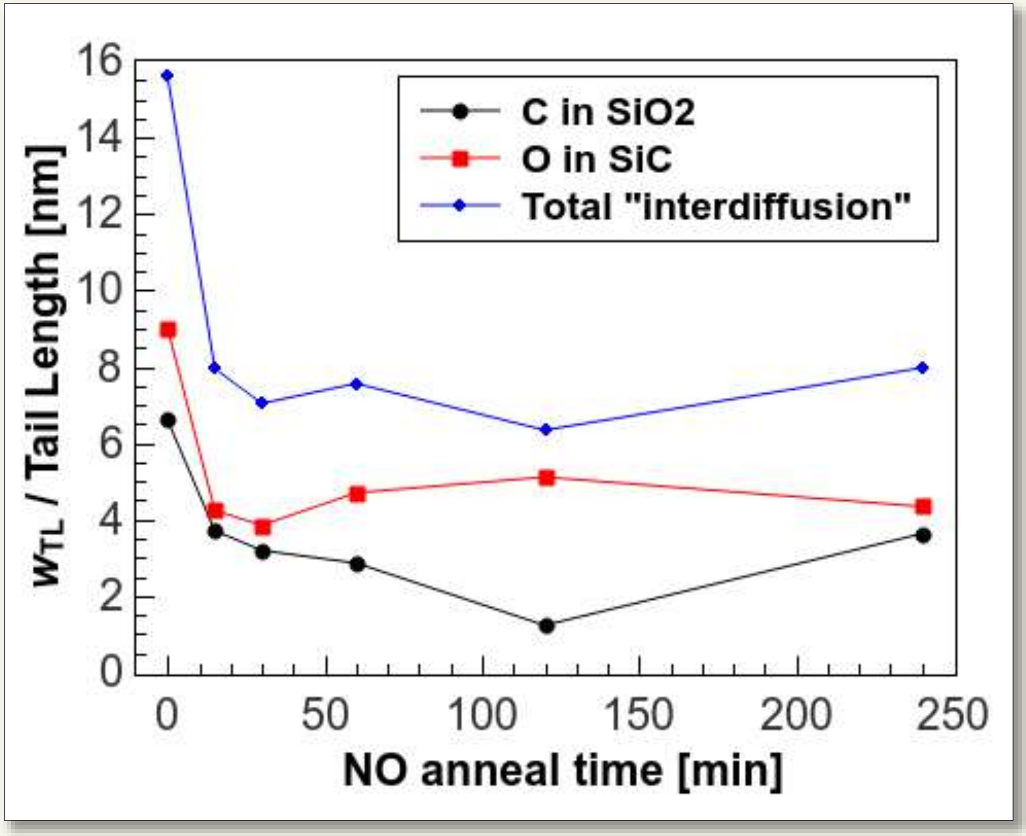
[1] P. Tanner, *et al.* Journal of Electronic Materials 28, 109–111 (1999).

"Interdiffusion" lengths

- Useful to see tails of C concentration in SiO_2 and O in SiC
  - Normalized bulk concentrations and measured tails with derivative



Results (“Interdiffusion”)

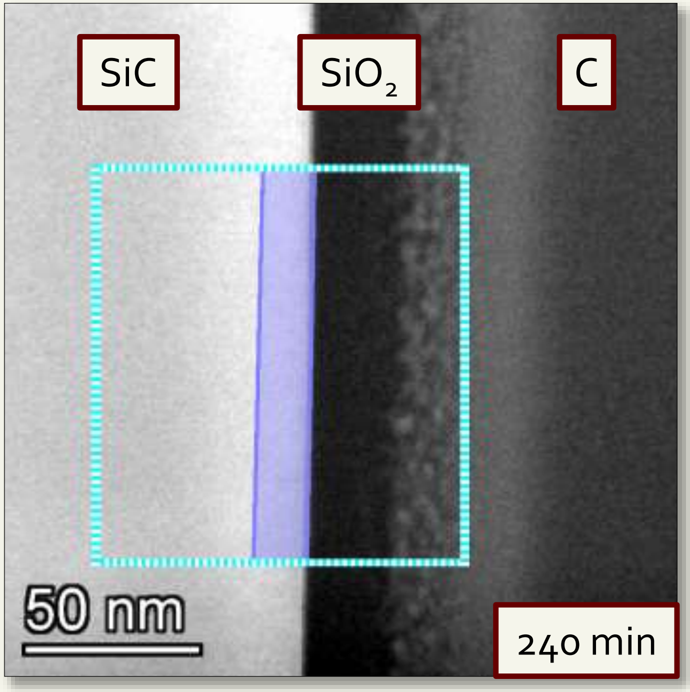


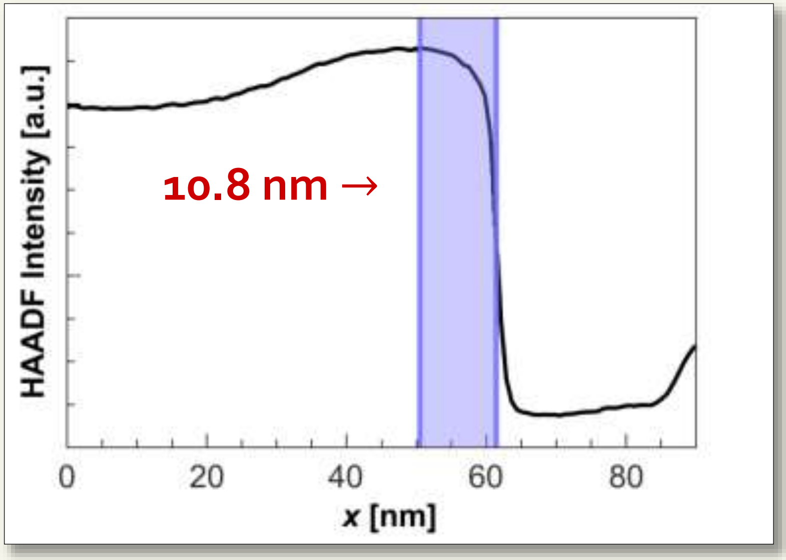
- Again, NO anneal shows significant improvement
- O tail in SiC always larger than C tail in SiO_2
- Why?
  - C efficiently removed during oxidation[1]
  - O solubility in SiC very low[2]

[1] M. Di Ventra and S. Pantelides, Phys. Rev. Lett. **83**, 1624 (1999).
[2] M. Di Ventra and S. Pantelides, J. Electro. Mater. **29**, 353 (2000).

HAADF-STEM Image Intensity[1]

- Z-contrast from enhanced scattering cross-sections of heavier elements
  - w_{TL} defined as width between peak and inflection point

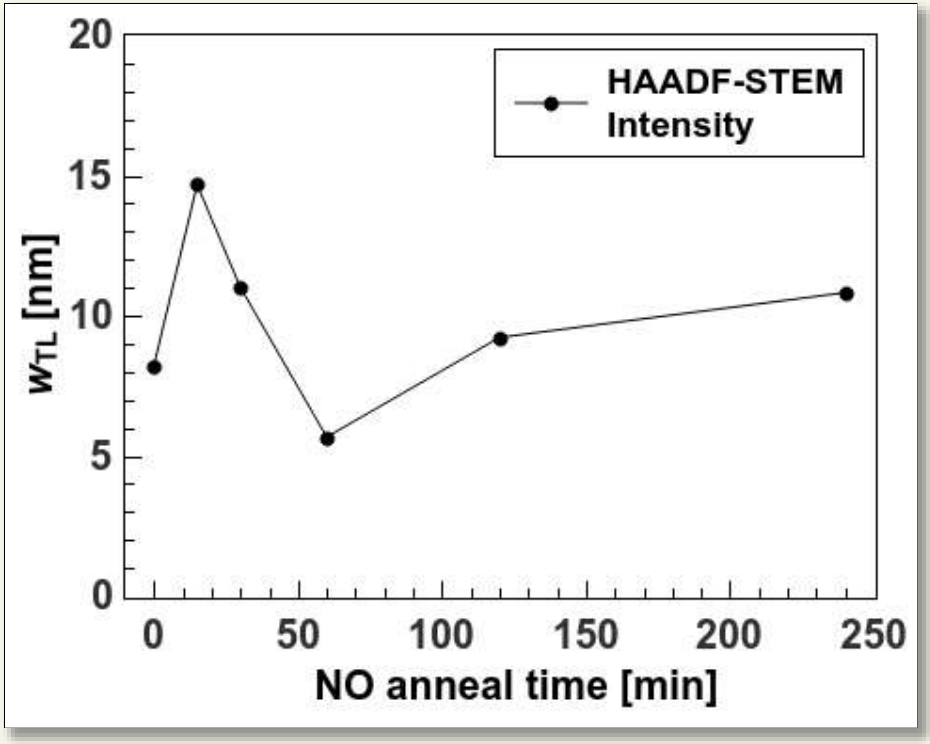


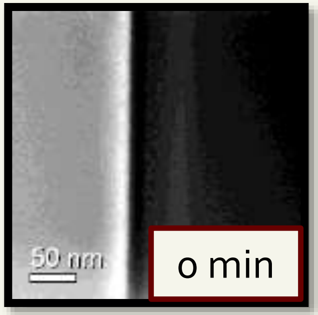


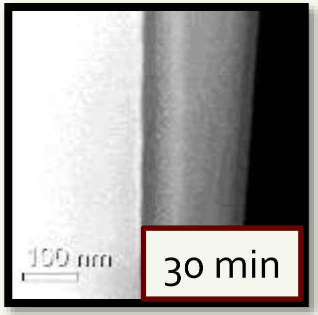
[1] After: T. Biggerstaff, *et al*. Appl. Phys. Lett. **95**, 032108 (2009).

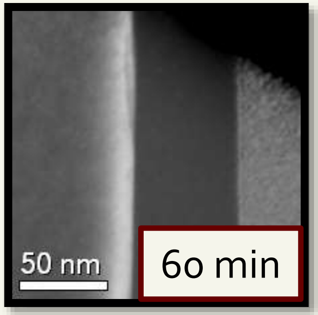
HAADF-STEM Results

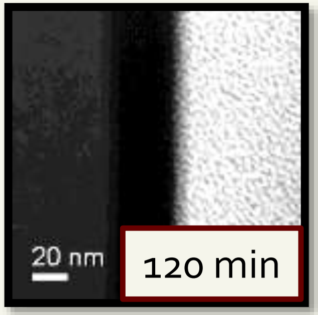
- w_{TL} results on same order as other methods
- Poorer overall trend in w_{TL}
  - HAADF images varied substantially between samples
- No excess C, but bright intensity line
  - Thickness variations?



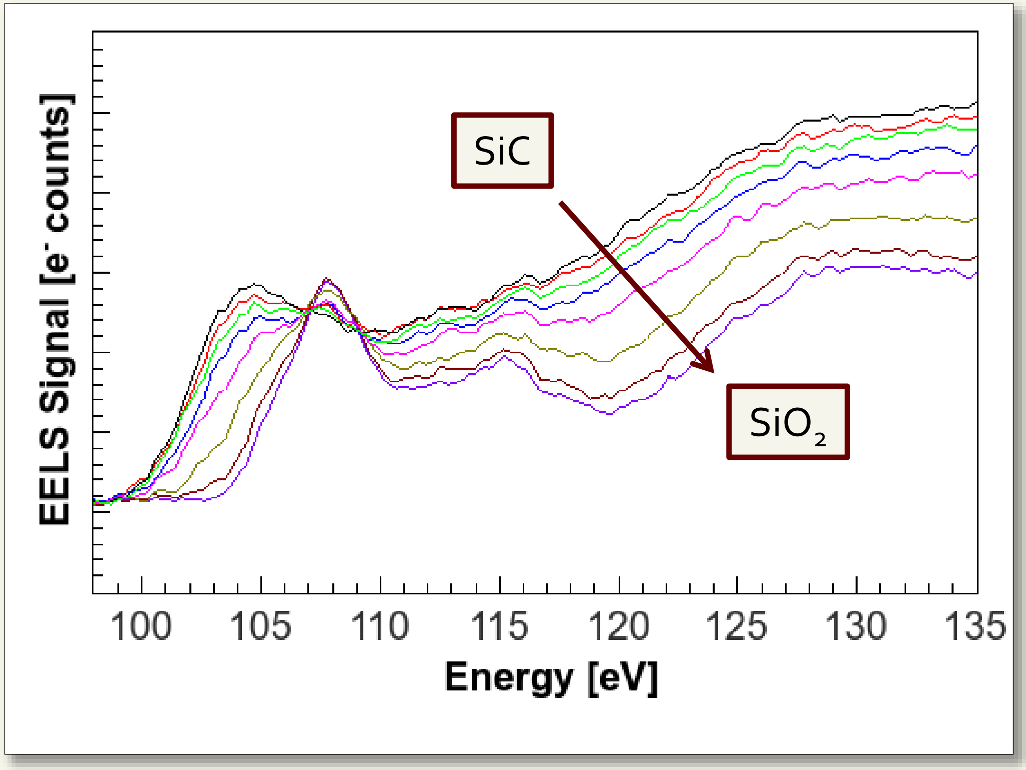








Si-$L_{2,3}$ Chemical Shift

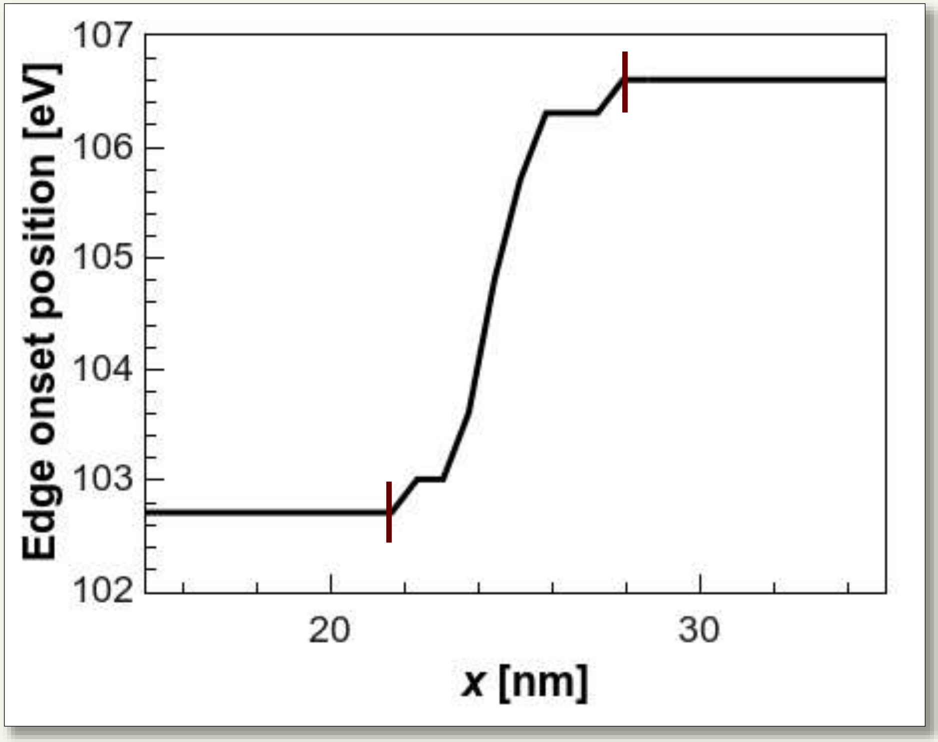


- EELS fine structure (ELNES) reflects local unoccupied density of states
  - Edge onset → minimum energy needed to excite core shell e⁻
  - Semiconductor → insulator
  - Band gap widens, core levels depressed relative to E_F[1]
    - Charge transfer from Si → C/O
    - Onset shifts to higher energy

[1] D. Muller, Ultramicroscopy **78**, 163 (1999).

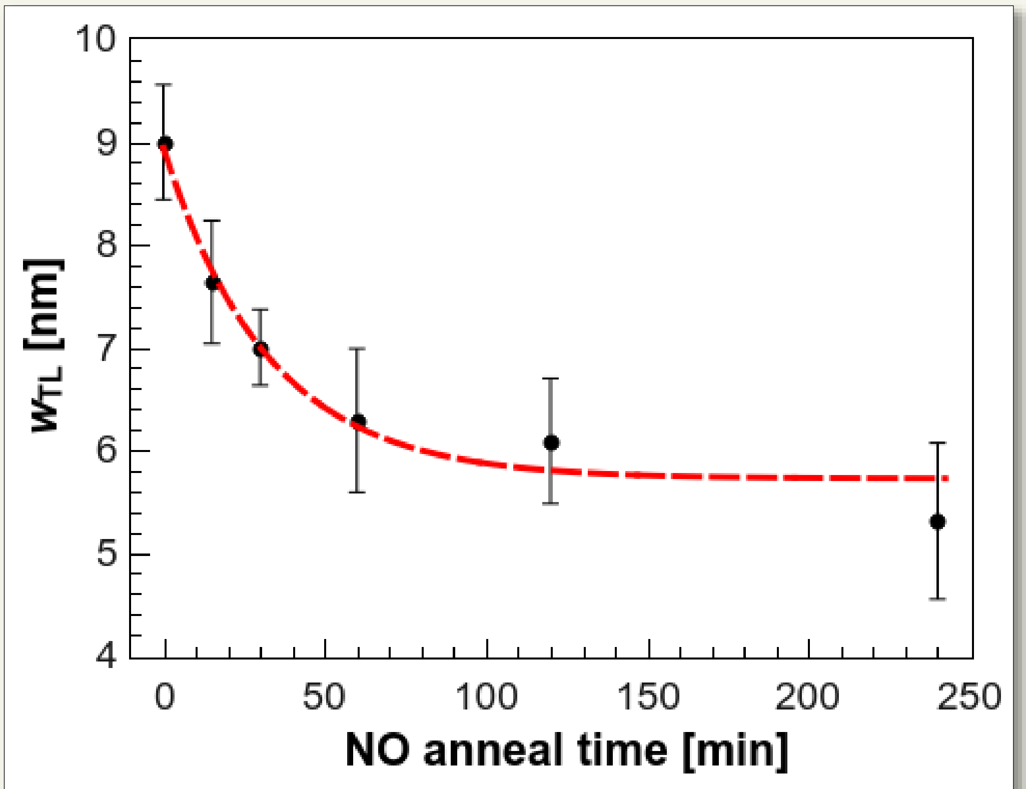
Si-$L_{2,3}$ Chemical Shift

- Wrote script to find inflection point of edge onset[1]
  - Sum spectra parallel to interface
  - Take second derivative
  - Find zero and output position as function of distance
- Gradual and monotonic shift
  - Bonding changes
  - Possible strain
  - Implies a mix of Si-C and Si-O
  - Modeling needed



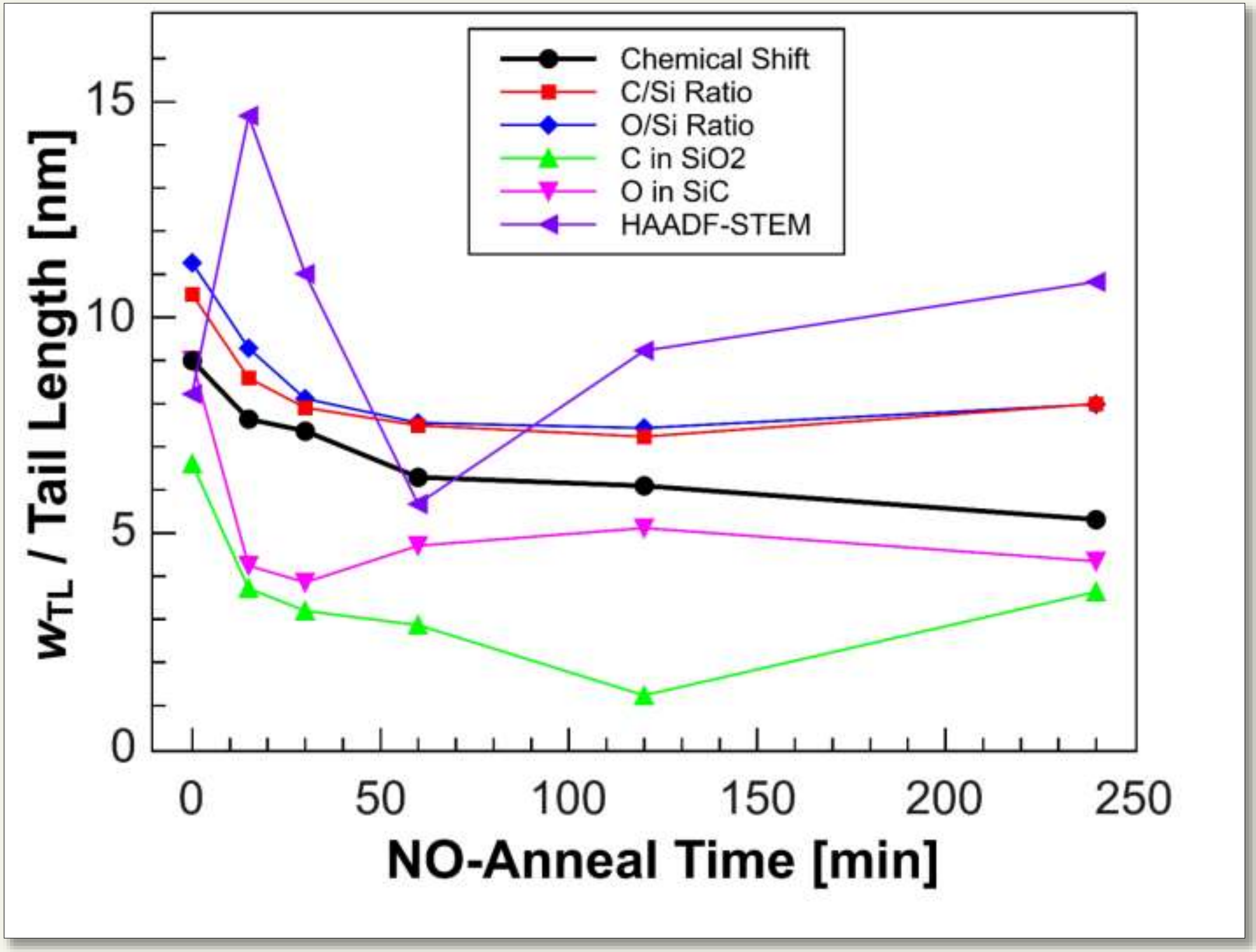
[1] 1 D. Muller, P. Batson, and J. Silcox, Physical Review B **58**, 11970 (1998).

Chemical Shift Results



- Significant NO anneal improvement
  - Best method to track transition layer
  - (Relatively) insensitive to spectral noise
    - 240 min now fits trend
  - Clear exponential decay
- Characterizes different phenomenon
  - Bonding vs. composition

All Results

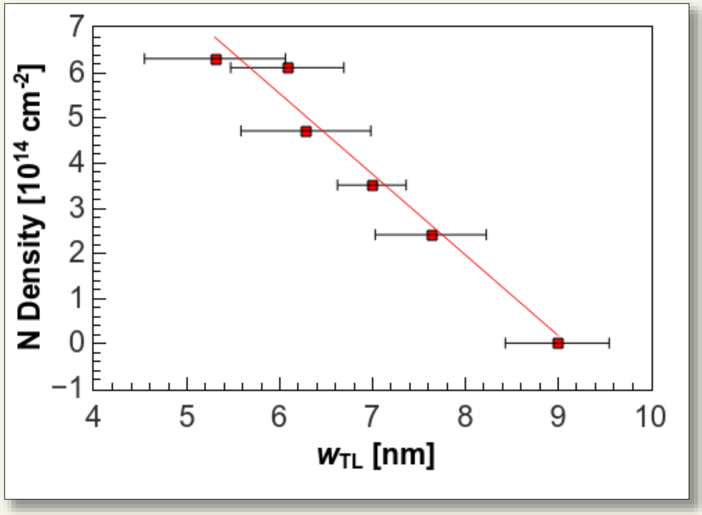


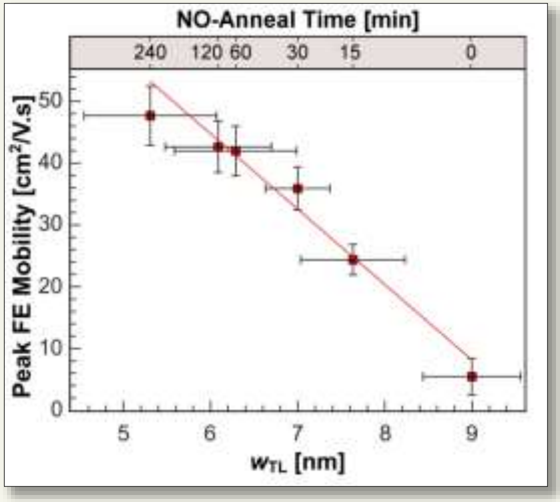
Chemical Shift
C/Si Ratio
O/Si Ratio
C in SiO2
O in SiC
HAADF-STEM
w_{TL} / Tail Length [nm]
0
5
10
15
0
50
100
150
200
250
NO-Anneal Time [min]

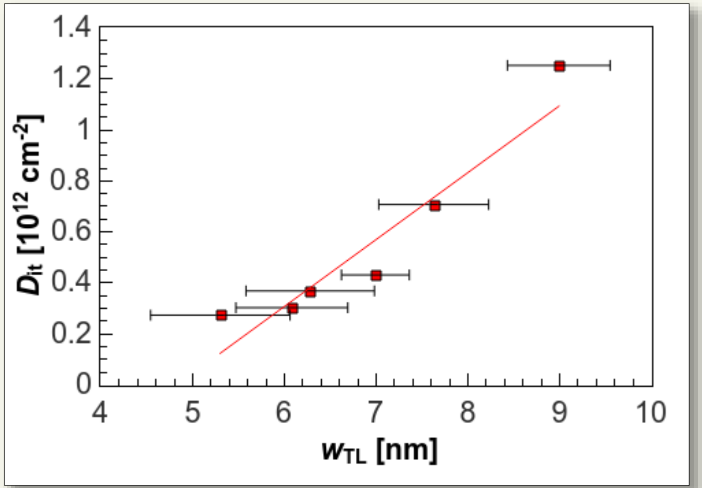
A. JAMES CLARK SCHOOL OF ENGINEERING

Electronic Measurements

- Data taken by J. Rozen
  - J. Rozen, *et al.* IEEE Trans. Electron. Dev. **58**, 3808 (2011).
  - J. Rozen, *et al.* J. Appl. Phys. **105**, 124506 (2009).
- w_{TL} correlates:
  - Linearly with N density
  - Inverse-linearly with D_{it} and μ_{FE}
- Effect of NO?
  - Active removal of C (and O) at interface
  - Removes defects associated with these species







Conclusions

- w_{TL} decreases nonlinearly with increasing NO anneal time
  - All w_{TL} determination methods agree!
  - Chemical shift of Si-$L_{2,3}$ edge onset was most reliable
  - Inverse linear correlation of w_{TL} with μ and N-density
  - Linear correlation of w_{TL} with D_{it}
- No excess C on either side of interface
- No layer of N detected at interface – but should be able to see it
- Monotonic chemical shift of Si-$L_{2,3}$ edge
  - Indicates gradual progression of Si bonding character
- HAADF-STEM images are least reliable in w_{TL} determinations
  - EELS is more consistent; more informative (chemistry and bonding)
- Smallest transition region for 4hr anneal → w_{TL} = 5.3 nm
- Described objective w_{TL} determination method for future comparison

Remaining Questions

- Structure and composition of interface, but what about bonding configurations?
  - High energy resolution ELNES study would give more bonding information
  - Could be correlated with XPS depth profiles across the interface
- How does NO treatment compare with N_2 and P passivation?
  - Do we observe the same $w_{TL} - \mu$ correlation and how do w_{TL} values compare?
- Can we detect N concentrations at the interface using EELS?
  - Longer collection times, thicker sample, and optimized collection parameters needed

Acknowledgements

- ARL Contracts W911NF-11-2-0044 and W911NF-07-2-0046.
- NispLab at UMD – supported by NSF and MRSEC
- Dr. Joshua Schumacher and Mike Hernandez at NIST

THANK YOU

Questions and comments?