MIAMI
Microscopes and
Ion Accelerators for
Materials Investigations

University of
HUDDERSFIELD

MIAMI Facilities

S E Donnelly
School of Computing and Engineering
University of Huddersfield, UK

EPSRC
Engineering and Physical Sciences
Research Council

The UK National Ion Beam Centre

In-situ TEM / ion accelerator facilities

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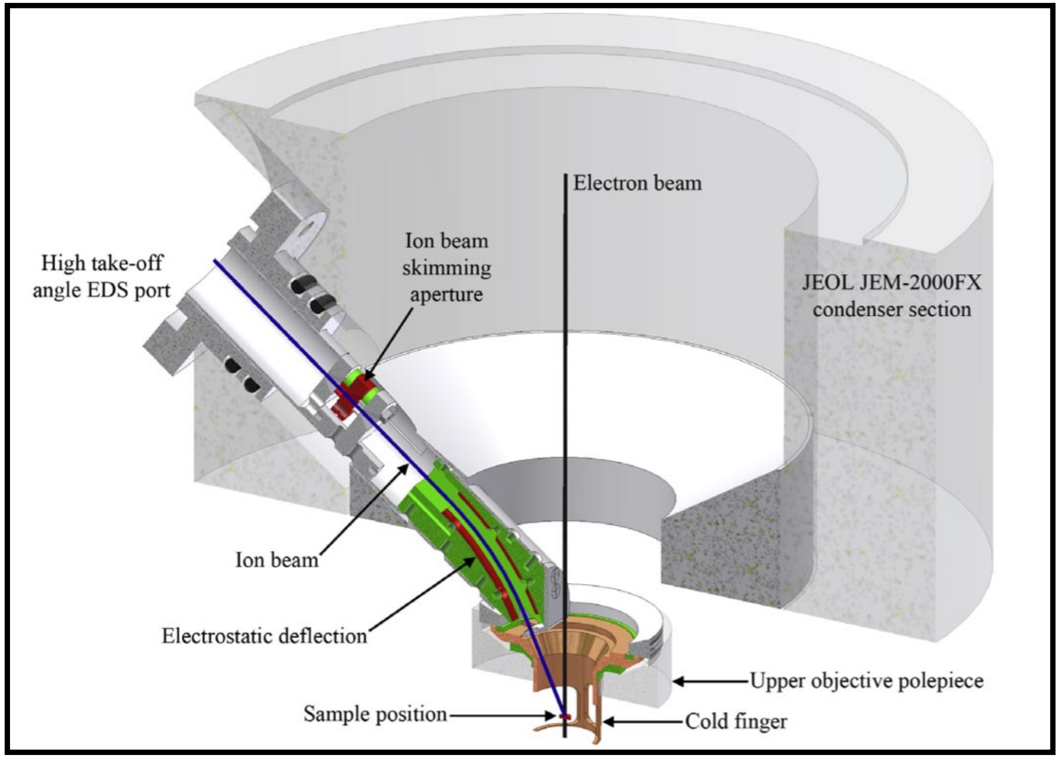

Experimental systems combining one or more ion-accelerators with a transmission electron microscope (TEM) enabling observation of ion-beam induced radiation damage at high magnification in real time.

Permit study of the formation and development of defects at the nanoscale during ion irradiation, often providing insights into fundamental properties and processes that are difficult to obtain by other means.

Applications

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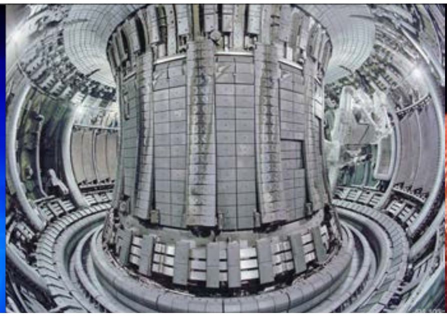
Investigations into any materials subjected to radiation damage from energetic particles:

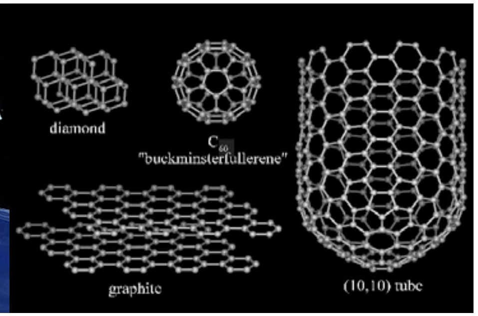
- Materials for nuclear fission (current and Gen IV), nuclear fusion (especially for plasma facing components) and glasses and ceramics for nuclear waste storage;

- Semiconductor processing and damage to microelectronic devices used in irradiating environments;

- Materials for space;

- Nanotechnology – e.g. effects of ion irradiation on nanostructures; use of ion beams for nano-patterning.

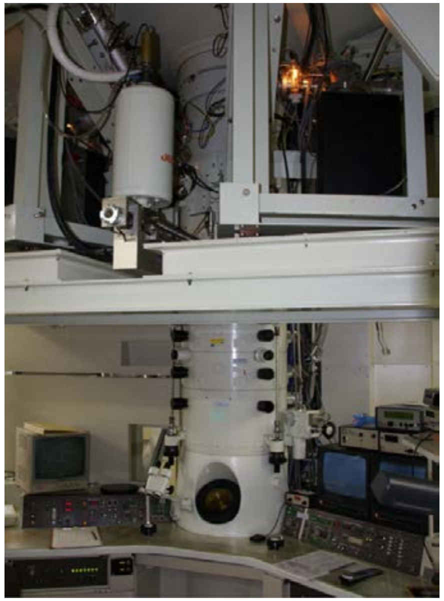
In-situ TEM / ion accelerator facilities

Kyushu, Japan

Argonne, USA

Hokkaido, Japan

Tsukuba, Japan

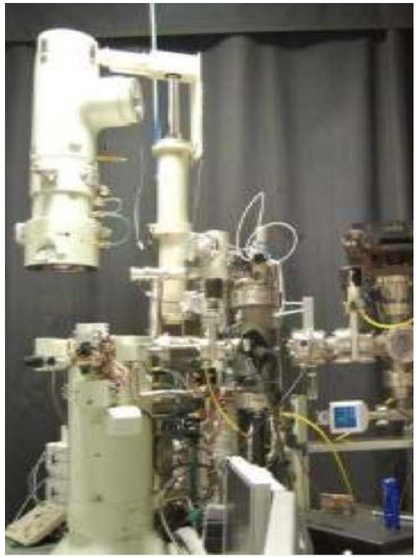


Shimane, Japan

Huddersfield, UK

Orsay, France

Wuhan, China



Sandia.USA

MIAMI-1

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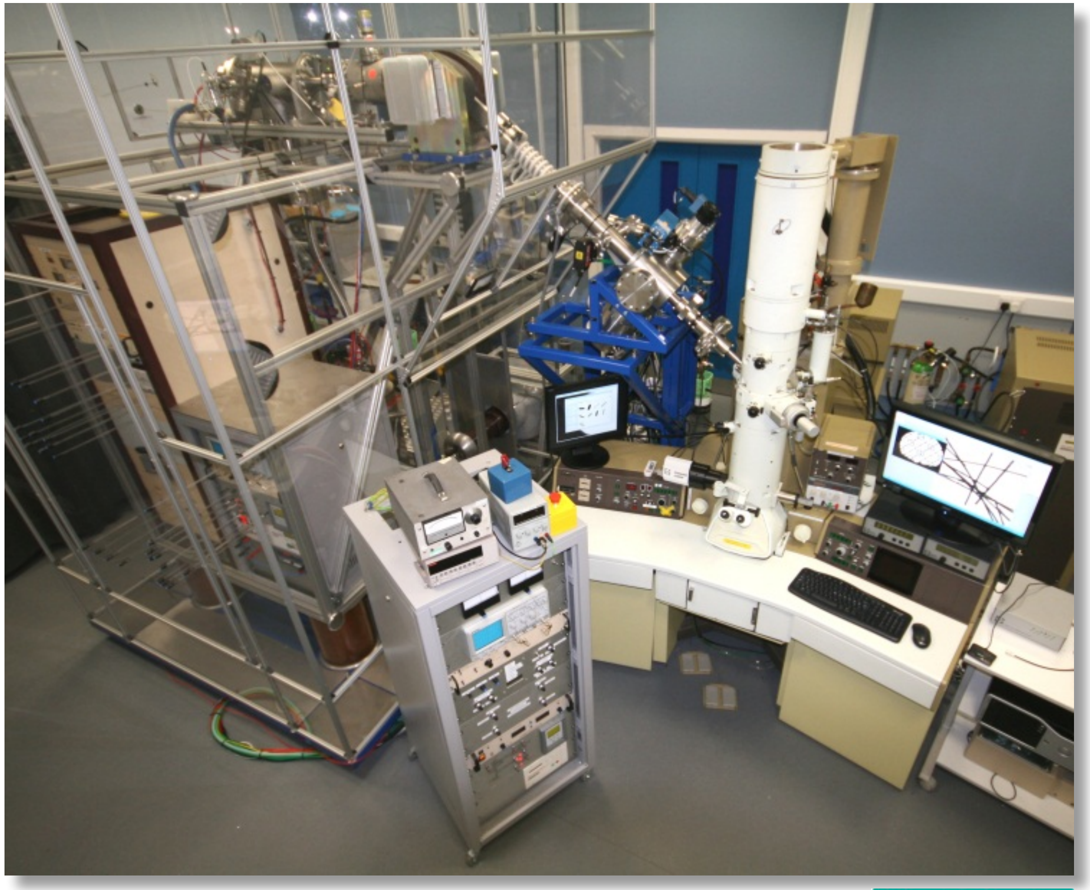
Specifications	
TEM	JEOL JEM-2000FX
e-Beam Accelerating Voltage	80 to 200 kV
Ion Beam Accelerating Voltage	1 to 100 kV
Ion Species	Most ions from H to W at all energies (limited by bending magnet)
Ion Flux	Fluxes of up to 1.5×10^{14} cm^{-2} s^{-1} for 6 kV He (for example)
Angle between Ion and Electron Beams	30°
Temperature	100 to 380 K or RT to 1270 K
Image Capture	Gatan ES500W Wide Angle CCD Gatan Orius SC200 (4 Megapixels)

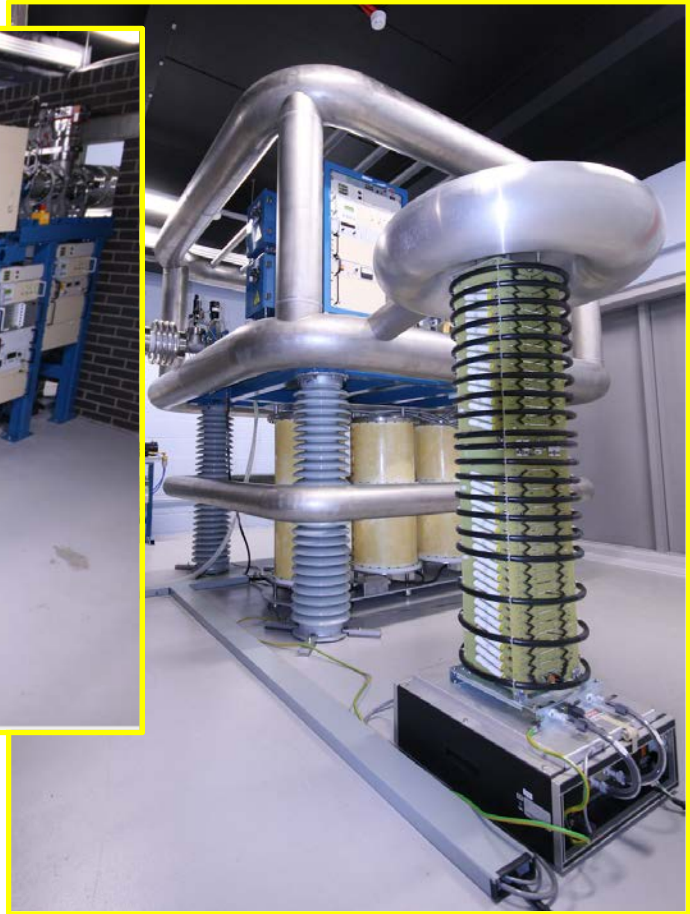


EPSRC

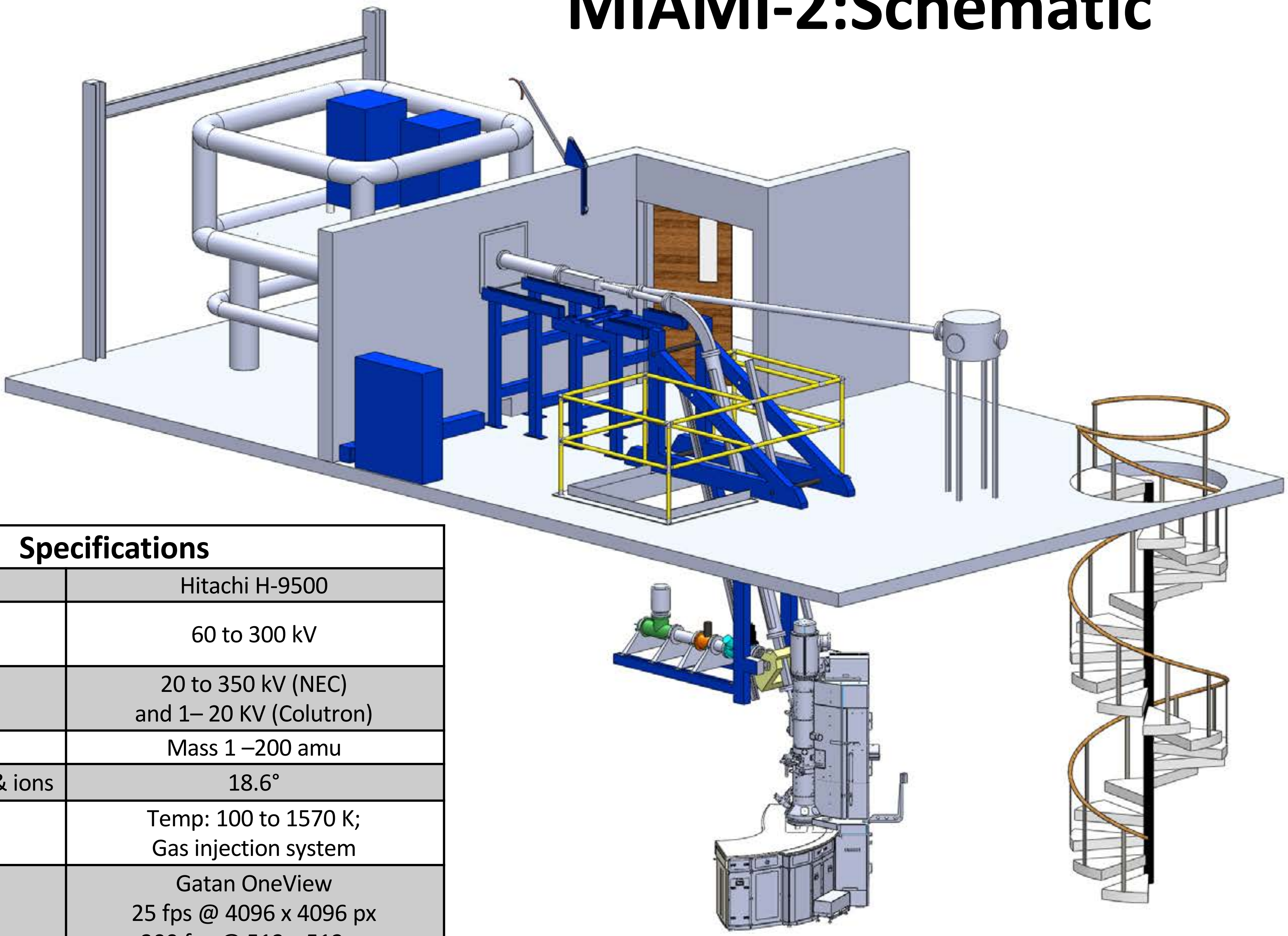
MIAMI-1 has been running, initially at the University of Salford and now at the University of Huddersfield, since about 2008. A particular focus has been irradiation with beams of low-energy inert-gas ions (particularly He).

For details of MIAMI-1, see: J. A. Hinks, J. A. van den Berg and S. E. Donnelly, J. Vac. Sci & Technol. *A* **29** (2011) 021003

MIAMI-2



MIAMI-2:Schematic



Specifications	
TEM	Hitachi H-9500
e-Beam Acc. Voltage	60 to 300 kV
Ion Beam Acc. Voltage	20 to 350 kV (NEC) and 1– 20 KV (Colutron)
Ion Species	Mass 1 –200 amu
Angle between e⁻s & ions	18.6°
Environment	Temp: 100 to 1570 K; Gas injection system
Image Capture	Gatan OneView 25 fps @ 4096 x 4096 px 300 fps @ 512 x 512 px
Analysis	EELS (Gatan Imaging Filter) and Bruker EDS
Tomography	Tomography holder and software

MIAMI-2: Holders and Analysis

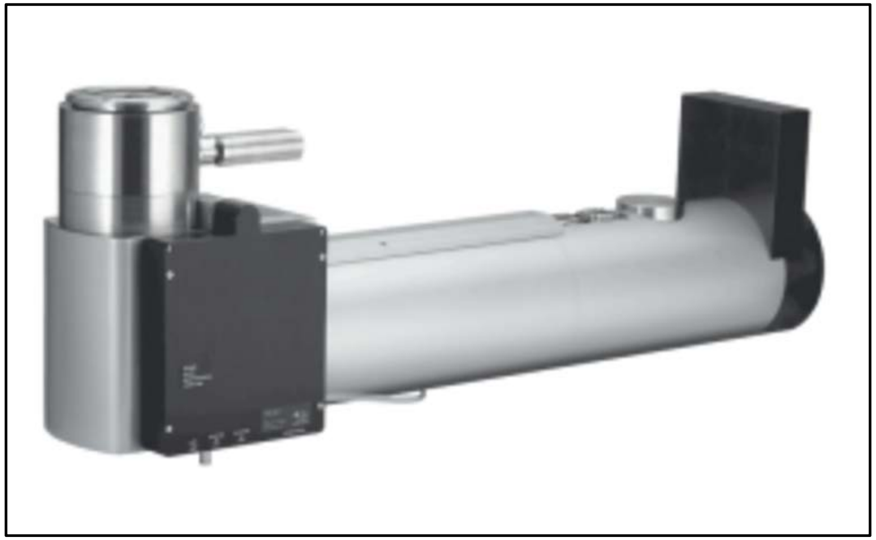
Holders:

- Hitachi Single tilt holder x 1
- Hitachi Double tilt holder x 1
- Hitachi Faraday cup holder x 1
- Gatan 628.TA single tilt heating holder x 2
- Gatan 652.MA double tilt heating holder x 2
- Gatan 636.MA double tilt liquid N_2 cooling holder x 1
- Gatan 901 temperature controller x 2
- Gatan 654.MA straining holder + 902 controller x 1
- Gatan 650 analytical tilt-rotate holder x 1

Heating up to 1300°C
Cooling down to ≈ 100°C
Straining

Analysis:

GATAN Imaging Filter (Quantum SE



Bruker Quantax 400 EDS

MIAMI-2: ETEM Capability

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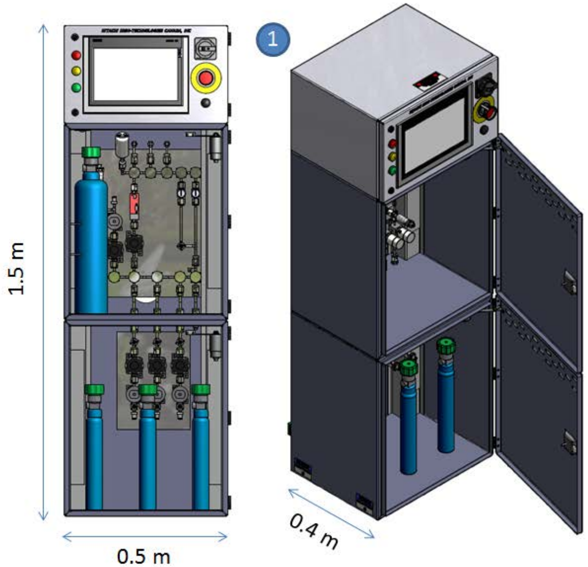
- Differentially-pumped specimen region;
- Can be used with any holder;
- Pressures to 1 Pa (10^{-2} mbar);
- Up to 4 gases.

3-2 Technologies Developed by HTC

HITACHI
Inspire the Next

Gas Handling System 3.0: standard



MAESTRO Package 3

- **4 gases**
- **Simple**
- **Safe**
- **Convenient interface**

- Up to 4 gases
- Ultimate safety: hardware and software
- Convenient user interface
- Reliable and easy to use
- Integrated with MAESTRO

Hitachi High-Tech

Examples of Experiments using MIAMI

Xe irradiation of SiO_2

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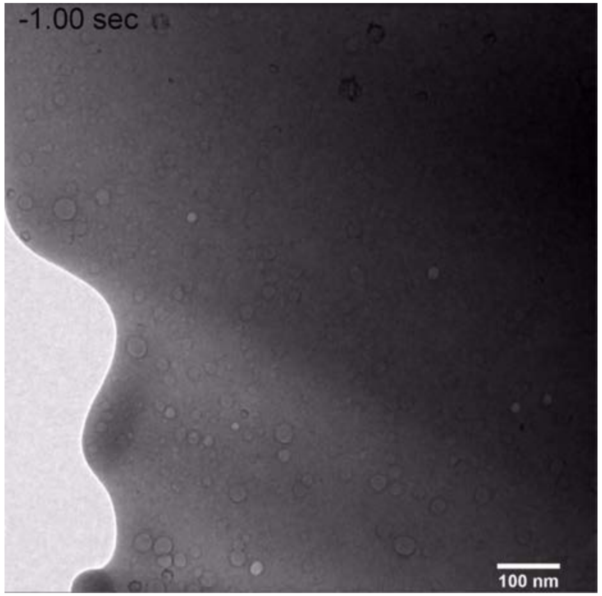
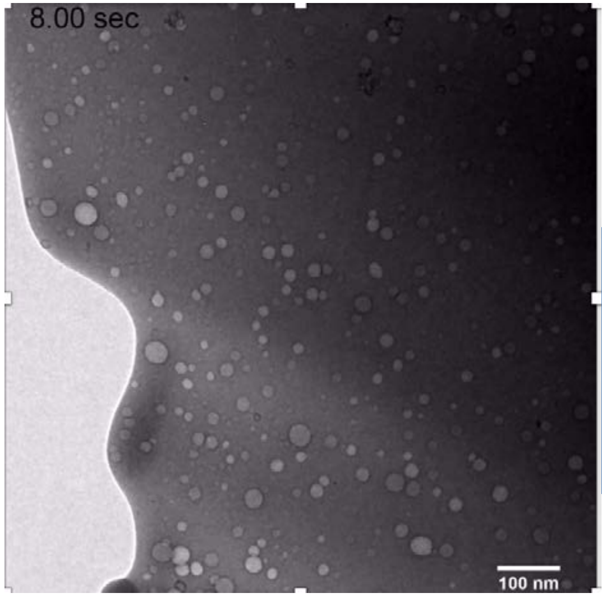
40 keV Xe ion irradiation (1080 K)
(pre-irradiated and saturated at 300 K already)





- Xe ion energy = 40 keV (@30 degree)
- Range ~ 27 ± 6 nm
- Electron energy =200 keV
- Flux ~ $1.3\text{x}10^{13}$ $\text{ions.cm}^{-2}.\text{s}^{-1}$
- Defocus~ 12.16 µm

Frames from video clip shown during presentation. Cavities "light-up" during irradiation. We believe that the enhanced contrast results from ion impacts removing Xe from cavities

Part of EPSRC funded project by Inamul Mir

Ion-Beam-Induced Bending of Nanowires

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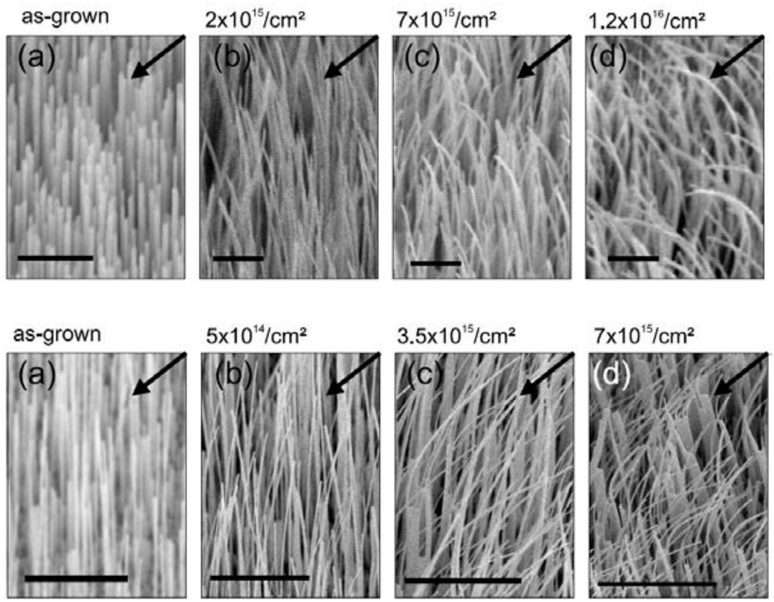
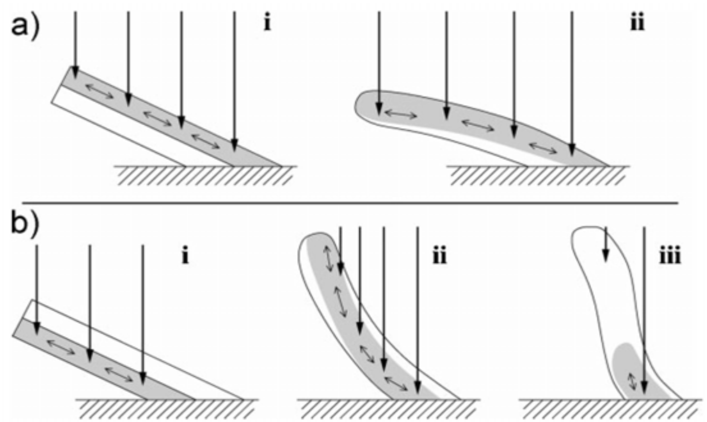
- Phenomenon reported in literature mainly in semiconductors nanowires (Si, Ge, GaAs, ZnO) but also Pt and W
- Various models have been proposed in the literature but the prevailing explanation is volume change due to damage accumulation
- Other mechanisms have been suggested including electronic-energy loss, thermal expansion and sputtering
- A general trend is observed that irradiation conditions with shallower damage profiles lead to bending away from the ion beam and deeper profiles cause bending towards the ion beam.



Nanotechnology **22** (2011) p185307



Small **22** (2009) p2576

Bending of Ge Nanowire under Irradiation with 30 keV Xe Ions

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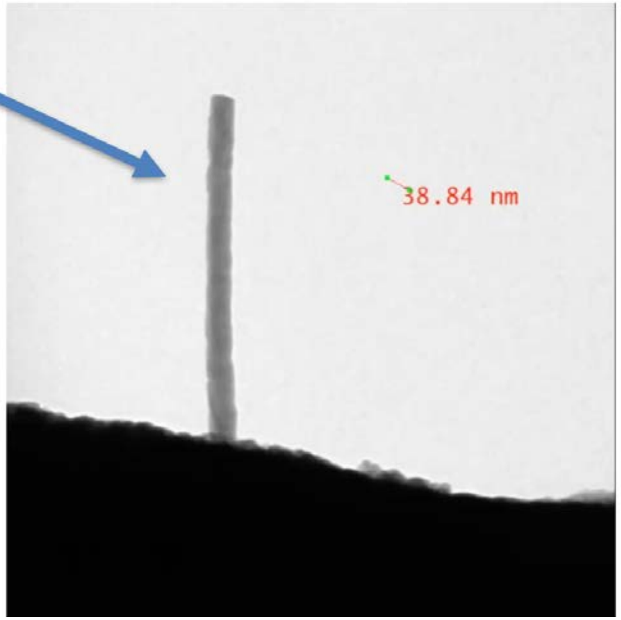
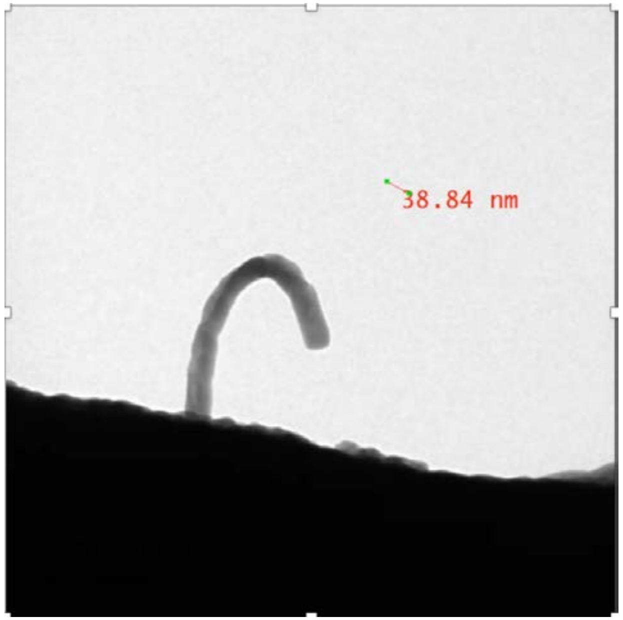
Before irradiation

Projected ion-beam direction.



Frames captured from video shown in presentation

During irradiation

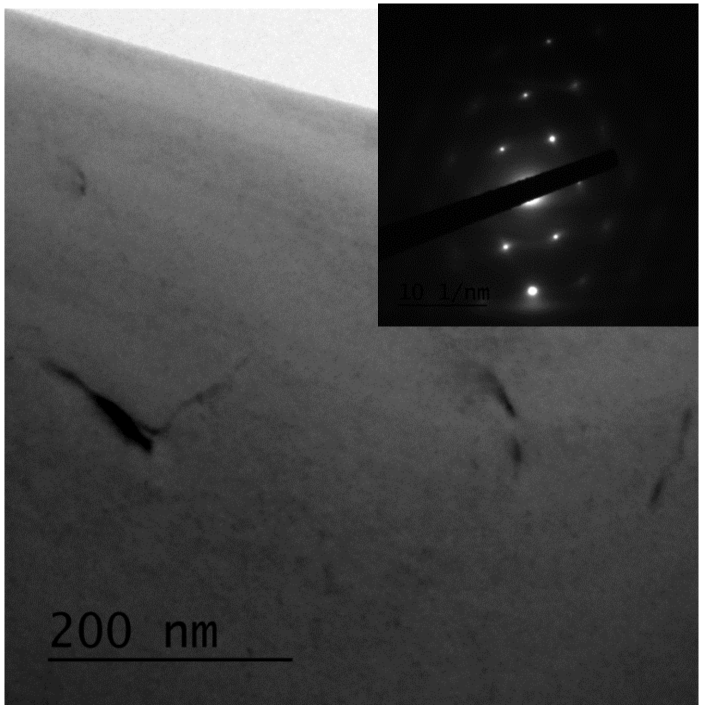


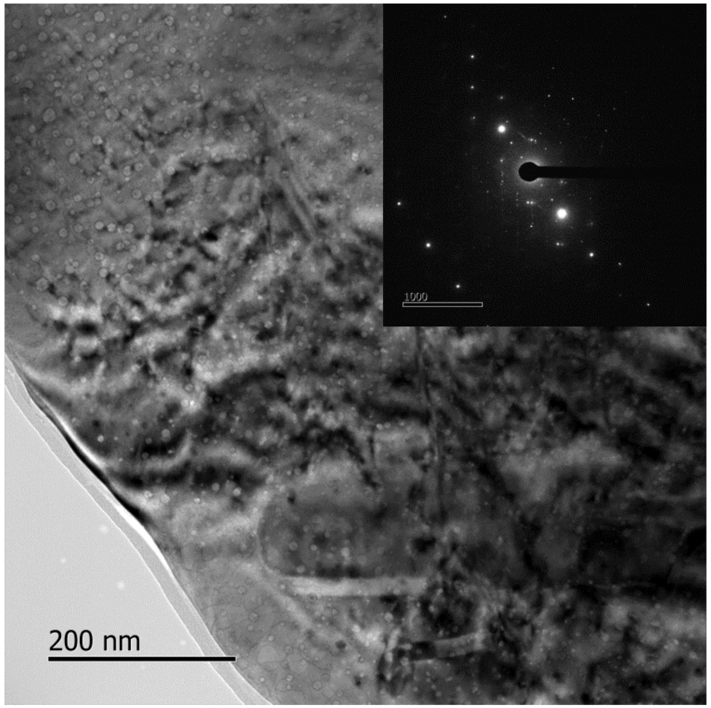
Temperature ≈ 20°C. Flux 1.68 x 10^{13} ions/cm^2/s

NW bending is PhD project work of Imran Hanif (Si) and Osmane Camara (Ge).

He ion irradiation of High Entropy Alloy (HEA)

- HEAs are potential candidates for use in nuclear power plants as fuel clad materials
- They are equiatomic alloys with 4 or more components
- FeCrCoNi HEA irradiated with 6 keV He ions in MIAMI-1 at 500°C to fluence of $7x10^{16}$ ions/cm^2
- Before irradiation material is single crystal and single phase (FCC)
- After irradiation He bubbles present and SADP indicates phase decomposition (polycrystalline and multiple phases)





He ion irradiation of High Entropy Alloy (HEA)

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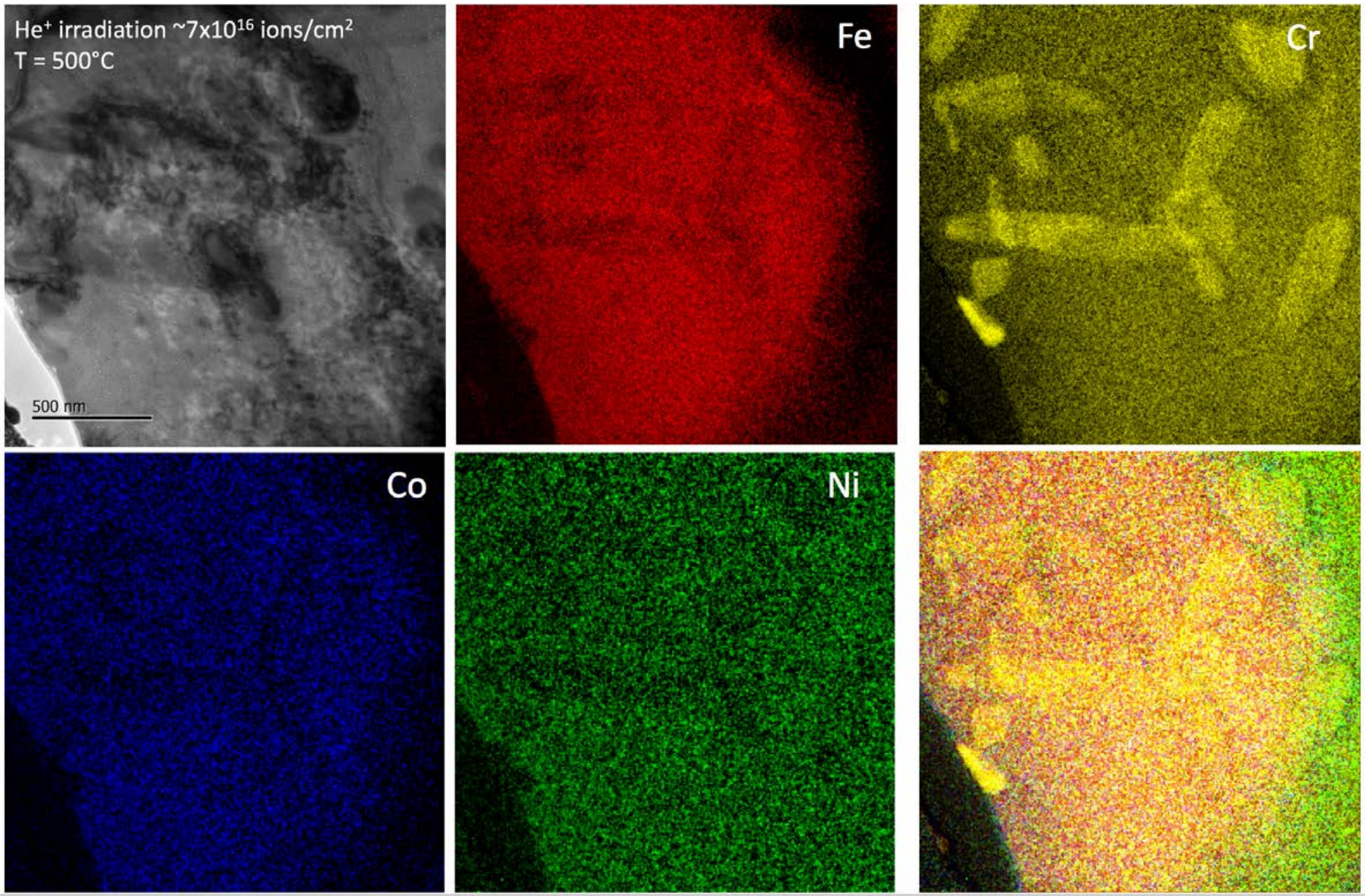
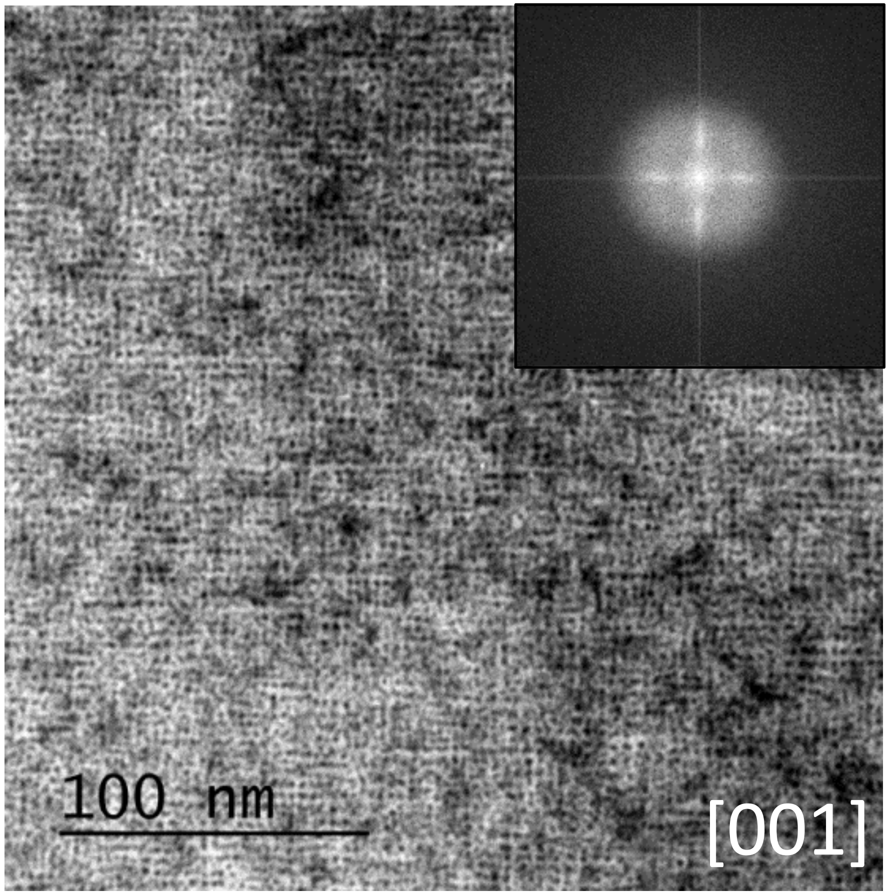
- Energy Filtered TEM (EFTEM) on MIAMI-2 used for further analysis of phase decomposition of HEA.
- Showed large amounts of Fe depletion and Cr precipitation after irradiation

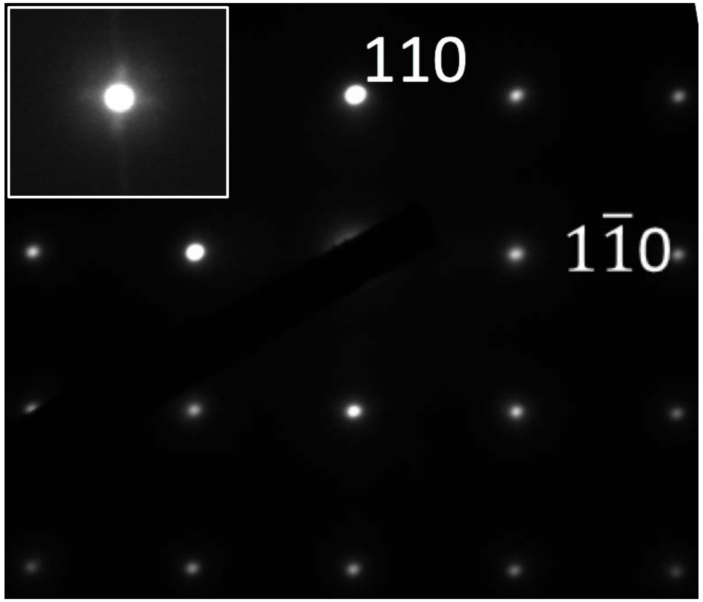


Research by Rob Harrison & Matt Tunes

He Bubble Superlattices in W

W: 15 keV He^{+}, T = 500°C. He appm/DPA ratio ~40,000





Lattice constant of helium bubble lattice,
a = 4.6 +/- 0.2 nm
Bubble density = 2.3 x 10^{19} He bubbles/cm^3

Part of EPSRC-funded project by Rob Harrison

Xe Ion Impacts on Au Nanorods

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PRL **111,** 065504 (2013) PHYSICAL REVIEW LETTERS week ending 9 AUGUST 2013

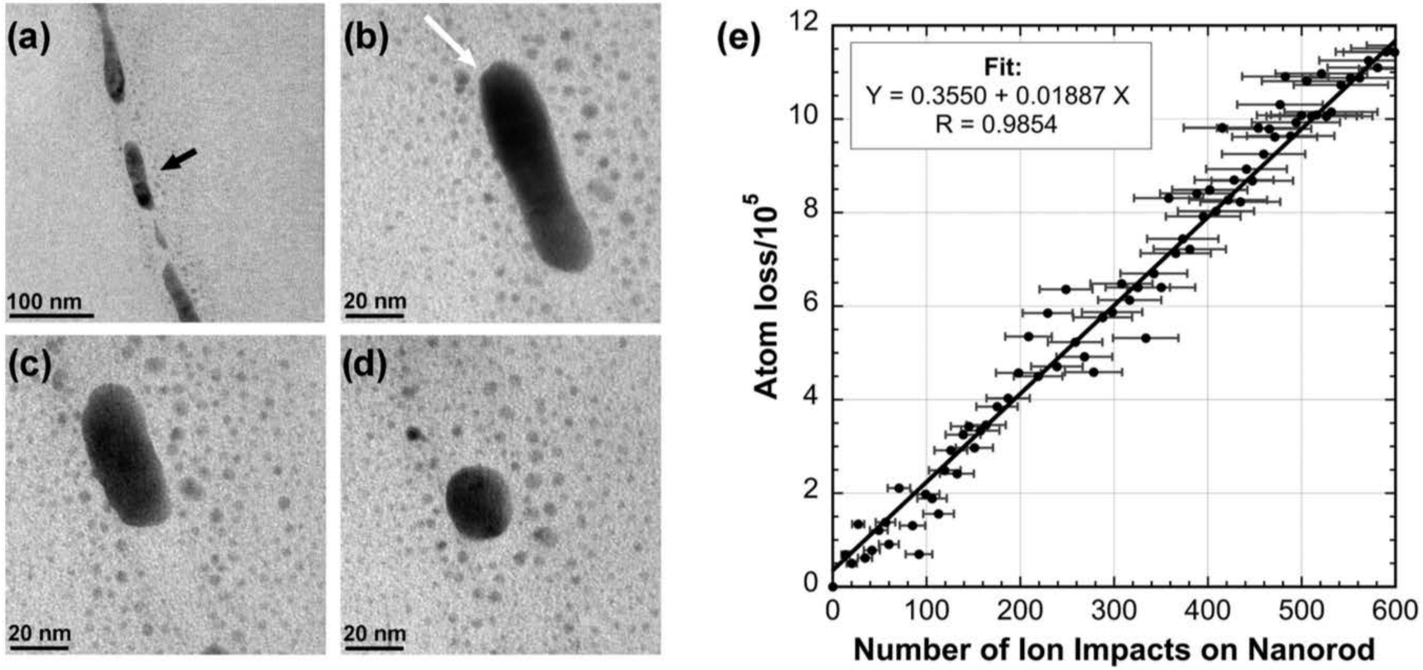
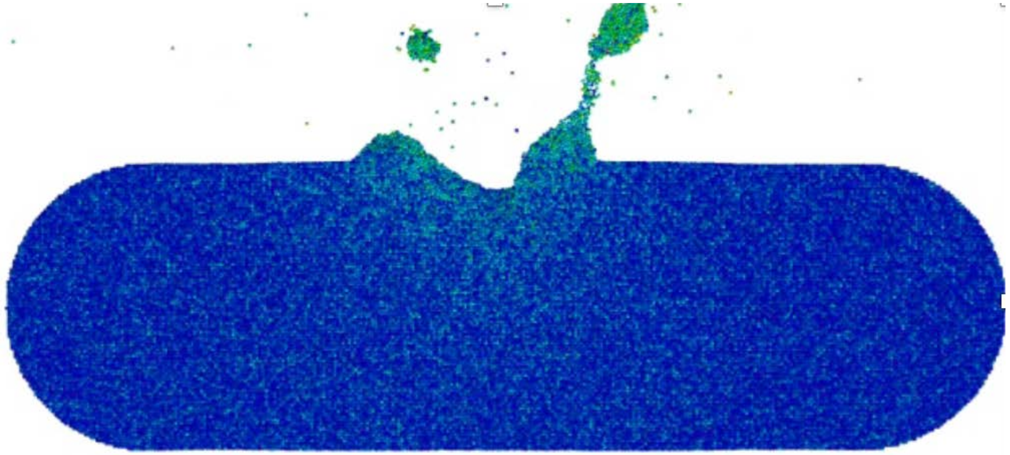


FIG. 1. Changes to Au nanowire due to irradiation with 80 keV Xe ions. (a) Segmentation due to "necking" and breaking of nanowire at grain boundaries following irradiation to a fluence of 2.1×10^{14} ions cm^{-2}. (b) Nanorod at starting point for volume measurements—white arrow indicates projected direction of the ion beam which was incident at 60° to the specimen plane. (c) Nanorod following irradiation to (additional) fluence of 1.6×10^{13} ions cm^{-2} ($\approx$ 227 impacts on nanorod). (d) Nanorod following irradiation to (additional) fluence of 5.5×10^{13} ions cm^{-2} ($\approx$ 316 additional impacts on nanorod). All are bright field TEM images. (e) Plot of atom loss versus ion impacts for Au nanorod shown in panels (b) to (d).

G. Greaves, J. A. Hinks, P. Busby, N. J. Mellors, A. Ilinov, A. Kuronen, K. Nordlund, and S. E. Donnelly, *Phys. Rev. Lett*. **111** (2013) 065504

MD Simulation of Xe Ion Impacts on Au Nanorods



"Explosive" cluster emission is additional to ballistic and thermal components of sputtering yield

Sputtering yield from impact shown: S=2560 atoms/ion

About 100 atoms sputtered due to "normal" ballistic and thermal processes – the rest due to cluster emission.

MD simulations by Kai Nordlund's group, University of Helsinki

CONCLUSIONS

Studies of radiation damage carried out in-situ in a TEM can yield significant insights into dynamic process that are difficult to obtain by other means. Areas of particular relevance include the formation and evolution of extended defects and overall morphological changes to nanostructures (shrinkage, bending).

With the installation of MIAMI-2 we add the following features:

- Dual ion beams and energies of up to 350 keV (singly charged);
- Electron Energy Loss Spectroscopy and Energy-Filtered TEM;
- Energy Dispersive X-Ray Spectroscopy;
- Environmental TEM
- Temperatures to 1300°C

This gives us a greatly enhanced capability and, in particular, the addition of EDS and EFTEM enables us to follow radiation-induced phase-changes, and changes to the chemical composition and electronic properties of materials.

Electron Microscopy and Materials Analysis Research Group

Prof Steve Donnelly, Dr Jonathan Hinks, Dr Vladimir Vishnyakov,
Prof John Colligon (emeritus), Dr Graeme Greaves, Dr Rob Harrison,
Dr Emily Aradi, Dr Inamul Mir

PhD Students

Imran Hanif, Osmane Camara, Matheus Tunes, Ewan Vanderboon,

MIAMI
Microscopes and Ion Accelerators for Materials Investigations

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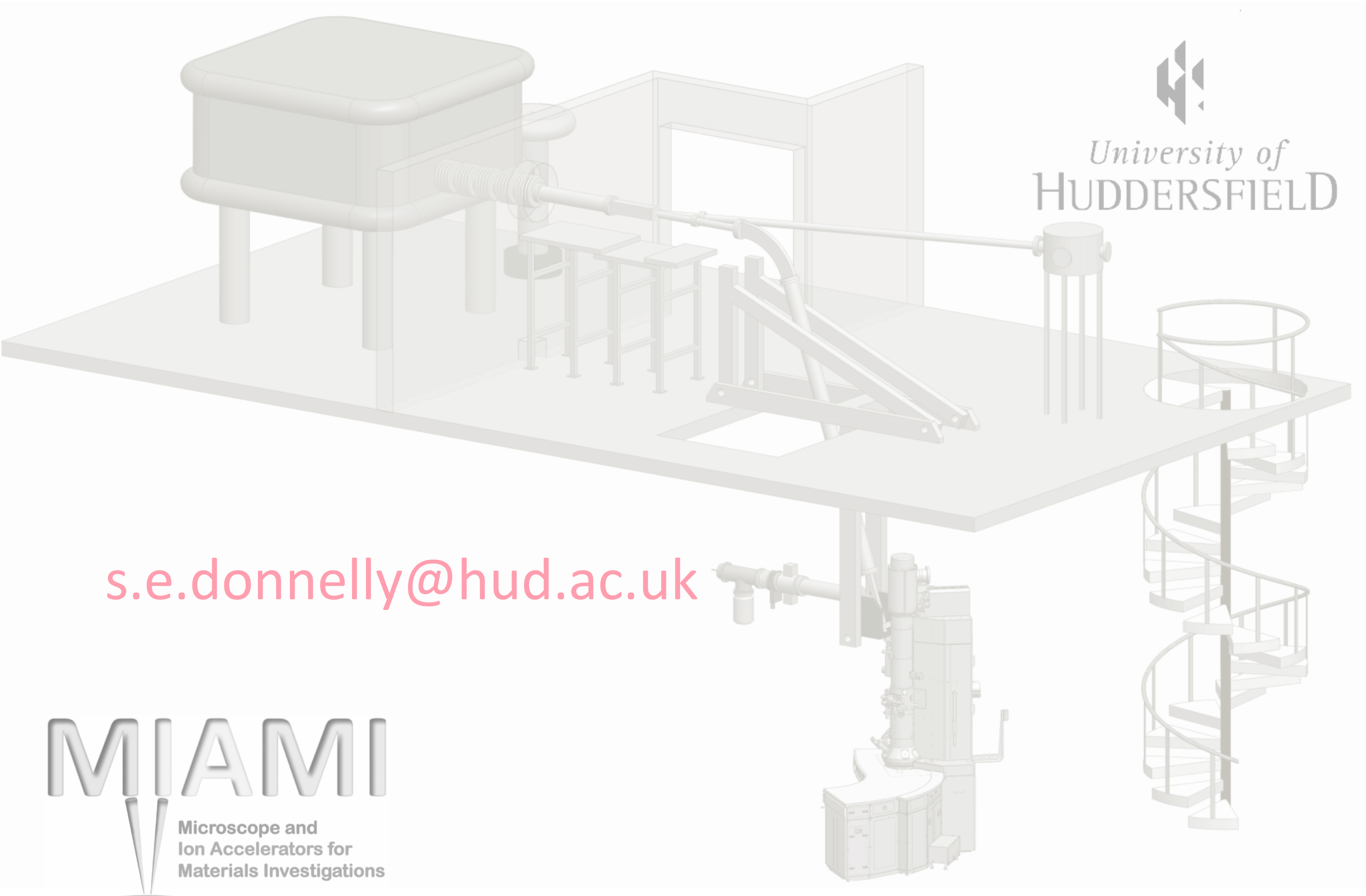
Thank you
for
your attention



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MIAMI

Microscope and
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