Full field chemical imaging of buried native sub-oxide layers on doped silicon

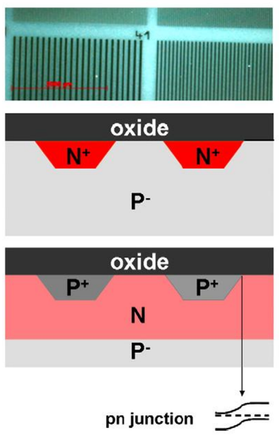
Luiz F. Zagonel[1,2,3], F. de la Peña[1], Nicholas Barrett[2], M. Walls[1], Olivier Renault[4]

1- Univercité Paris-Sud, Laboratoire de Physique des Solides, CNRS/UMR 8502, F-91405, Orsay, France
2- CEA, IRAMIS, SPCSI, LENSIS, F-91191 Gif-sur-Yvette, France
3- Current address: **Brazilian Nanotechnology National Laboratory, Campinas, Brazil**
4- CEA, LETI, MINATEC, 38054 Grenoble Cedex 9, France

ABSTRACT

We use fully energy-filtered X-ray photoemission electron microscopy to analyze the spatial distribution of the silicon sub-oxide structure at the SiO_2/Si interface as a function of underlying doping pattern. A native oxide layer was present during the analysis. The data was acquired in spectrum imaging mode, ie.: a sequence of energy filtered images is recorded so that spectra can be extracted for each pixel of the image stack. Using a spectroscopic pixel-by-pixel curve fitting analysis, we obtain the sub-oxide binding energies and intensity distributions over the full field of view. Binding energy maps for each oxidation state are obtained with a spatial resolution of 120 nm. The experimental data are used to obtain quantitative maps of the sub-oxide layer thickness and also their spatial distribution over the p–n junctions.

Sample

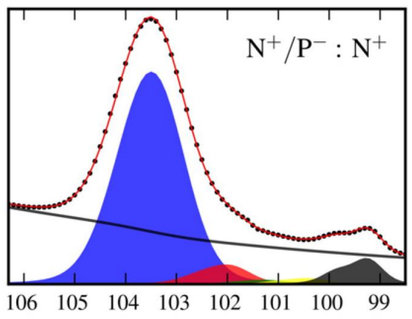


The samples consisted of two-dimensional doped patterns implanted into silicon with a native oxide layer. One region is heavily doped while the other is lightly doped.

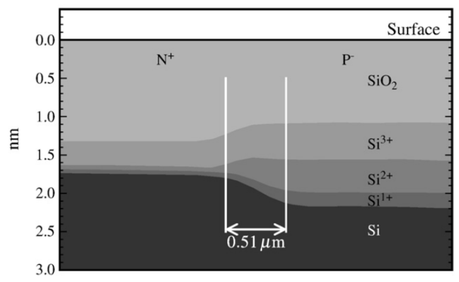
Sample	n-type (at/cm^3)	p-type (at/cm^3)
N^+/P^-	10^{20}	1×10^{15}
P^+/N	2.5×10^{16}	10^{20}

Layer	$\sigma_{Layer}/\sigma_{Si}$	$\lambda_{Layer}/\lambda_{Si}$	n_{Layer}/n_{Si}	$I^{\infty}_{Layer}/I^{\infty}_{Si}$
Si^{1+}	1	1.28	0.86	1.10
Si^{2+}	1.10	1.56	0.73	1.25
Si^{3+}	1.60	1.84	0.59	1.74
Si^{4+}	2.08	2.12	0.46	2.03

Results at a glance

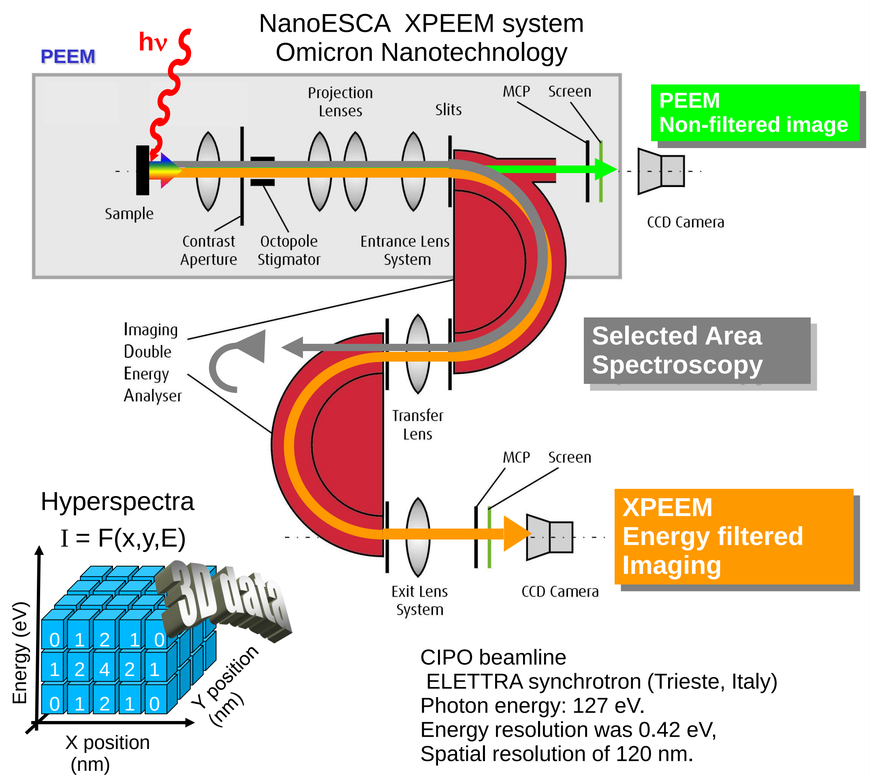


Spectrum from a selected area and its decomposition in silicon sub-oxides.



Reconstructed depth profile of silicon oxides.

Experimental Design

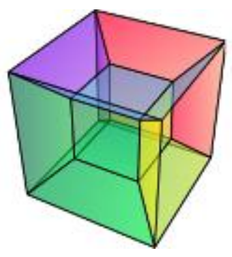


CIPO beamline
ELETTRA synchrotron (Trieste, Italy)
Photon energy: 127 eV.
Energy resolution was 0.42 eV,
Spatial resolution of 120 nm.

Find out more:

Full field chemical imaging of buried native sub-oxide layers on doped silicon patterns
F. de la Peña et al., Surface Science 604 (2010) 1628–1636. doi:10.1016/j.susc.2010.06.006

Spatially resolved, energy-filtered imaging of core level and valence band photoemission of highly p and n doped silicon patterns
N. Barrett et al., J. Phys.: Condens. Matter 21 (2009) 314015. doi:10.1088/0953-8984/21/31/314015



Hyperspy:

a Python toolbox for (hyper)spectroscopy

Hyperspy has been developed as a tool to facilitate hyperspectral data analysis. Specifically, it provides easy access to multidimensional curve fitting, peak analysis and machine learning algorithms, as well as a viewing framework for navigating data and reading and writing capabilities for some popular hyperspectral formats.

http://www.hyperspy.org

Free your data

- Hyperspy can read and write to multiple formats (see table)
- As much as possible, all original file information (metadata included) is retained
- The default data format for output, HDF5, is based in a widespread open standard, facilitating data exchange with other software

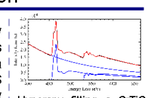
Format	Read	Write
Gatan's dm3	Partial	-
FEI's emi and ser	Partial	-
HDF5	Complete	Complete
Image	Complete	Complete
MRC	Complete	-
EMSA/MSA	Complete	Complete
NetCDF	Complete	-
Ripple	Complete	Complete

n-dim curve fitting

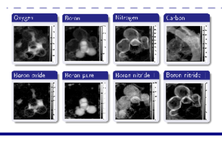
- Hyperspy can easily build additive 1D models from easy to define components
- It can fit a model to hyperspectral data of arbitrary dimensions
- Supports coupling, fixing, bounding, weights, maximum likelihood estimation...
- It comes with many predefined components: from lines and Gaussian peaks up to EELS ionisation edges.

Easy EELS quantification

Hyperspy includes not only components for EELS edges and background but also a special mode for EELS quantification with many functions to make the process of building and fitting the model as automatic as possible.



Hyperspy fitting a $SrTiO_3$ EEL spectrum with Hartree-Slater ionisation edges, a power law background and multiple scattering by LL convolution

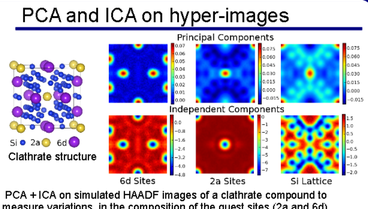


Elemental and bonding maps of a boron nitride nano particle quantification by fitting a model that includes the ELNES to a 2D hyperspectrum.

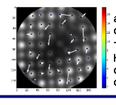
Statistical analysis

- Hyperspy makes it easy to perform PCA for noise or dimensionality reduction
- It includes algorithms for non heteroscedastic noise: weighted PCA and MLPCA
- It integrates ICA algorithms from MDP in an easy to follow workflow
- It includes tools to pre-process the data in order to reduce the artefacts that can arise

PCA and ICA on hyper-images



PCA + ICA on simulated HAADF images of a clathrate compound to measure variations in the composition of the guest sites (2a and 6d)



PCA + ICA employed to study the variation in position and number of atomic species through several images of a dislocation core in an $SrTiO_3$ grain boundary.

The image vectors superimposed on the average HAADF image represent the position changes. The colored spots and corresponding colorbar indicate changes in peak height.

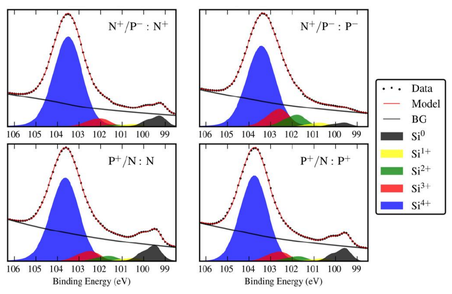
[1] de la Peña, F. *et al. Ultramicroscopy* ***111, 169-176 (2011).***
[2] Arenal, R. *et alUltramicroscopy* ***109, 32-38 (2008).***
[3] Sarahan, M.C., Chi, M., Masiel, D.J. & Browning, N.D *Ultramicroscopy* ***111, 251-257 (2011).***

Intensity and Binding energy: From pixel by pixel fitting to maps

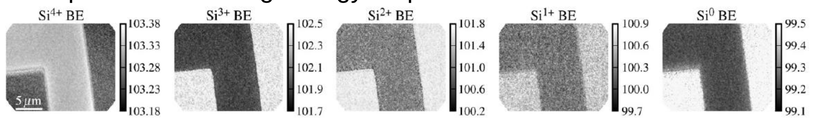
By fitting each pixel of the dataset it is possible to map the binding energy of each silicon-sub-oxide and to map the intensity of each component.

Average Binding energy of each region

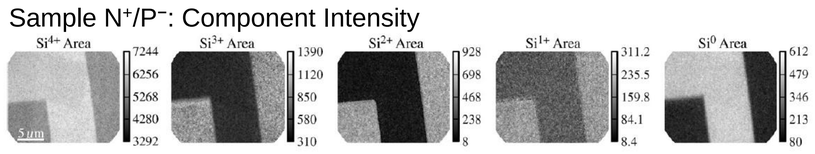
Layer	N^+/P^-		P^+/N	
	N^+	P^-	N	P^+
Si^0	99.24 ± 0.03	99.59 ± 0.09	99.47 ± 0.03	99.57 ± 0.03
Si^{1+}–Si^0	1.05 ± 0.15	1.08 ± 0.17	1.05 ± 0.29	1.05 ± 0.32
Si^{2+}–Si^0	1.82 ± 0.12	2.12 ± 0.12	2.13 ± 0.24	2.02 ± 0.24
Si^{3+}–Si^0	2.70 ± 0.06	2.87 ± 0.12	2.95 ± 0.16	2.78 ± 0.13
Si^{4+}–Si^0	4.10 ± 0.01	3.67 ± 0.09	4.00 ± 0.04	4.01 ± 0.04



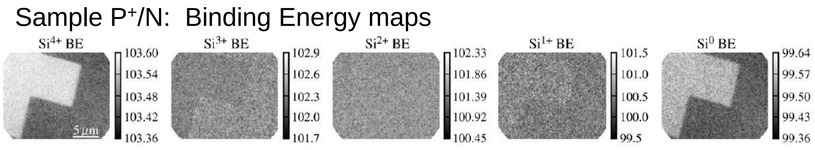
Sample N^+/P^-: Binding Energy maps



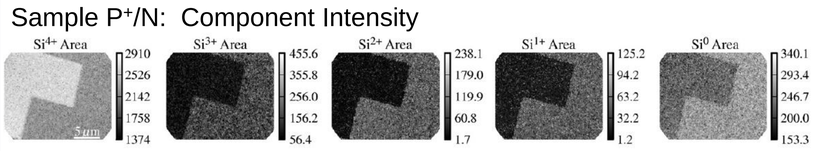
Sample N^+/P^-: Component Intensity



Sample P^+/N: Binding Energy maps

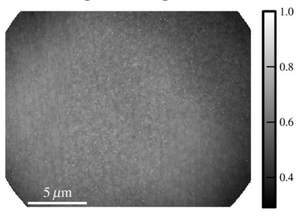


Sample P^+/N: Component Intensity

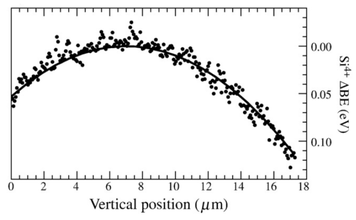


Data correction

Intensity mapping: Vignetting free

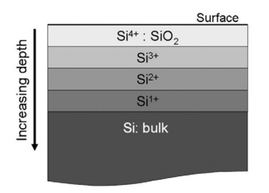


Energy mapping: Isochromaticity



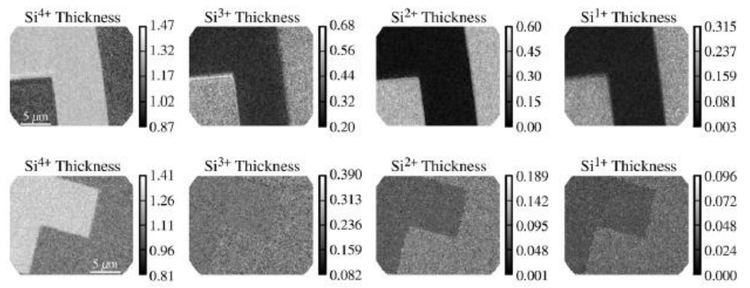
Also: Background removal in each pixel
Photon beam flux decay from electron beam current monitor.

Thickness Maps

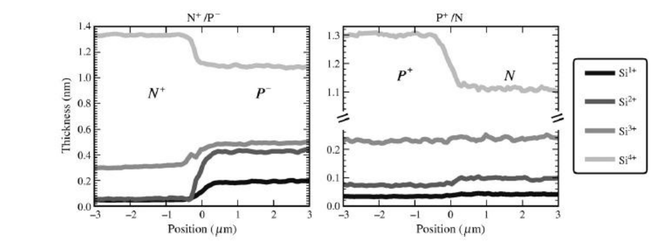


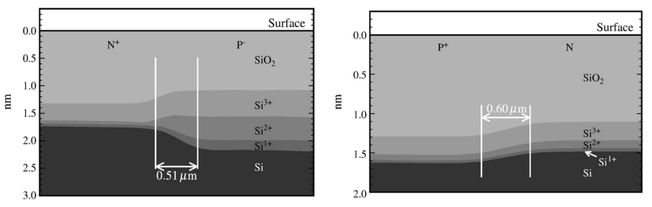
A 5 layer model is used to calculate the thickness of each sub-oxide.

Layer	N^+/P^-		P^+/N	
	N^+	P^-	N	P^+
Si^0	–	–	–	–
Si^{1+}	0.05 ± 0.02	0.19 ± 0.03	0.04 ± 0.01	0.03 ± 0.01
Si^{2+}	0.06 ± 0.03	0.49 ± 0.06	0.10 ± 0.02	0.07 ± 0.02
Si^{3+}	0.31 ± 0.06	0.43 ± 0.05	0.24 ± 0.04	0.23 ± 0.03
Si^{4+}	1.34 ± 0.03	1.08 ± 0.06	1.11 ± 0.05	1.31 ± 0.04



Thickness profiles





Summary

Hyperspy proved to be suitable of

- **Instrumental artefact correction**
- **Pixel-by-pixel curve fitting with multi-component model**
- **Binding energy and Intensity Mapping**
- **Calculated thickness mapping**

P doped oxide is always thicker than N doped.

P^+/N shows little dependence while N^+/P^- has bigger oxide thickness variation. N^+ is the most abrupt interface.