IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

X-ray absorption spectroscopy characterisation of irradiation damage in REBCO coated conductors

S. Speller, J. Lewis, K. Adams, J. Tufnail, R. Nicholls, C. Grovenor, S. Diaz-Moreno, F. Mosselmans, W. Iliffe

OxfordMaterials

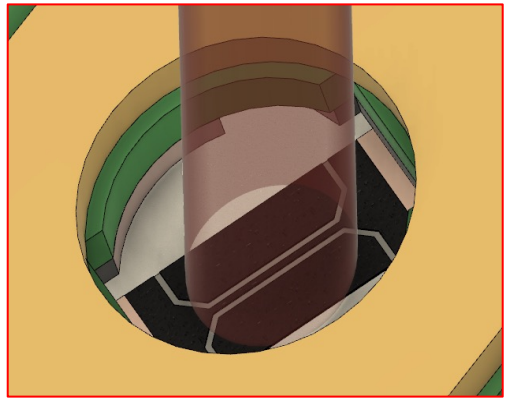
IREF, Arona, Nov 2023

UNIVERSITY OF OXFORD

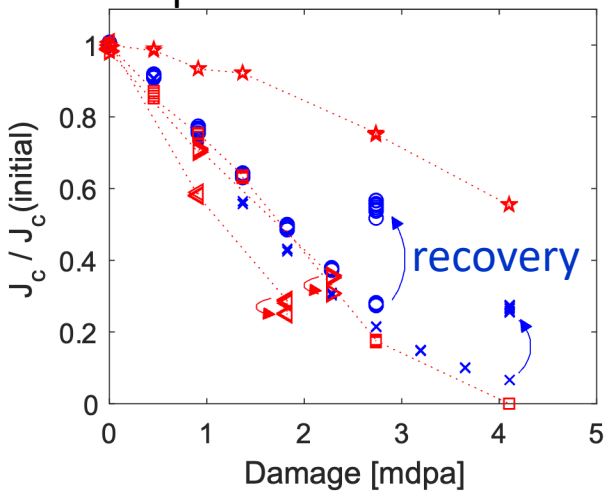
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Oxford University research on radiation damage of HTS

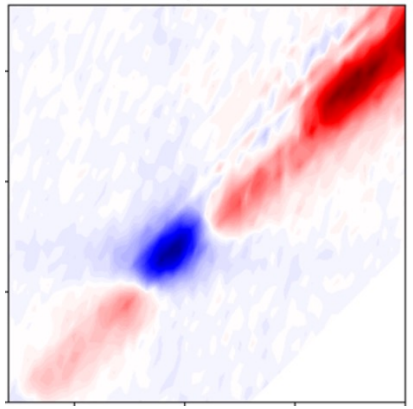
In situ cold irradiation experiments

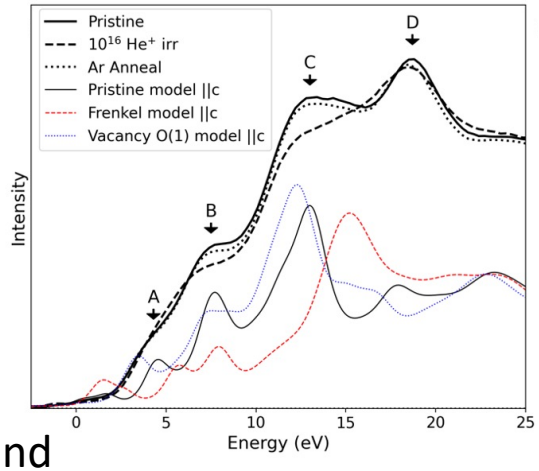


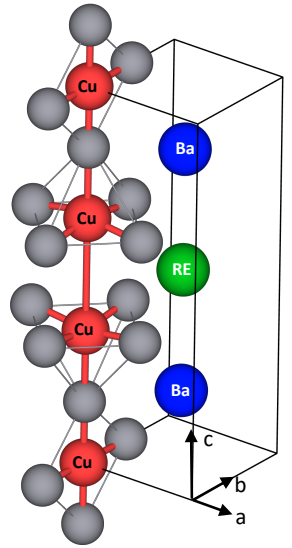
Surrey Ion Beam Centre



X-ray absorption spectroscopy and DFT modelling

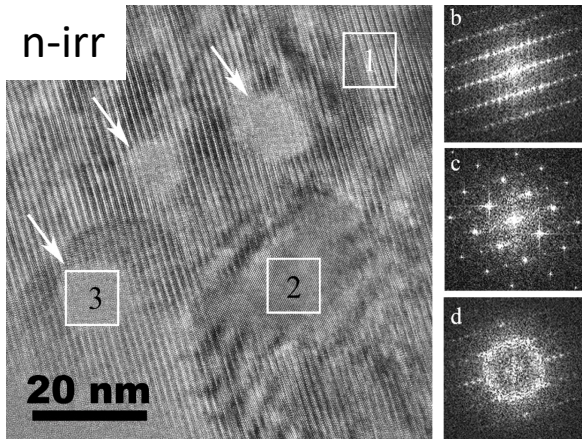


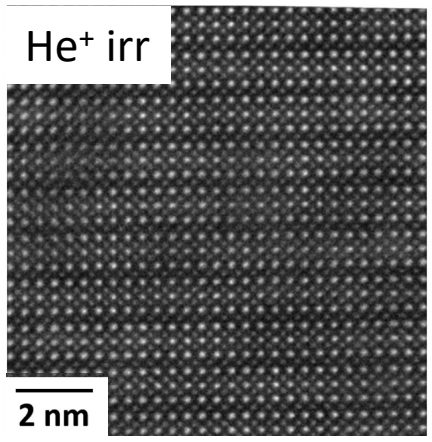


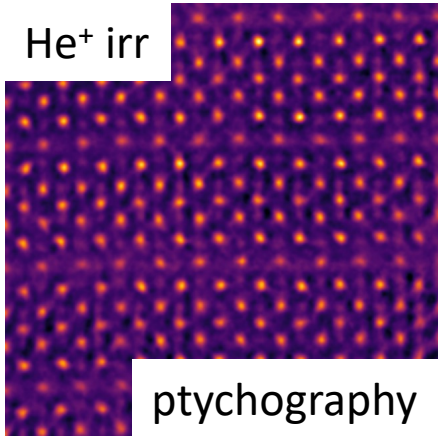


I20 beamline at Diamond

Transmission electron microscopy in Oxford materials and at ePSIC







UNIVERSITY OF OXFORD

DEPARTMENT OF MATERIALS

diamond

UNIVERSITY OF SURREY Ion Beam Centre

UK Atomic Energy Authority

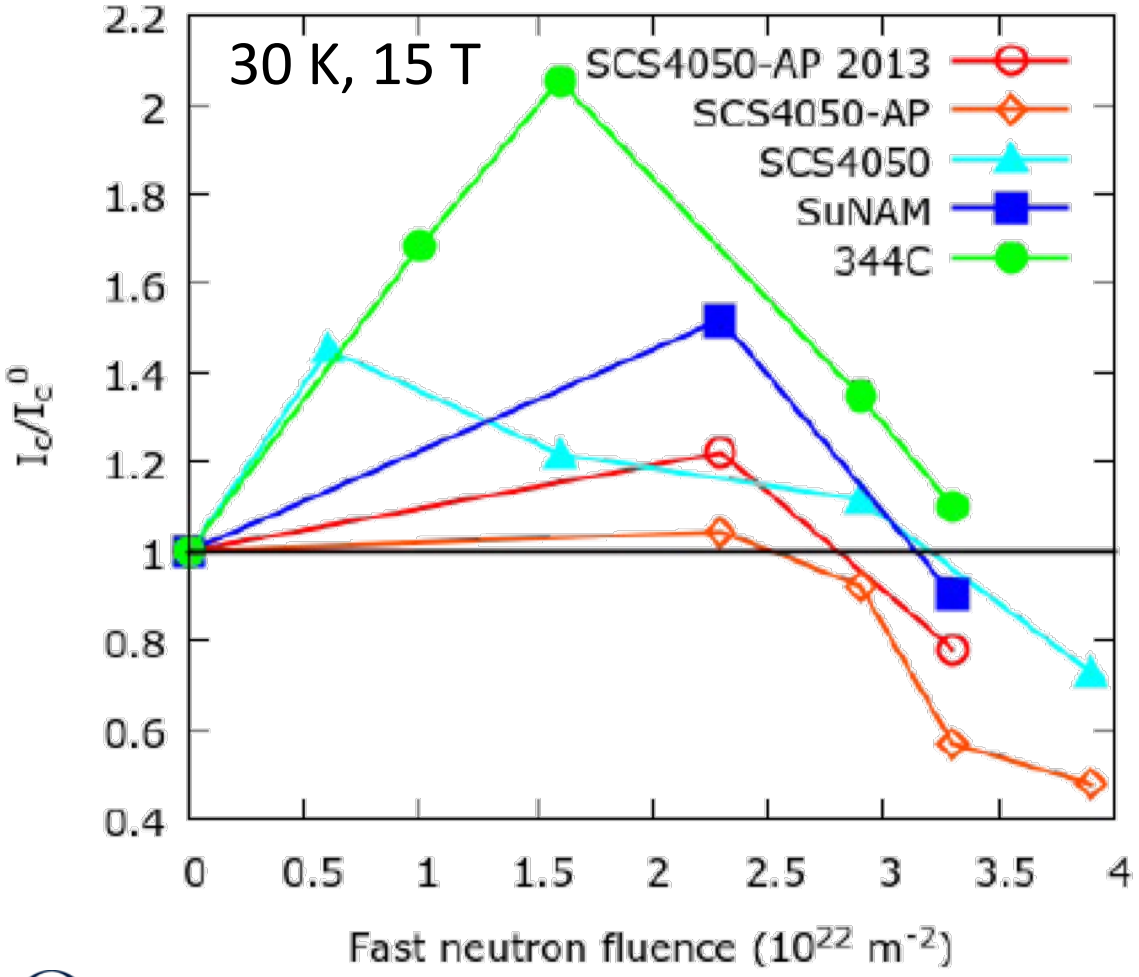
TU WIEN

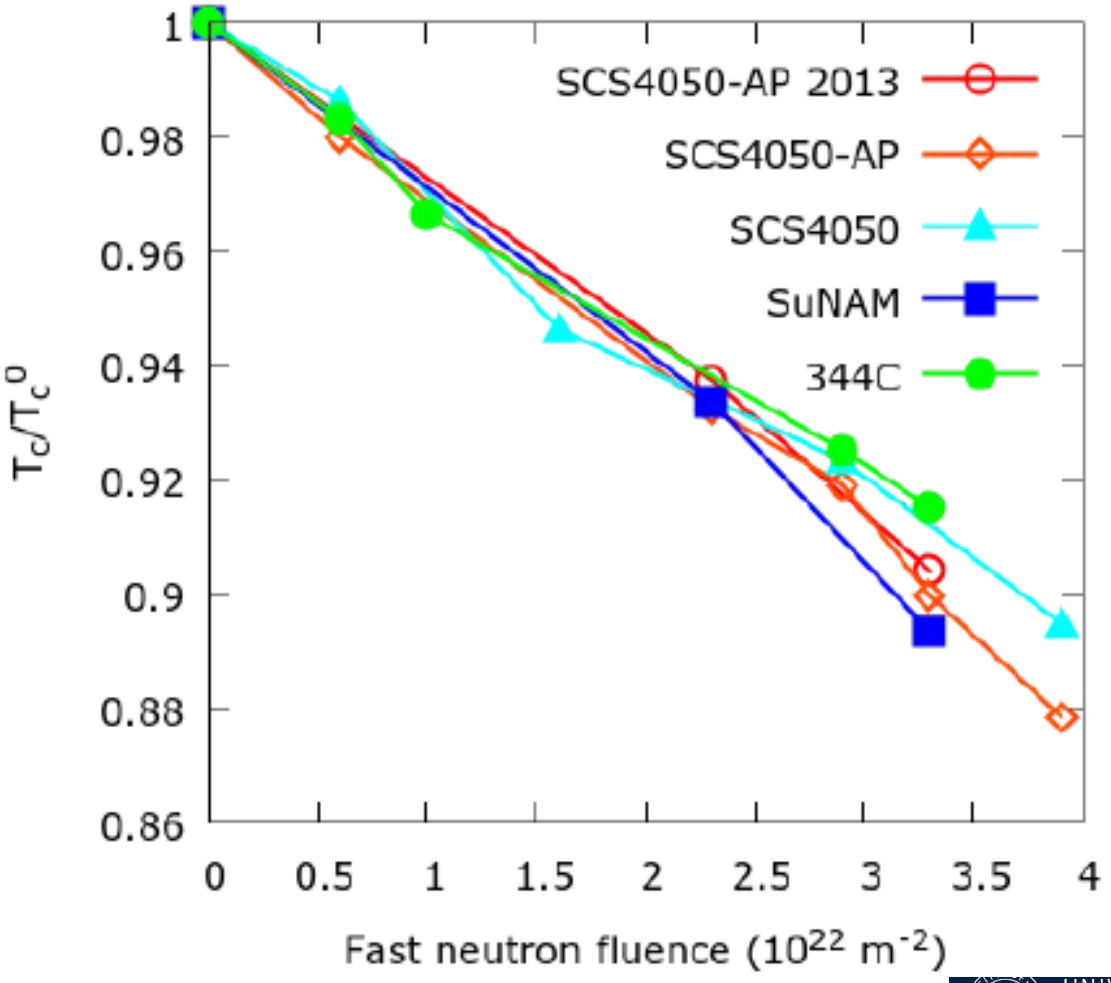
ePSIC electron Physical Sciences Imaging Centre

Tokamak Energy

Effect of n-irr on superconducting properties

Superpower SCS4050-AP coated conductor irradiated in TRIGA reactor (Vienna) to a neutron fluence of **3.3×10^{22} m^{-2}**



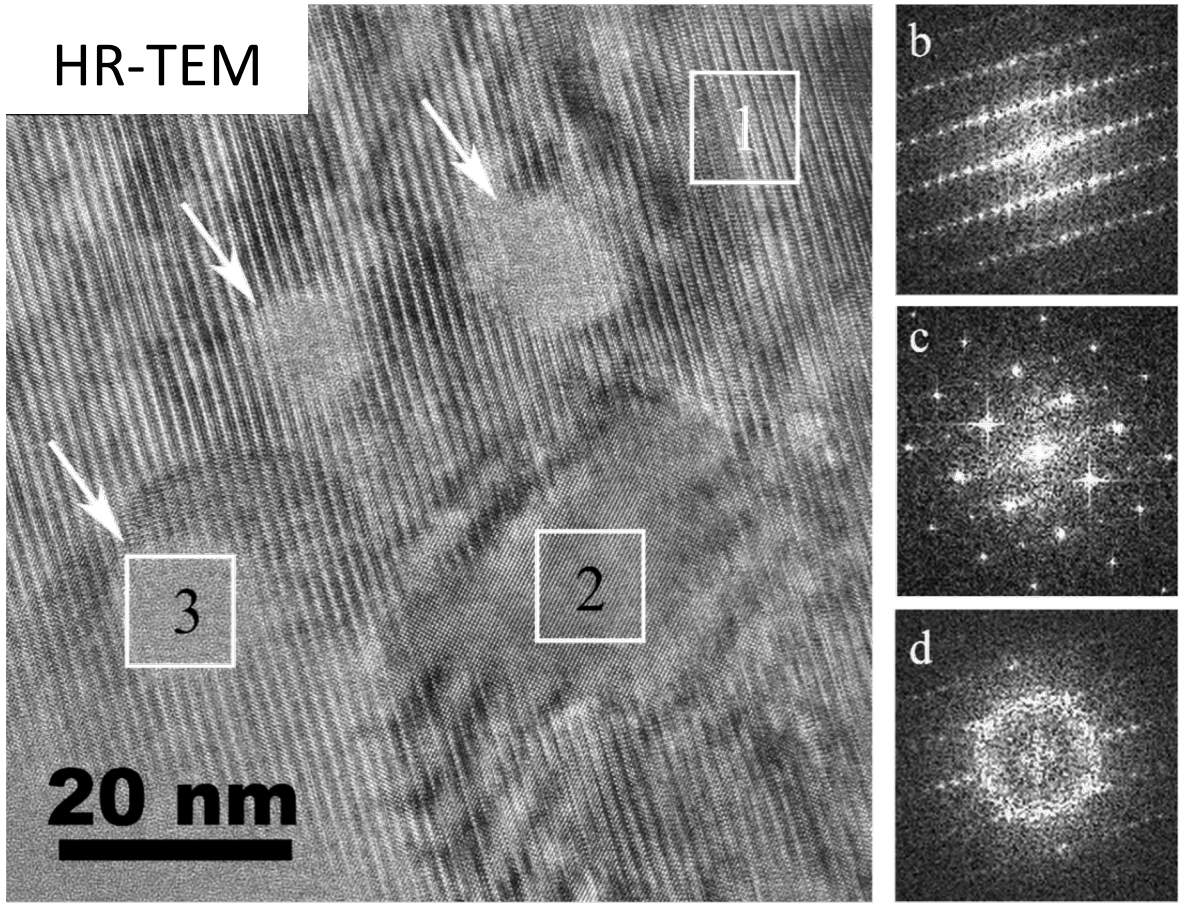


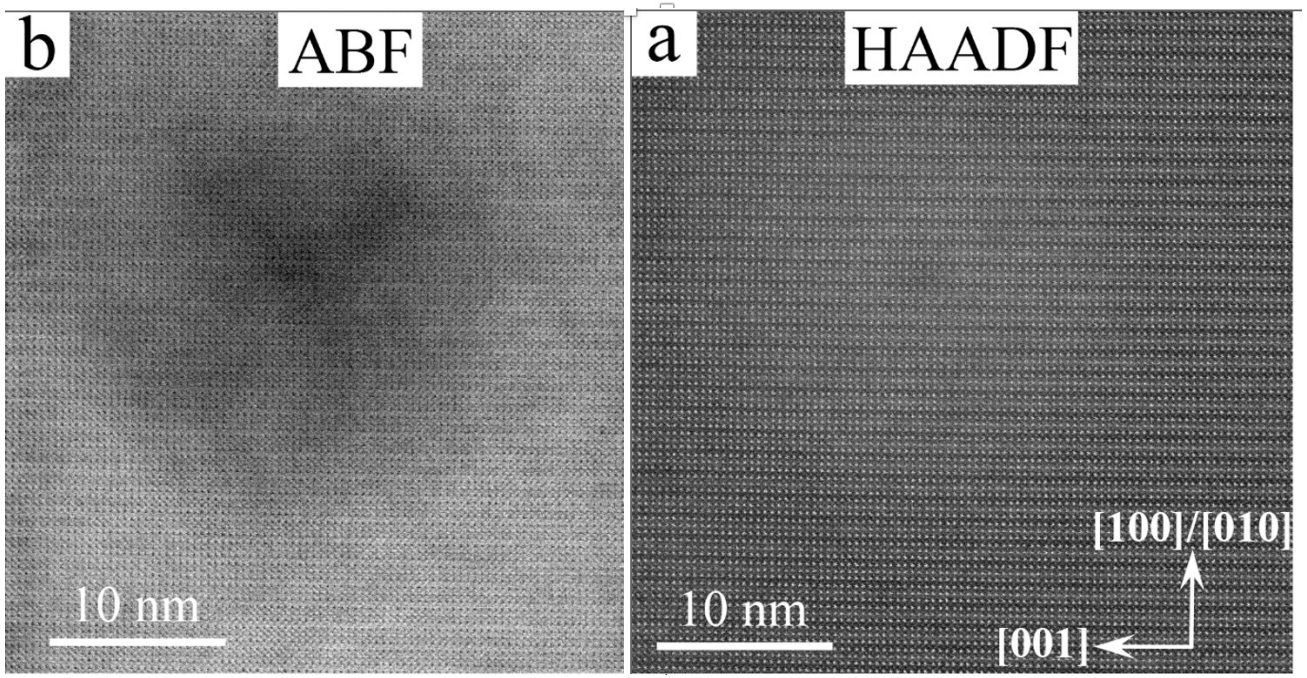
[Fischer et al (2018) *SUST* 31 044006]

IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Damage cascade damage in neutron irradiated sample

Superpower SCS4050-AP coated conductor irradiated in TRIGA reactor (Vienna) to a neutron fluence of **3.3×10^{22} m^{-2}**



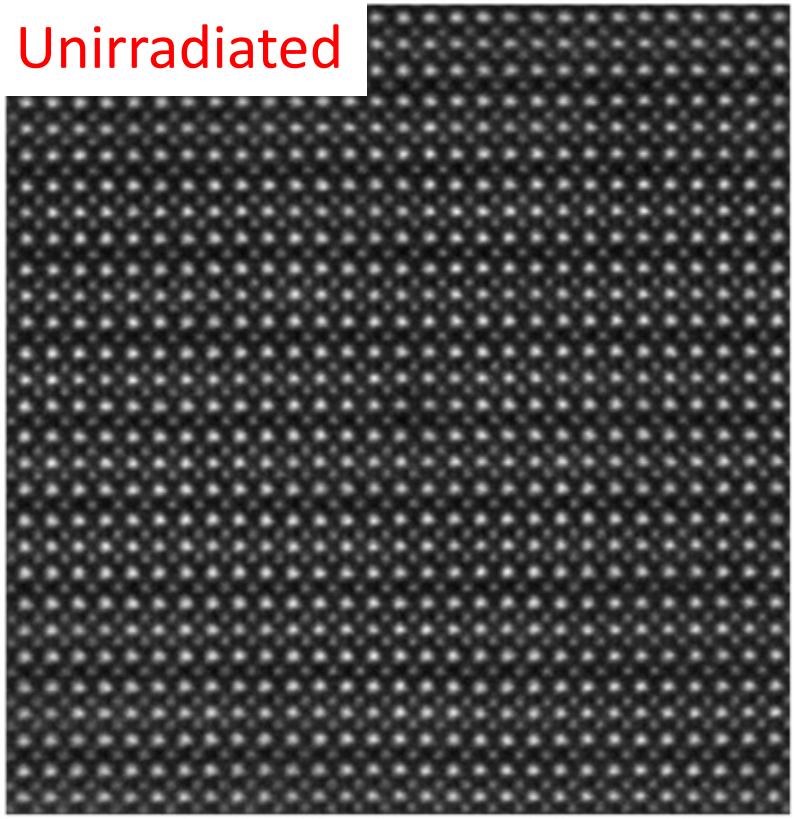


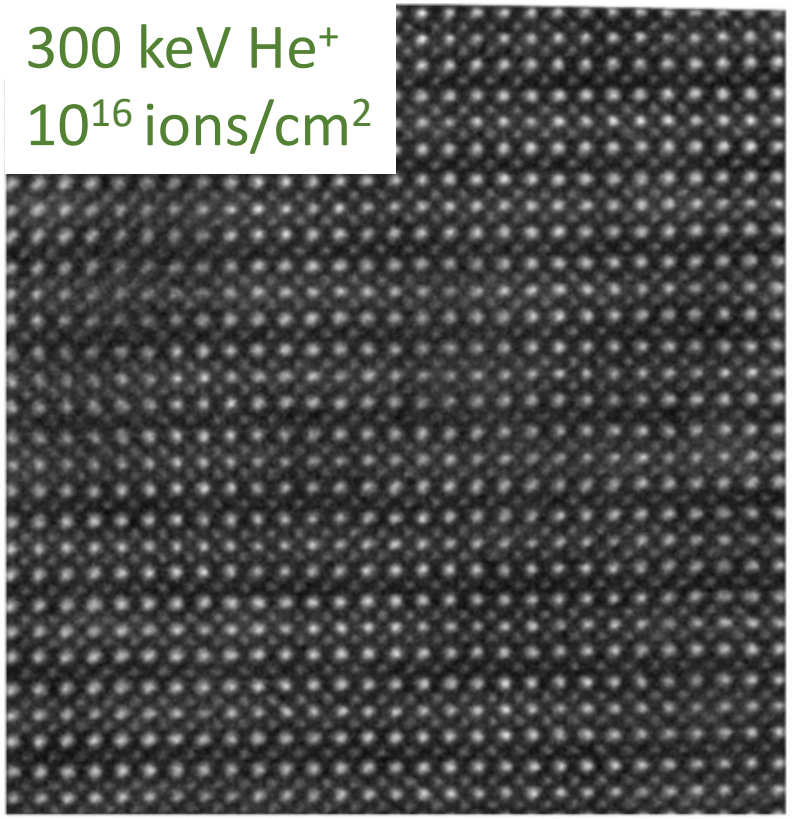
Scanning transmission electron microscopy (STEM): ABF = bright field, HAADF = dark field)

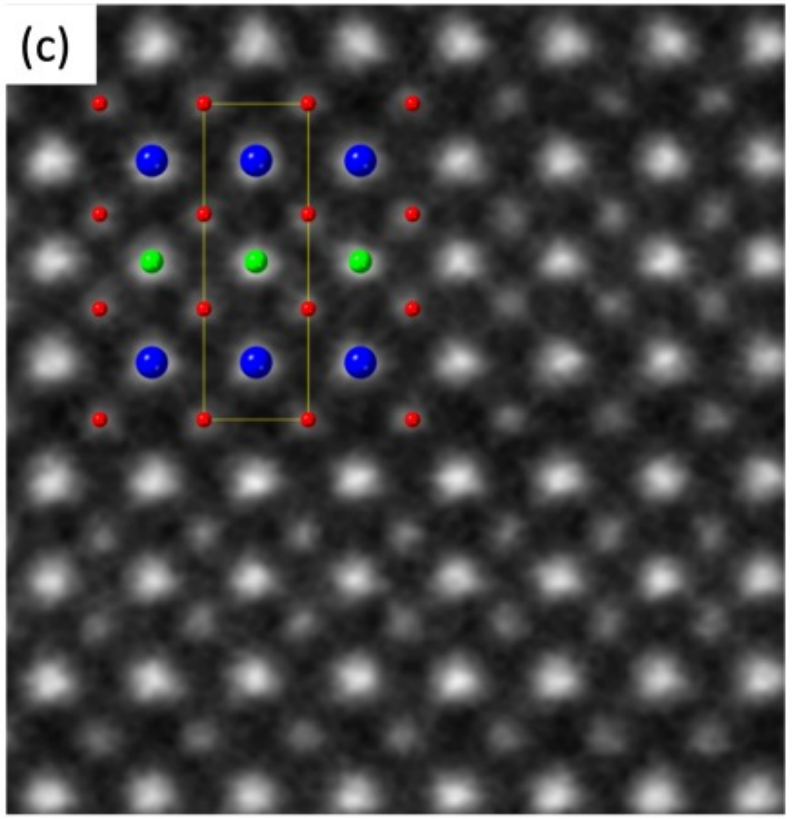
[Linden et al, *Journal of Microscopy*, 0 1– 10. https://doi.org/10.1111/jmi.13078]

What causes T_c degradation?

- Atomic resolution HAADF STEM images of SuperPower 4050 AP – heavy cations visible

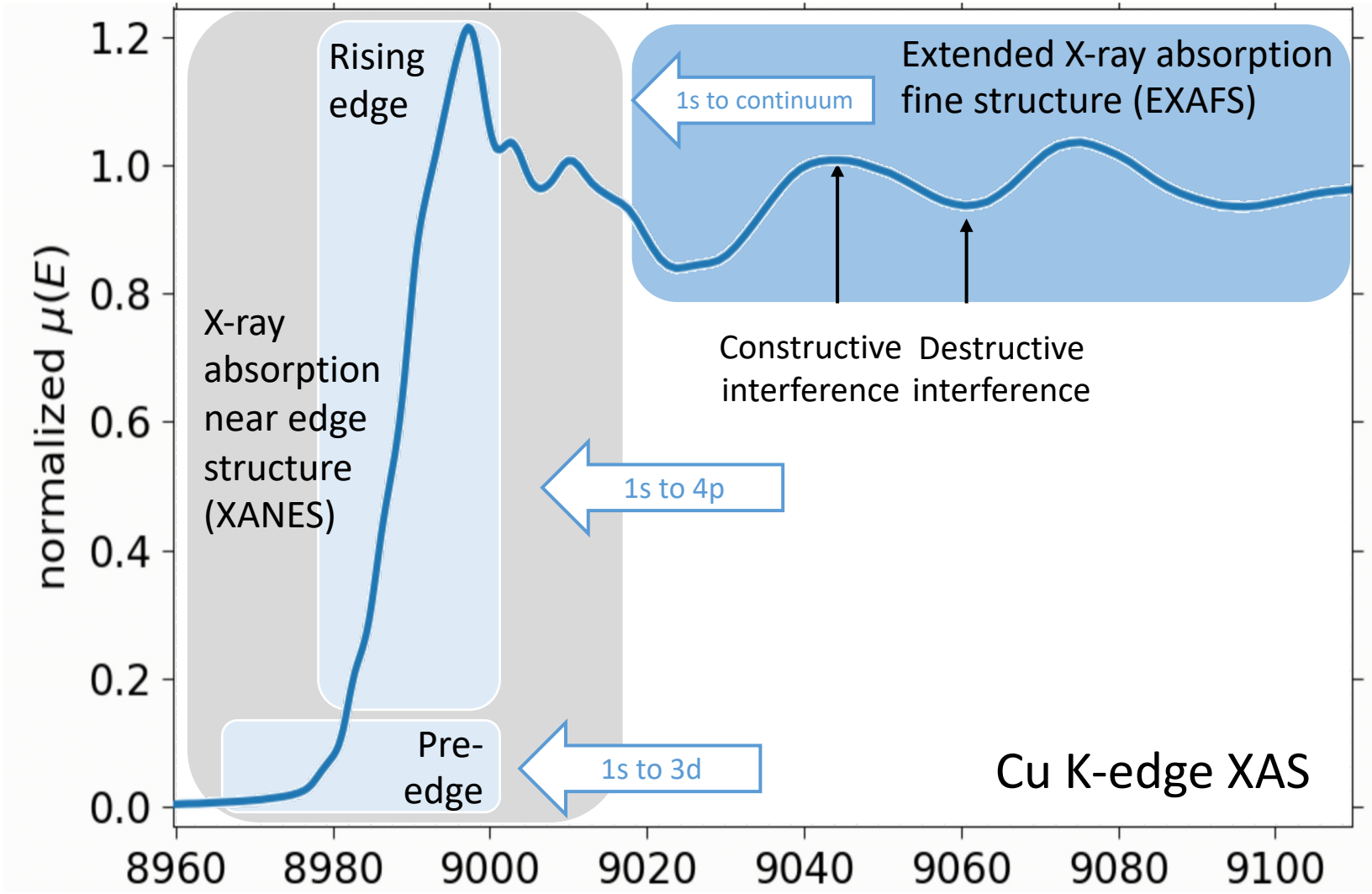




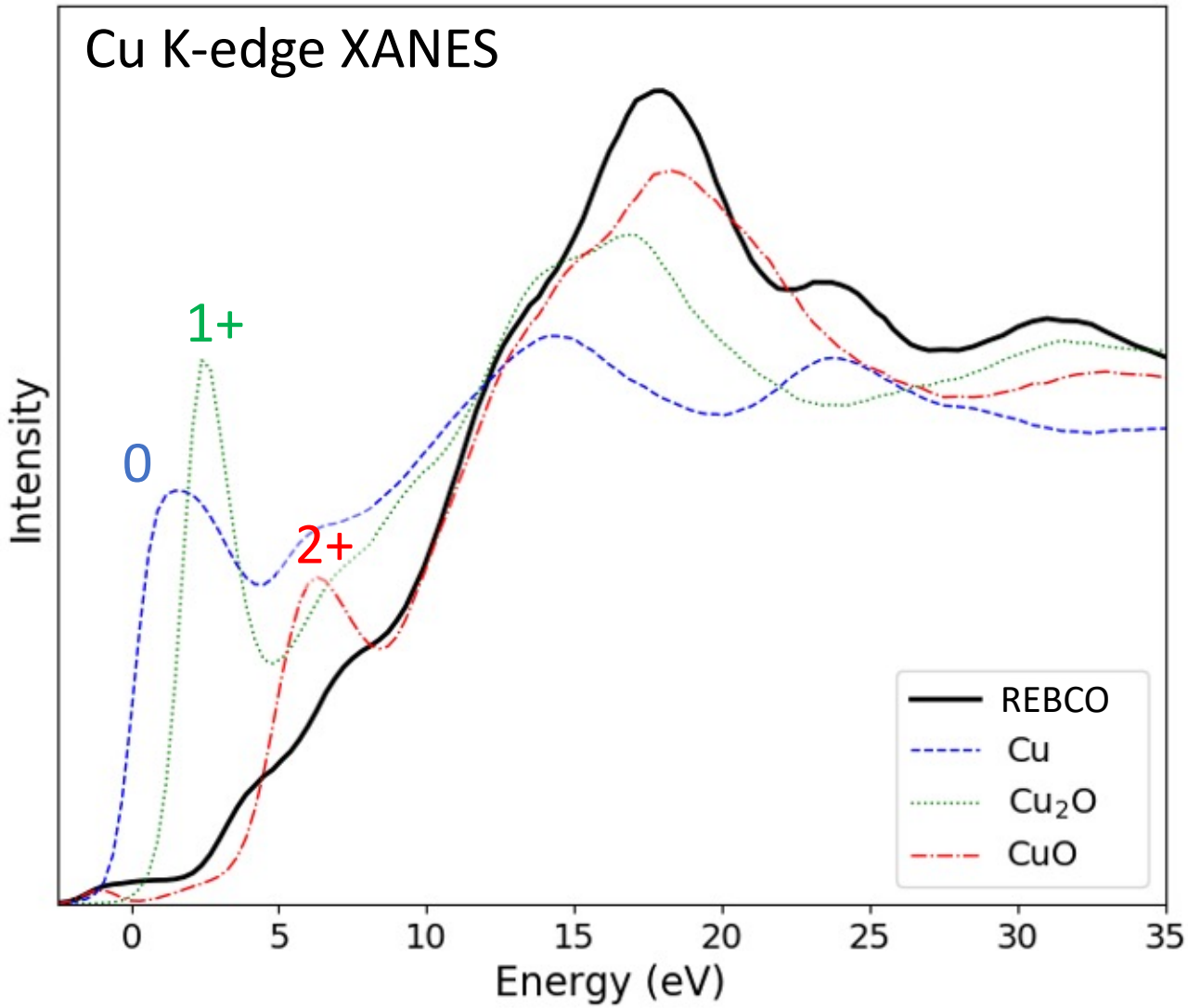


Nicholls et al, *Communications Materials* (2022) **3**:52
Linden et al, *Journal of Microscopy*, 0 1– 10. https://doi.org/10.1111/jmi.13078

X-ray absorption spectroscopy



XANES: effect of oxidation state

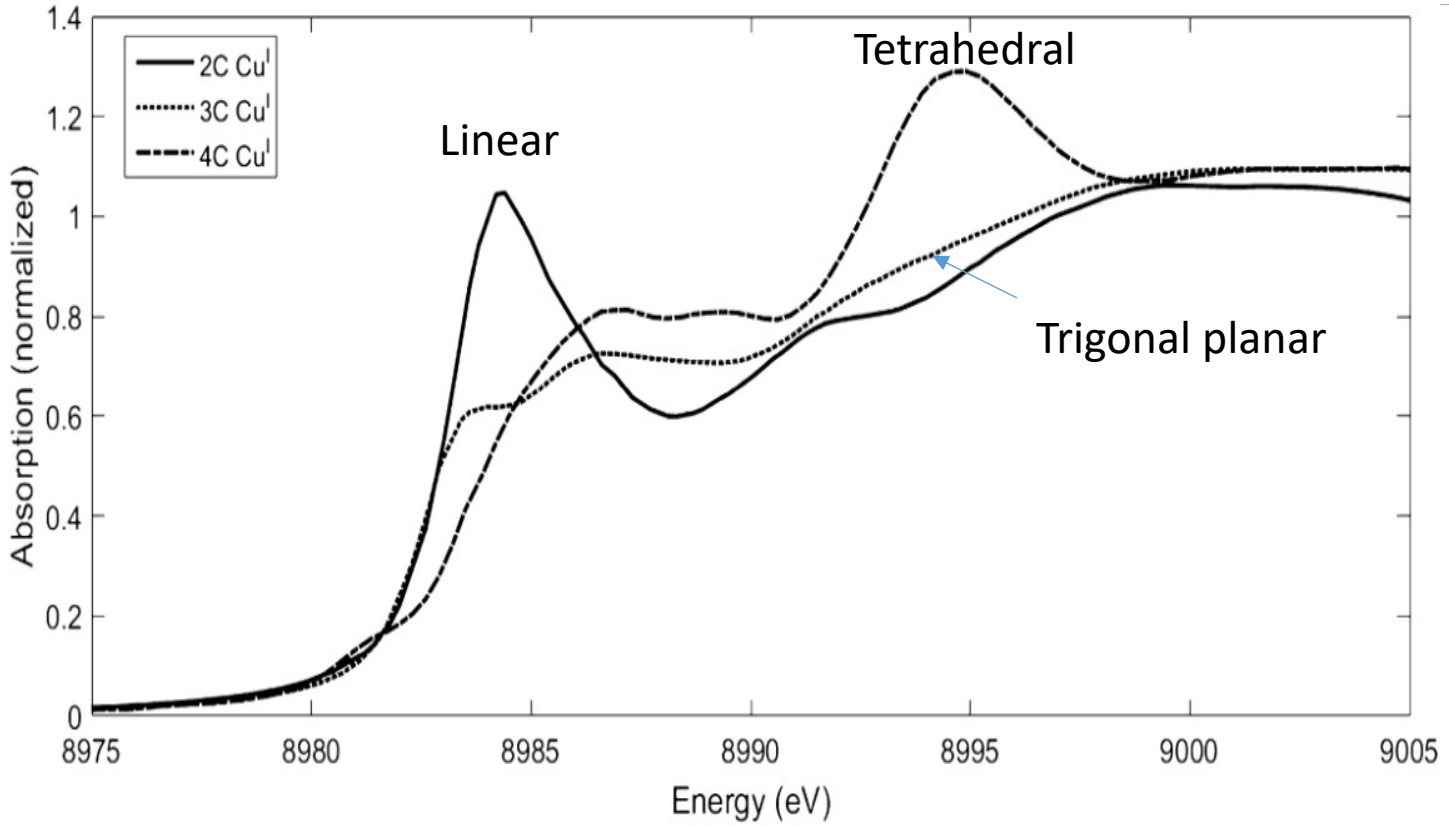


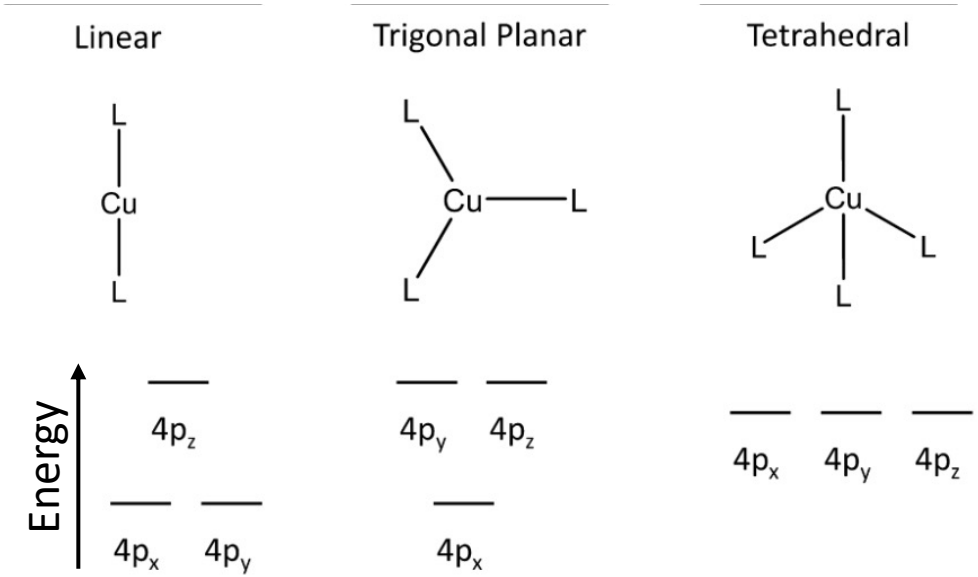
Data obtained at I20-scanning Diamond Light Source

Absorption edge shifts to higher energy as oxidation state increases

XANES: effect of coordination

Effect of bonding environment on Cu^1 complexes





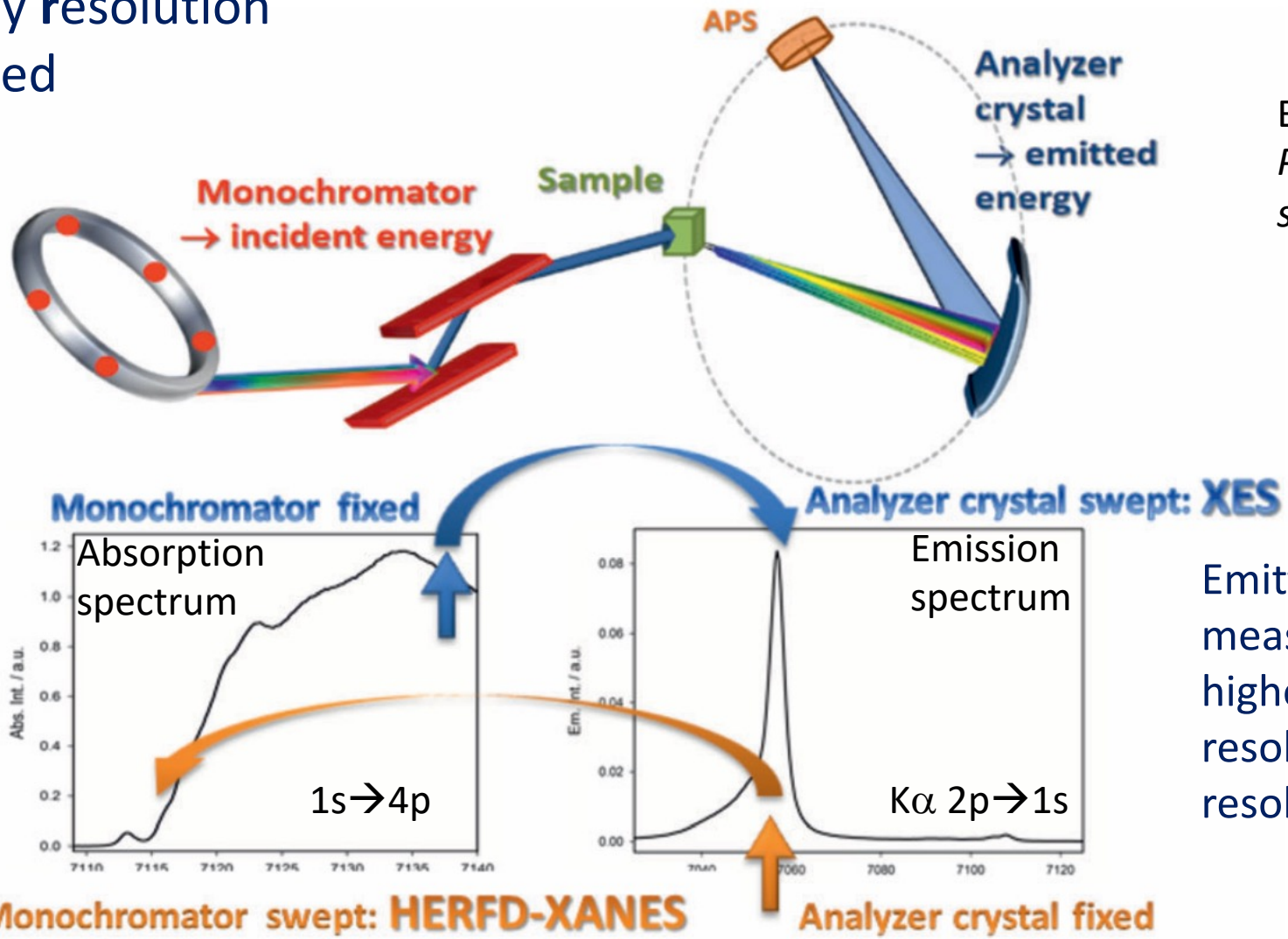
High energy resolution needed to analyse complicated spectra of complicated structures like REBCO.

M.L. Baker et al. *Coordination Chemistry Reviews* 345 (2017) 182–208

Cu K-edge HERFD-XANES

HERFD = **h**igh **e**nergy **r**esolution **f**luorescence **d**etected

Hard X-rays probe entire thickness of REBCO layer



Bauer et al *Phys.Chem.Chem.Phys.*, 2014, 16, 13827

K-edge absorption spectrum energy resolution usually limited by core hole lifetime broadening

Emitted beam can be measured with much higher energy resolution → increases resolution of XANES

OxfordMaterials

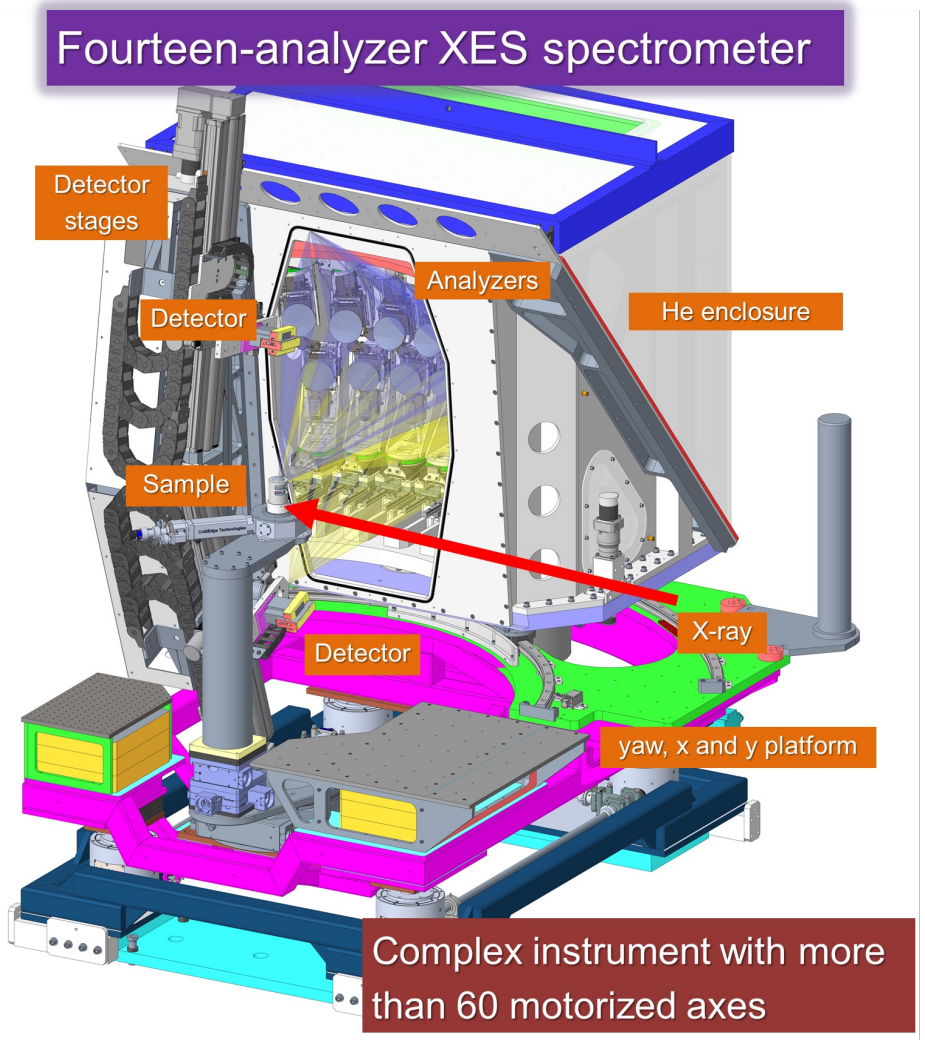
UNIVERSITY OF OXFORD

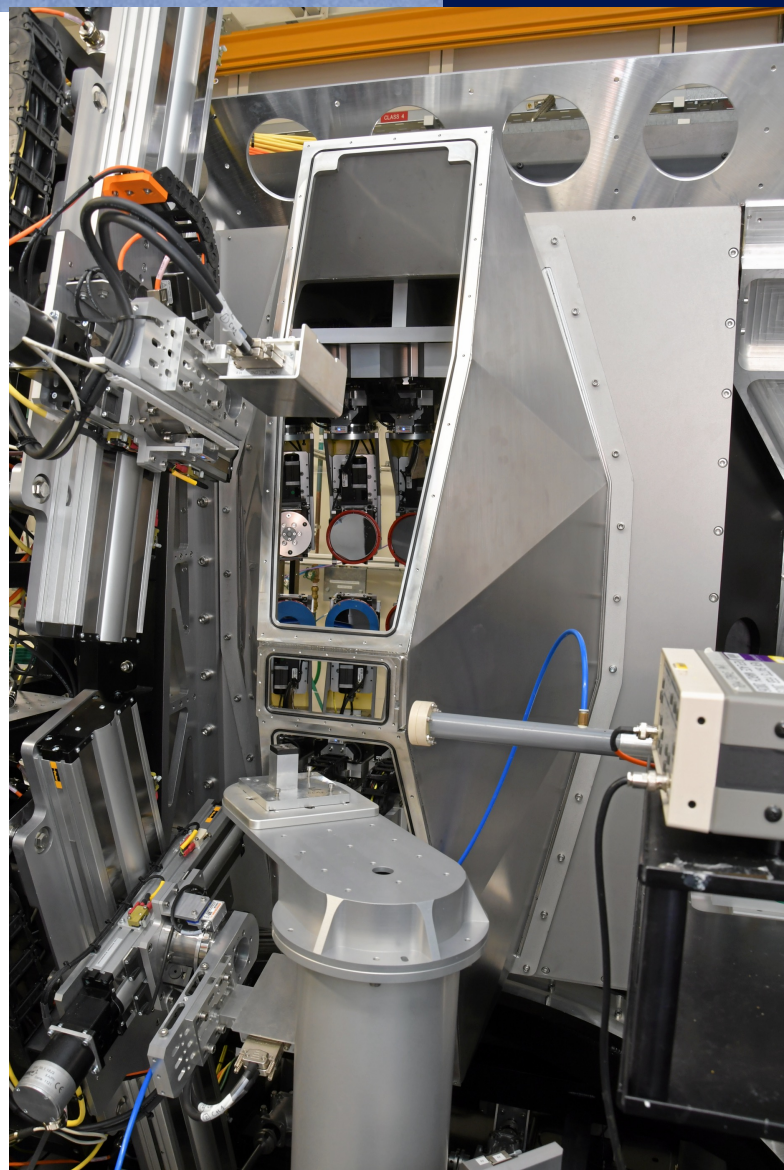
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

HERFD-XANES at Diamond Light Source

I20-scanning beamline

- HERFD, XES, RXES, V2C
- New analyser with 14 crystals
- 2 rows of crystals allows 2-colour measurements
- Energy resolution ~1 eV
- Energy range 5-18 keV





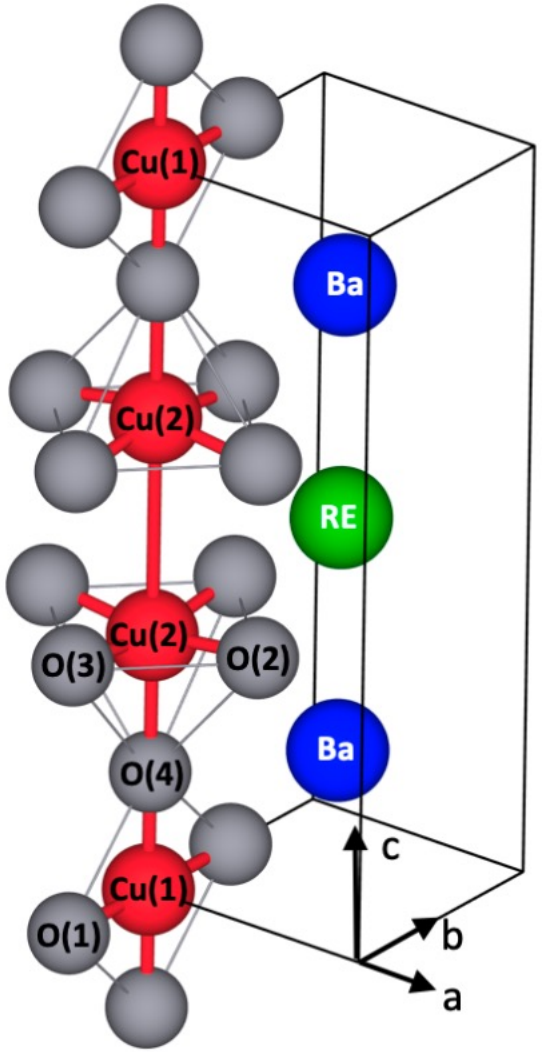
OxfordMaterials

Cu sites in REBCO

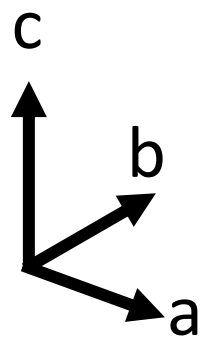
REBCO has orthorhombic unit cell with

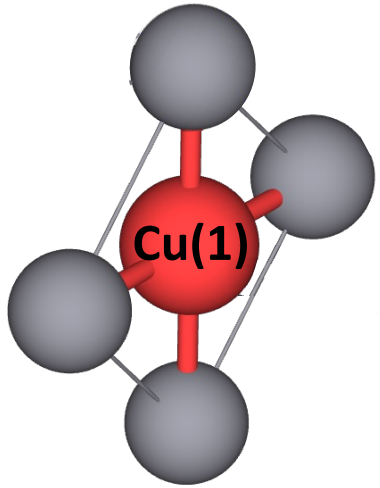
$a \approx b$

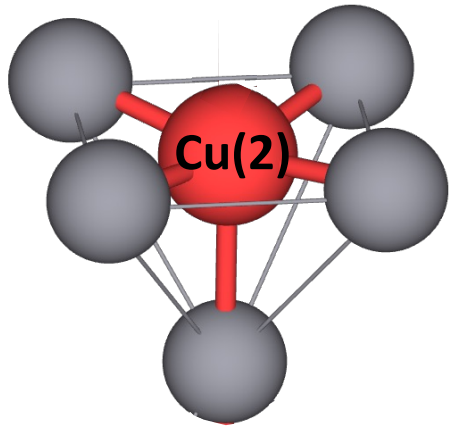
$c \approx 3a$



Two copper sites in REBCO structure







Chain site

- Cu(1) is 4-fold coordinated by O
- 2 Cu-O bonds along b
- 2 Cu-O bonds along c

Plane site

- Cu(2) is 5-fold coordinated by O
- 2 Cu-O bonds along a
- 2 Cu-O bonds along b
- 2 Cu-O bonds along c

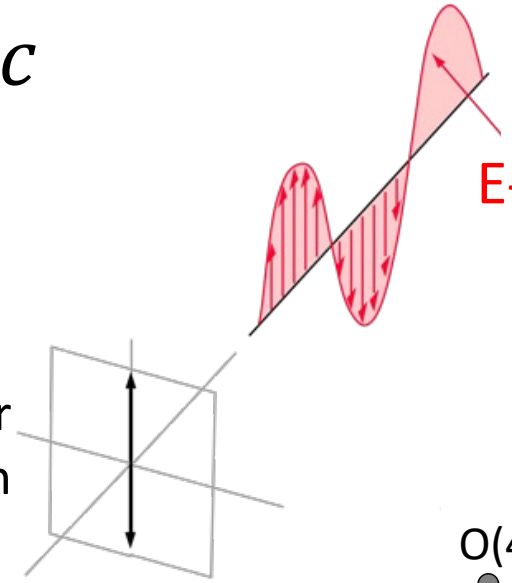
OxfordMateri

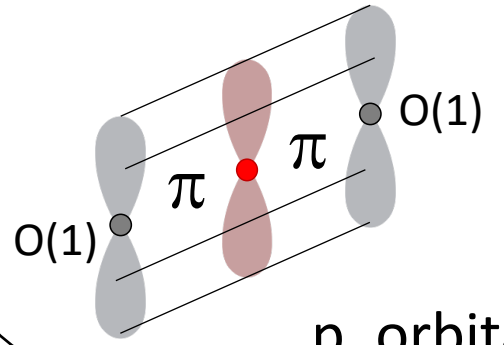
UNIVERSITY OF OXFORD

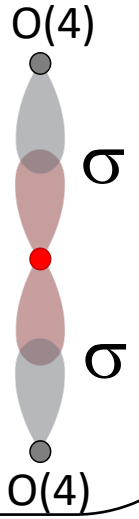
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Polarisation direction affects which bonds are probed

$\hat{E} \parallel c$

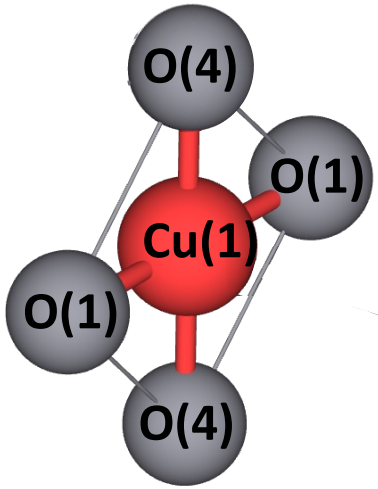


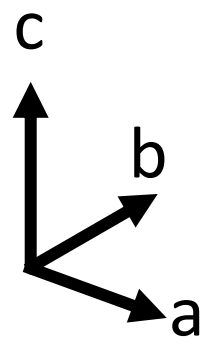




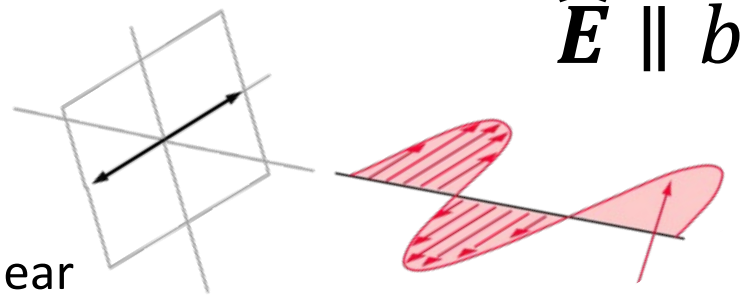
p_z orbitals

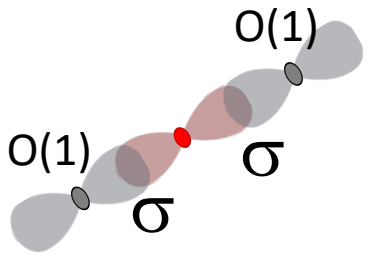
Chain site Cu

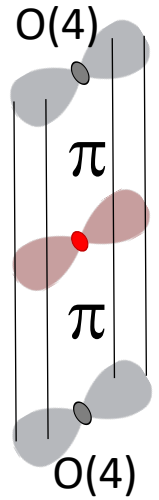




$\hat{E} \parallel b$





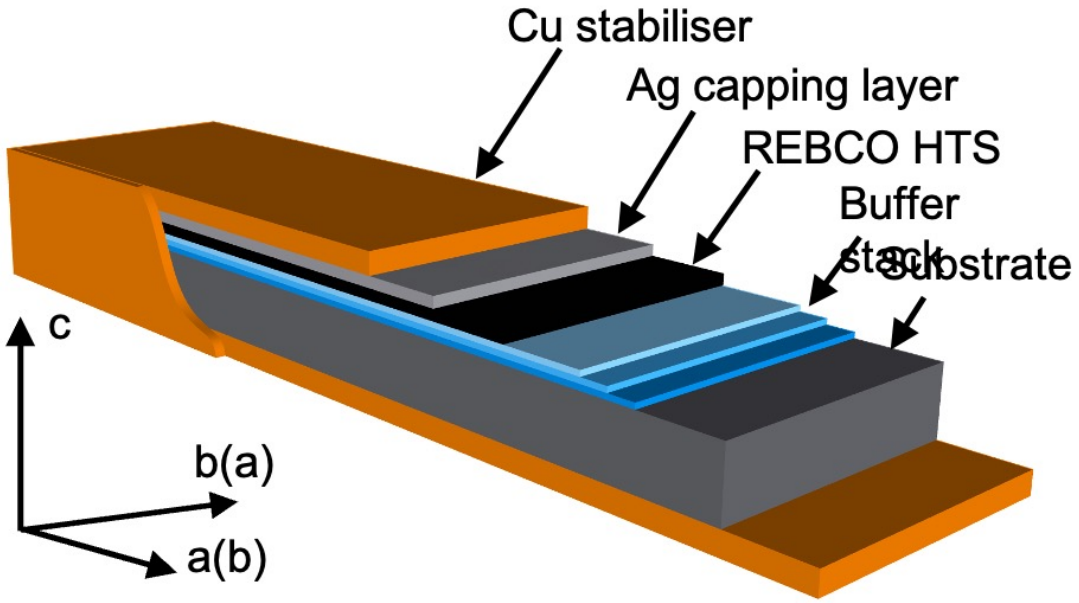


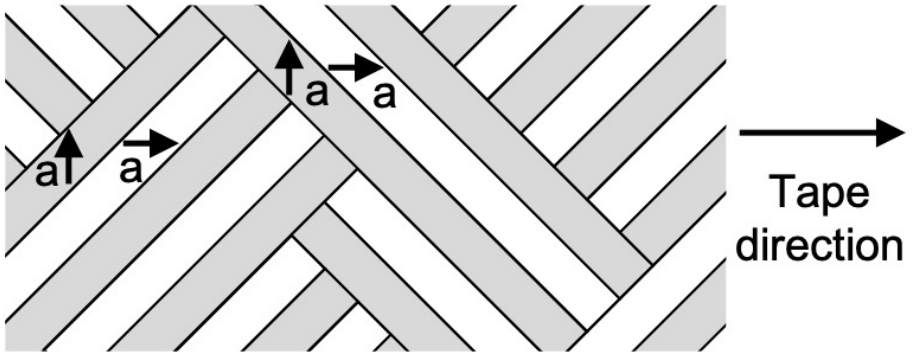
p_y orbitals

OxfordMaterials

UNIVERSITY OF OXFORD

Coated conductor texture



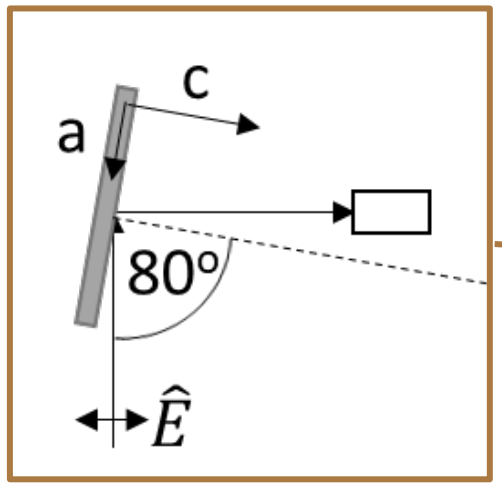


- REBCO layer is highly textured in coated conductor with c-axis perpendicular to tape surface
- REBCO has a slight orthorhombic distortion with b slightly larger than a
  - material is naturally twinned
  - a/b polarisations will be averaged in the spectroscopy measurements

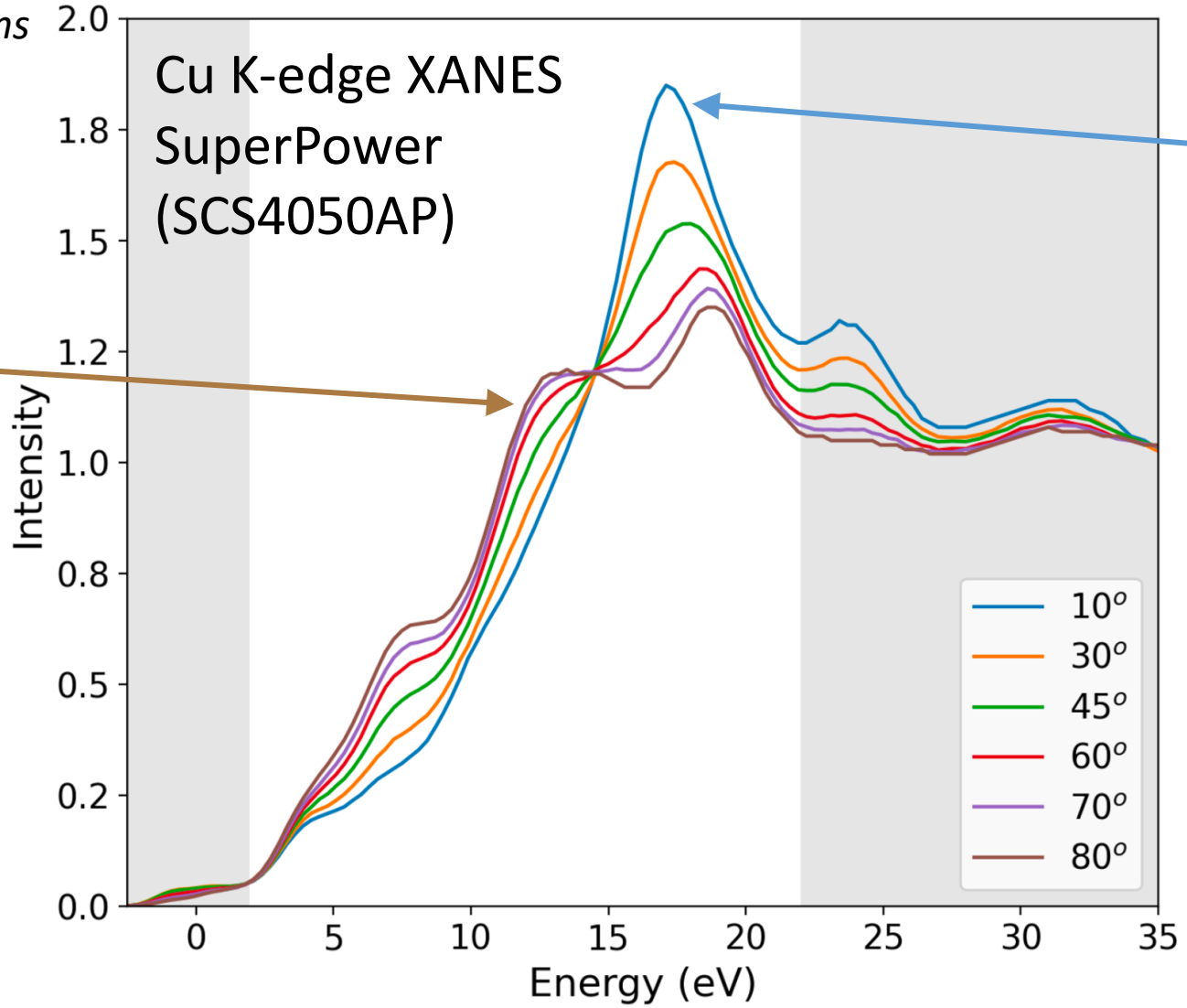
UNIVERSITY OF OXFORD

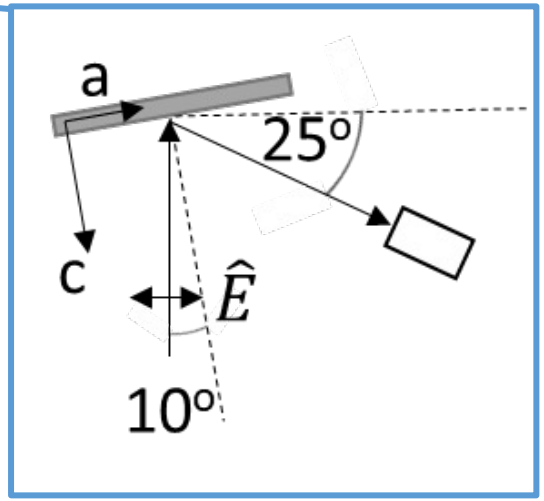
Orientation dependence

Nicholls et al, *Communications Materials* (2022) **3**:52



Polarisation is close to c-axis oriented





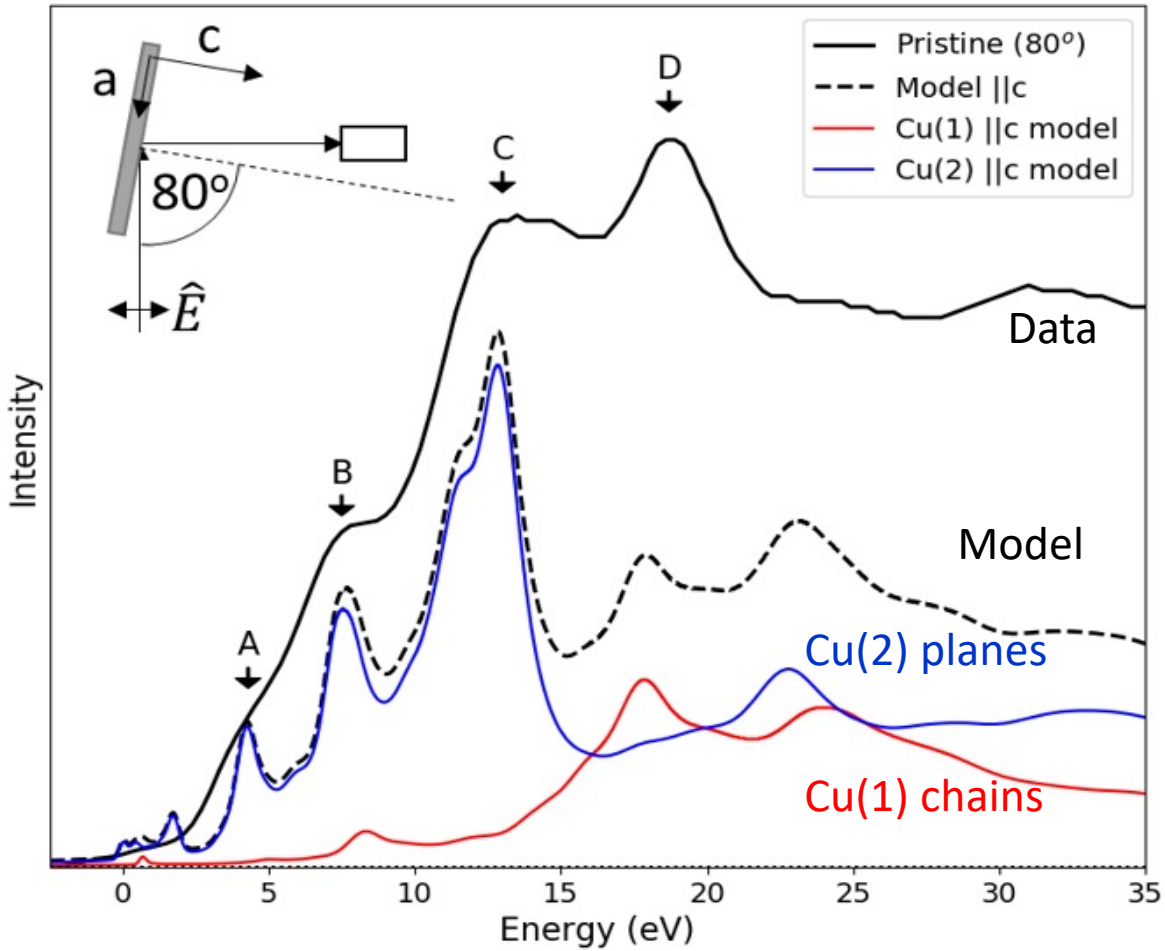
Polarisation is close to a/b-axis oriented

OxfordMaterials

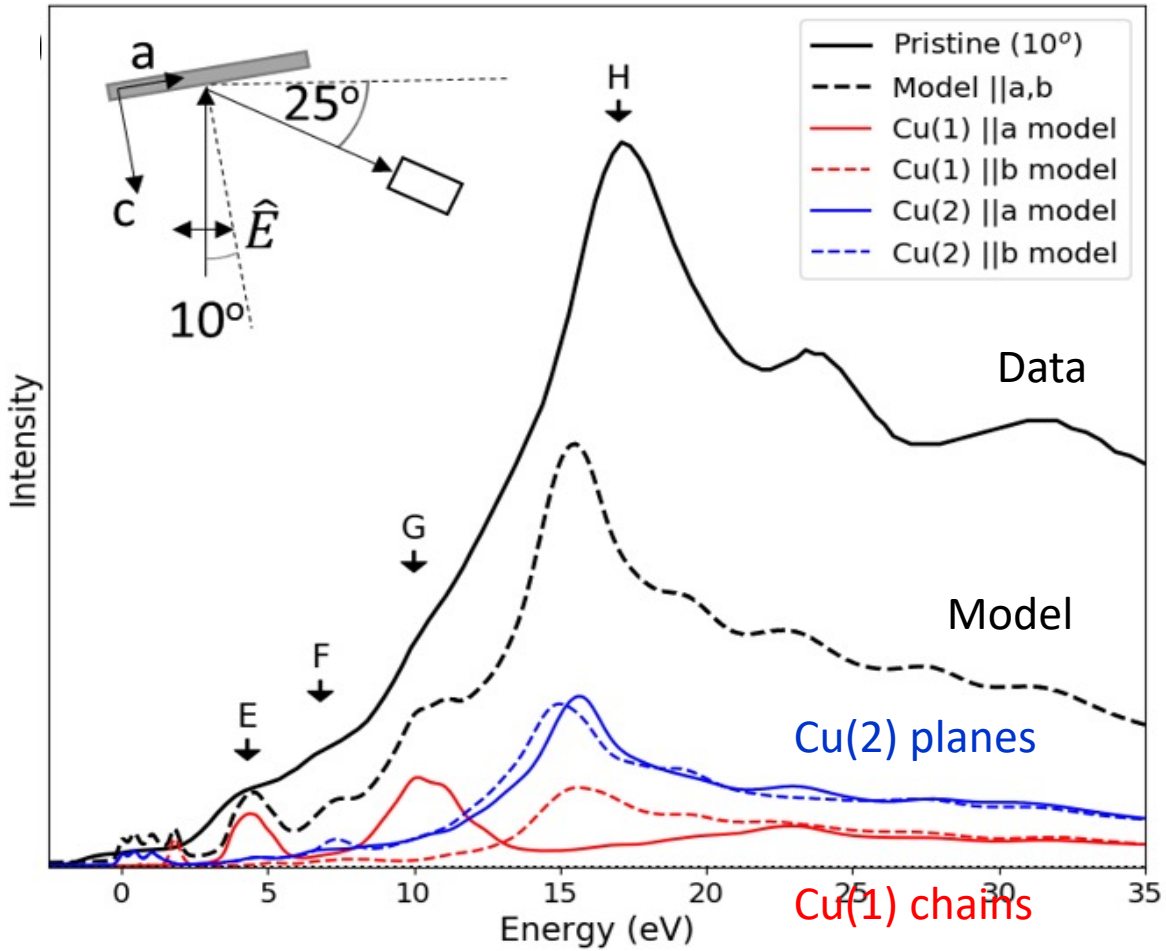
UNIVERSITY OF OXFORD

Pristine (unirradiated) coated conductor

Polarisation || c-axis



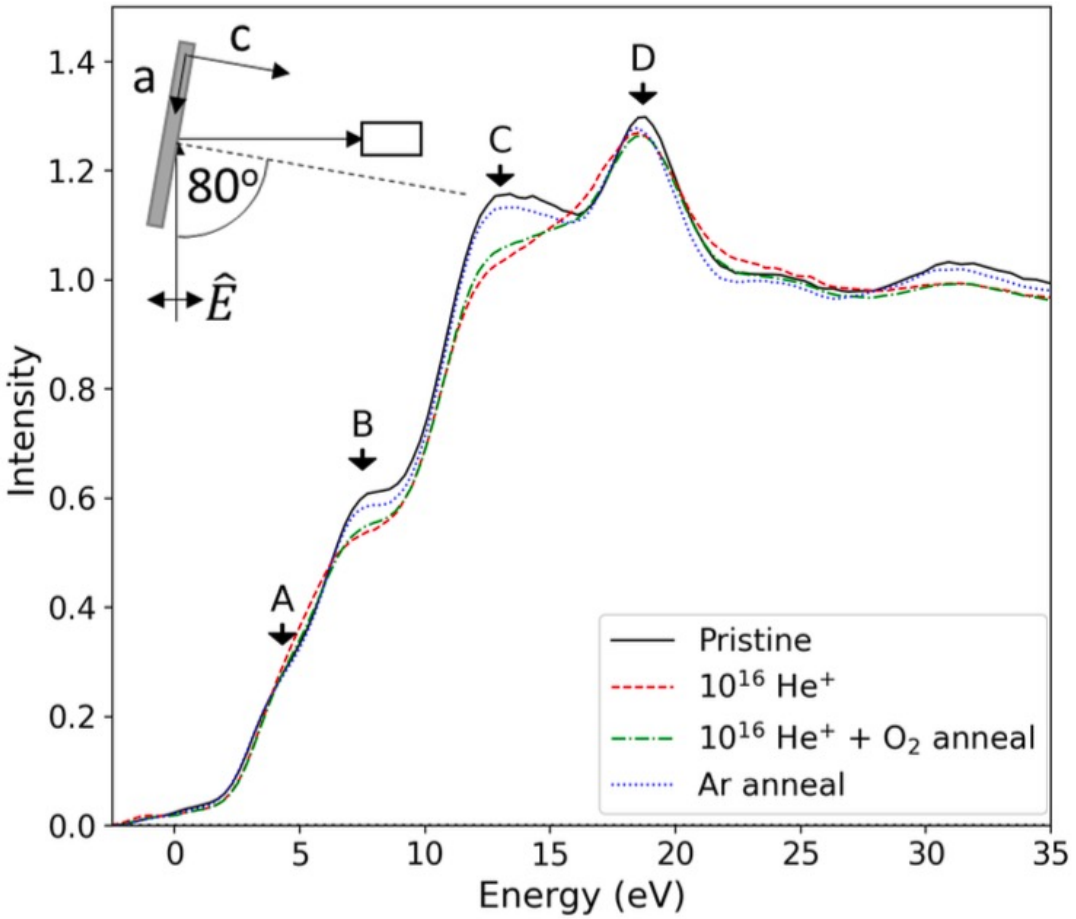
Polarisation || a(b)-axis



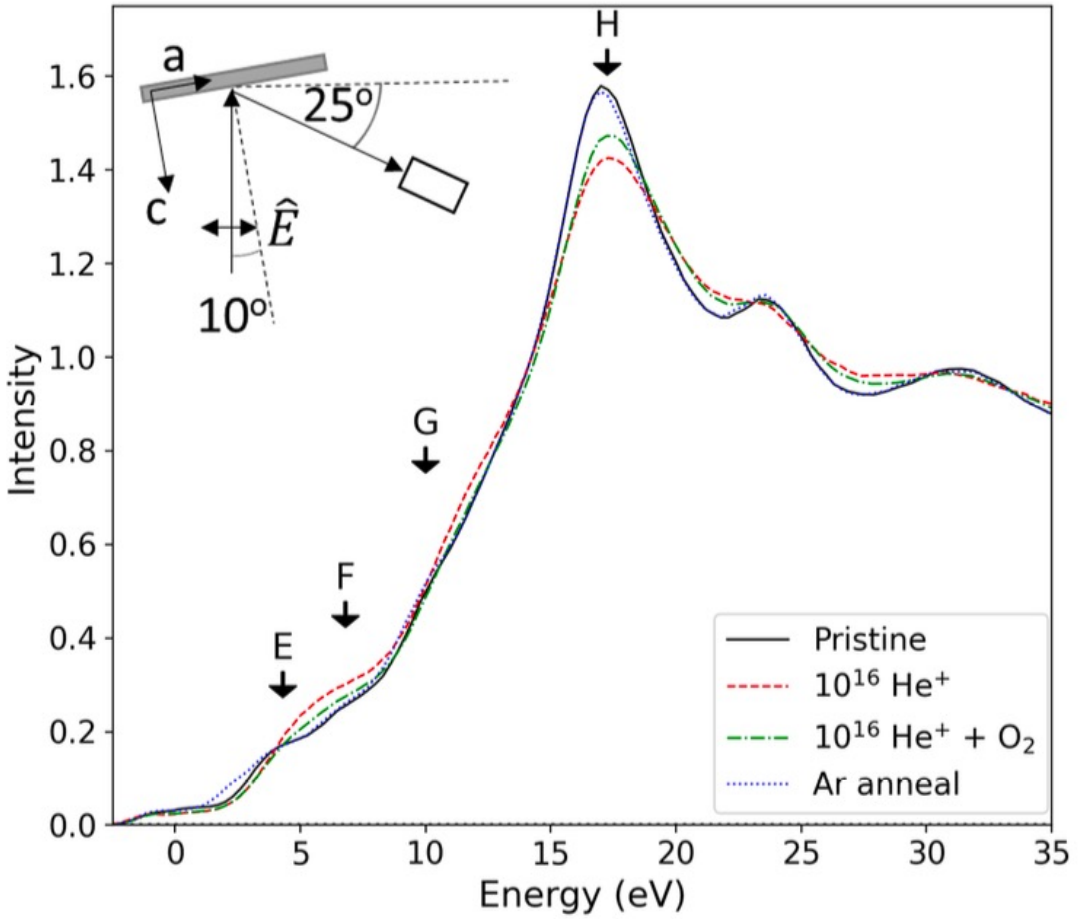
Nicholls et al, *Communications Materials* (2022) **3**:52

300 keV He^+ irradiation

Polarisation || c-axis



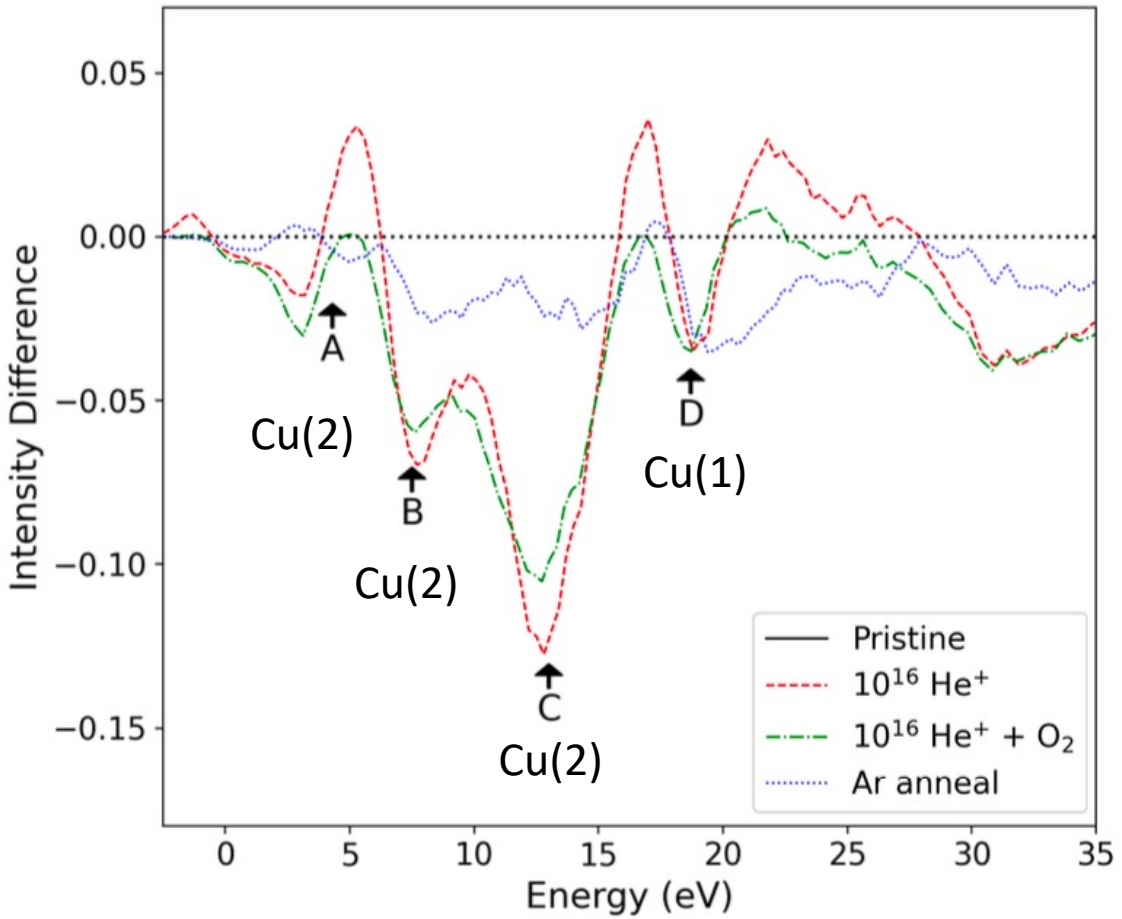
Polarisation || a(b)-axis



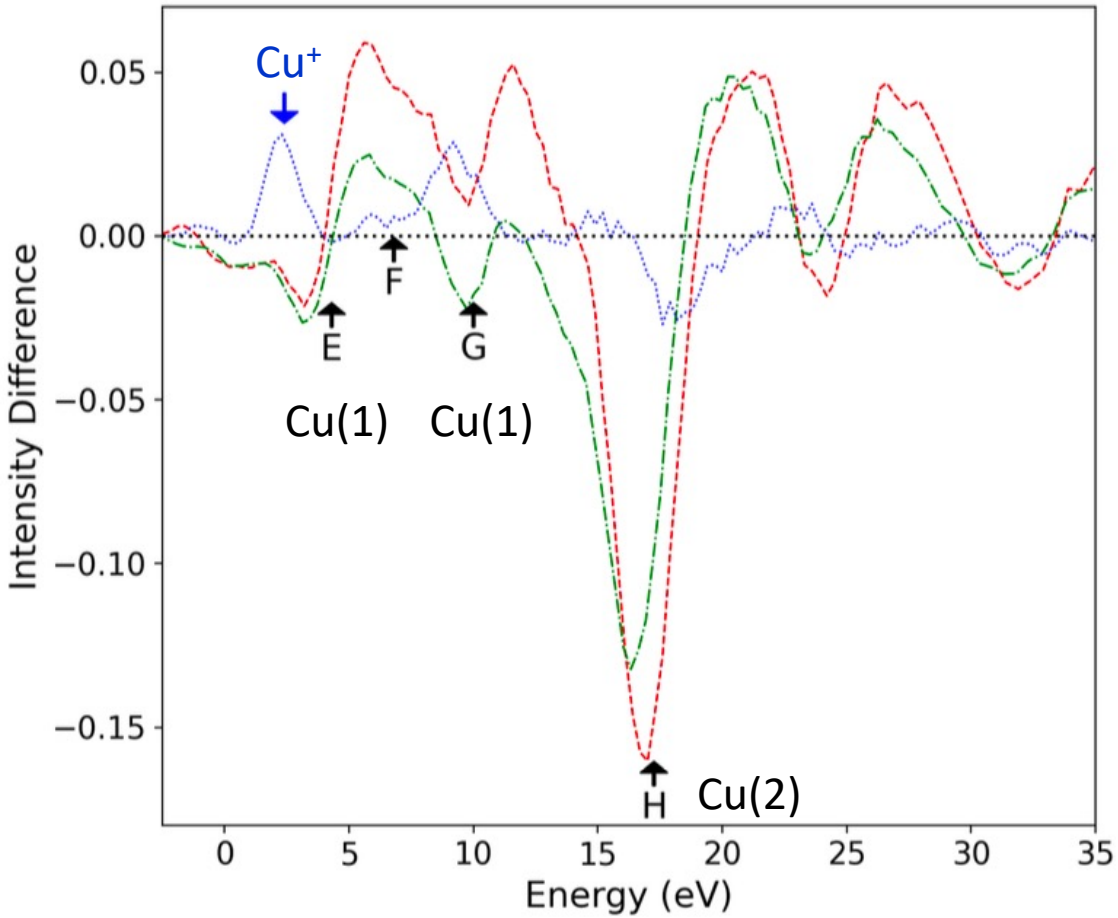
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

300 keV He^+ irradiation Difference spectra

Polarisation || c-axis



Polarisation || a(b)-axis

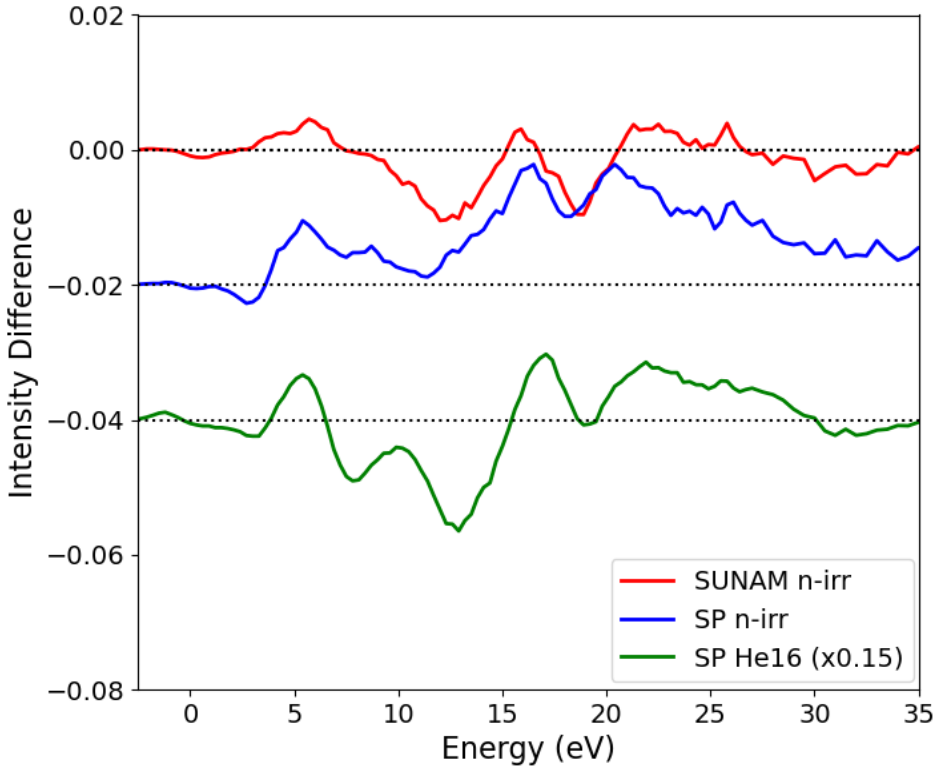


Considerable changes to the Cu(2) spectral features on irr

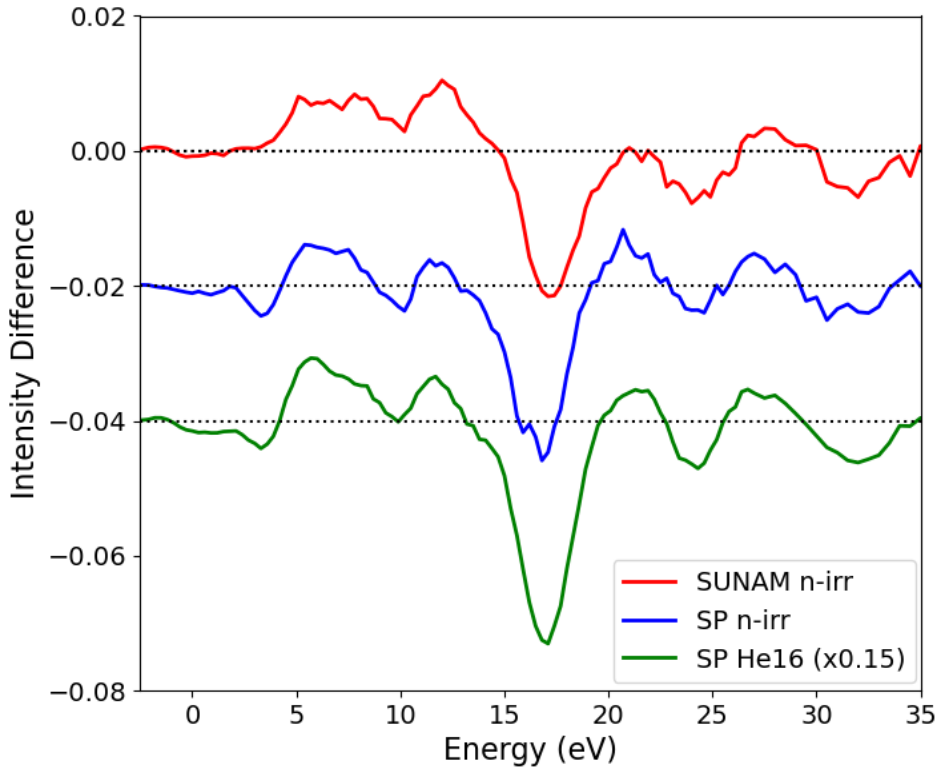
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Comparison with neutron irradiation

Polarisation || c-axis



Polarisation || a(b)-axis



n-irr
(3.3×10^{18} n cm^{-2} in Vienna Triga reactor)

He-irr
(10^{16} cm^{-2}, 300 keV, at SIBC)

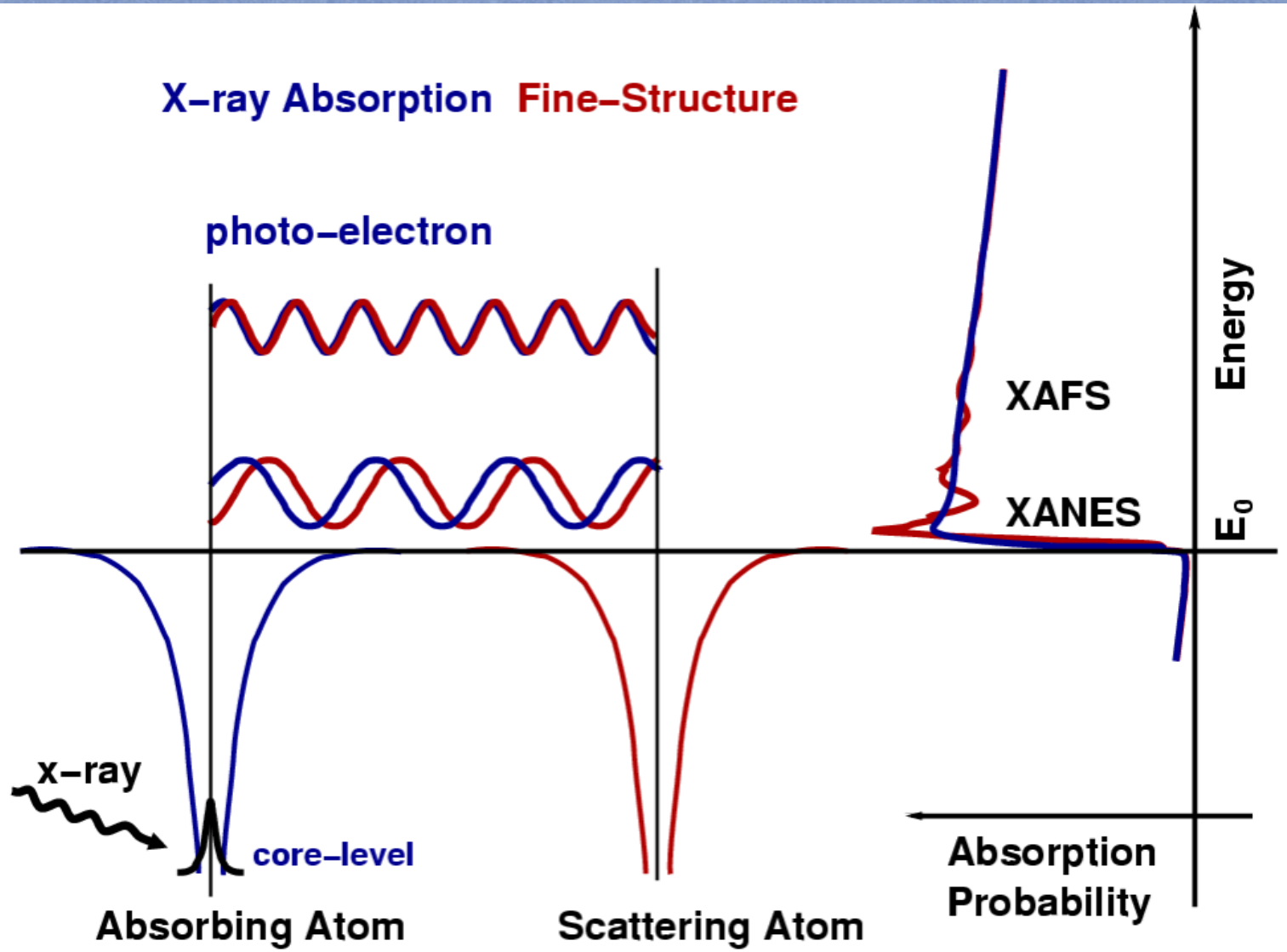
K Adams *et al* 2023 *Supercond. Sci. Technol.* **36** 10LT01

300 keV He^+ and fast neutrons produce similar changes to XANES spectra → similar defects produced?

Extended X-ray Absorption Fine Structure (EXAFS)

Oscillations in post-edge region caused by photoelectrons being scattered by surrounding atoms and returning to the absorbing atom

The EXAFS signal is the modulation of the amplitude of the photoelectron wavefunction at the absorbing atom due to the scattered photoelectron



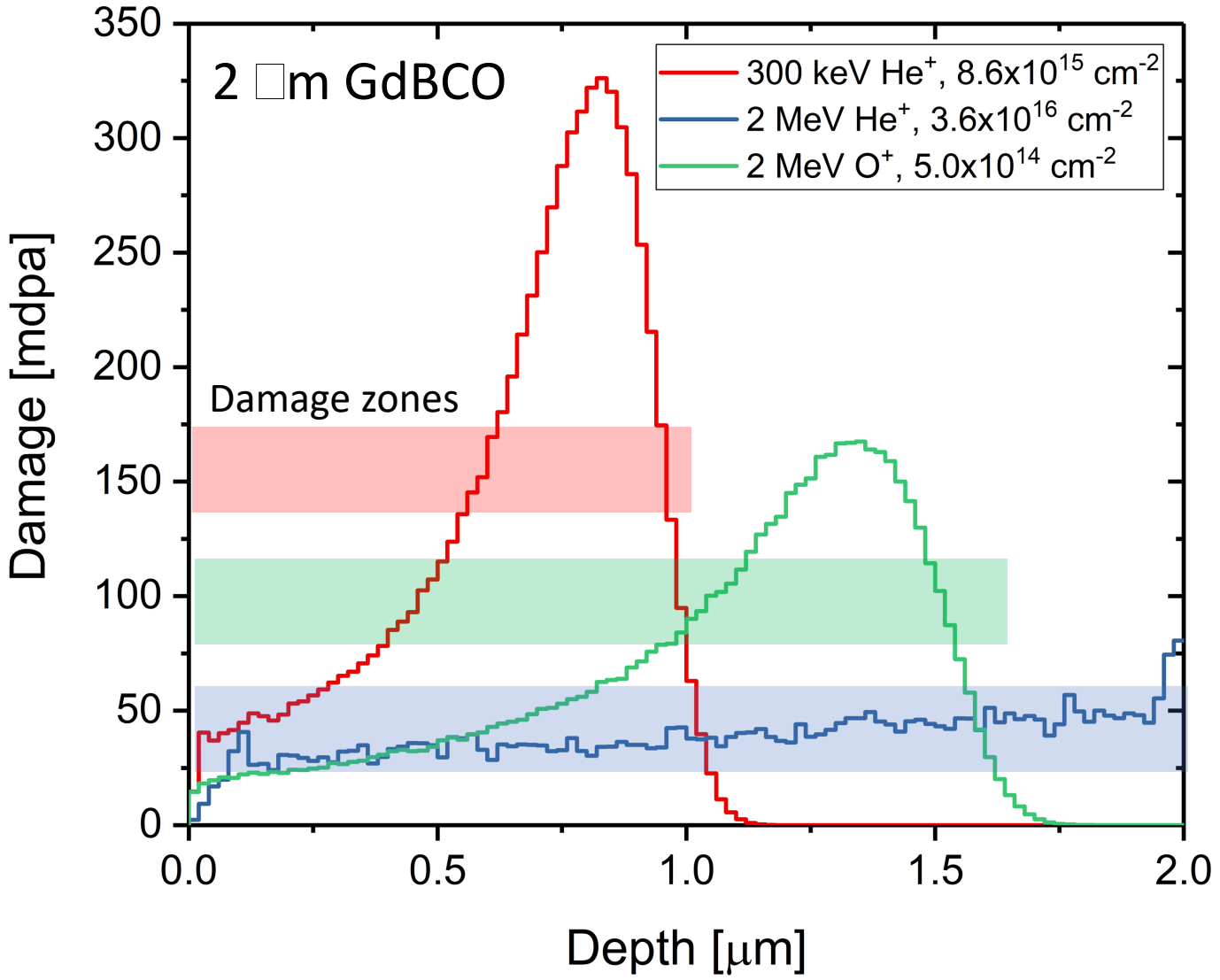
M. Newville, Fundamentals of XFAS

OxfordMaterials

UNIVERSITY OF OXFORD

IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Fujikura GdBCO coated conductor ion irradiations



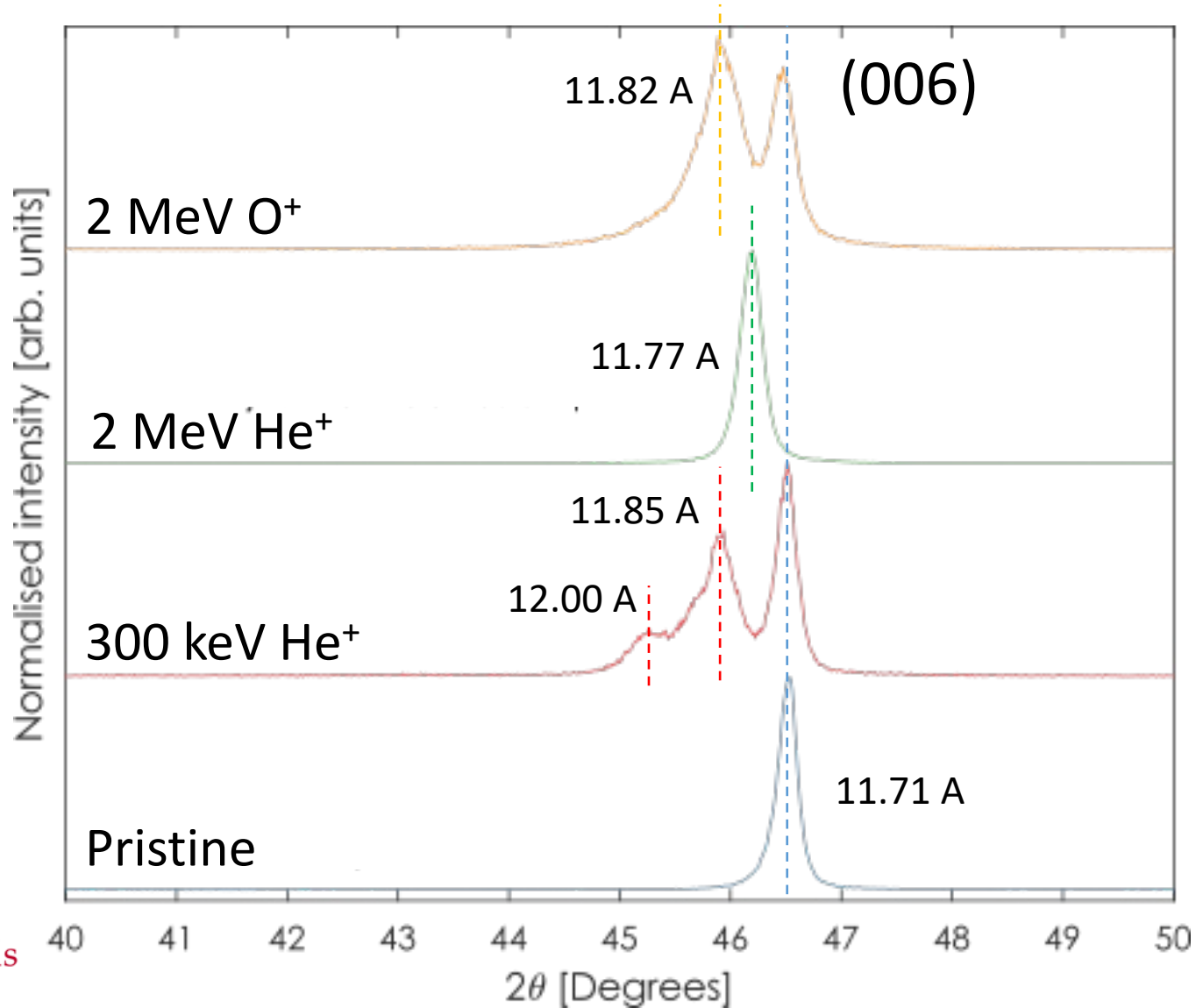
Fujikura CC selected to avoid elements that overlap with Cu, Ba, Gd edges

- GdBCO/CeO_2/MgO (IBAD)/Y_2O_3/Al_2O_3/Hastelloy
- No $BaZrO_3$ APCs

Ion	Average damage [mdpa]	Max damage at Bragg peak [mdpa]	Depth of Bragg peak [□m]
300 keV He+	72	~325	~0.6
2 MeV He+	39	~160	~1.3
2 MeV O+	59	-	-

UNIVERSITY OF OXFORD

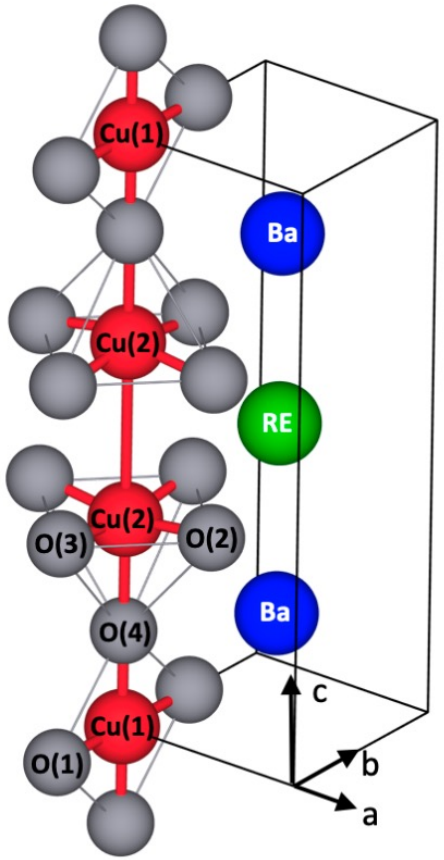
θ–□θ XRD analysis

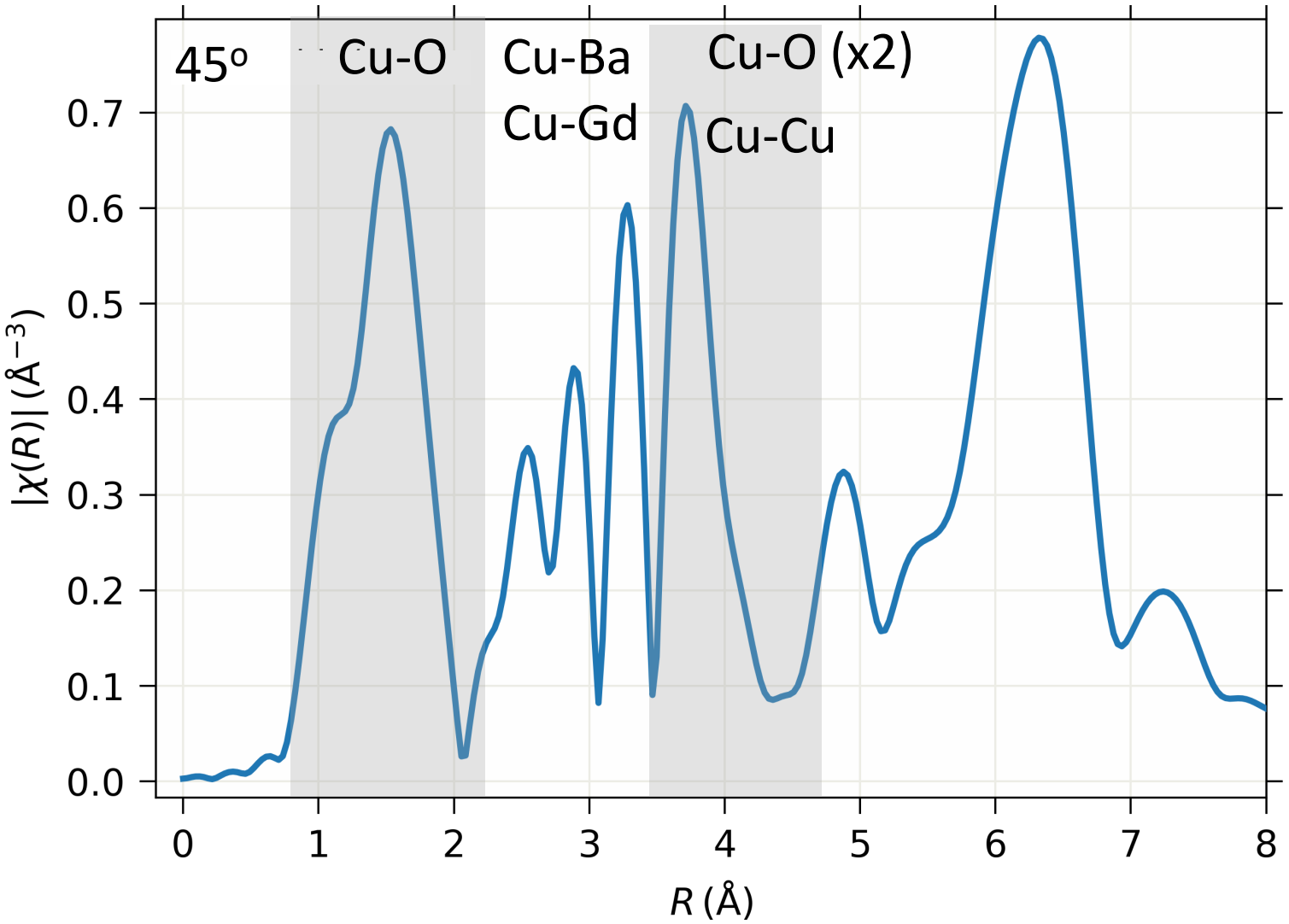


- Irradiation increases c-axis lattice parameter
- 2 MeV O^+ and 300 keV He^+ have 2 peaks → top layer is damaged and bottom layer is pristine, as expected from SRIM
- Damaged layer peaks are broader

IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Cu K-edge EXAFS of pristine Fujikura GdBCO sample





Many bonds of similar lengths overlap making it difficult to analyse the data

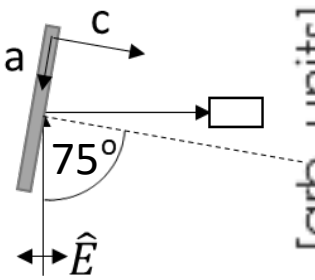
OxfordMaterials

UNIVERSITY OF OXFORD

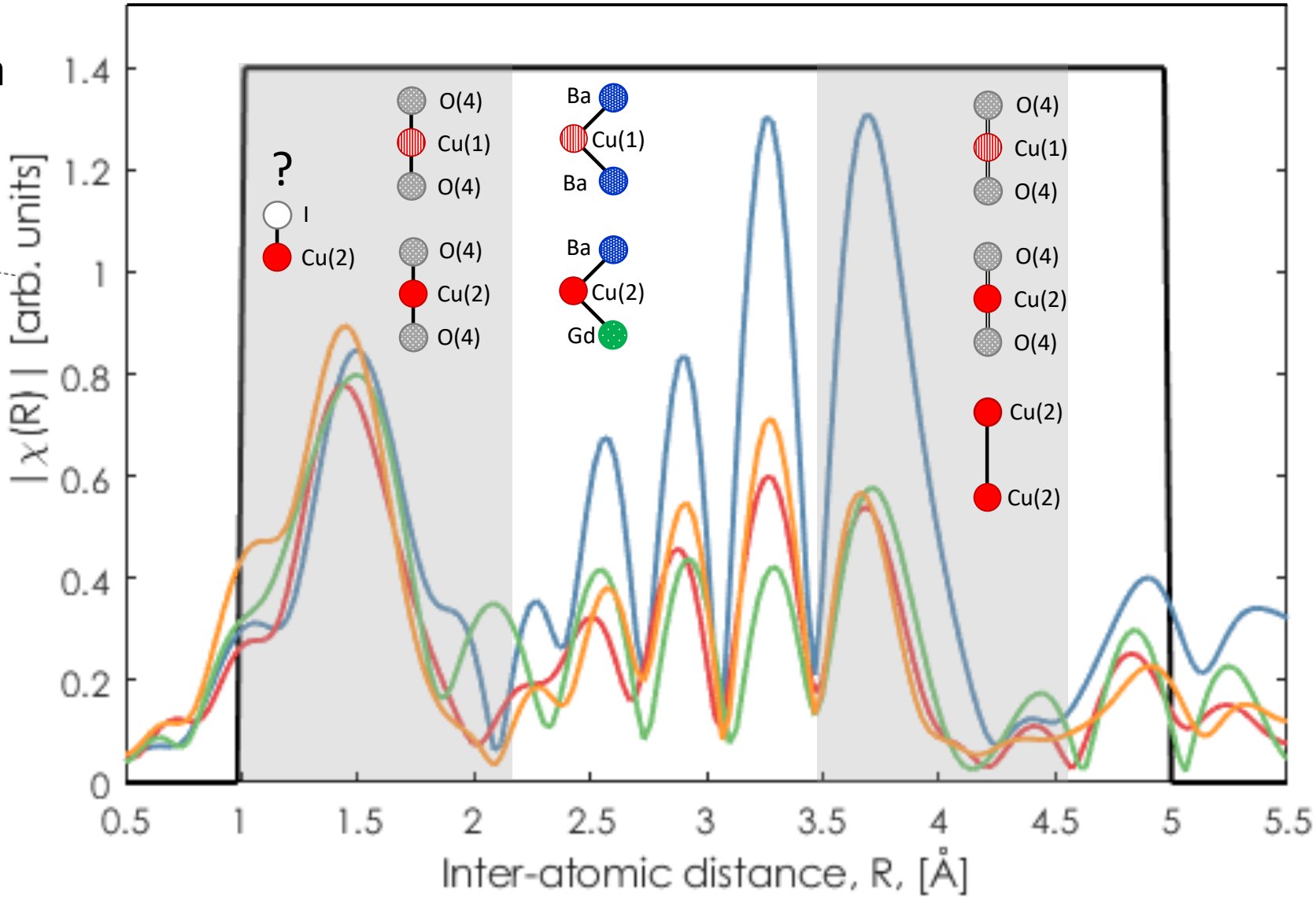
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Effects of irradiation on bonding in c direction

75° spectra



mainly probes bonding along c direction



diamond

I20 beamline

Pristine

300 keV He^+ (av mdpa = 79)

2 MeV He^+ (av mdpa = 39)

2 MeV O^+ (av mdpa = 59)

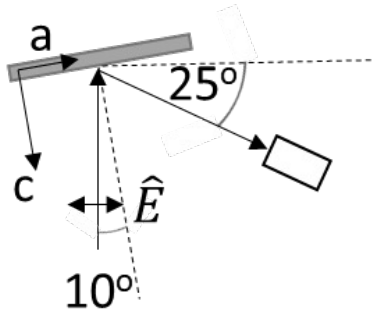
Oxfo

UNIVERSITY OF OXFORD

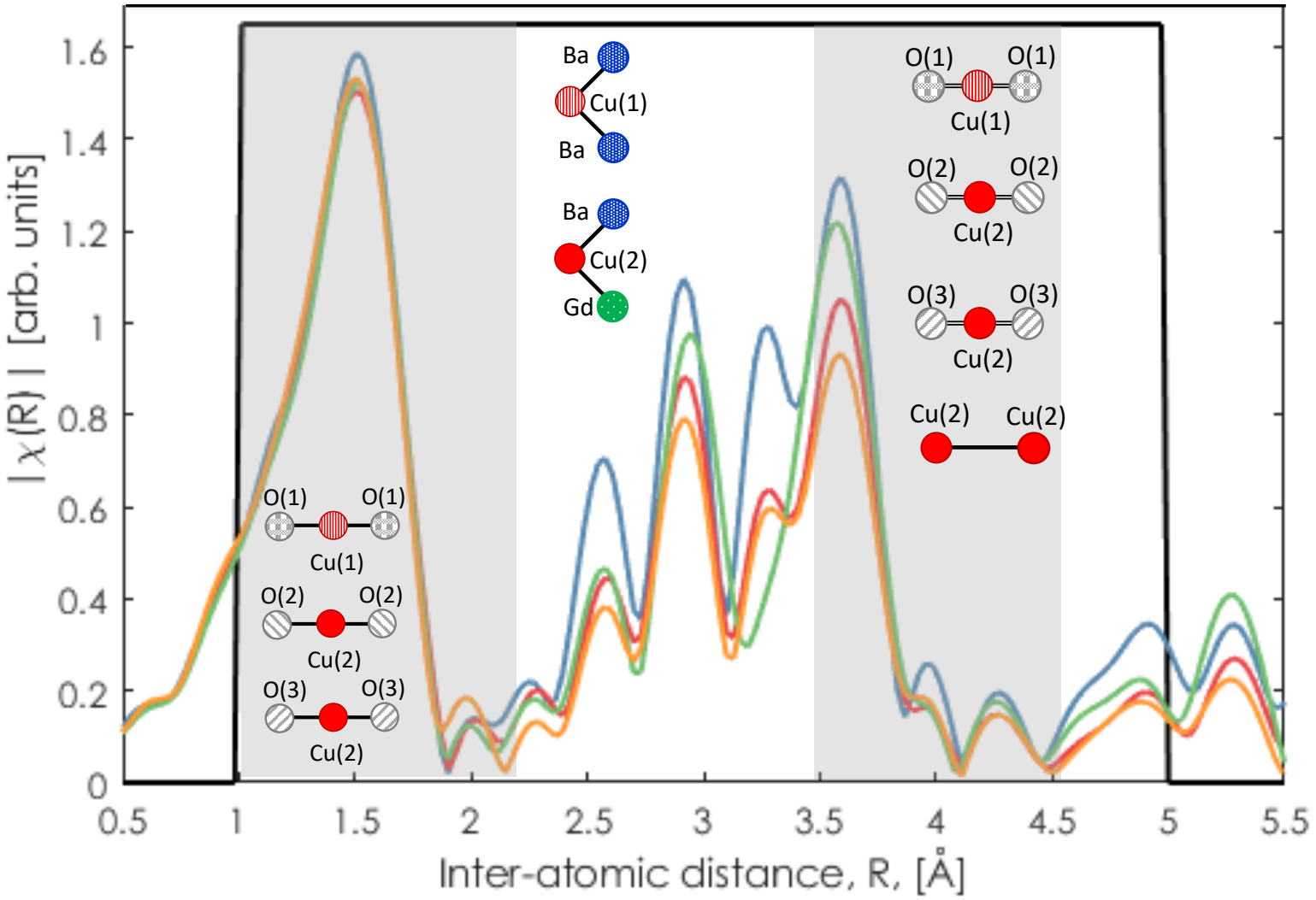
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Effects of irradiation on bonding in (a,b) plane

10° spectra



mainly probes bonding in (a,b) plane



diamond

I20 beamline

- Pristine
- 300 keV He^+ (av mdpa = 79)
- 2 MeV He^+ (av mdpa = 39)
- 2 MeV O^+ (av mdpa = 59)

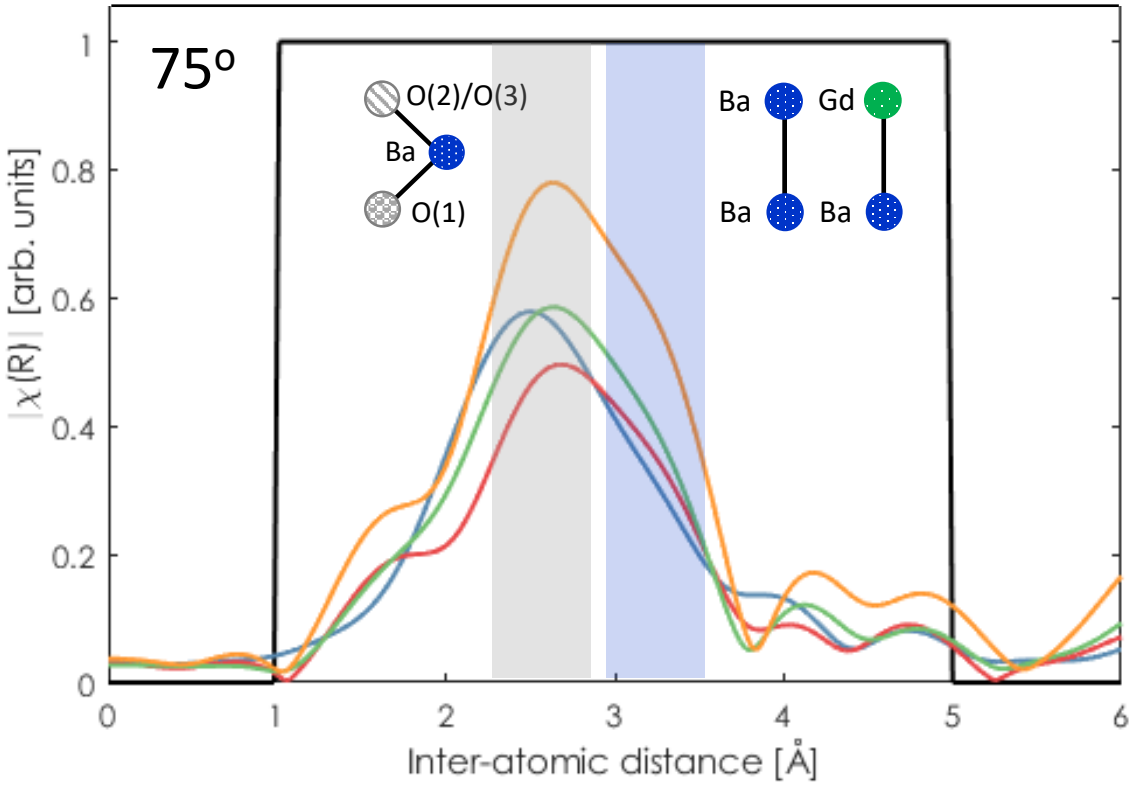
OxfordMat

UNIVERSITY OF OXFORD

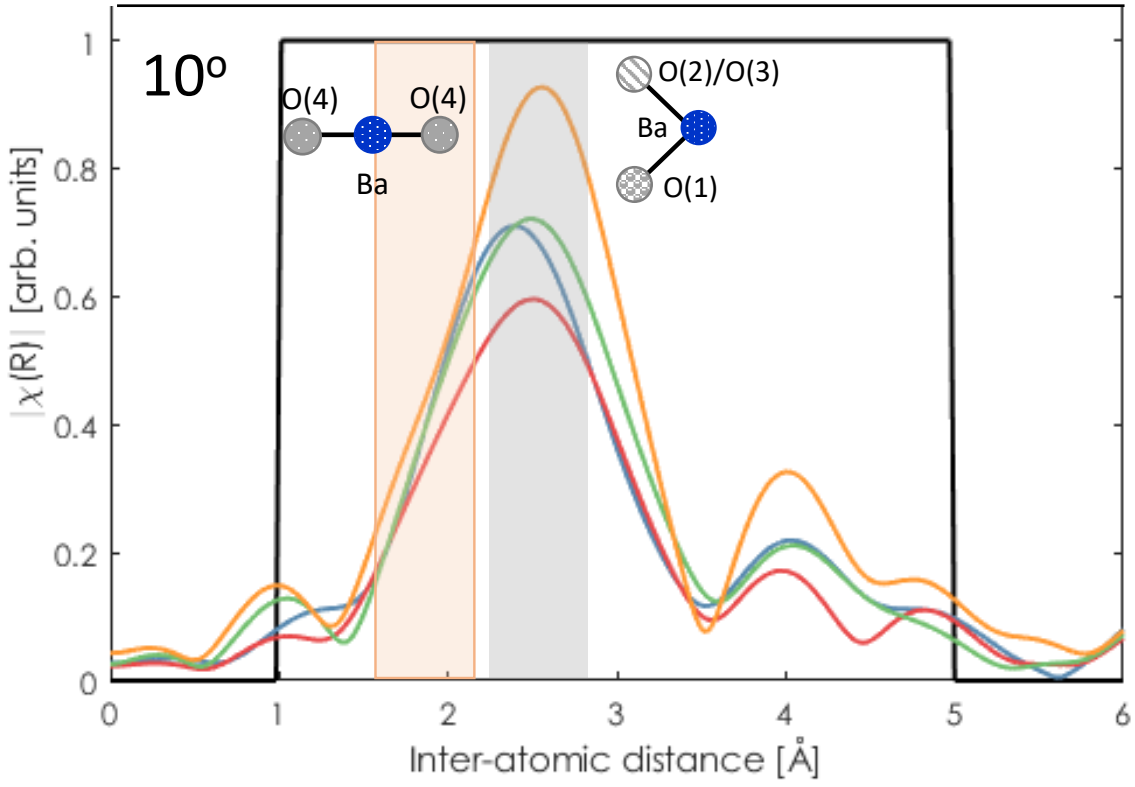
IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Ba L-edge EXAFS

Probing direction close to c-axis



Probing direction close to (a,b) plane



B18 beamline

diamond

Pristine

300 keV He^+ (av mdpa = 79)

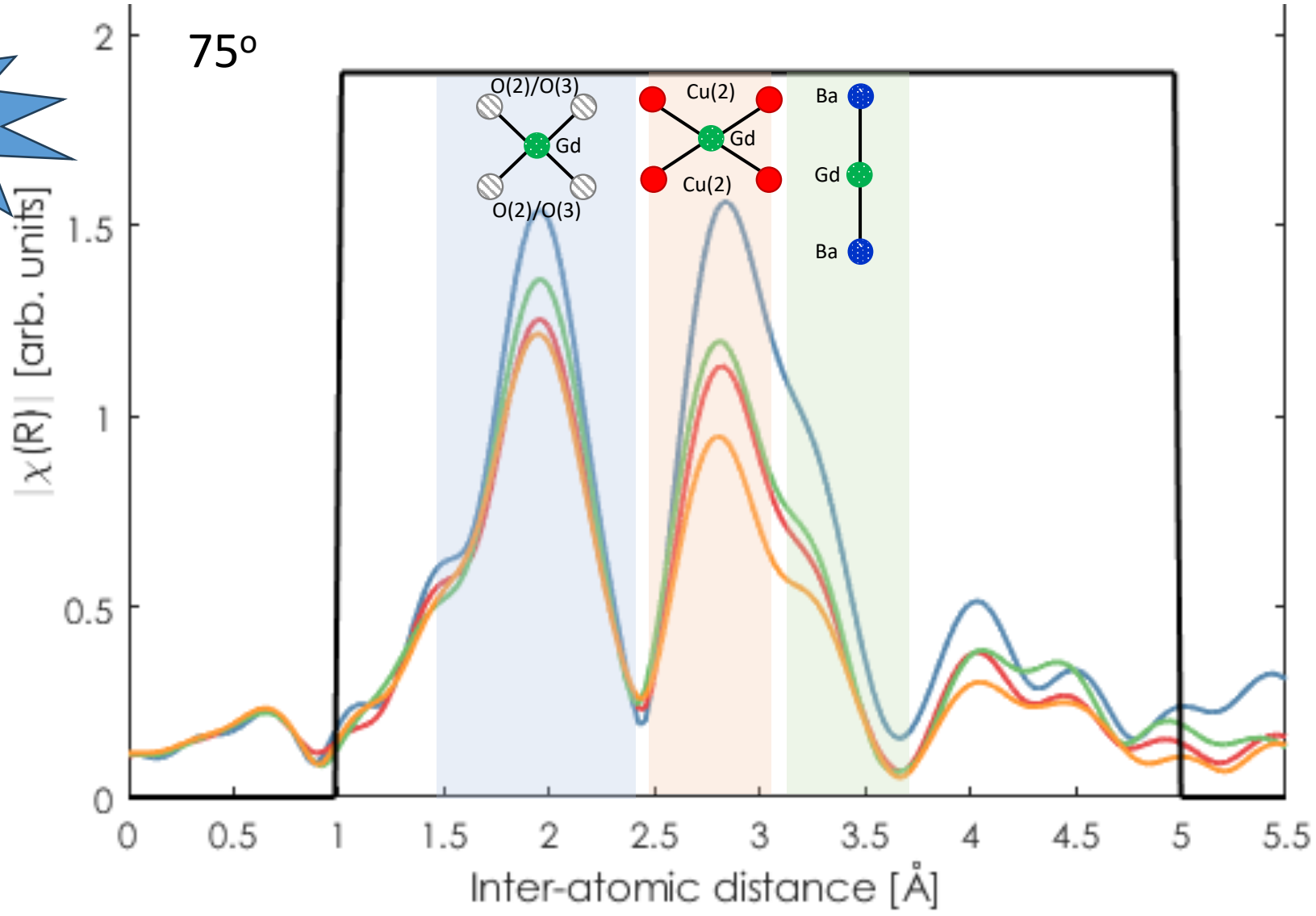
2 MeV He^+ (av mdpa = 39)

2 MeV O^+ (av mdpa = 59)

IEEE-CSC, ESAS and CSSJ SUPERCONDUCTIVITY NEWS FORUM (global edition), Issue No. 55, January, 2024. Invited presentation given at IREF 23, November 14, 2023, Arona, Italy

Gd L-edge EXAFS

Hot off the press



Pristine

300 keV He^+ (av mdpa = 79)

2 MeV He^+ (av mdpa = 39)

2 MeV O^+ (av mdpa = 59)

I20 beamline

diamond

Next steps

- Cryogenic transfer of ion irradiated samples to enable XAS of cold irradiated samples before warming up and controlled annealing experiments
- Full analysis of the EXAFS data
- Analysis of the pre-edge region of the XANES spectra
- Polarisation dependent Raman spectroscopy

Summary

- X-ray absorption spectroscopy is a useful tool for probing the local bonding and structure averaged over entire REBCO layer
- High energy resolution Cu K-edge XANES is complicated by presence of two distinct copper sites.
  - Modelling is required to interpret the spectra
  - Can see that O in plane sites are affected by irradiation
- EXAFS tells us about changes in bond lengths and disorder on irradiation
  - Orientation dependence helps distinguish between scattering paths of similar lengths
  - Full interpretation requires data from Cu, Ba and RE edges and careful fitting

Acknowledgements

Oxford University
- Jarrod Lewis
- Kirk Adams
- James Tufnail
- Yatir Linden
- Tayebeh Mousavi
- Chris Grovenor
- Rebecca Nicholls

Diamond Light Source
- Sofia Diaz-Moreno
- Fred Mosselmans
- Matteo Aramini

UKAEA (Culham)
- Will Iliffe

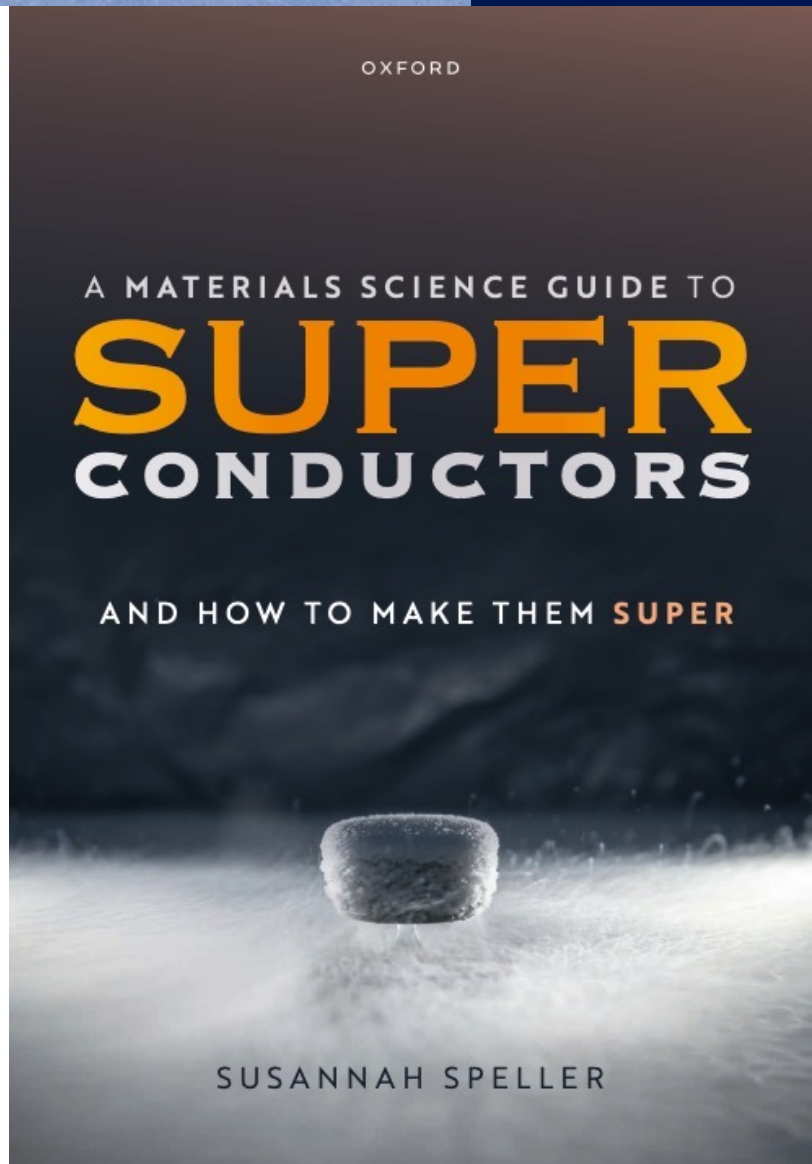
ePSIC facility (Harwell)
- Mohsen Danaie

Surrey Ion Beam Centre
- Nianhua Peng
- Roger Webb

TU Wien
- Michael Eisterer

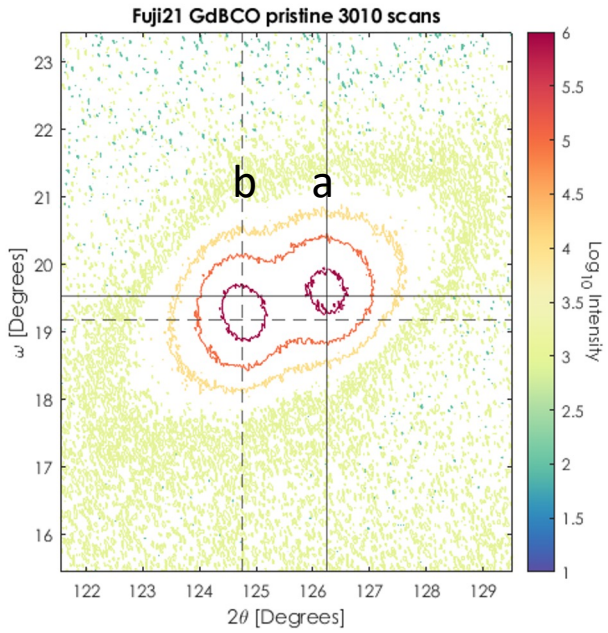
UKRI Engineering and Physical Sciences Research Council

OxfordMaterials



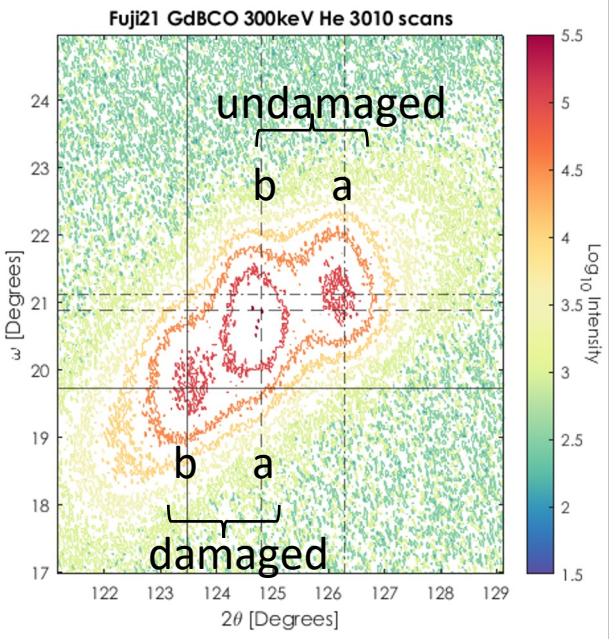
XRD: off-axis geometry

Pristine



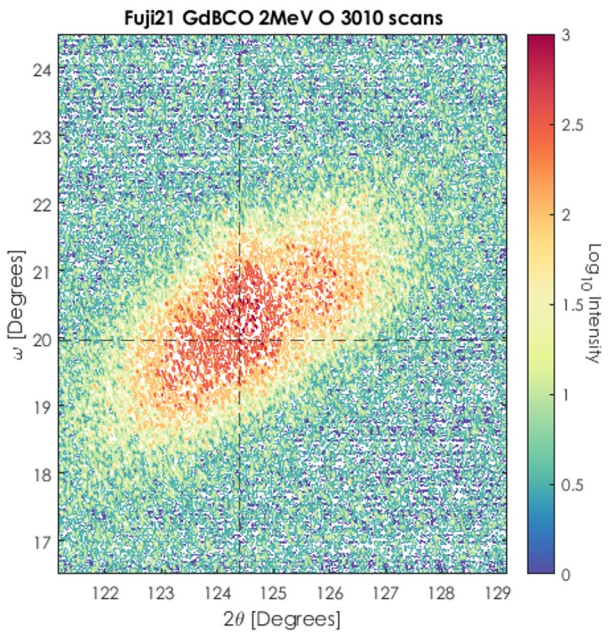
2 peaks
→ different a/b lattice parameters

300 keV He^+



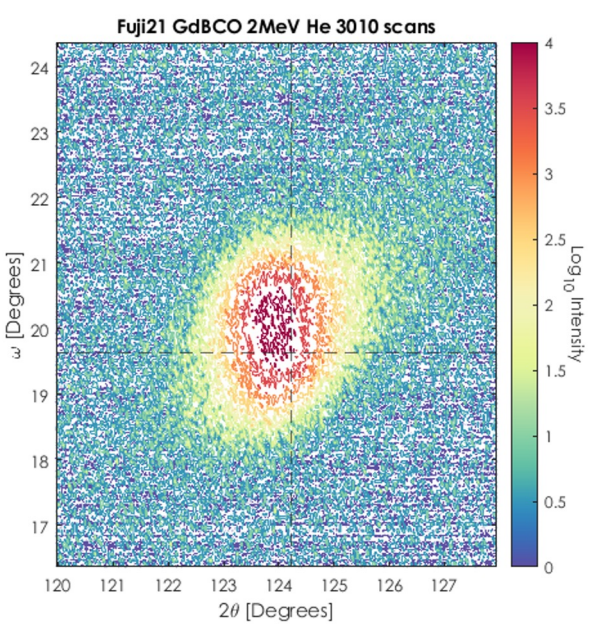
2 sets of 2 peaks
→ Damaged layer has different lattice parameters (lower 2θ values)

2 MeV O^+



Less distinct version of the 300 keV He^+
More intensity in damaged position – Bragg peak is broader deeper

2 MeV He^+



Single peak – chain disorder?

(3 0 10) planes

OxfordMaterials

UNIVERSITY OF OXFORD