

Semiconductor characterisation in a state-of-the-art electron microscope.

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Francisco de la Pena, Adeline Grenier,
Jean-Luc Rouvi re and Amal Chabli

Outline of talk

- Introduction
 - An introduction to the characterisation platform at Minatec
 - The transmission electron microscope
 - Aberration correction in the TEM
- Aberration corrected TEM for the semiconductor industry
 - Dopant atom counting in GaN quantum dots
 - Strain mapping by geometrical phase analysis
- Field Mapping in the TEM
 - Electron holography for nanofield mapping
 - Strain mapping with nm scale resolution
 - Dark Holography V NBED for strain mapping
 - Field Mapping with 1 nm spatial resolution
- MultiTEM
 - Applied to 28 nm HKMG nMOS device with CESL stress liner
 - EDX for the next generation of electron microscopes

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Minatec - focus on nano and microtechnologies

Education - 1200 Students

**Research - 1200 applied researchers
500 fundamental researchers**

Concentration of tools and competences.

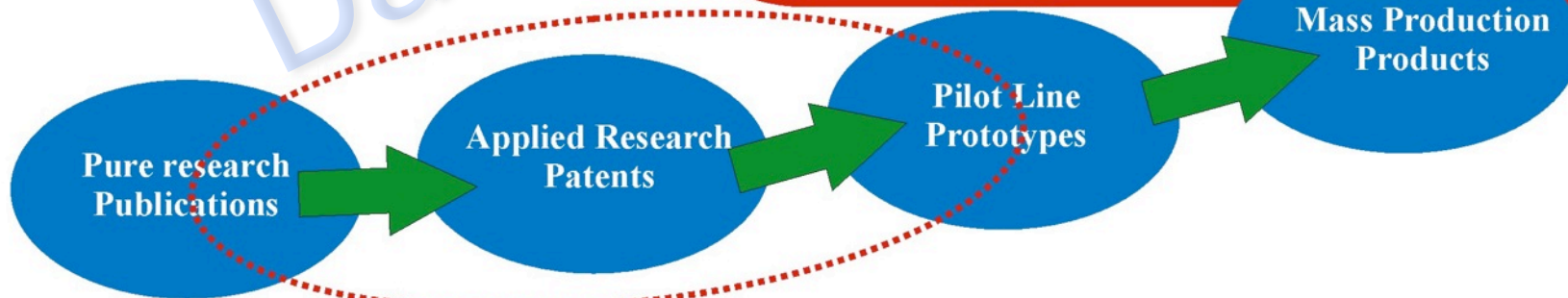


A single mission - Create innovation & transfer it to industry

**A clear focus - 1. Micro and nanotechnologies with critical mass in Si
2. Advanced devices for new applications**

Our activities include
Si technologies 22 nm node and beyond
MEMS
Photonics
Wireless and multimedia
Biology and healthcare

Nano-characterisation
Ion beam analysis - *Atom Probe, TOF SIMS, MEIS*
Scanning probe - *SSRM, SCM, KFM, 3DAFM*
TEM - *FEI Titan Pico, FEI Titan, Tecnai, JEOL 2010FEF*
FIB - *2 FEI dual beam, Zeiss dual beam*
Surface - *XPEEM, XPS, Nano-Auger*
Optical - *FTIR-IR, UV Raman*



Aberration correction in the TEM

- "Chromatic and spherical aberrations of rotationally symmetric lenses are unavoidable"

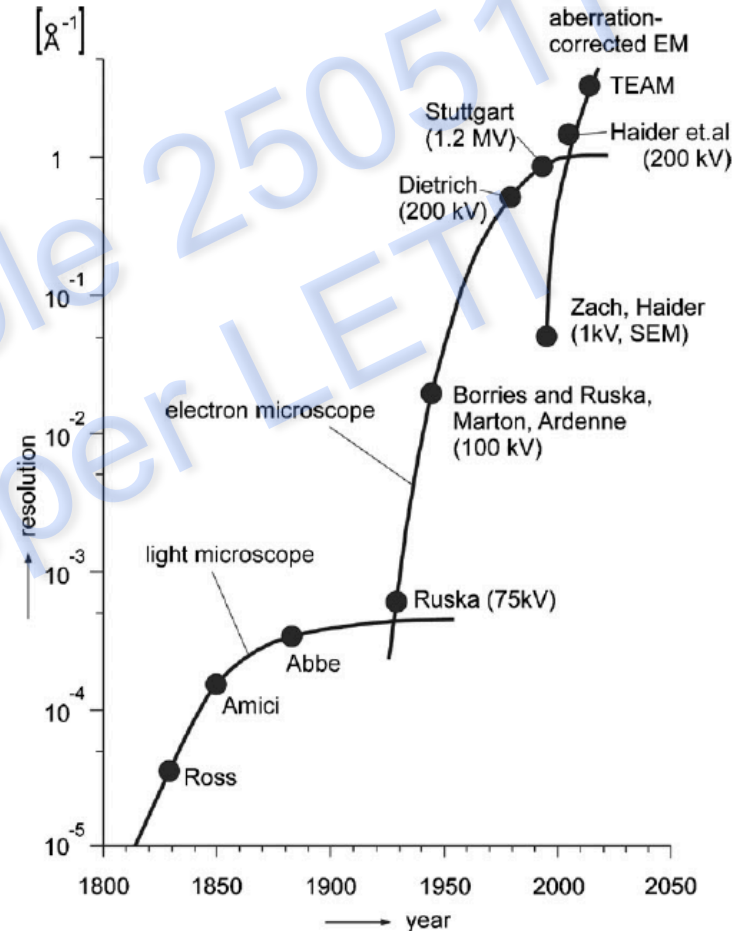
Otto Schertzer, 1936

- "It would be very easy to make an analysis of any complicated chemical substance; all one would have to do would be to look at it and see where the atoms are. The trouble is that the electron microscope is 100 times too poor. Is there any way to make the electron microscope more powerful?"

Richard Feynmann, 1959



Aberration corrector on the Hubble Space Telescope

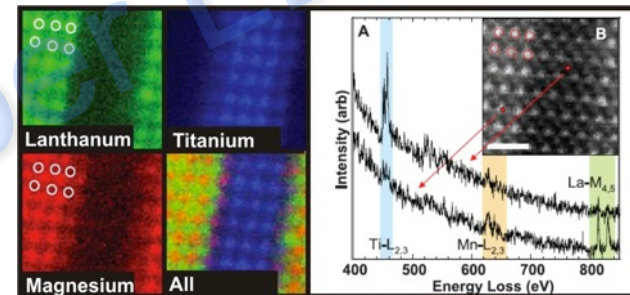
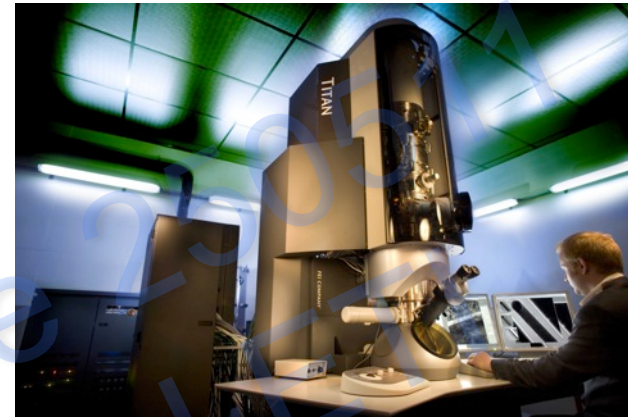


Rose, J. Electron Microscopy 58 p77 (2009)

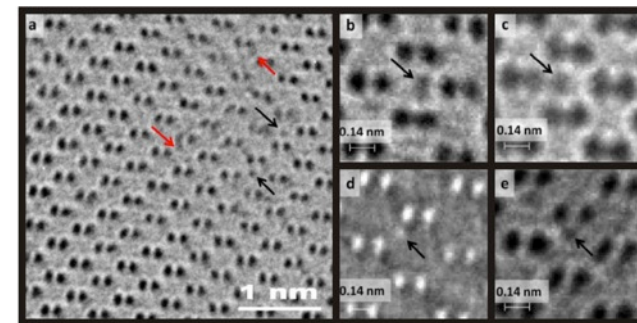
www.ncem.lbl.gov/TEAM-project

State-of-the-art transmission electron microscopes.

- Cost between 1 and 10 million Euros.
- 60 – 300 kV electron beam ($\lambda = 2.5 \text{ pm}$ at 200kV).
- Cs correction to improve spatial resolution to 50 pm.
- Monochromators provide an energy spread of 0.15 eV.
- The TEM is an extremely versatile tool, multiple techniques can be performed on the same sample in a single session.
- Specimen preparation is key for most applications.



Muller et al. Science, 219, 22nd Feb, (2008).



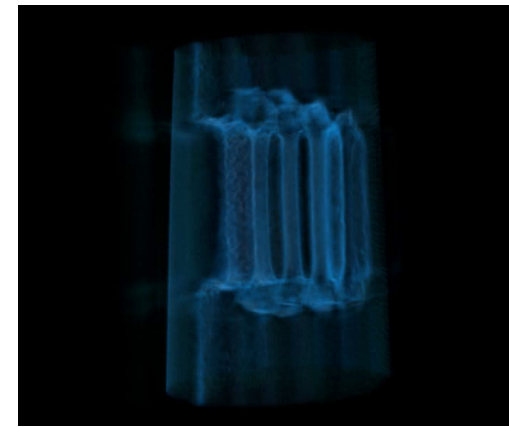
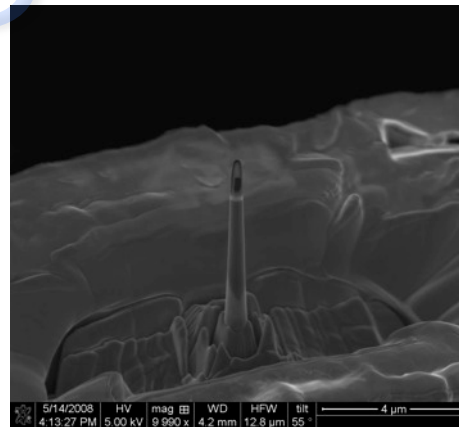
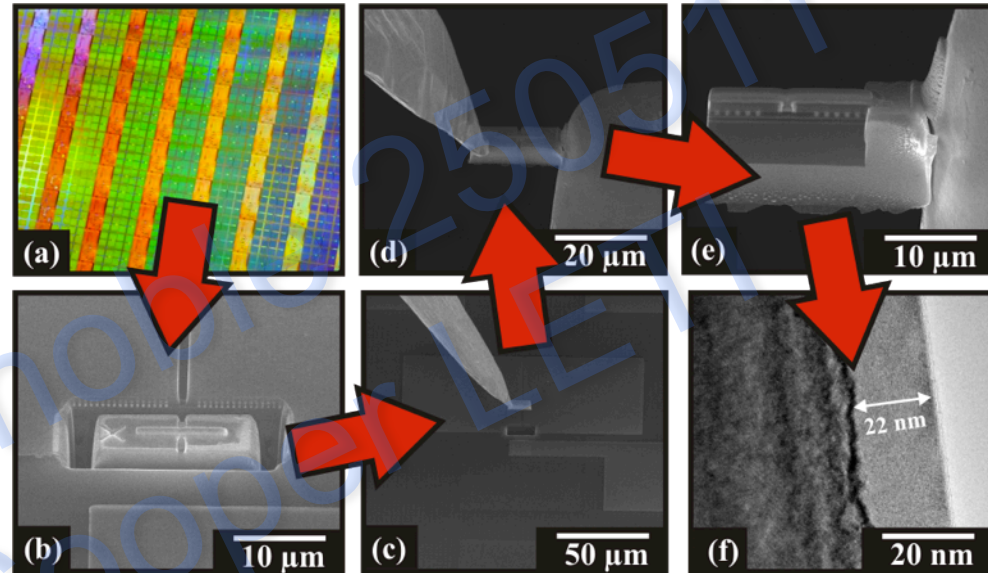
Alloyeau et al. PRB 80, 014114 (2009)

Specimen preparation by *in-situ* liftout

The limiting factor for all TEM-based techniques is the quality of the specimen!



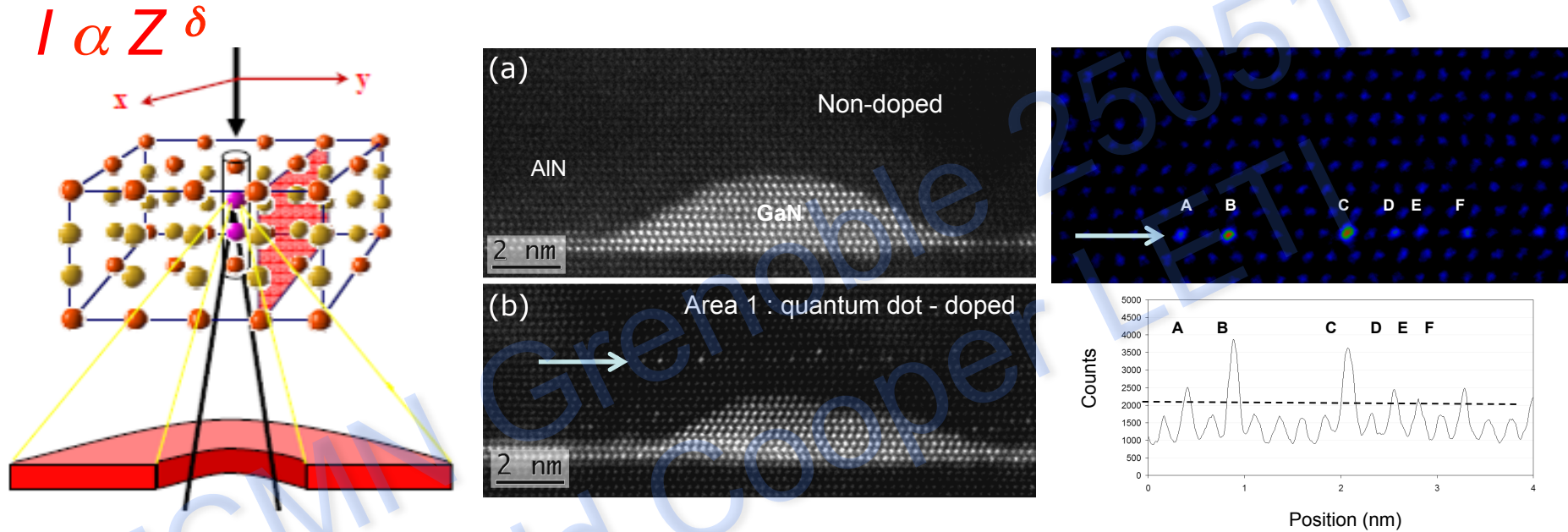
We need parallel-sided specimens prepared using low energy ions.



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HAADF STEM imaging of GaN Quantum dots.



Important to know the dopant position to control device properties
 Visualization of dopant by HAADF-STEM imaging

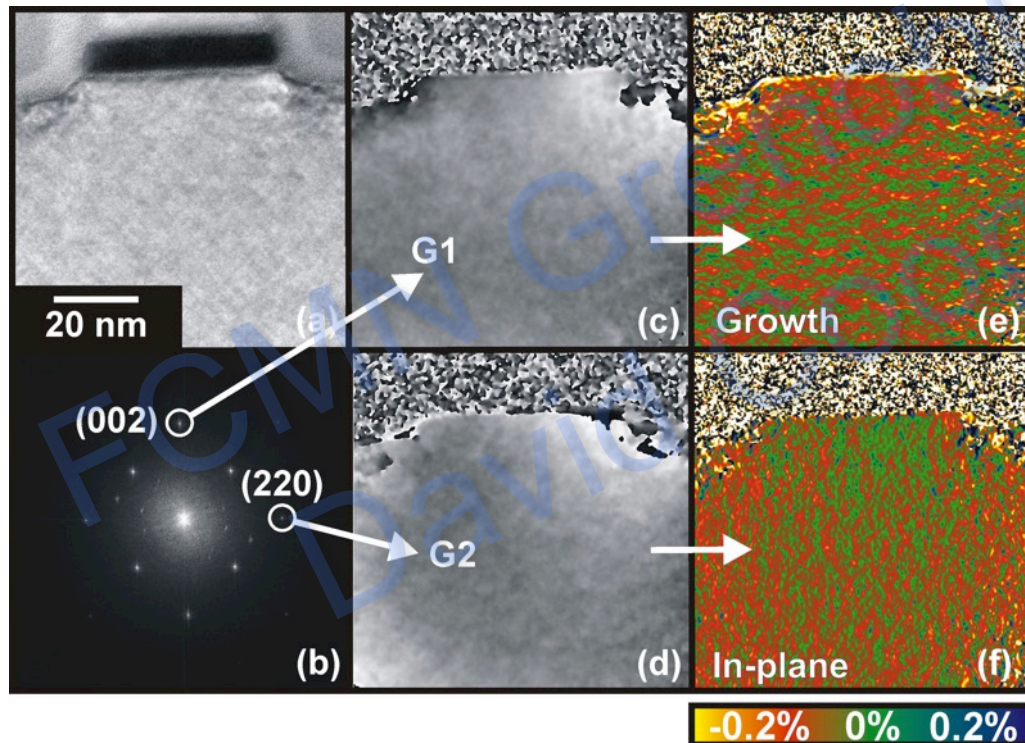
Application to Tm doping in GaN/AlN
 $Z(\text{Tm}) = 69$
 $Z(\text{Al}) = 13$
 $Z(\text{Ga}) = 31$

Rare earth atom doping
 Eu, Pr, Sm \rightarrow red
 GaN + Tm \rightarrow blue \rightarrow Produce full color device
 Er, Ho, Tb \rightarrow green
 A. J. Steckl and R. Birkhahn, Appl. Phys. Lett. **73**, 1700 (1998)

Okuno et al, Appl. Phys. Lett. **96**, 251908 (2010)

Strain Mapping from high resolution images.

Strain mapping performed from a Cs corrected high resolution image of a 28 nm HKMG nMOS device strained using CESL technology



$$\nabla_{\mathbf{r}}\phi_{GPA} = 2\pi\Delta\mathbf{g} = 2\pi(\mathbf{g}_{obj} - \mathbf{g}_{ref})$$

$$d_{ref} = 1/g_{ref}$$

$$\varepsilon = \frac{d_{obj} - d_{ref}}{d_{ref}}$$

- Small field of view (60 nm)
- High levels of noise
- Need reference in the field of view

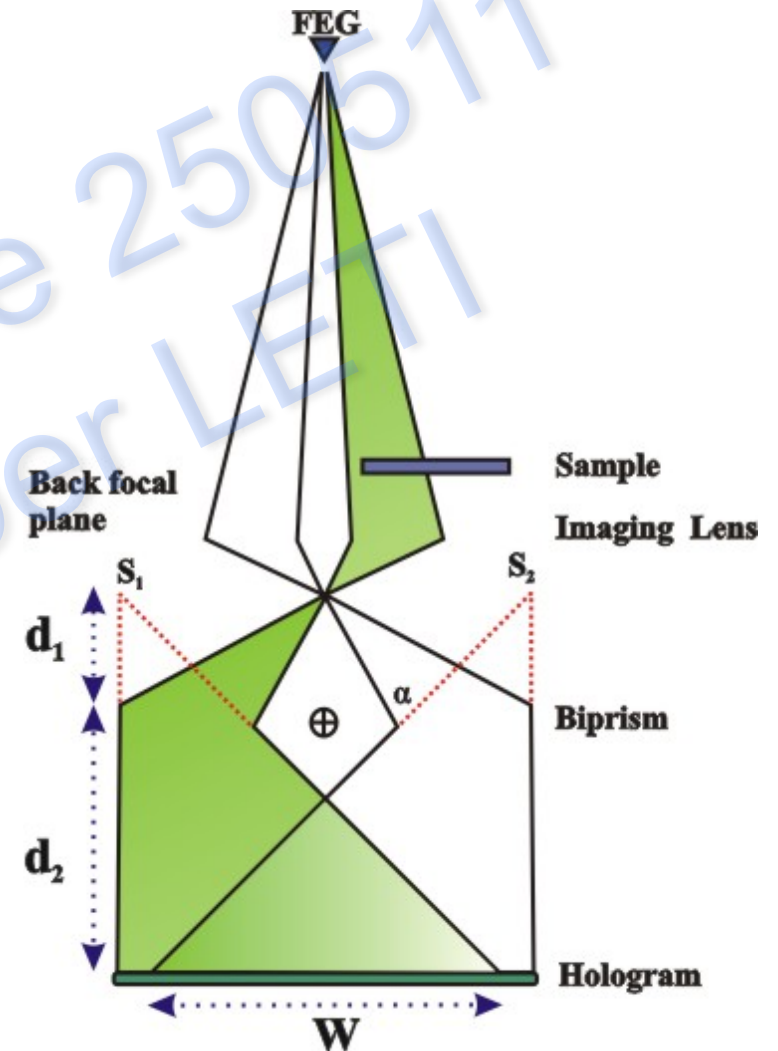
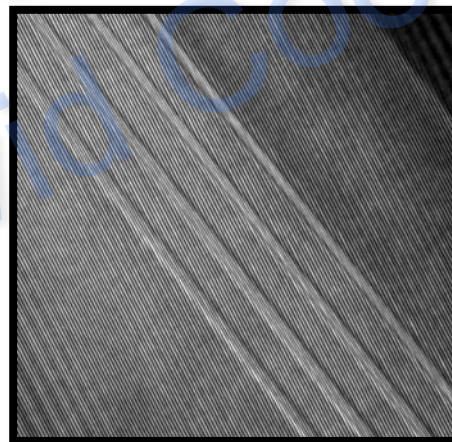
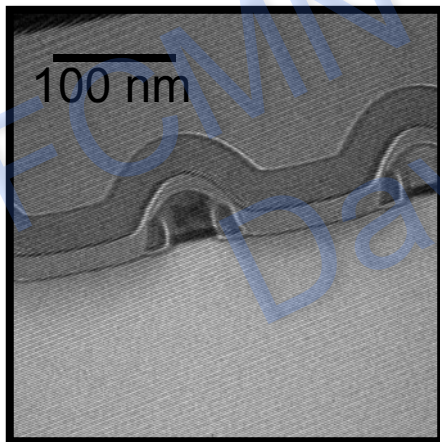
Hytch et al., Ultramicroscopy 74, 131 (1998)

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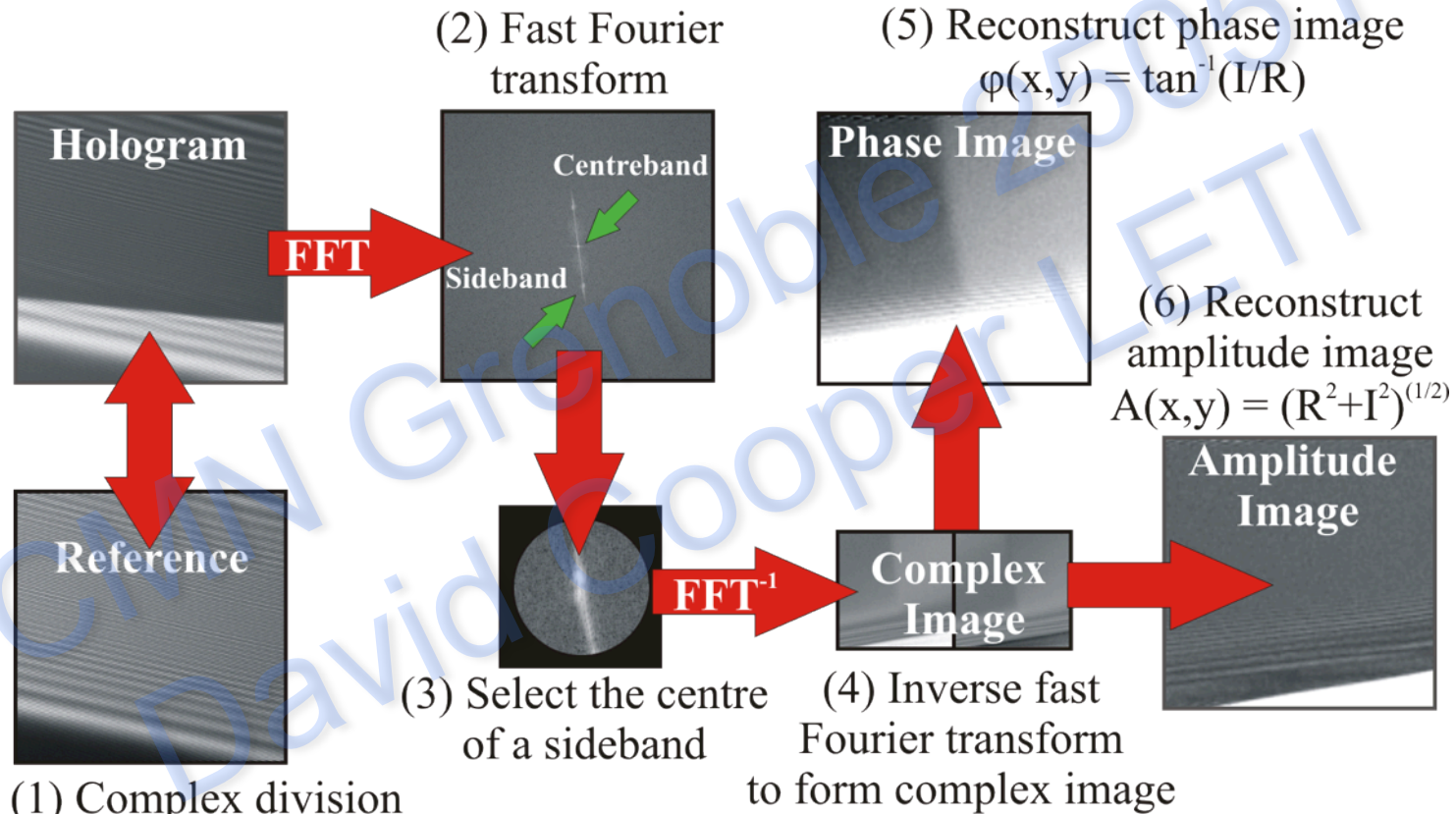
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Off-axis electron holography.

- Coherent electron source (FEG)
- Rotatable Möllenstedt-Düker biprism
- Lorentz lens provides a large field of view
- Digital image acquisition
- Off-line accurate phase reconstruction



Hologram reconstruction



$$\Delta\varphi(x, y) = C_E \int V(x, y, z) dz - \frac{e}{h} \iint B_{\perp}(x, y) dx, dy$$

Requirements for electron holography of semiconductor specimens

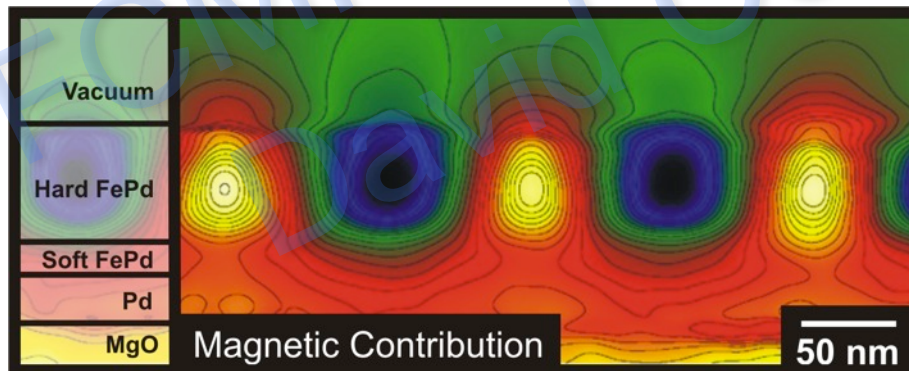
- Wide field of view - *flexibility*
- Good hologram contrast – *phase resolution – sensitivity*
- Small fringe spacing – *spatial resolution*
- Many electron counts – *phase resolution – sensitivity*
- Low intensity electron beam – *reduce artifacts from charging*

$$\delta\phi \propto \frac{1}{\mu\sqrt{N}}$$

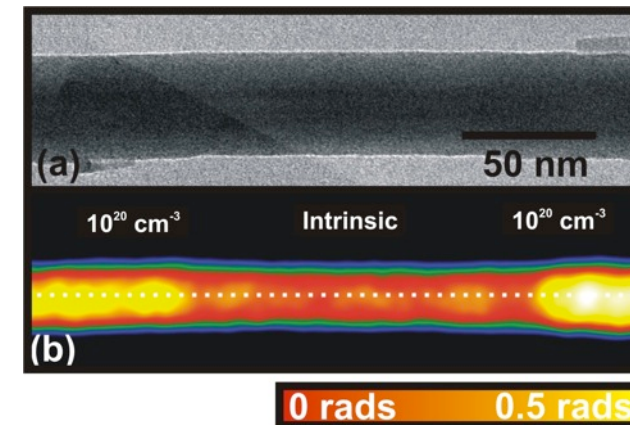
μ = fringe contrast
 N = electron counts

Electron holography on the FEI Titan

The mechanical and electrical stability of the Titan allows us to acquire electron holograms for long time periods with high contrast levels using a low electron beam intensity.

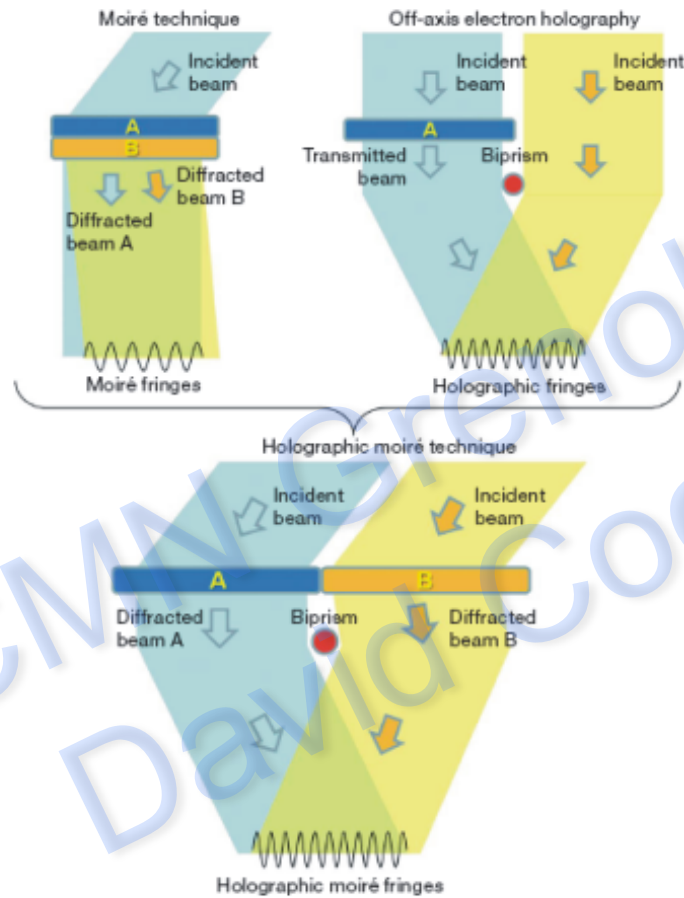


Massebouef et al, Nanoletters 9, 2803 (2009)

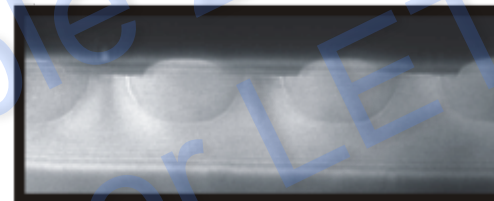


Den Hertog et al, Nanoletters 9, 3837 (2009)

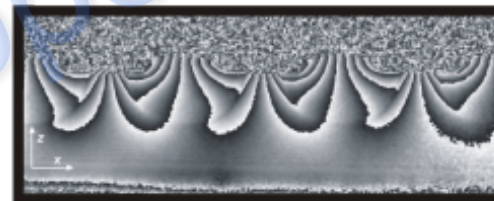
A new way to measure strain - dark field holography



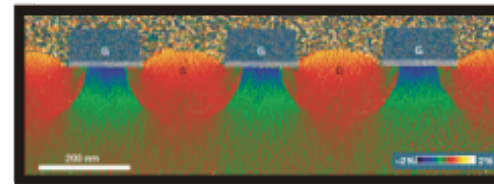
Bright Field Image



220 Dark Field Image



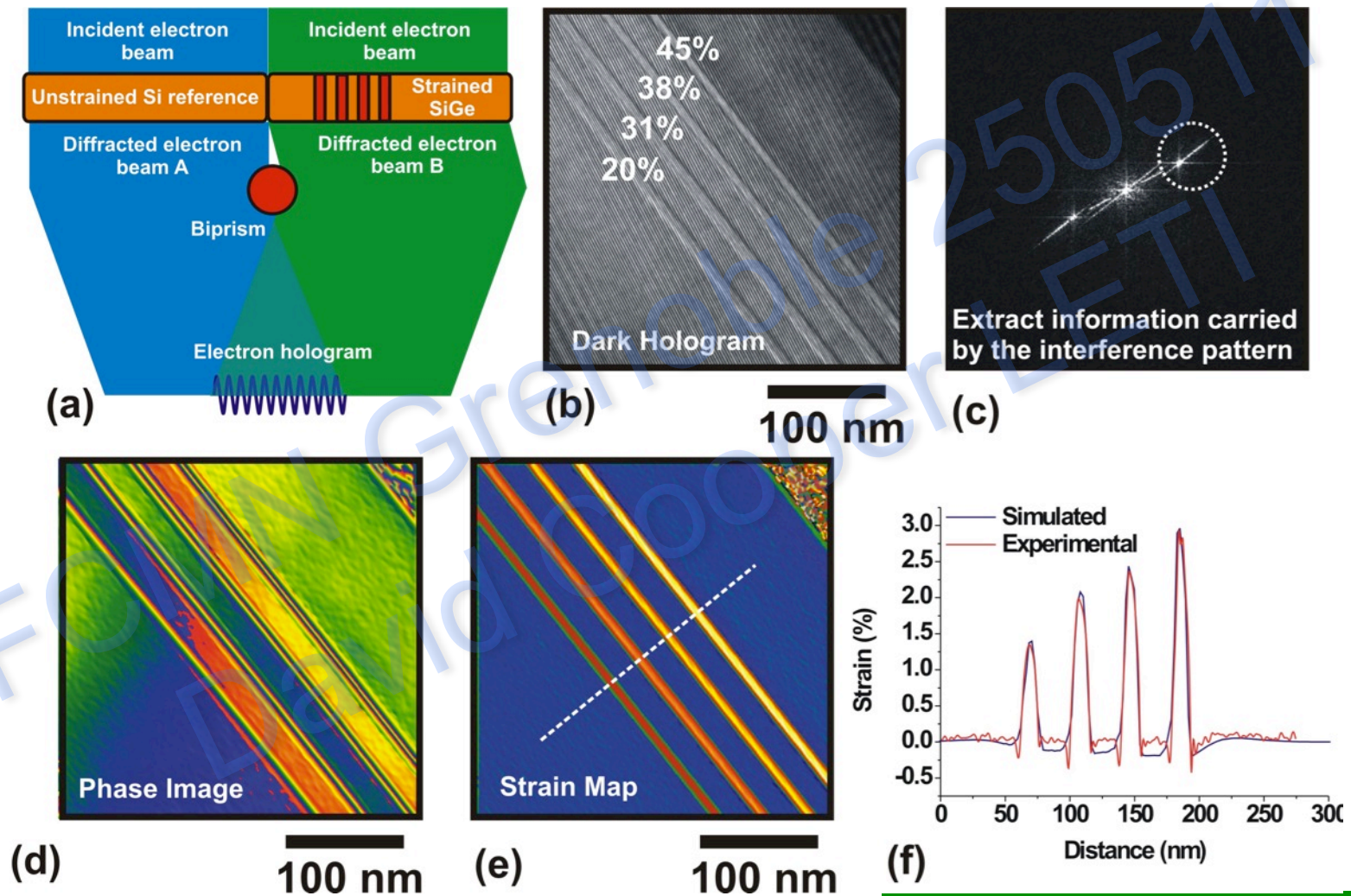
Phase Image



Strain map in E_{xx} direction

Hytch et al Nature 453 1086 (2008)

Dark field holography for quantitative strain measurements.

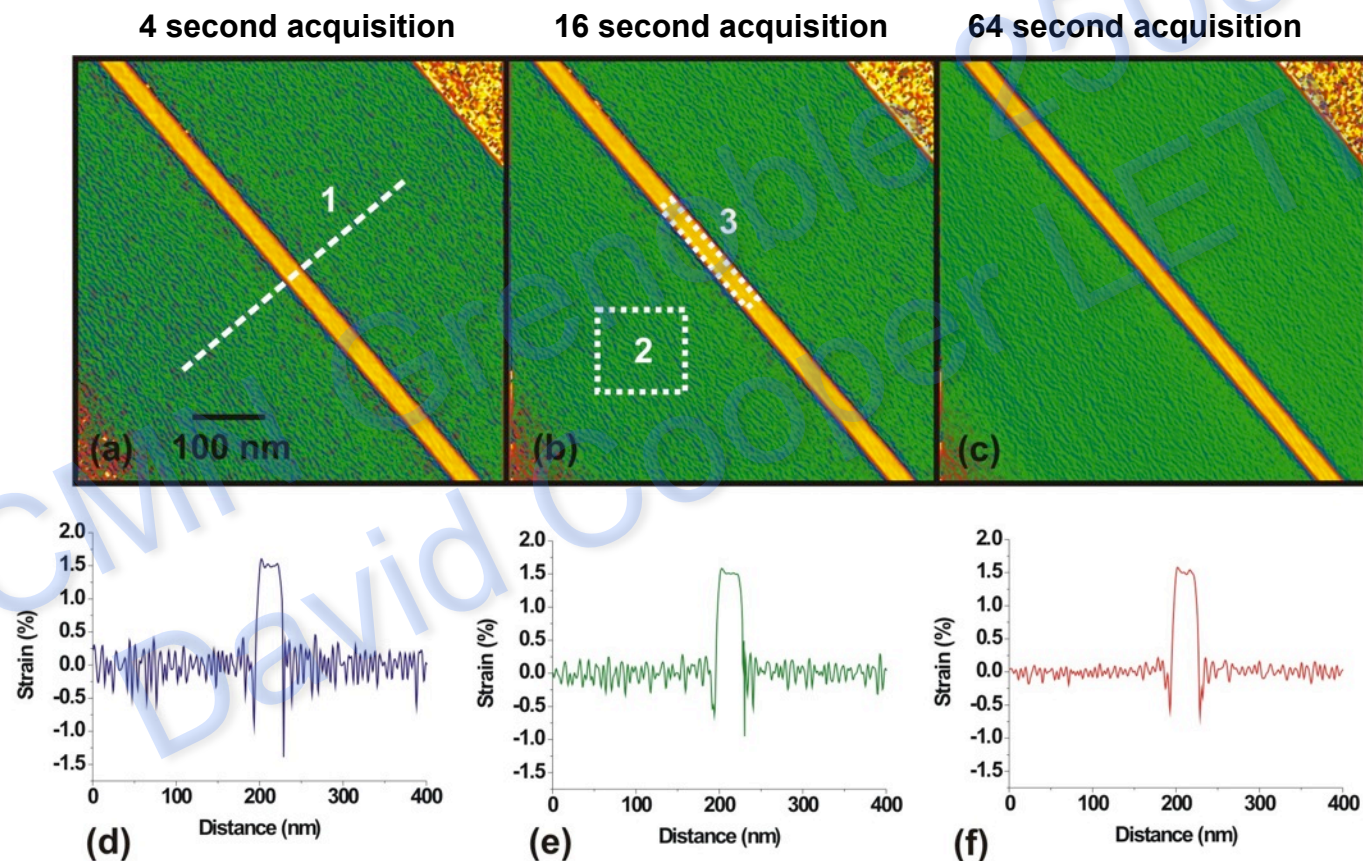


Cooper et al., Appl. Phys. Lett. 95, 053501, (2009)

Remember that the measured values of strain are relative to the reference!

Dark Holography using the Titan TEM

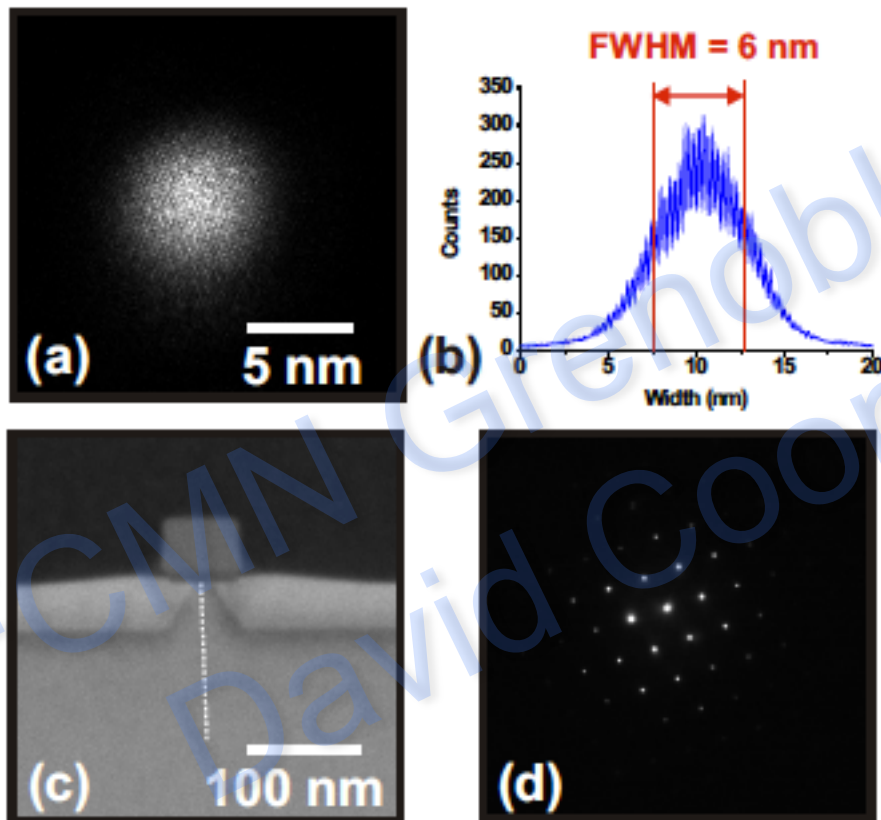
Test sample grown by epitaxy with a 30-nm-thick $\text{Si}_{0.77}\text{Ge}_{0.23}$ layer in Si.



Cooper et al., Appl. Phys. Lett. 95, 053501, (2009)

A sensitivity of 0.02 %
has been demonstrated

Nanobeam Electron Diffraction



- Using the 3 condenser lens system in the Titan, a near-parallel beam is formed with a convergence angle of 0.2 mrad and a diameter of 6 nm.
- The beam is scanned across the specimen in STEM mode and a diffraction pattern is acquired at each point.
- Values of strain can be determined by measuring the shift of the diffraction spots relative to a reference.

Nanobeam Electron Diffraction

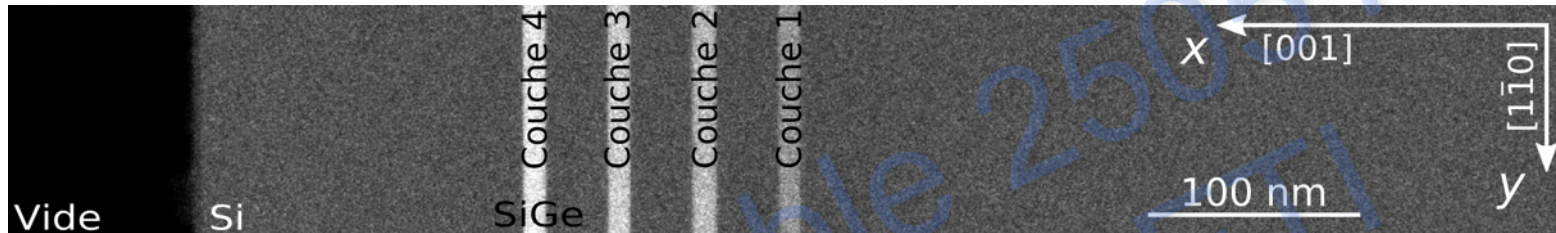
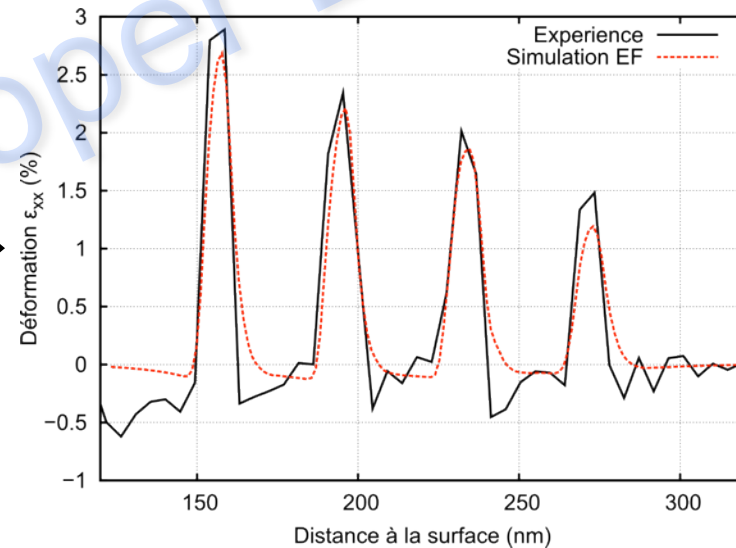
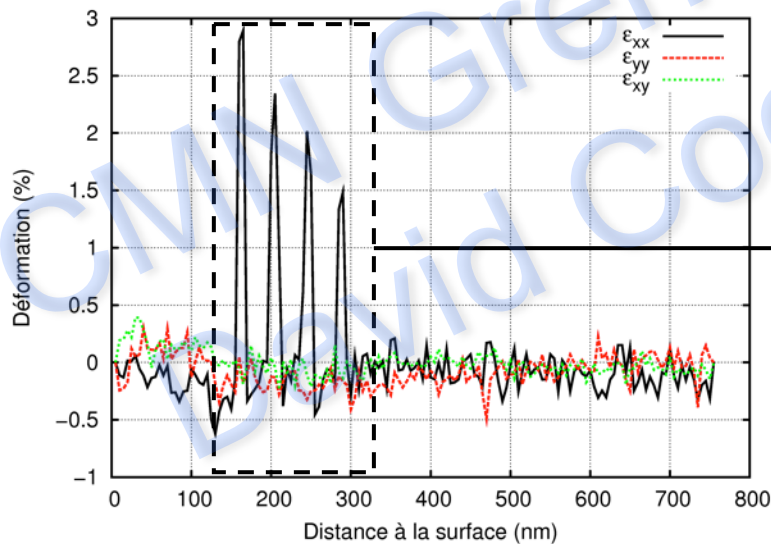


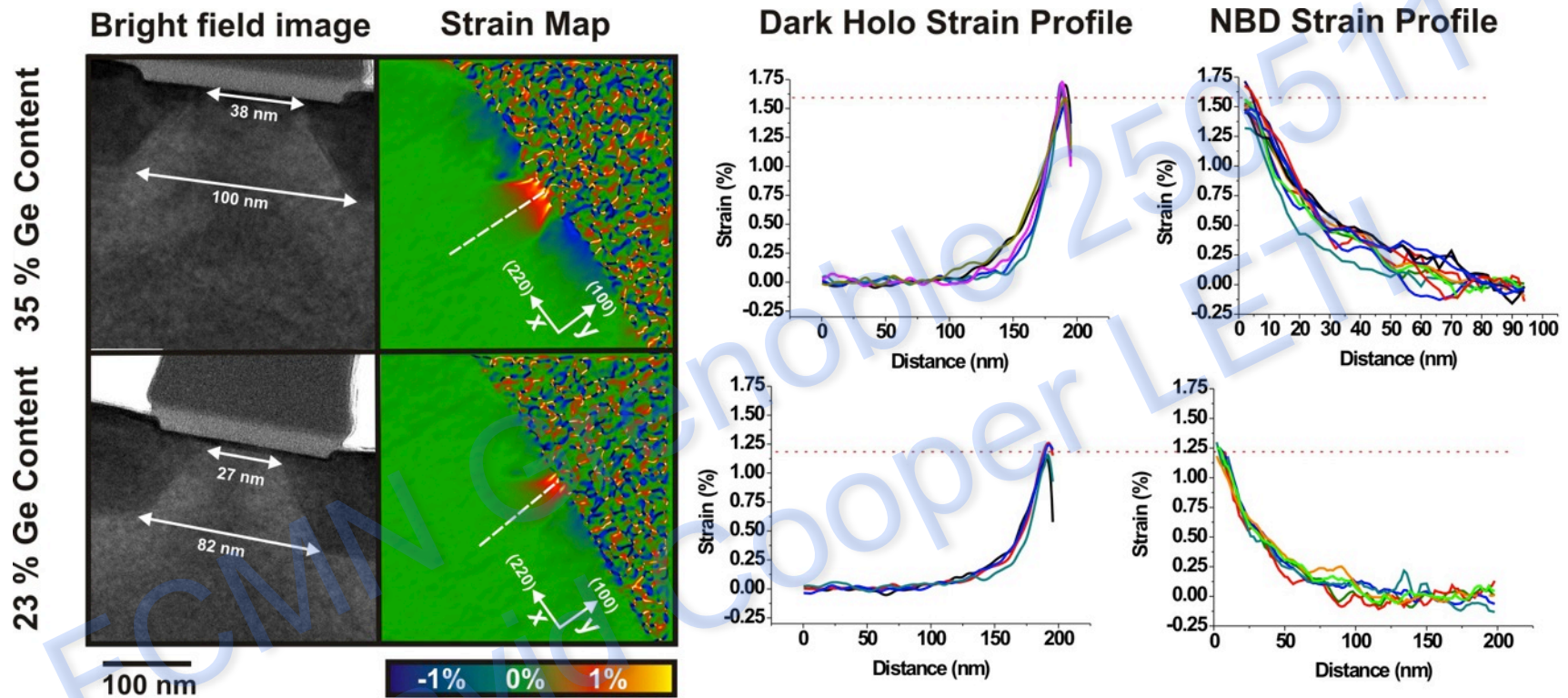
Image STEM/
HAADF



Béché et al., Appl. Phys. Lett. 95 123114, (2009)

A sensitivity of 0.06 %
has been demonstrated

Strain measurement in SiGe device structures.



The strain maps and profiles are all for the {220} direction and describe the compression in the conduction channel. The dark holography and NBD results seem to be consistent with each other.

Cooper et al., Semicond. Sci. Tech. 25 095012 (2010)

Each graph shows 6 different profiles in order to show the reproducibility of the techniques.

Silicidation of SiGe for electrical contacts

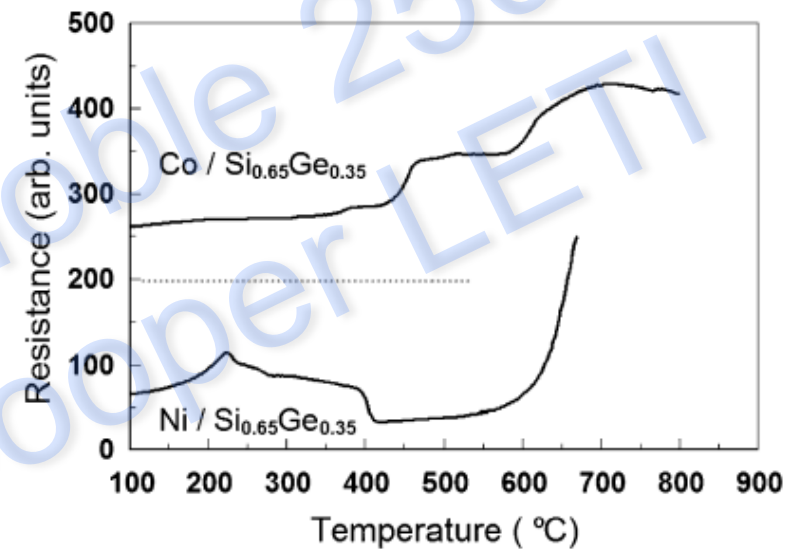
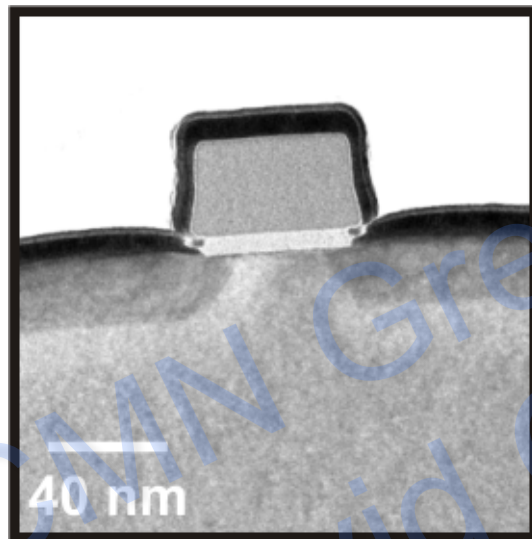
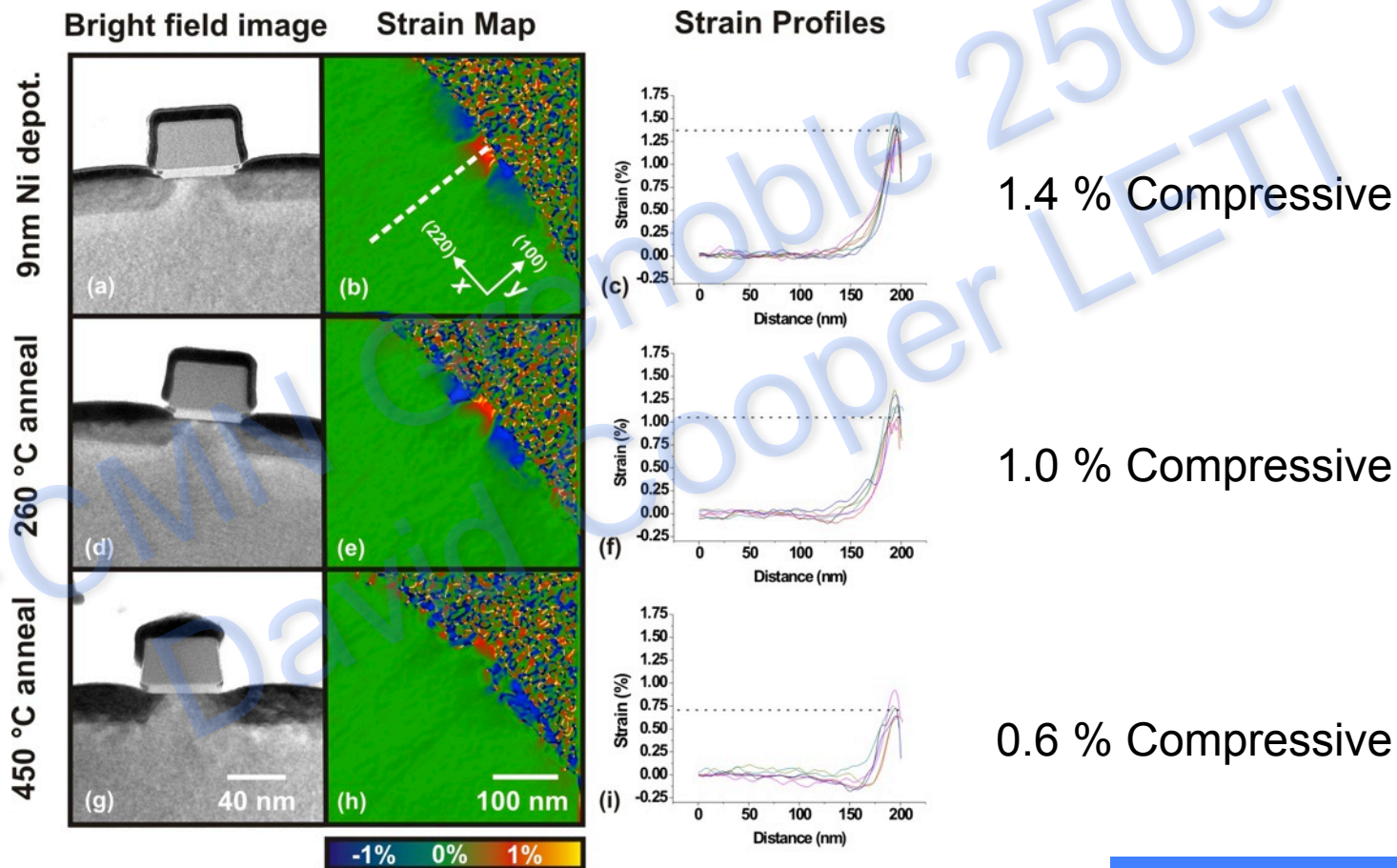


Fig. 3. Evolution of film resistance for Ni and Co deposited on Si (35 at.% Ge) on insulator substrate. While the formation of low resistivity CoSi₂ is pushed to higher temperature in the presence of Ge, the region of low resistivity for NiSi is similar to that of Fig. 1.

Lavoie et al. Micro. Eng. 70, 144-157 (2003).

Silicidation of SiGe for electrical contacts

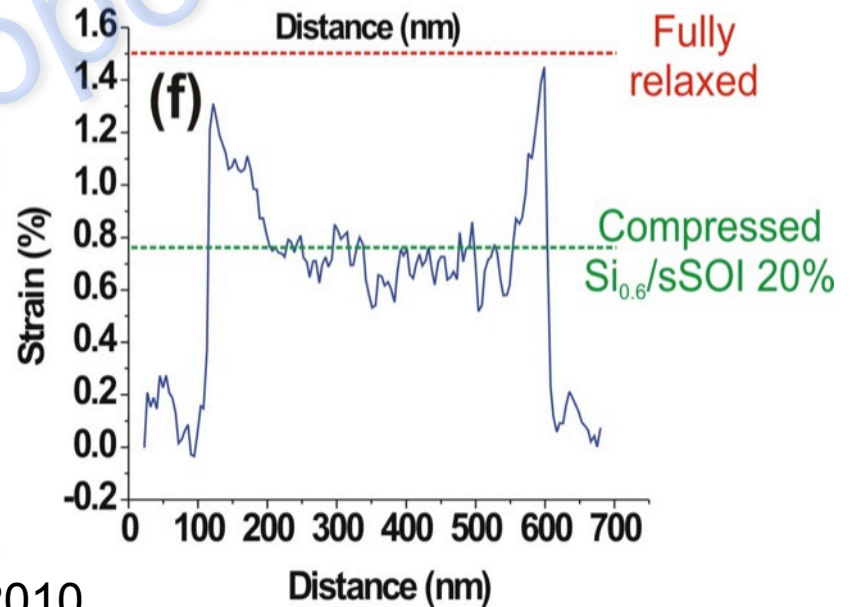
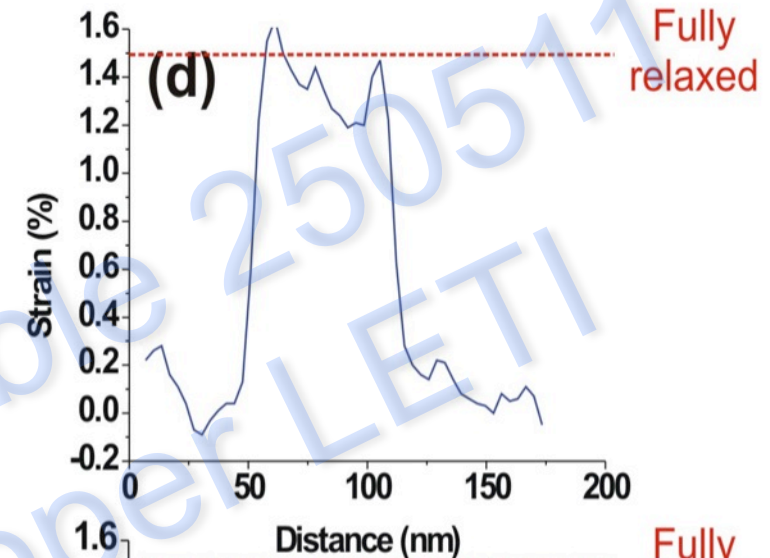
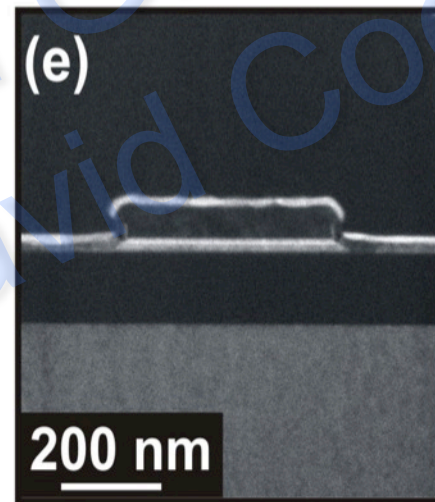
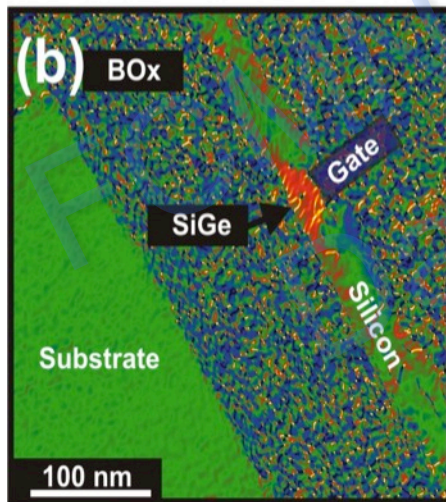
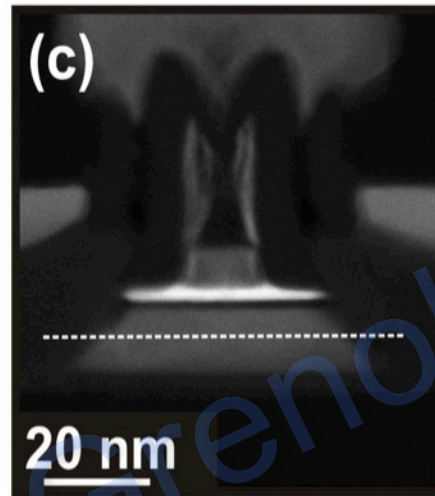
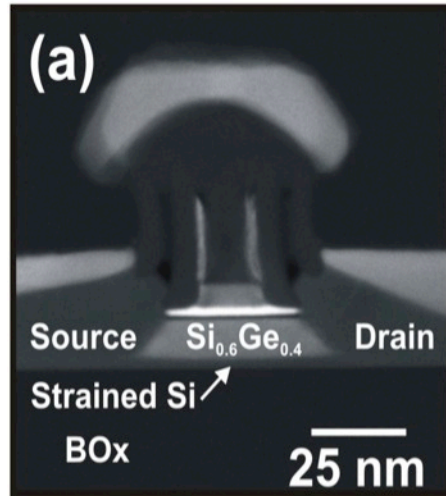
Recessed source and drain: 35 % Ge content and 38 nm gate length.



Cooper et al., Appl. Phys. Lett. 96 113506 (2010)

Maps show the strain in the (220) in-plane direction.

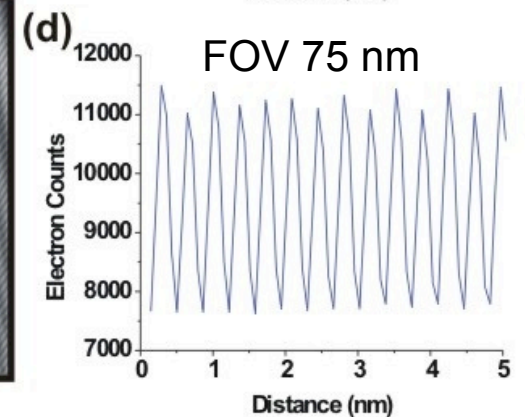
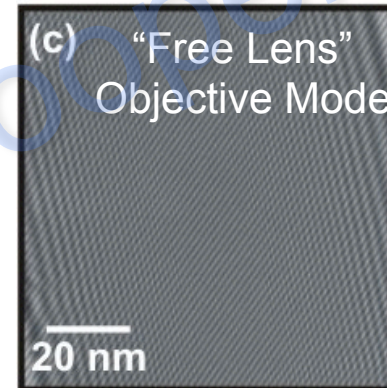
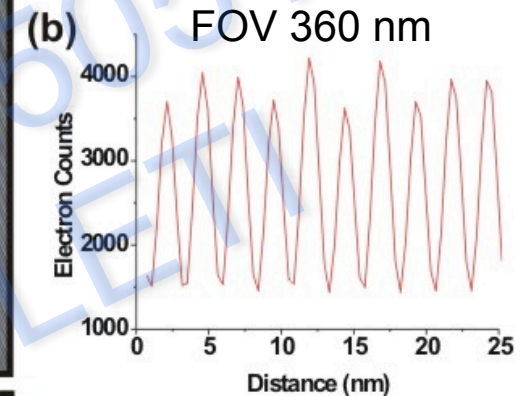
Strain Measurement of a functional sSOI pMOS device.



Hutin et al. IEDM 2010

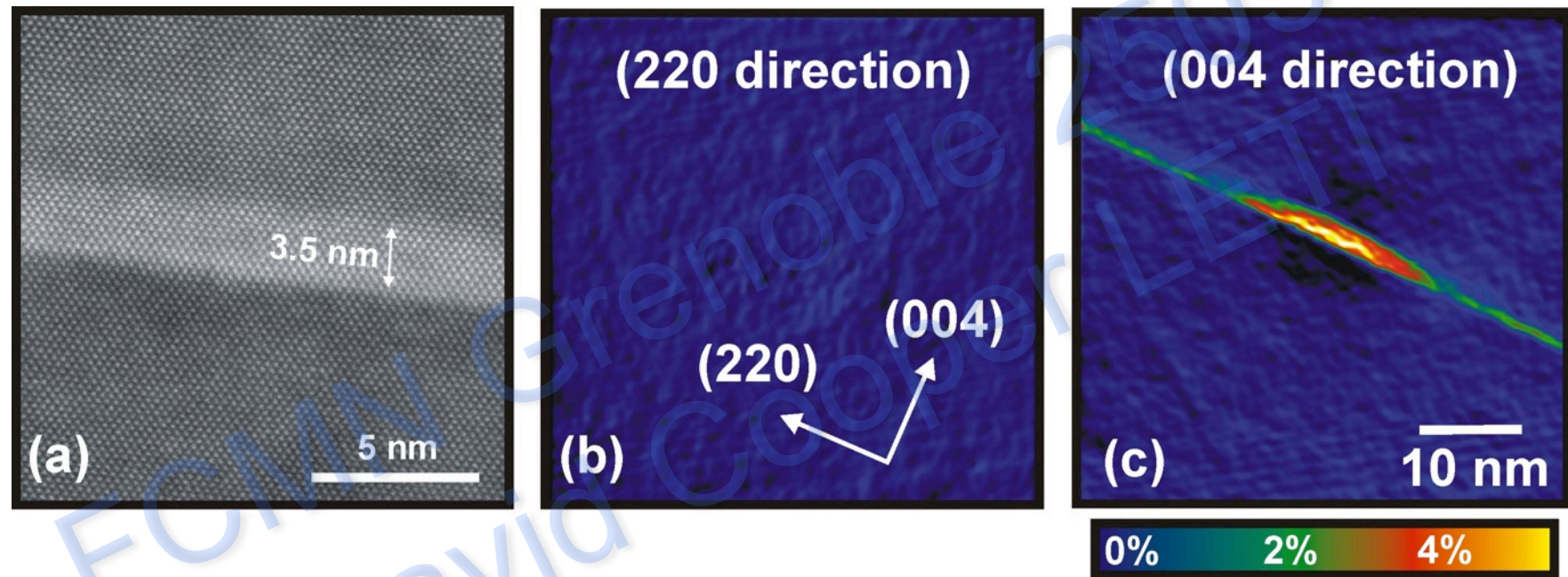
Field mapping with 1 nm spatial resolution

- Electron holography in the Titan using the Lorentz lens has a spatial resolution of around 6 nm for an acceptable value of the signal to noise ratio.
- The spatial resolution using a Lorentz lens is around 2 nm.
- The Titan can be operated in "Free lens control" in order to improve the spatial resolution.
- Now 0.3 nm fringe spacing can be obtained in the Titan to provide phase images with a spatial resolution of 1 nm and a field of view of 75 nm.



The field of view will always scale with the spatial resolution due to the sampling requirements on the CCD camera.

Dark Holography with 1 nm spatial resolution.



(a) STEM image showing height of InAs QD in a InP lattice. (b) Strain map in the (220) in-plane direction. (c) Strain map for the (004) growth direction.

Strain measurement by dark holography and nanobeam electron diffraction.

Dark Holography

Spatial resolution of 1 to 10 nm ☺

Sensitivity of +/- 0.02 % ☺

Micron-scale field of view ☺

Reference needed in hologram field of view ☹

Small specimen tilts are required ☹

Simple data analysis ☺

Tricky to perform ☹

Instant 2D imaging (no scanning required) ☺

Reference must be aligned to region of interest ☹

Biprism required ☹

NBED

Spatial resolution of 3 to 6 nm ☹

Sensitivity of +/- 0.06 % ☺

Flexible field of view ☺

Reference can be taken from anywhere ☺

Performed down the zone axis ☺

Automated data analysis ☺

Extremely easy to set up and perform ☺

Difficult and time consuming to produce 2D maps ☹

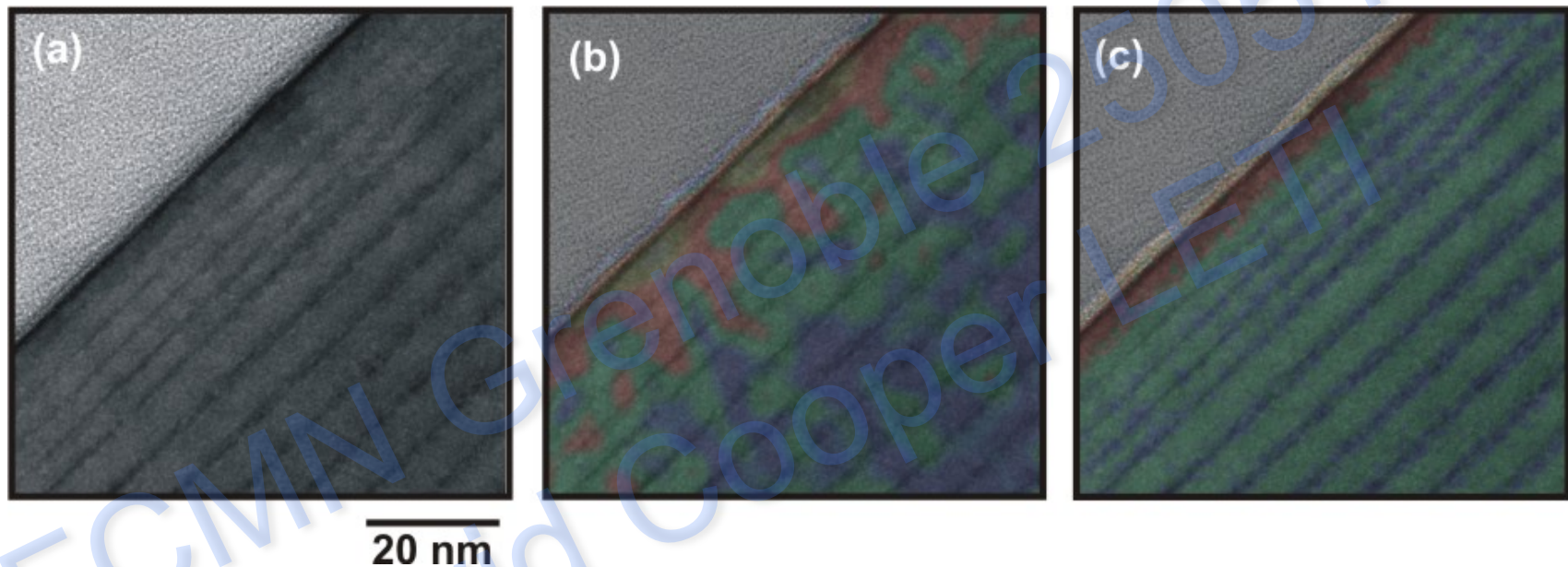
Very large data sets ☹

Three condenser lenses required ☹

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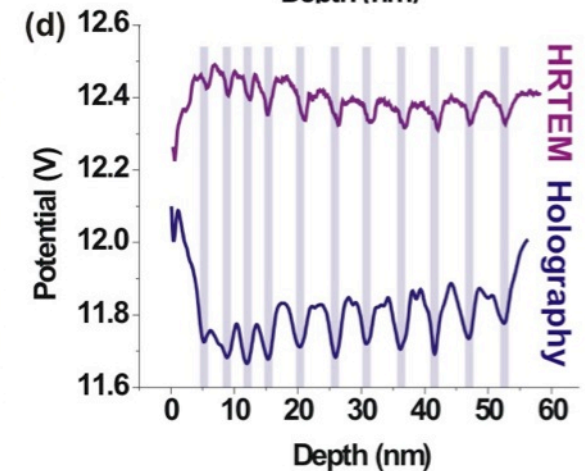
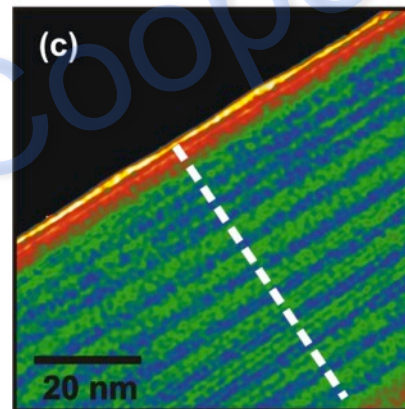
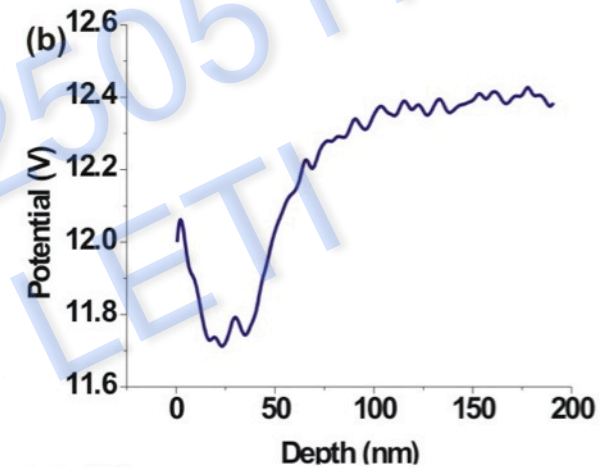
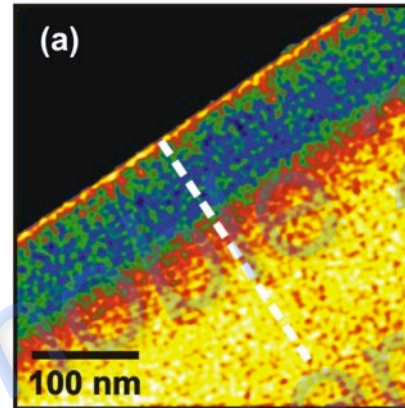
Dopant profiling with 1 nm spatial resolution



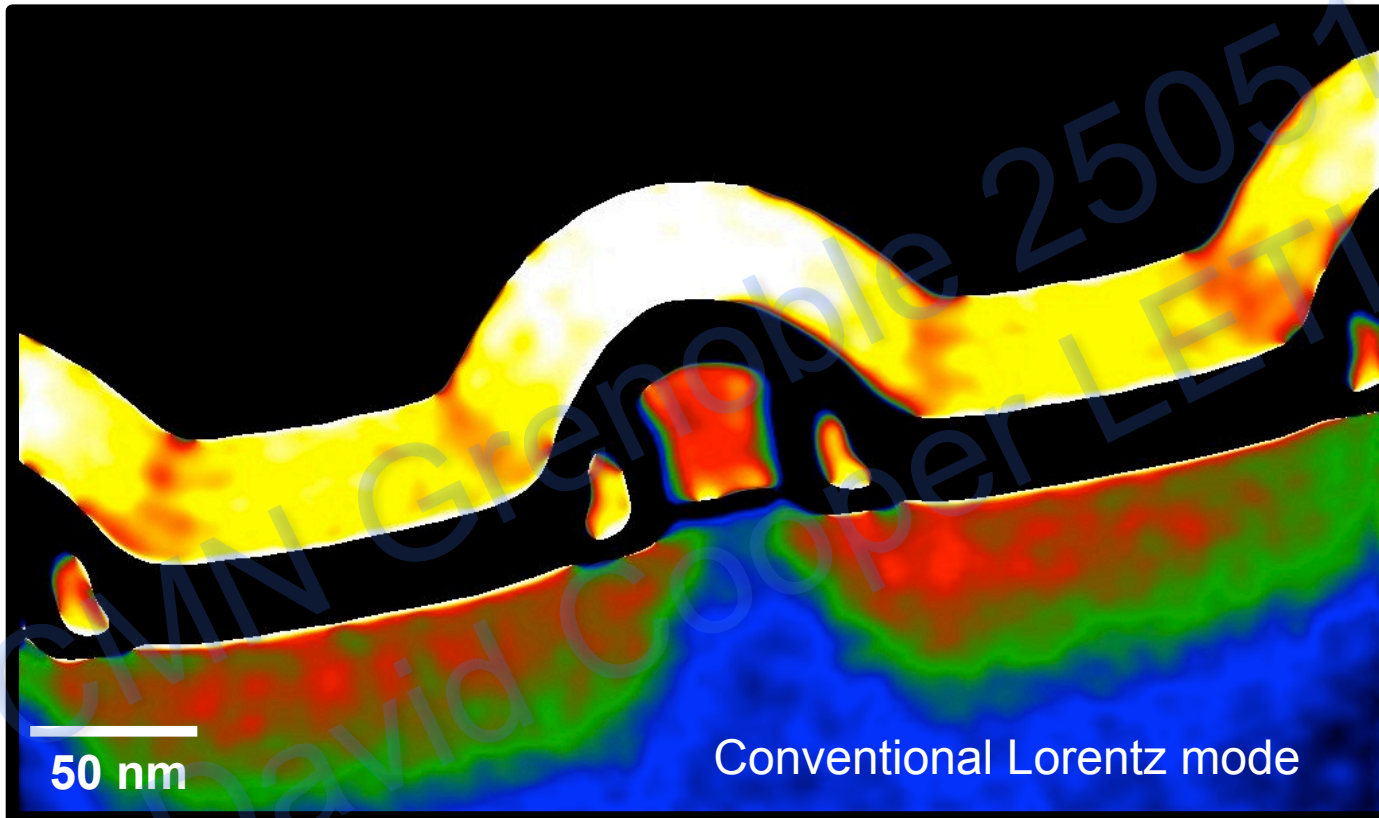
- Demonstration of 1 nm spatial resolution.
- Fig (a) shows TEM image of highly-doped delta layers in boron, separated by 3.5 nm and 7 nm. The dark contrast in the doped layers has been increased by using a small objective aperture.
- Fig (b) shows active dopant map acquired using Lorentz holography overlaid onto the TEM image, the deltas have not been resolved.
- Fig (c) shows active dopant map acquired using free lens control objective mode overlaid onto the TEM image, the delta layers are clearly resolved.

Dopant profiling with 1 nm spatial resolution

- (a) Shows the potential map of boron delta layers acquired using Lorentz mode holography, here the spatial resolution is 6 nm and the field of view is 270 nm. The layers are clearly not resolved (b) Shows a profile acquired across the delta layers.
- (c) Shows the potential map of the boron delta layers acquired in free lens objective mode. (d) Shows a profile acquired across the delta layers compared to a profile acquired from the HRTEM image. Here the position of the deltas has been mapped to an accuracy of 0.35 nm.
- The profile in the Lorentz potential map has been averaged over 5 nm and the profile in the free lens potential map over only 1 nm.



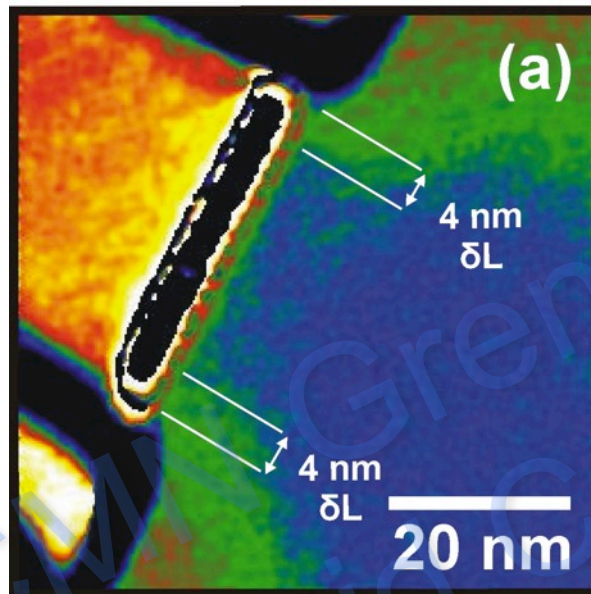
Electron holography of real nMOS devices.



- 40 nm gate test structure supplied by ST Microelectronics
- Prepared using back side milling in the FIB at low operating voltage.
- Examined using Lorentz mode electron holography
- Use of substrate as reference, signal to noise improved by stability of Titan TEM.
- Shows positions of active dopants with a spatial resolution of 6 nm.

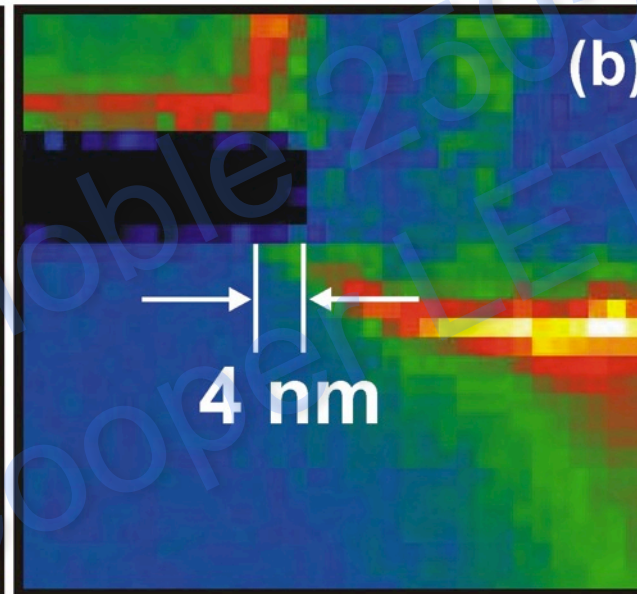
Compare electron holography to EELS

Holography - Potential



1 nm spatial resolution

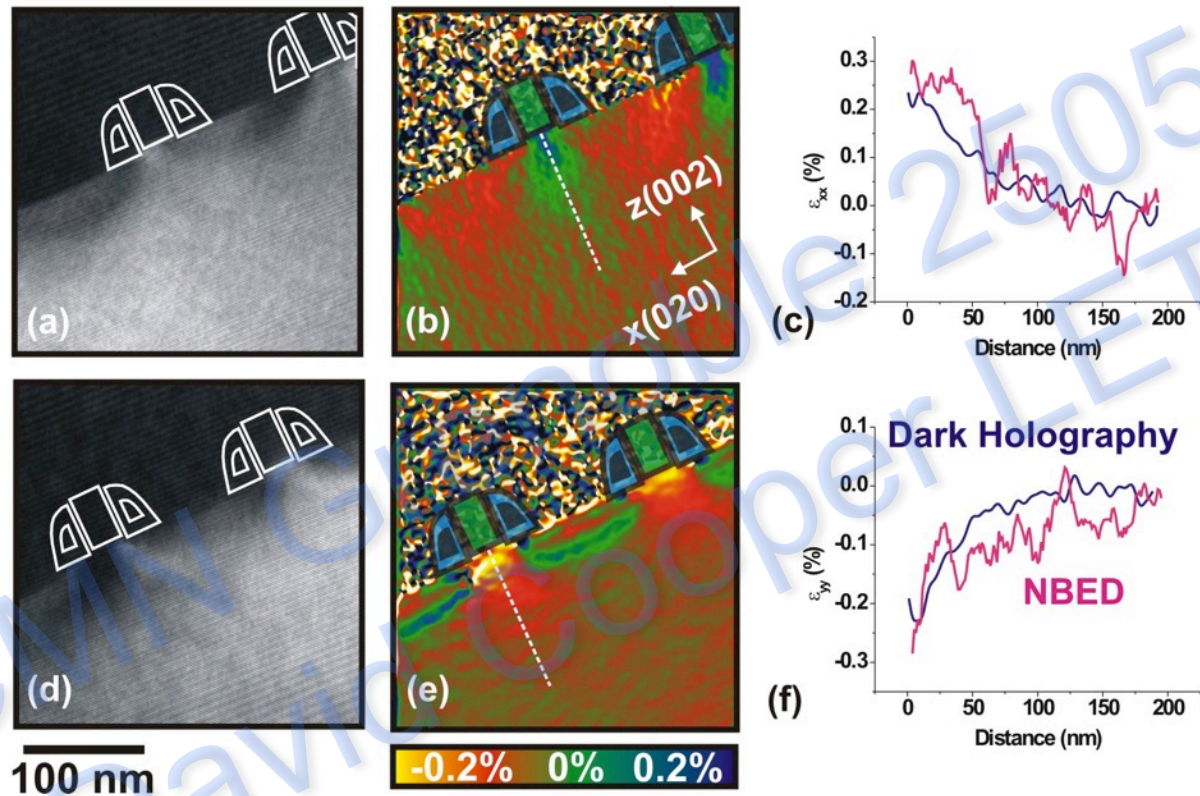
EELS - Atoms



2 nm spatial resolution

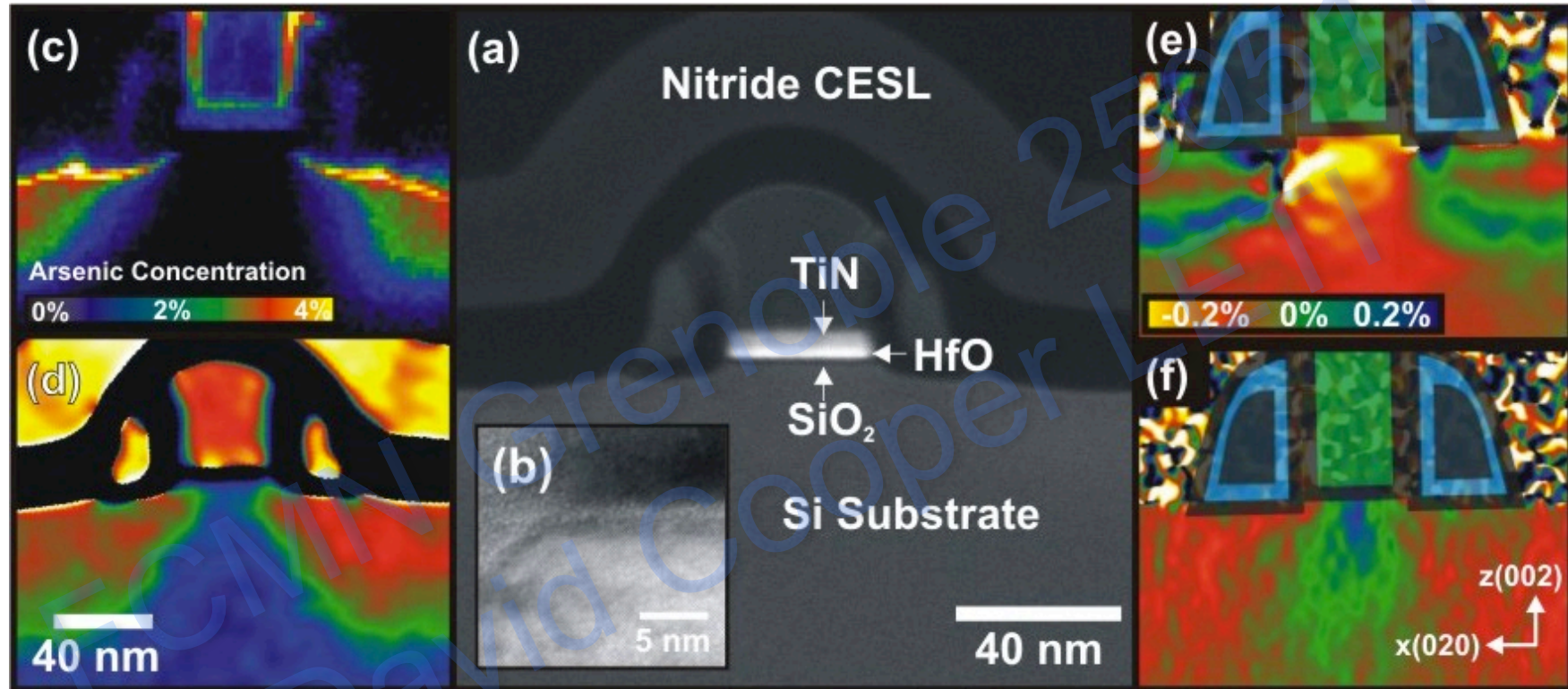
- By using free lens objective mode holography we can see the position of the electrical junction with 1 nm spatial resolution.
- This can be compared to EELS results which show the position of the dopant atoms (EELS performed by Germain Servanton at ST)
- Difficult to see boron and phosphorus using EELS
- Phosphorous detection possible by EDX
- Boron has now been detected by EELS (Using PCA data analysis).

Strain mapping for CESL devices.



- (a) Shows a dark field hologram and (b) a strain map for the (220) in-plane direction. (c) Shows profiles extracted from under the gate showing the strain measured by dark holography and NBED.
- (d) and (e) shows a dark field electron hologram and strain map for the (004) growth direction and (f) profiles showing the values of strain under the gate for the growth direction.
- The stability of the Titan allows excellent sensitivity to detect these low values of strain. These values are just about detectable by NBED when everything is perfectly optimised.

Nanofield mapping in the TEM



- Now from the same semiconductor sample we can get information about the structure by (a) STEM and (b) image corrected TEM.
- We can measure (c) the positions of the dopant atoms by EELS and the electrical potential by electron holography
- The strain can also be measured using dark field electron holography and NBED.

Conclusions

- Working in a multidisciplinary team that mixes pure researchers, academics and applied engineers is a very effective way of developing advanced characterisation techniques.
- Aberration correction makes high resolution imaging fast and easy. It will also be absolutely essential for low energy microscopy.
- We have a very stable TEM platform that can be used for electron holography.
- We have an excellent tool kit for measuring strain for the next generations of semiconductor devices (Holography, NBED, GPA of TEM and HAADF Images).
- **Sample preparation is absolutely key**
- TEM is a very versatile tool that can be used to acquire different types of quantitative information directly from the same TEM sample.

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*... thank you for
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