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

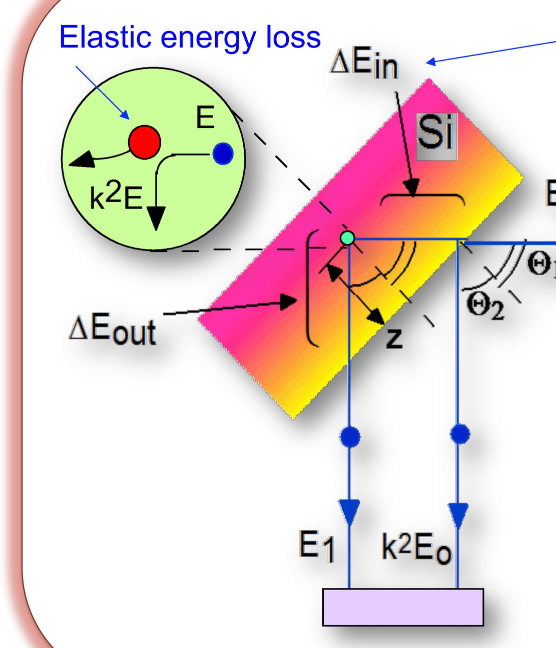
J. A. van den Berg[a], P. Bailey[a], R. Barlow[a], T. C. Q. Noakes[b], S. H. Kilcoyne[a] and R. Cywinski[a].
[a] International Institute for Accelerator Applications, School of Applied Sciences, University of Huddersfield, Huddersfield, HD1 3DH, UK.
[b] STFC Daresbury Laboratory, Warrington, WA4 4AD, 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:



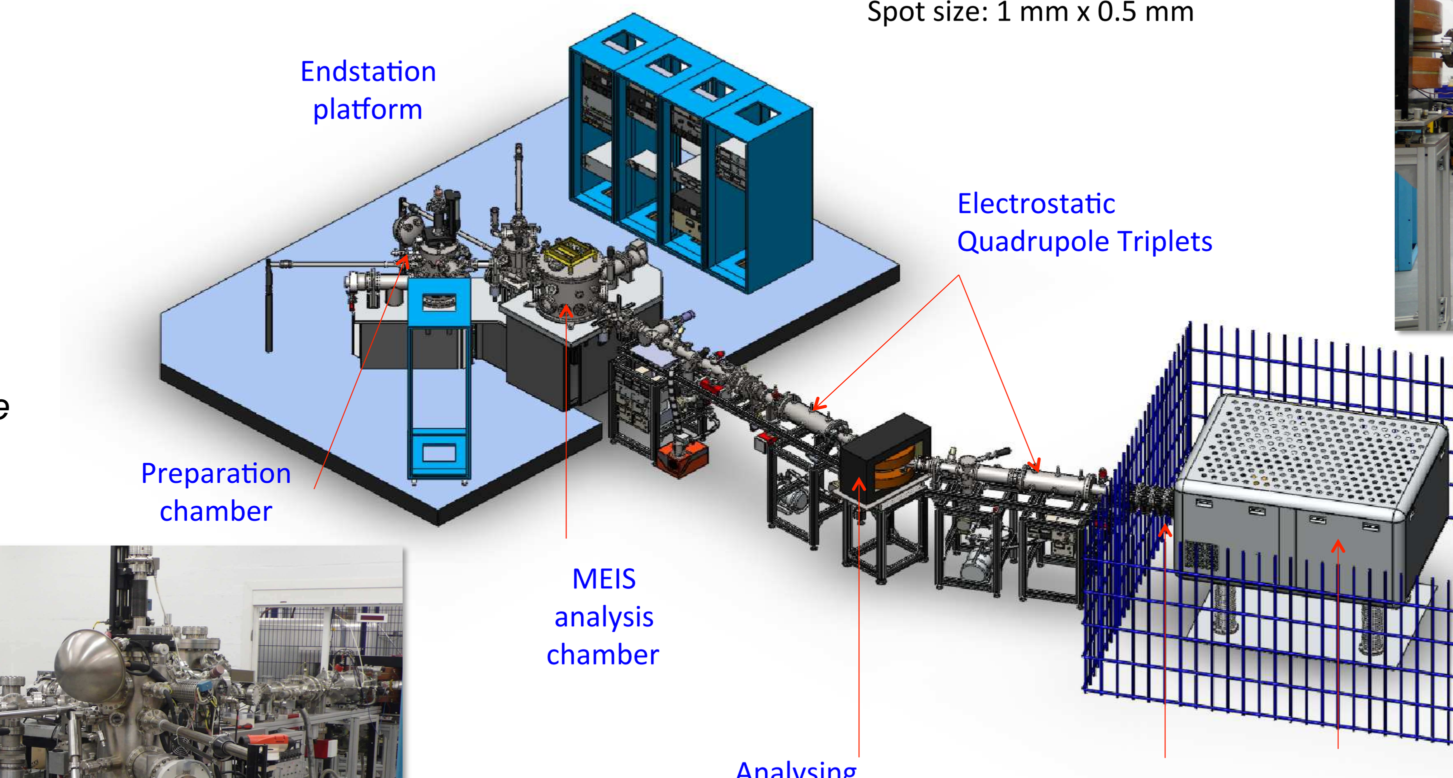
- **Elastic energy loss** yields the **mass** of the atom scattered off
- **Inelastic energy loss** within the sample enables **depth analysis**

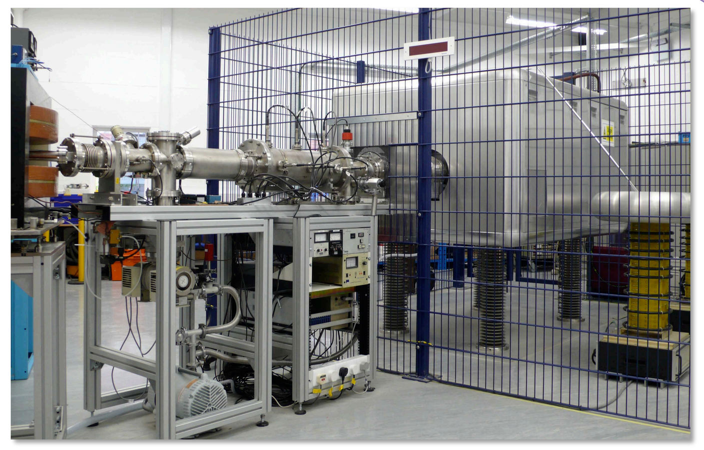
Backscattering of 50 - 200 keV H^+ or He^+ ions off nanometre thin overlayers / films on top of a Si (100) surface.

MEIS facility at IIAA

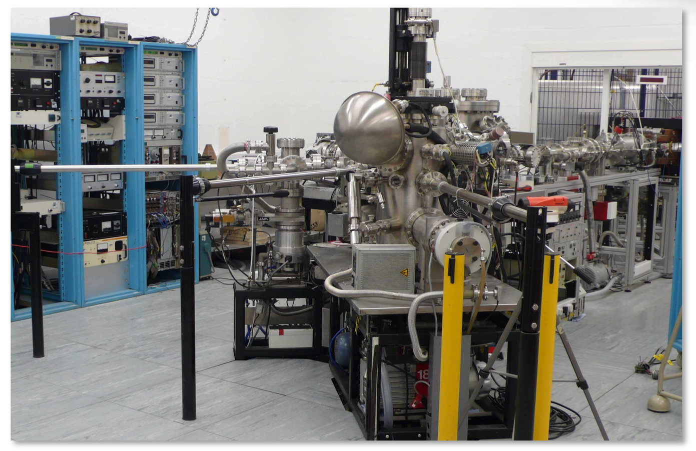
Ion beam: 100-200 keV H^+ or He^+
Ang. divergence: < 0.1
Spot size: 1 mm x 0.5 mm

MEIS experimental endstation fitted with Surface Science preparation chamber





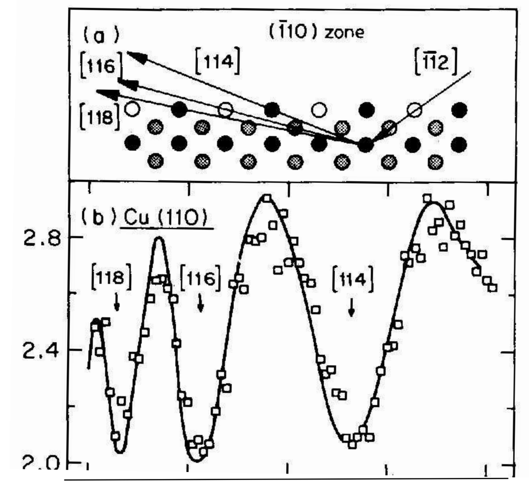
Newly built 200 keV ion accelerator



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)

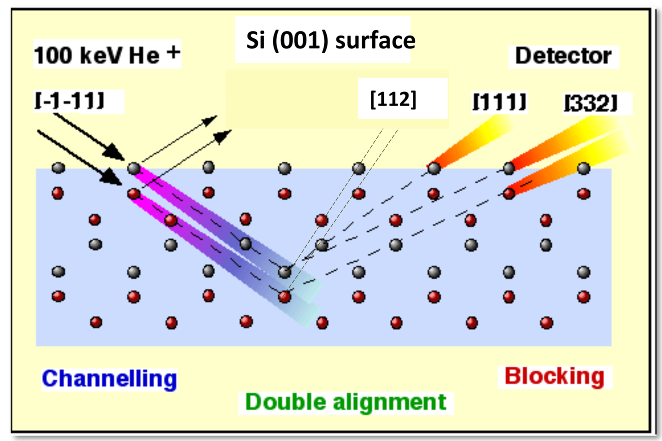


(after Tim Noakes, Daresbury Laboratory)

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.

Scattering configuration & Data output

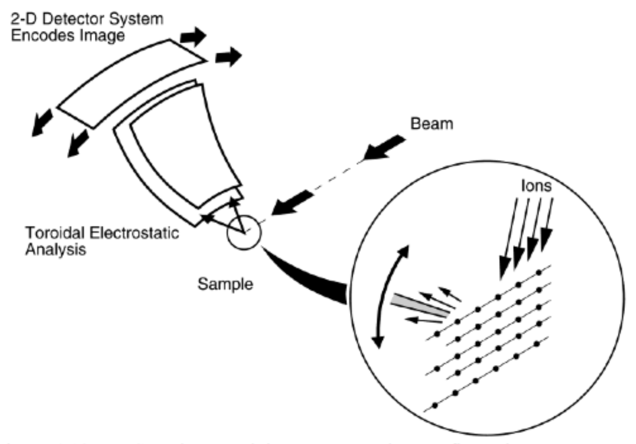


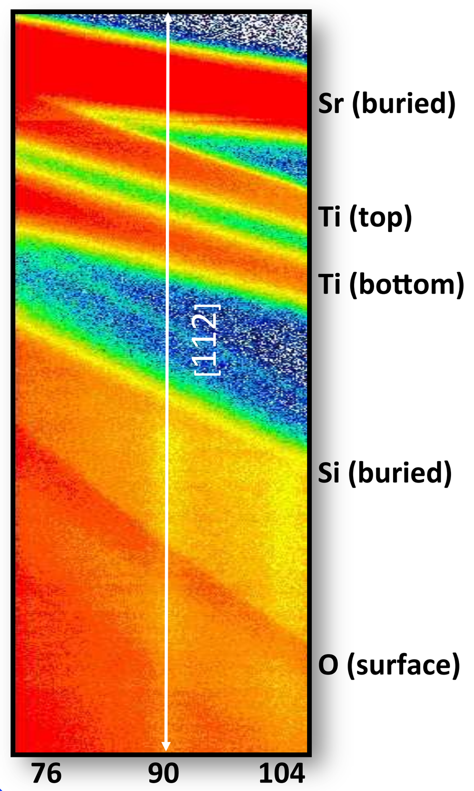
Channeling & Blocking (**Double alignment**)

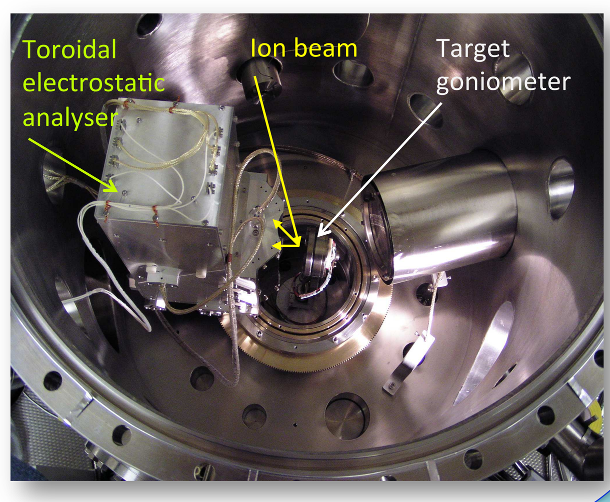
is used to suppress scattering from the underlying Si(100) crystal matrix and maximise the scattering yield from overlayers, amorphous or crystalline.

Data output:

2-D ion scattering yield
Spectrum as function of energy and scattering angle

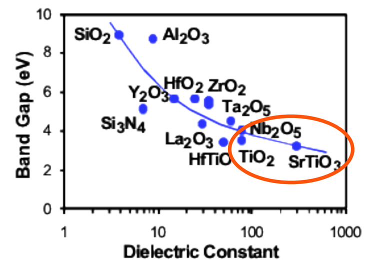






High-*k* STO MIMcap layers

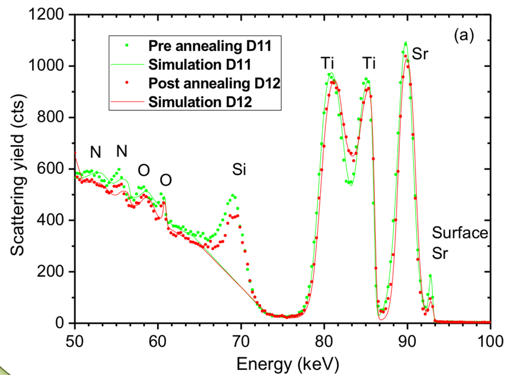
Ongoing scale reduction in μ-electronics as SiO_2 fails.



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.

DRAM MIMcap structure

2 nm	TiN
3 nm	STO
3 nm	TiN
	SiO_2



MEIS analysis of the effect of thermal processing

Ti & N peaks due to 2 TiN layers; Sr & O peaks

- Spectrum simulation yields depth profile: **layer thicknesses verified to within 0.2 nm.**

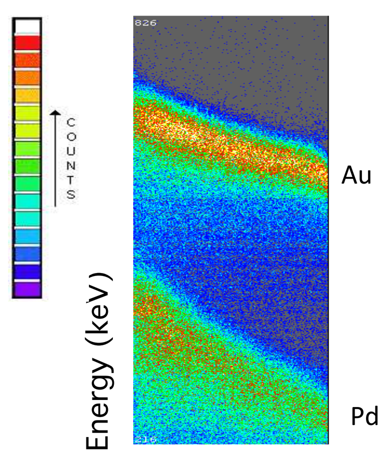
Red MEIS spectrum taken after heating to 650 °C

- 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)

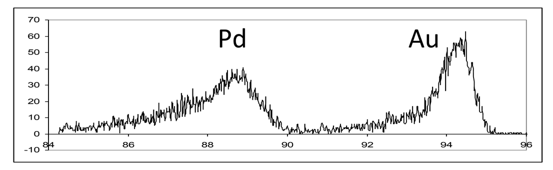
Au/Pd nanoparticles for catalysis

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



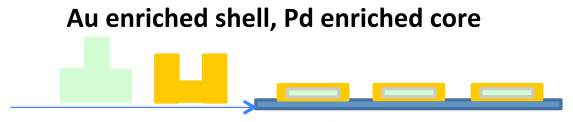
A R Haire et al,
Surface Science 605 214 (2011)

MEIS backscattering peak shape studies & extensive model simulations



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

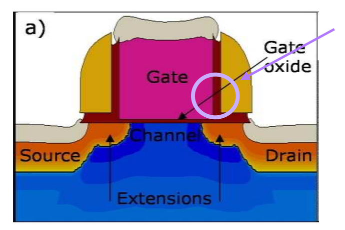
Au enriched shell, Pd enriched core



Base Core Surface

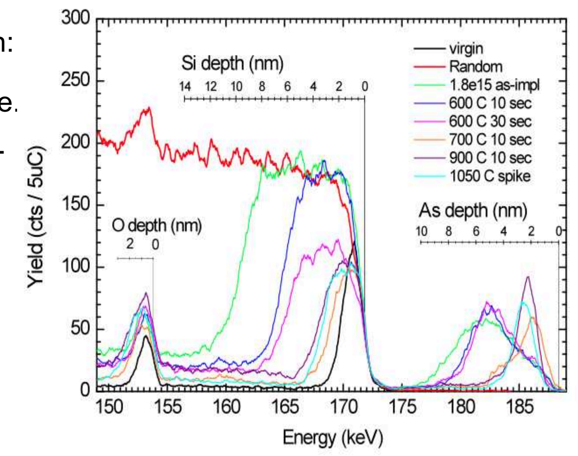
- Au/Pd interdiffusion at high temperatures
- MEIS can see through absorbate layers

Shallow As implant & Si disorder annealing

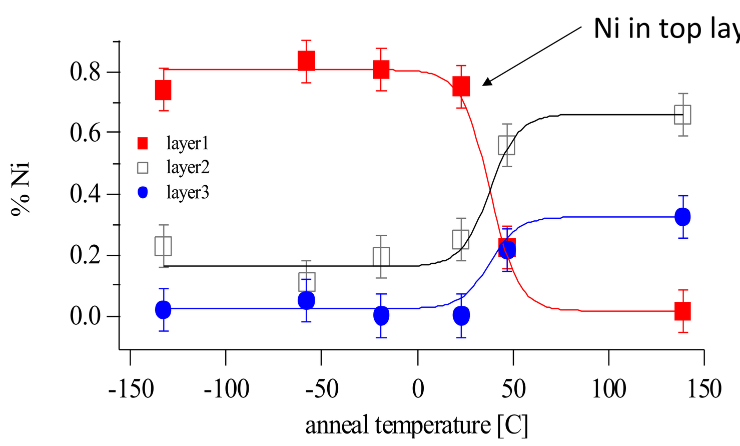


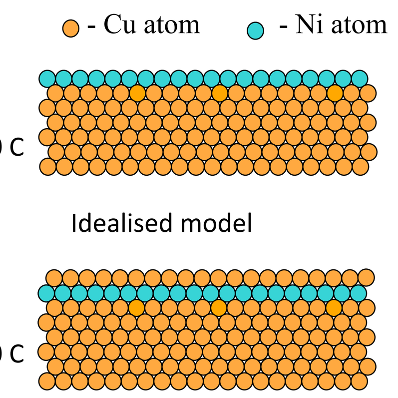
CMOS transistors now require ultra shallow (< 15 nm) S/D extension junctions.
Ion implantation causes Si lattice disorder.
Annealing produces reordering & dopant activation
MEIS can probe dopant and disorder build-up and annealing for a 2.5 keV As^+ @ 1.8 E 15 cm^{-2} implant.

- **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 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).



Spintronics layers



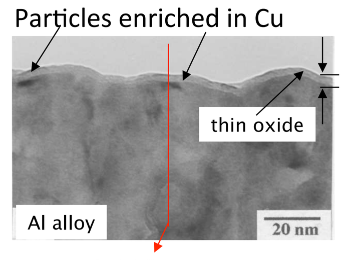


T C Q Noakes, P Bailey et al. PRB 67 (2003) 153401

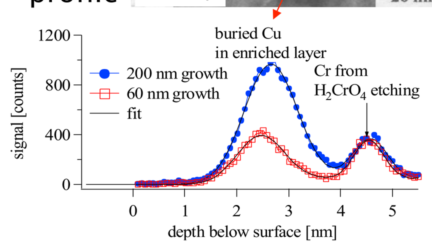
MEIS determines:

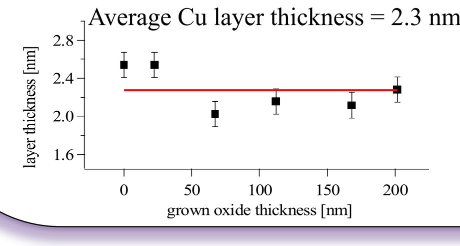
- The instability of a 0.18 nm thick Ni layer just above room temperature (50 °C).
- An idealised model of the rearrangement of the Cu and Ni atoms, i.e. the formation of a monolayer of Cu on top, shadowing of the Ni atoms.

Industrial Al -1% Cu oxide growth

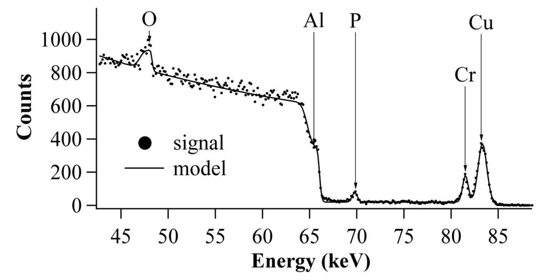


MEIS depth profile





- 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.



- 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

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.

Contact: j.vandenberg@hud.ac.uk