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Van den Berg, Jaap, Bailey, Paul, Barlow, Roger, Noakes, T.C.Q., Kilcoyne, Susan H. and Cywinski, Robert

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# The UK MEIS facility - a new future at the IIAA, Huddersfield

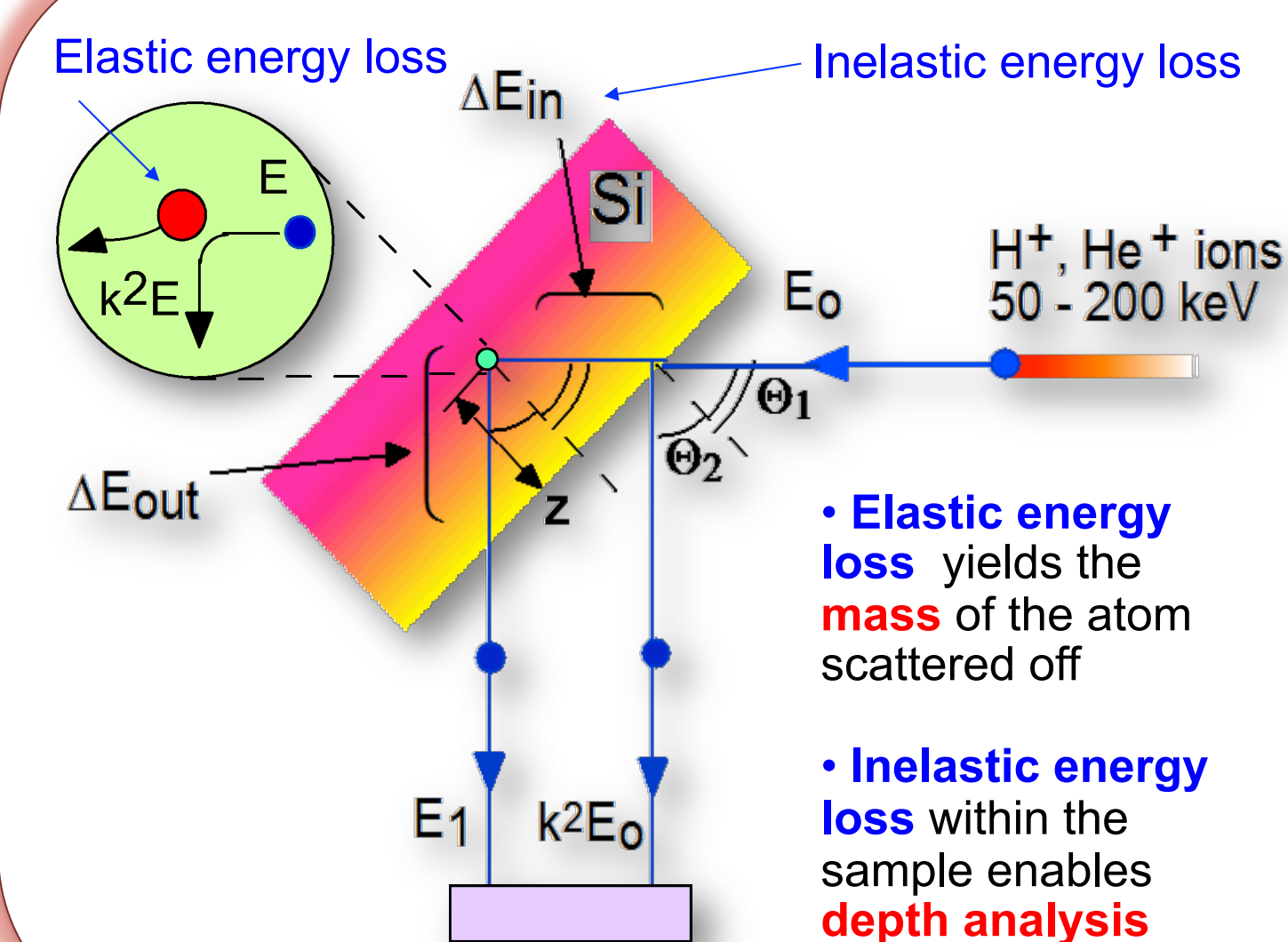J. A. van den Berg[a], P. Bailey[a], R. Barlow[a], T. C. Q. Noakes[b], S. H. Kilcoyne[a] and R. Cywinski[a].

[a] International Institute for Accelerator Applications, School of Applied Sciences, University of Huddersfield, Huddersfield, HD1 3DH, UK.
[b] STFC Daresbury Laboratory, Warrington, WA4 4AD, UK.

*University of* **HUDDERSFIELD**

## Introduction

The MEIS facility, formerly at STFC's Daresbury Laboratory and used by a wide range of UK and foreign research groups, has moved to the **IIAA** at the University of Huddersfield. It has been fitted with a new 200 keV ion accelerator. MEIS is a powerful tool for the structural and compositional characterisation of nanolayers, including depth profiling. Further to basics of the MEIS technique, examples of its analytical capability are presented from the area of Ion beam crystallography but mainly from Depth profiling analysis.
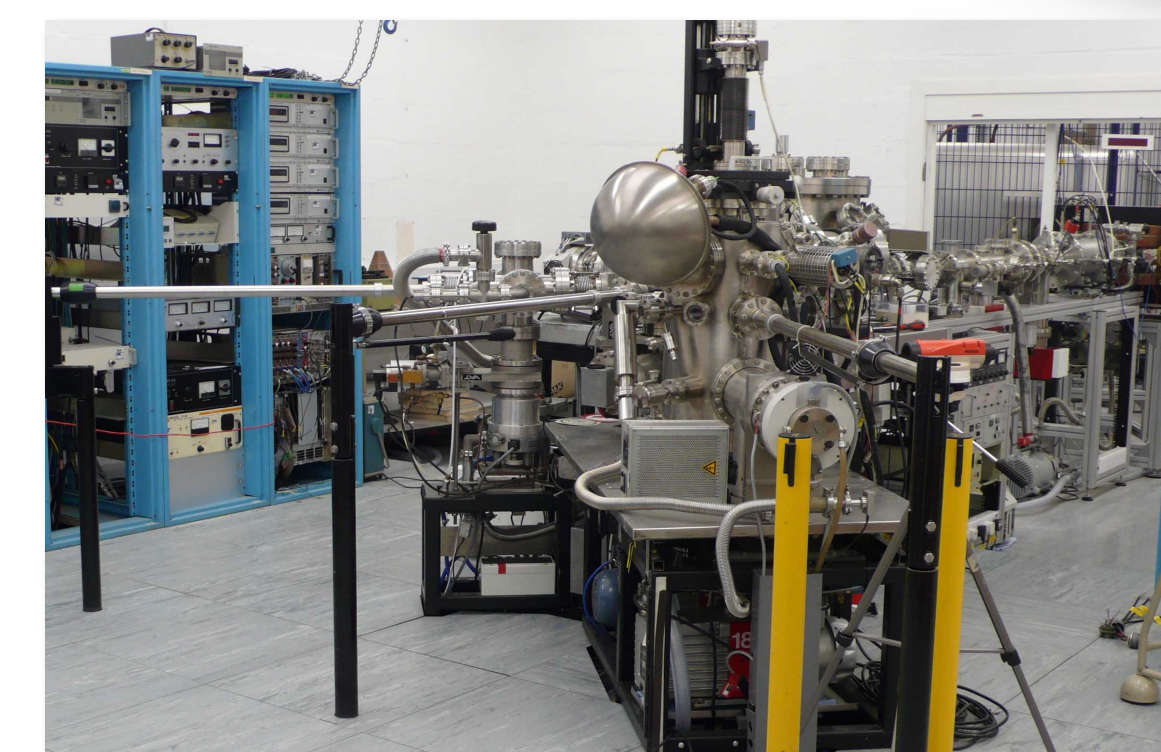
### MEIS principle:

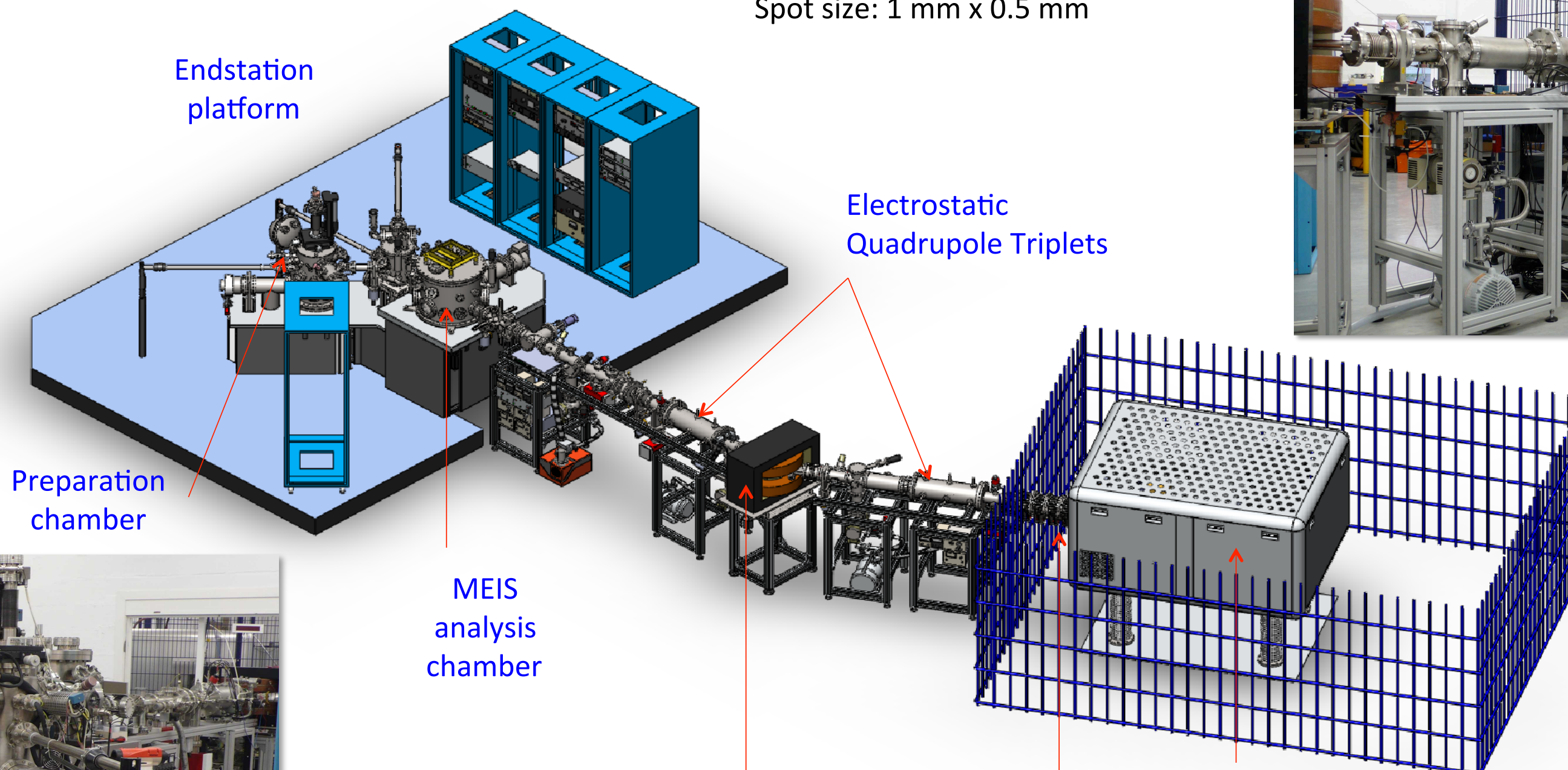Elastic energy loss — Inelastic energy loss

$\Delta E_{in}$   Si

$k^2E$   E

$\Delta E_{out}$   z   $\Theta_1$   $\Theta_2$

$E_1$   $k^2E_0$

$E_0$

H+, He+ ions
50 - 200 keV

- **Elastic energy loss** yields the **mass** of the atom scattered off
- **Inelastic energy loss** within the sample enables **depth analysis**

Backscattering of 50 - 200 keV H+ or He+ ions off nanometre thin overlayers / films on top of a Si (100) surface.

## Scattering configuration & Data output

100 keV He +   Si (001) surface   Detector
[-1-11]   [112]   [111]   [332]

**Channeling & Blocking**
**(Double alignment)**

is used to suppress scattering from the underlying Si(100) crystal matrix and maximise the scattering yield from overlayers, amorphous or crystalline.

Channeling   Double alignment   Blocking

### Data output:
2-D ion scattering yield Spectrum as function of energy and scattering angle

2-D Detector System Encodes Image

Toroidal Electrostatic Analysis

Beam   Sample   Ions

Energy (keV)

Sr (buried)
Ti (top)
Ti (bottom)
[112]
Si (buried)
O (surface)

76   90   104
Scatt angle (degr)

Toroidal electrostatic analyser   Ion beam   Target goniometer

## MEIS facility at IIAA

Ion beam: 100-200 keV H+ or He+
Ang. divergence: < 0.1
Spot size: 1 mm x 0.5 mm

Endstation platform

Electrostatic Quadrupole Triplets

*MEIS experimental endstation fitted with Surface Science preparation chamber*

Preparation chamber

MEIS analysis chamber

Analysing magnet

Acceleration lens

High Voltage ion source platform

*Newly built 200 keV ion accelerator*

## High-k STO MIMcap layers

Ongoing scale reduction in μ-electronics as $SiO_2$ fails.

| DRAM MIMcap structure | |
|---|---|
| 2 nm | TiN |
| 3 nm | STO |
| 3 nm | TiN |
| | SiO₂ |

New high-k capacitor layer dielectrics are needed. $SrTiO_3$ (STO) and $TiO_2$ are promising materials. STO has a high dielectric constant, k ≥ 200.

Band gap (eV) vs Dielectric Constant
$SiO_2$   $Al_2O_3$   $Y_2O_3$   $ZrO_2$   $HfO_2$   $Ta_2O_5$   $Si_3N_4$   $La_2O_3$   $Nb_2O_5$   HfTiO   $TiO_2$   $SrTiO_3$

### MEIS analysis of the effect of thermal processing
Ti & N peaks due to 2 TiN layers; Sr & O peaks

Pre annealing D11
Simulation D11
Post annealing D12
Simulation D12

Scattering yield (cts)
N N   O O   Si   Ti Ti Ti   Sr   Surface Sr
50   60   70   80   90   100
Energy (keV)

- **Spectrum simulation** yields depth profile: **layer thicknesses verified to within 0.2 nm.**
- Red MEIS spectrum taken after heating to 650 °C
  - Ti diffuses into intermediate STO layer during annealing.
  - Some Sr segregation and oxidation on surface (narrow peaks).

J.A. van den Berg et al., Appl Surface Sci 281, 8 (2013)

## Spintronics layers

% Ni vs anneal temperature [C]
Ni in top layer
- Cu atom   - Ni atom
< 50 C
Idealised model
> 50 C

layer1   layer2   layer3
-150   -100   -50   0   50   100   150
anneal temperature [C]

T C Q Noakes, P Bailey et al. PRB 67 (2003) 153401

**MEIS determines:**
➢ The instability of a 0.18 nm thick Ni layer just above room temperature (50 °C).
➢ An idealised model of the rearrangement of the Cu and Ni atoms, i.e. the formation of a monolayer of Cu on top, shadowing of the Ni atoms.

## Au/Pd nanoparticles for catalysis

MEIS analysis of Au/Pd alloy nanoparticles supported on silica planar oxide films (AuPd bimetallic catalysts are used in e.g. Vinyl acetate synthesis)

Energy (keV)   Au   Pd
Scattering Angle
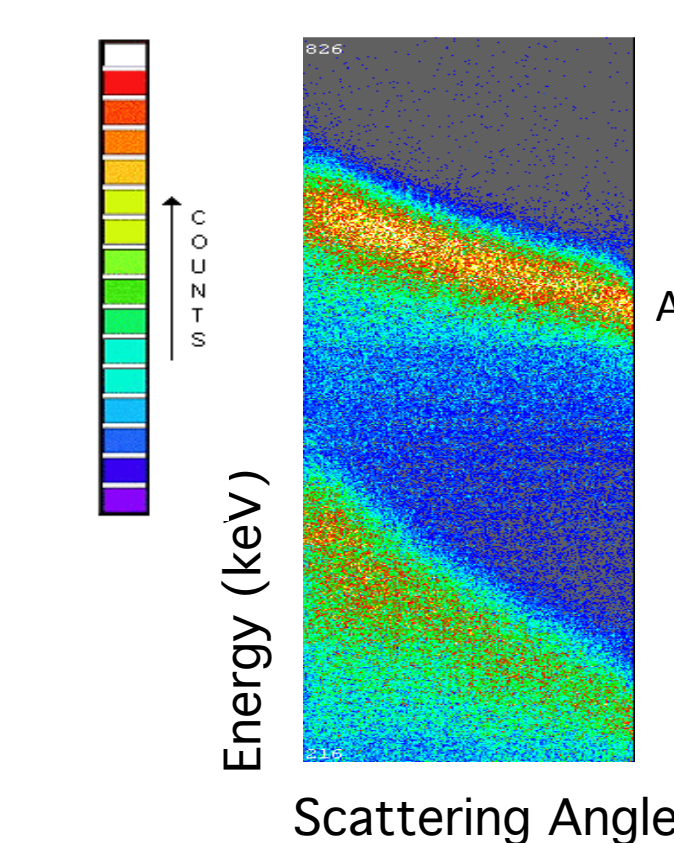
### MEIS backscattering peak shape studies & extensive model simulations

Pd   Au
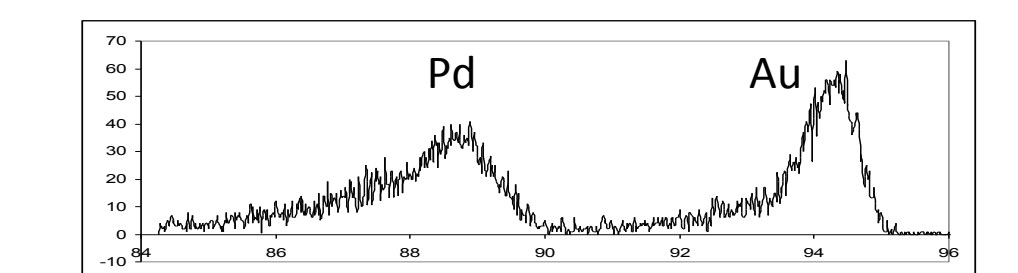
- particles are formed with a gold-enriched shell and palladium-enriched core even at room temp.

**Au enriched shell, Pd enriched core**

Base   Core   Surface

- Au/Pd interdiffusion at high temperatures
- MEIS can see through absorbate layers

A R Haire et al,
Surface Science 605 214 (2011)

## Industrial Al -1% Cu oxide growth

TEM section   Particles enriched in Cu
thin oxide
MEIS depth profile   Al alloy   20 nm
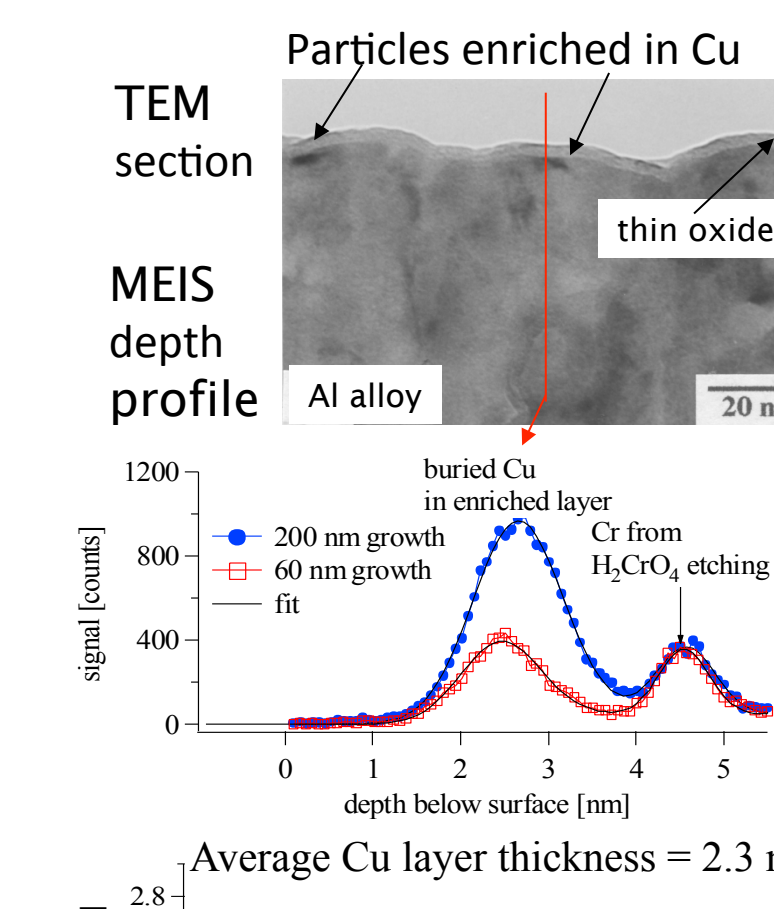
- Oxidation of Al-1% Cu produces a Cu-enriched layer, which has major effects on both the corrosion and growth of oxide films.
- Experimentally, thick oxides 20-200 nm thick are grown anodically; then etched away with chromic acid to enable MEIS analysis.

buried Cu in enriched layer
200 nm growth   60 nm growth   Cr from $H_2CrO_4$ etching   fit
signal
depth below surface [nm]

Average Cu layer thickness = 2.3 nm
layer thickness vs grown oxide thickness [nm]

O   Al P   Cu   Cr
Counts   signal   model
45   50   60   70   80   85
Energy (keV)

- Cu enrichment occurs not through film growth but by the generation of increasing numbers of Cu-rich nano-cluster of constant thickness.

Carcia-Vergara et al. Appl Surf Sci 205 (2003) 121
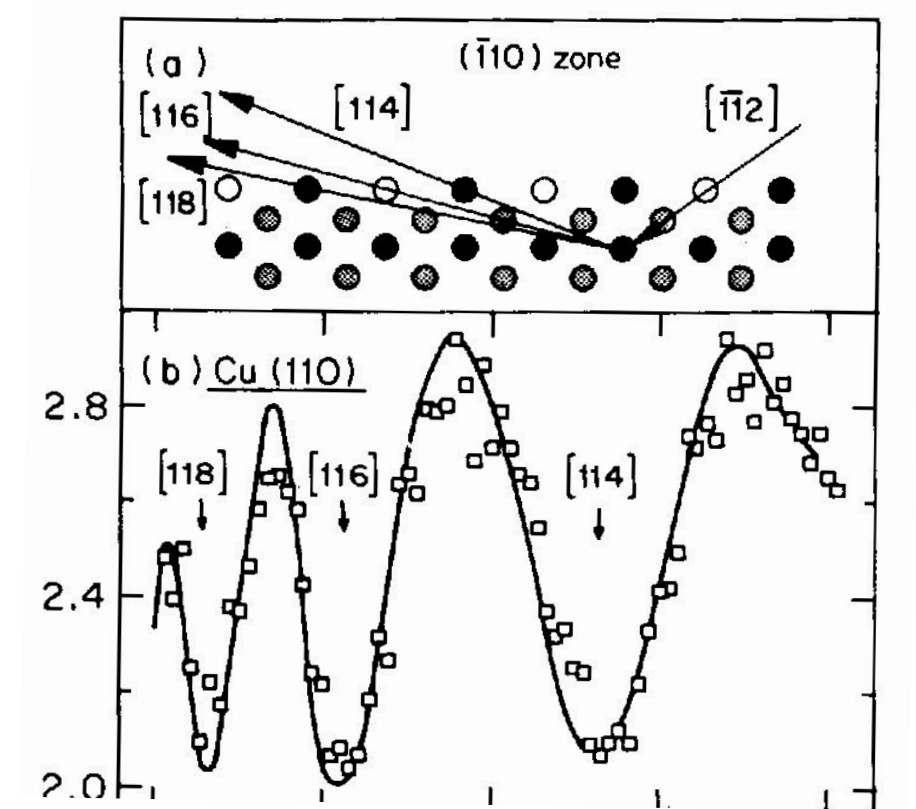
## Ion beam crystallography

### Angular Intensity Variation on Cu (110) surface beam along [-1-12]

- Shadowing effects used to select number of layers illuminated
- Blocking effects reveal relative positions of the atoms (i.e. the layer structure)
- Shifts in blocking dips related to changes in layer spacing (surface relaxation, strain)
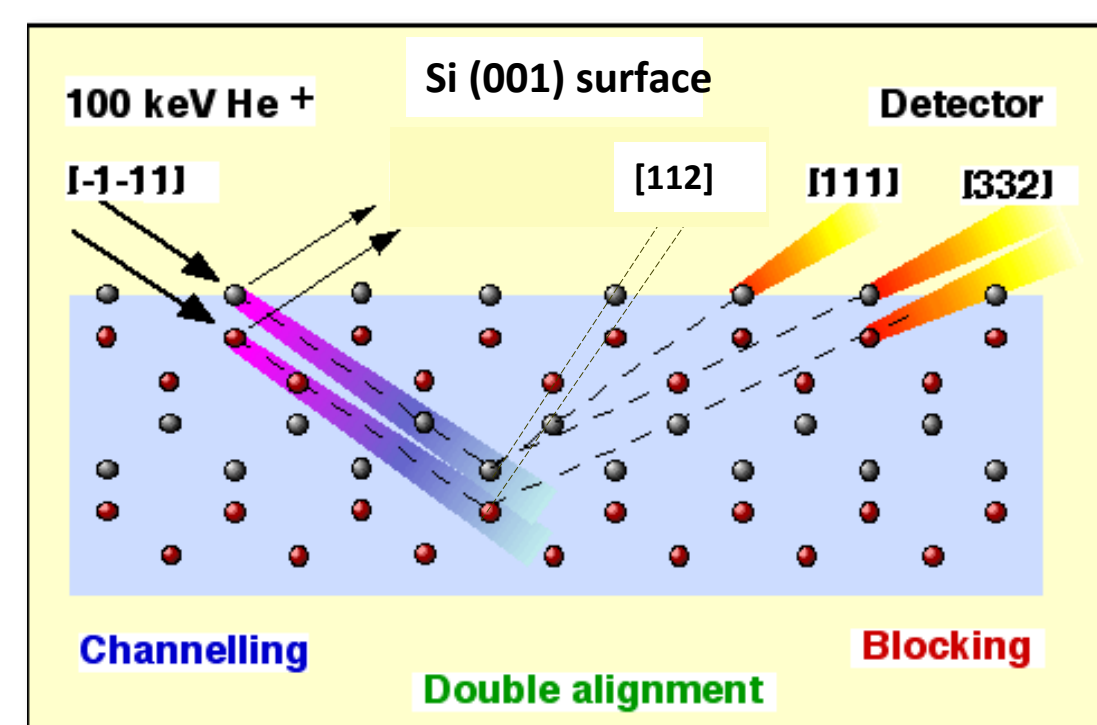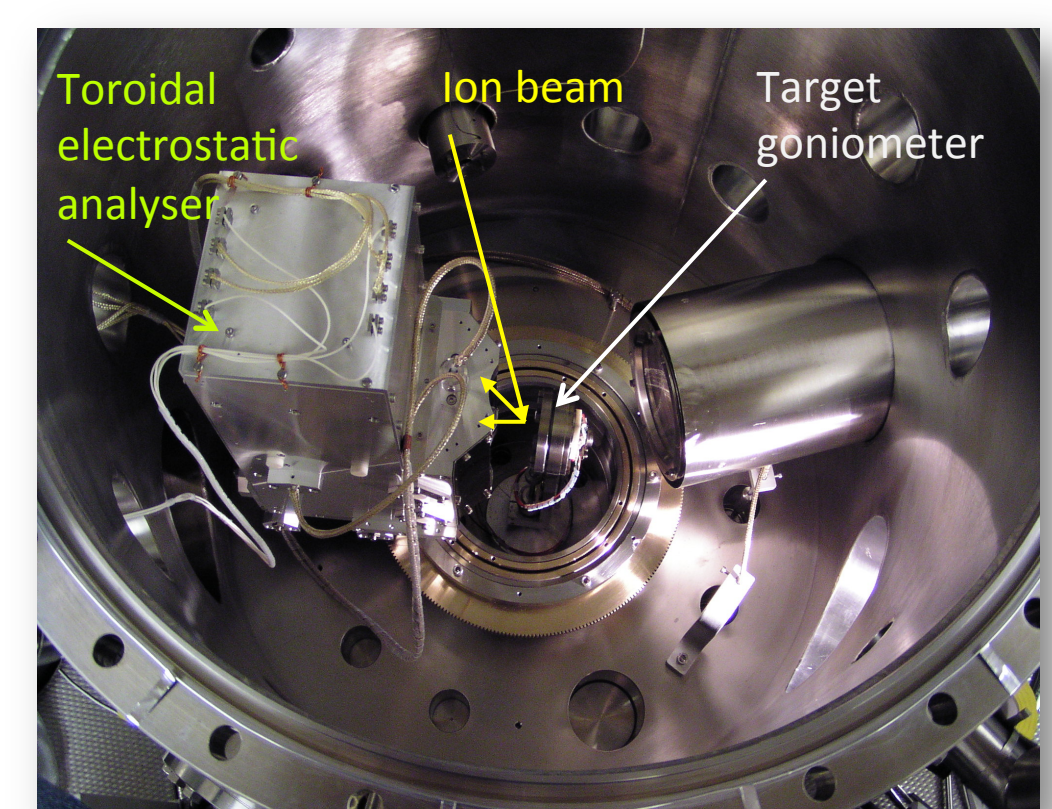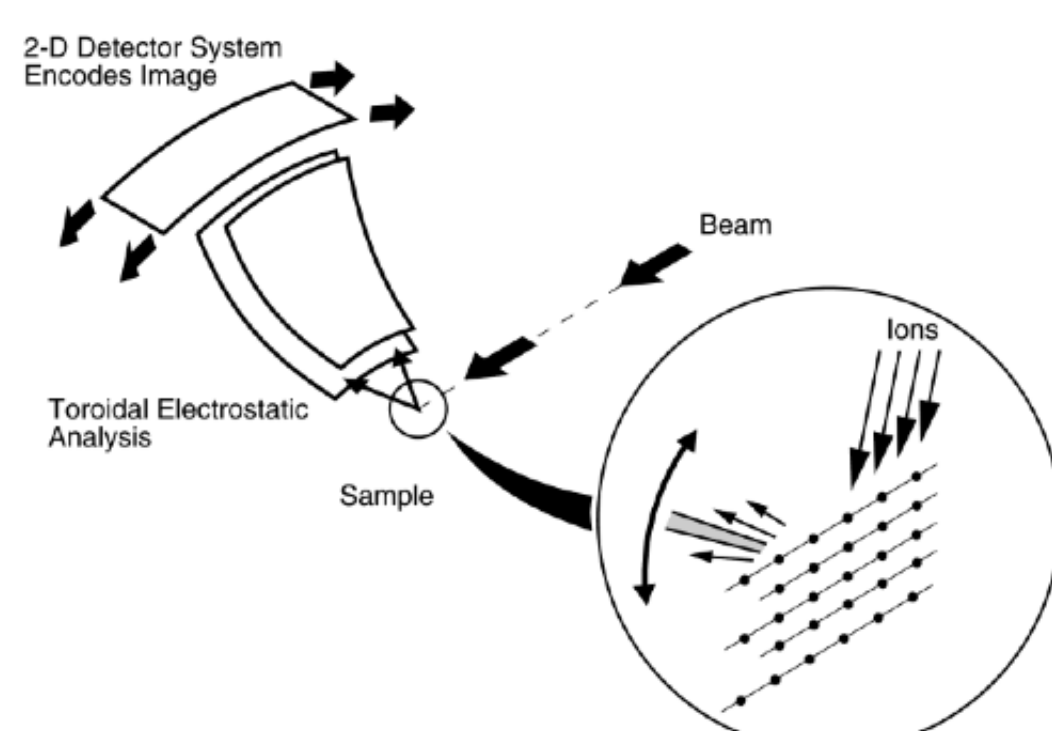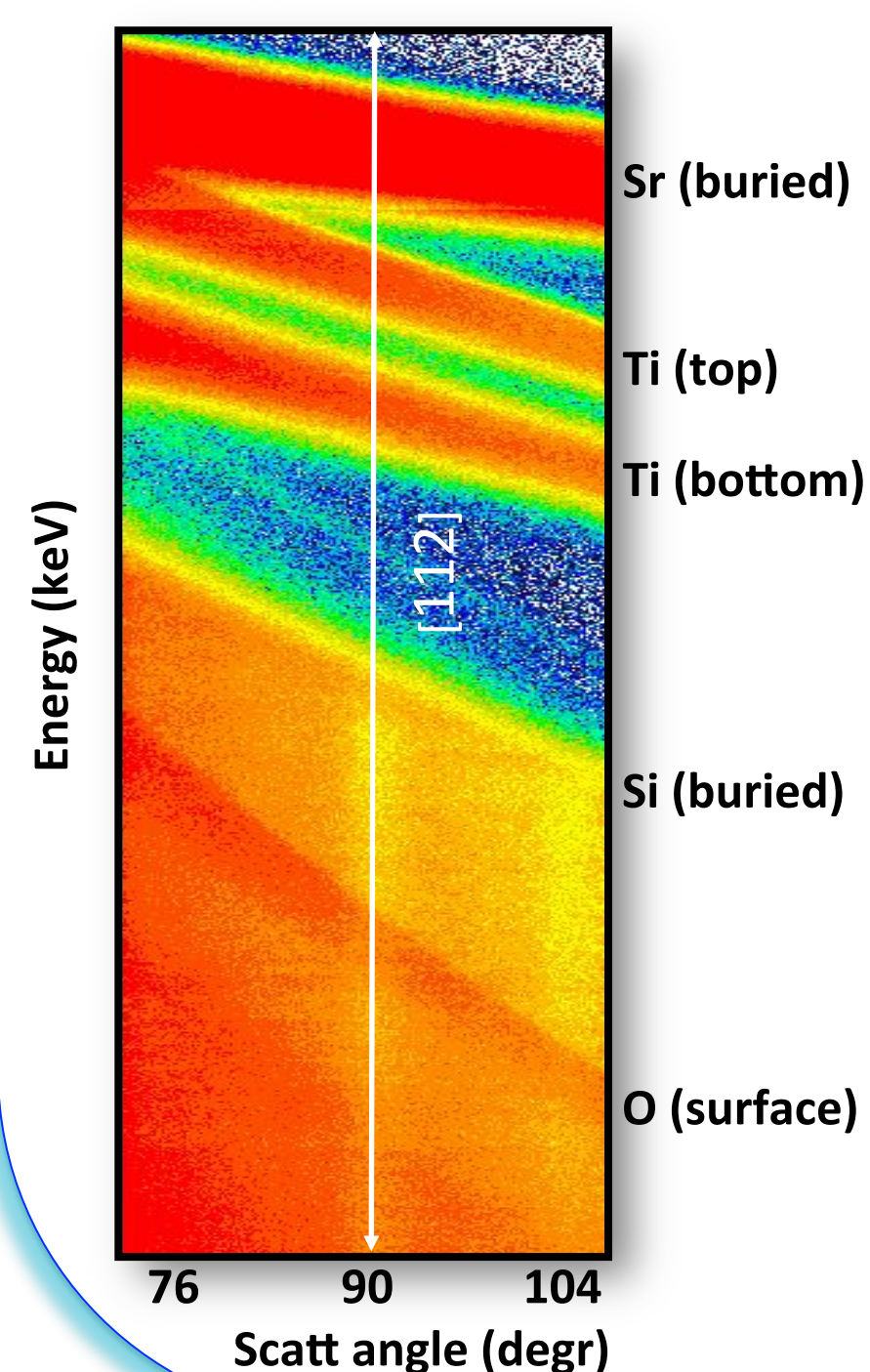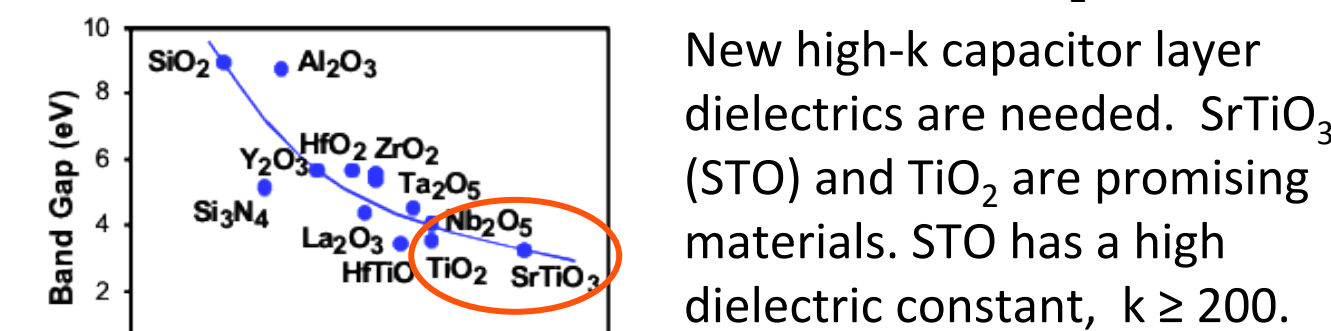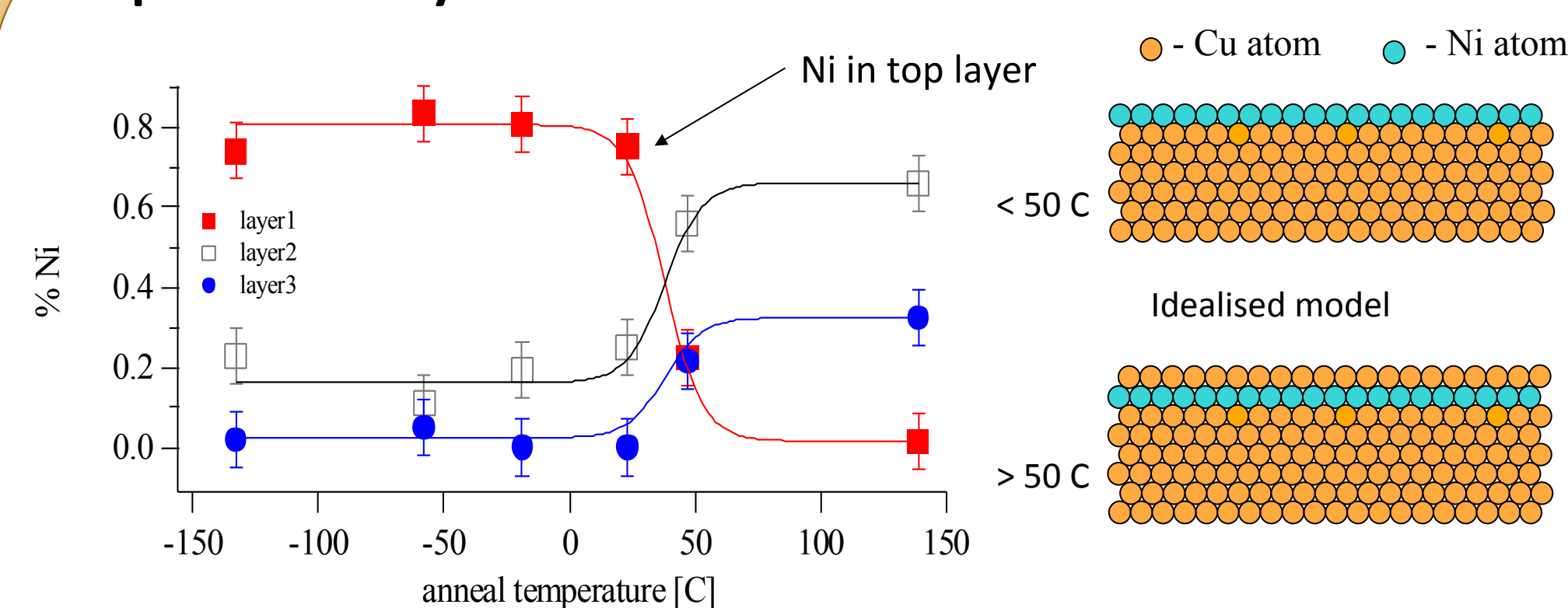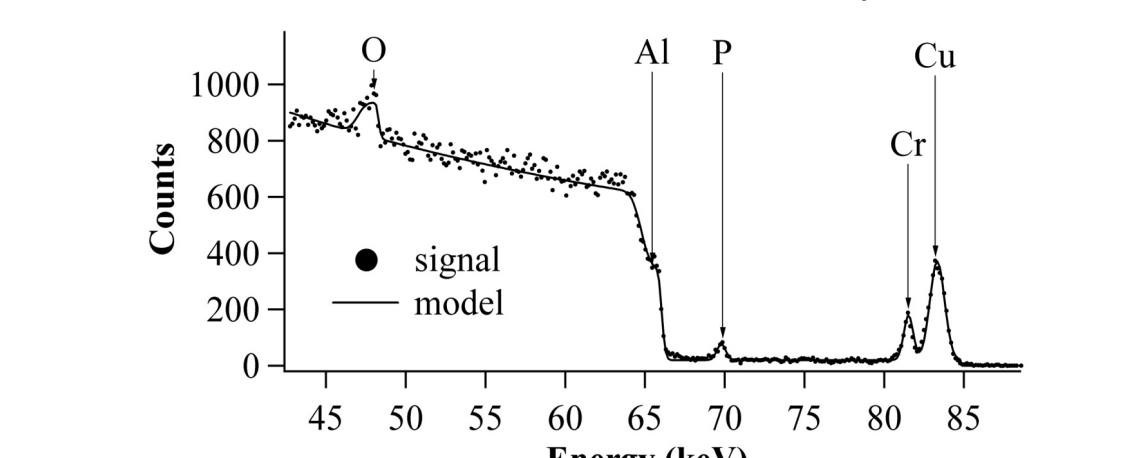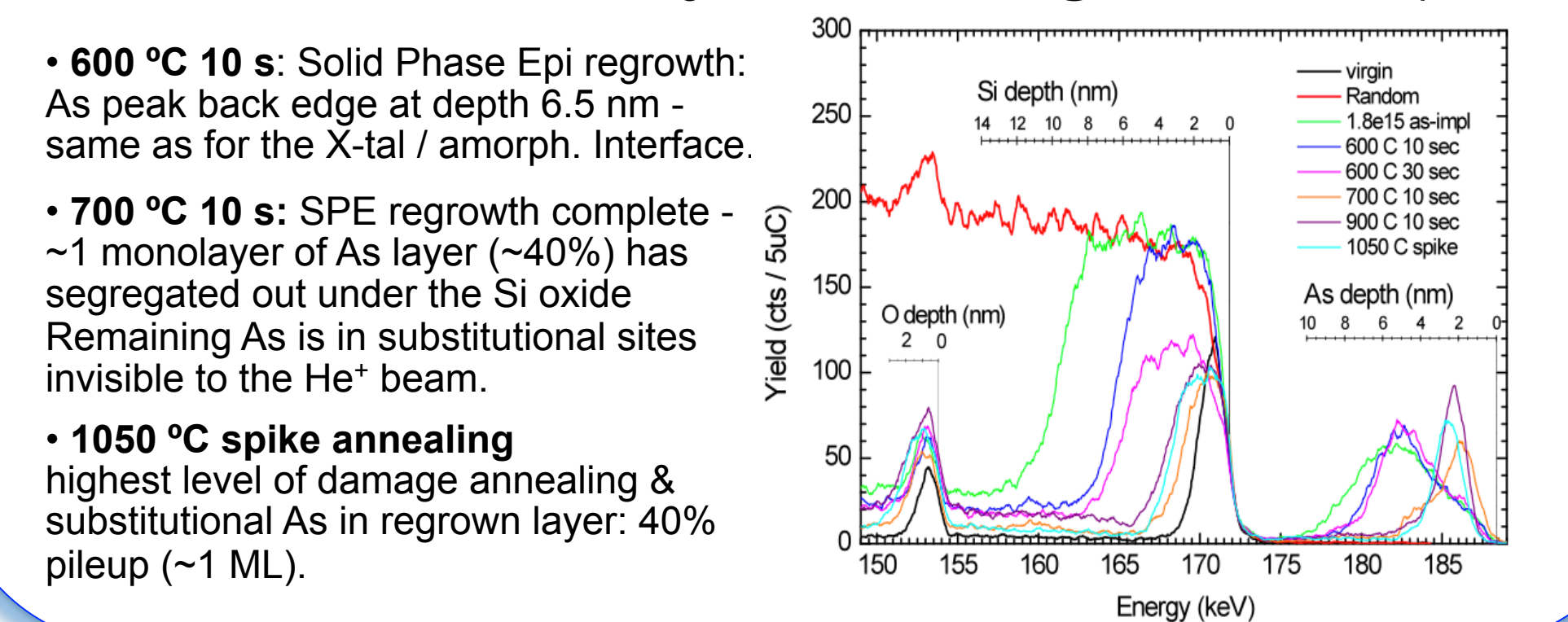- Amplitudes of dips indicate additional illumination (thermal vibrations, disorder)

(a)   (\overline{1}10) zone
[116]   [114]   [\overline{1}12]
[118]
(b) Cu (110)
[118]   [116]   [114]
2.8   2.4   2.0

(after Tim Noakes, Daresbury Laboratory)

## Spectrum simulation

Whether MEIS is used for Ion beam crystallography on the basis of angular intensity spectra or Depth profiling using energy spectra, extensive and detailed spectrum simulation is an indispensible part of obtaining the information presented. Simulation programs include VEGAS (Warwick Uni group) for Ion beam crystallography or SIMNRA (M Maier) or IGOR® based (P Bailey) and others for quantitative depth profiling.

## Shallow As implant & Si disorder annealing

CMOS transistors now require ultra shallow (< 15 nm) S/D extension junctions.
Ion implantation causes Si lattice disorder. Annealing produces reordering & dopant activation MEIS can probe dopant and disorder build-up and annealing for a 2.5 keV As+ @ 1.8 E 15 cm⁻² implant.

Gate   Gate oxide
Source   Channel   Drain
Extensions

- **600 °C 10 s:** Solid Phase Epi regrowth: As peak back edge at depth 6.5 nm - same as for the X-tal / amorph. Interface.
- **700 °C 10 s:** SPE regrowth complete - ~1 monolayer of As layer (~40%) has segregated out under the Si oxide Remaining As is in substitutional sites invisible to the He+ beam.
- **1050 °C spike annealing** highest level of damage annealing & substitutional As in regrown layer: 40% pileup (~1 ML).

virgin   Random
14 12 10 8 6 4 2 0
Si depth (nm)
1.8e15 as-impl
600 C 10 sec
600 C 30 sec
700 C 10 sec
900 C 10 sec
1050 C spike
O depth (nm)
As depth (nm)
Yield (cts / SμC)
Energy (keV)
150   155   160   165   170   175   180   185

## Conclusions

MEIS provides unique compositional and structural information on crystalline, metal, alloy, oxide and semi-conductor surfaces and amorphous overlayers.

*Surface Crystallography*
➢ Adsorbed atom position
➢ Over layer registry
➢ Outer layer expansion / contraction

*High depth resolution profiling*
➢ Micro (nano) electronics: Shallow ion implant and plasma doping: dopant profiles; disorder annealing; High-k gate oxide and DRAM MIMcap dielectric nanolayers
➢ Atomic layer deposition film growth
➢ Corrosion protection of light alloys
➢ Spintronics
➢ Model catalytic systems - nanoparticles

The re-established MEIS facility is now operational and open for collaborative research in existing and new areas.