



Probing disorder in ion irradiated REBCO coated conductor

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&
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Irradiation Effects in HTS for Fusion (IREF) 2025

Acknowledgements

University of Oxford:

- Prof Susie Speller
- Prof Rebecca Nicholls
- Prof Chris Grovenor
- Dr Clara Barker
- Kirk Adams
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Talk structure

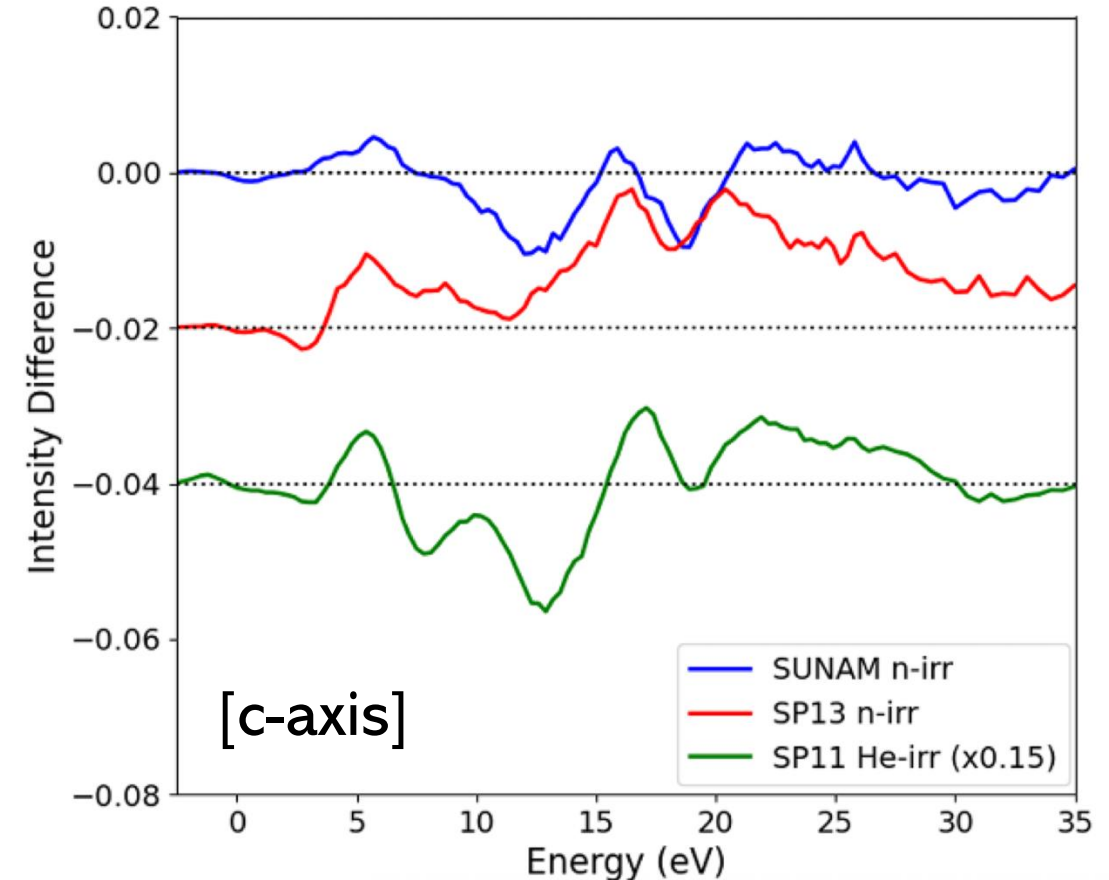
- Overview of He ion irradiation treatments
- Crystal structure studies of He⁺ irradiated REBCO cc
 - Characterisation with laboratory Cu-K source X-ray diffraction
- Local structure studies of He⁺ irradiated REBCO cc
 - Analysis of synchrotron Cu-K edge X-ray absorption spectroscopy
- Conclusions

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2 MeV He⁺ Irradiation

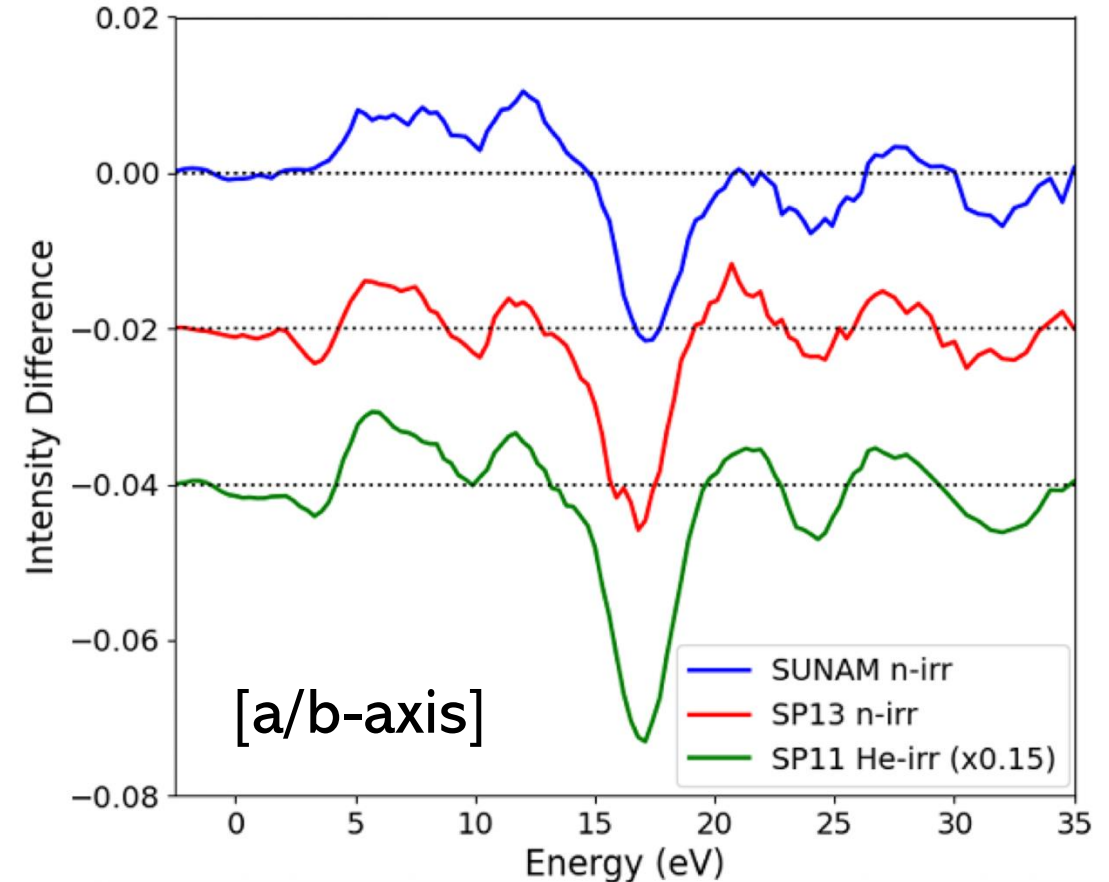
- He ion irradiation carried out at the Surrey Ion Beam Centre, UK.
- He⁺ chosen as a neutron proxy as similar irradiation features observed in previous studies [1].



[1] – Adams, K. *et al.*, *Superconductor Science & Technology*, 2023, Vol.36, 10LT01.

2 MeV He⁺ Irradiation

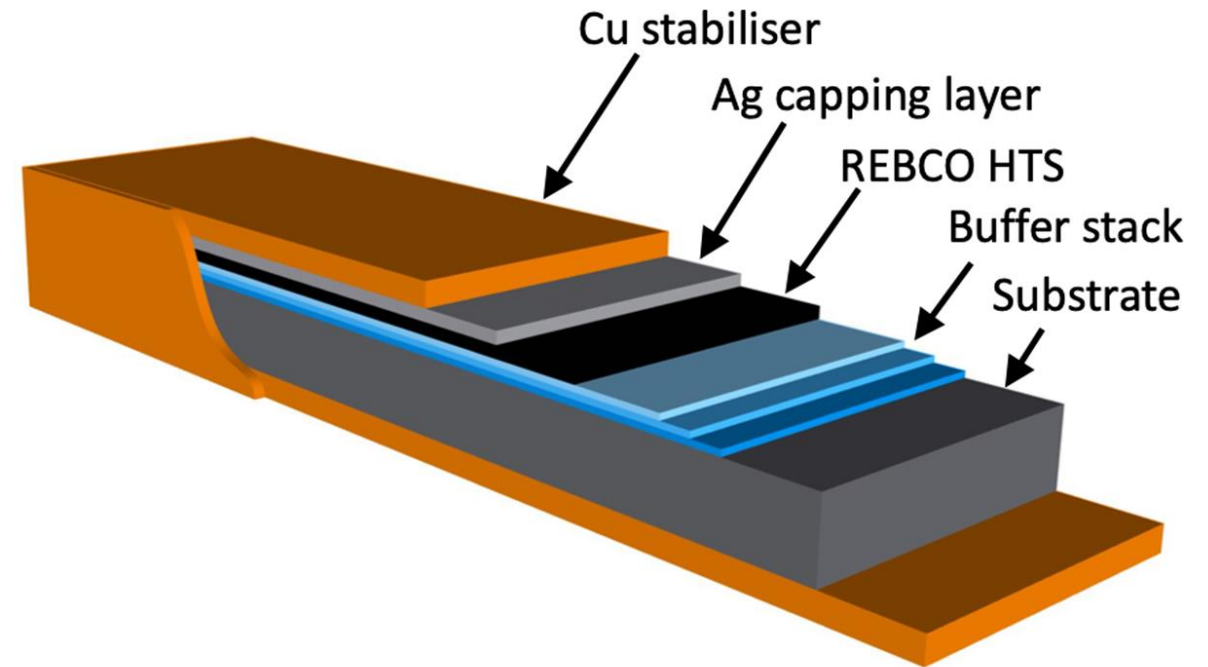
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2 MeV He⁺ Irradiation

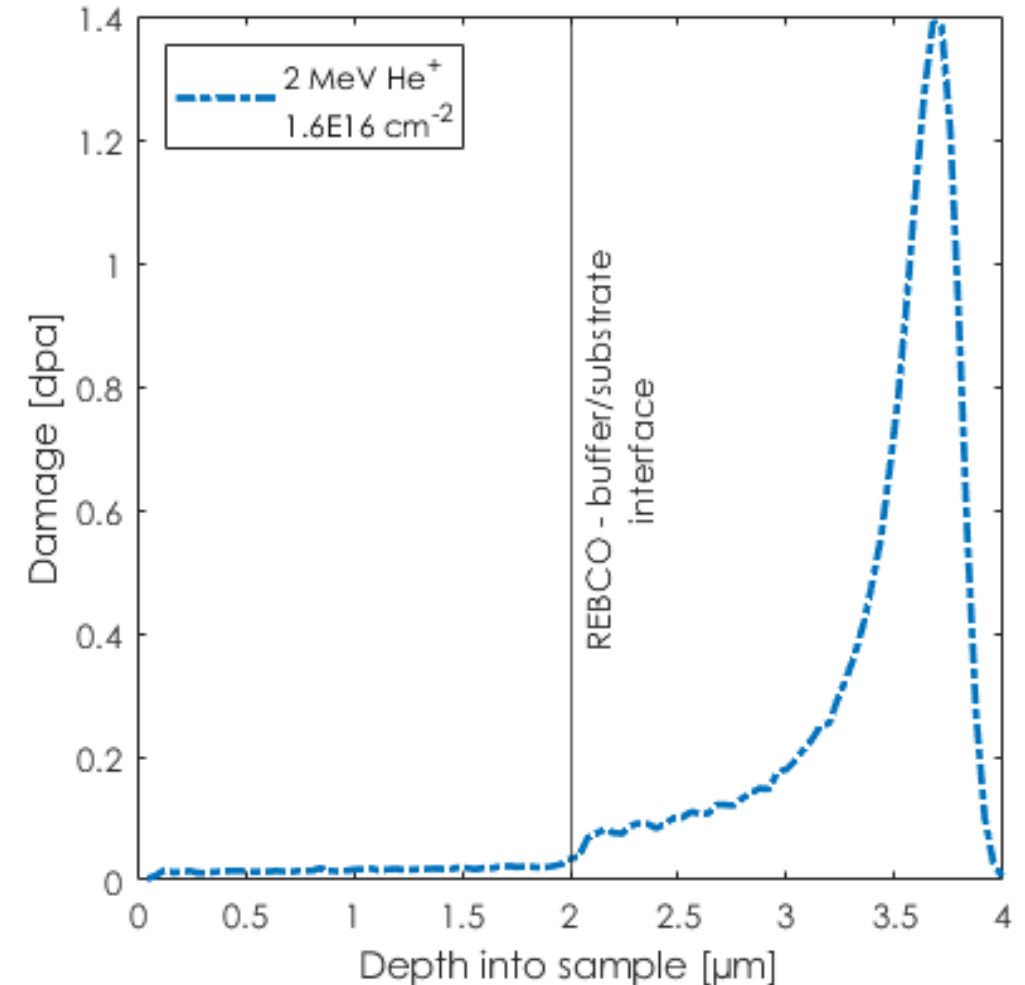
- Stabiliser and capping layers removed with wet chemical etching to expose HTS layer.
- Prevents ions being attenuated.



[2] – Nicholls, R. *et al.*, *Communications Materials*, 2022, Vol.3 (52).

2 MeV He⁺ Irradiation

- 2 MeV He⁺ chosen for relatively uniform damage in HTS layer.
- Fluence magnitudes of 1E16 ions/cm² at room temperature.
- Typically aiming for 10-40 mdpa.

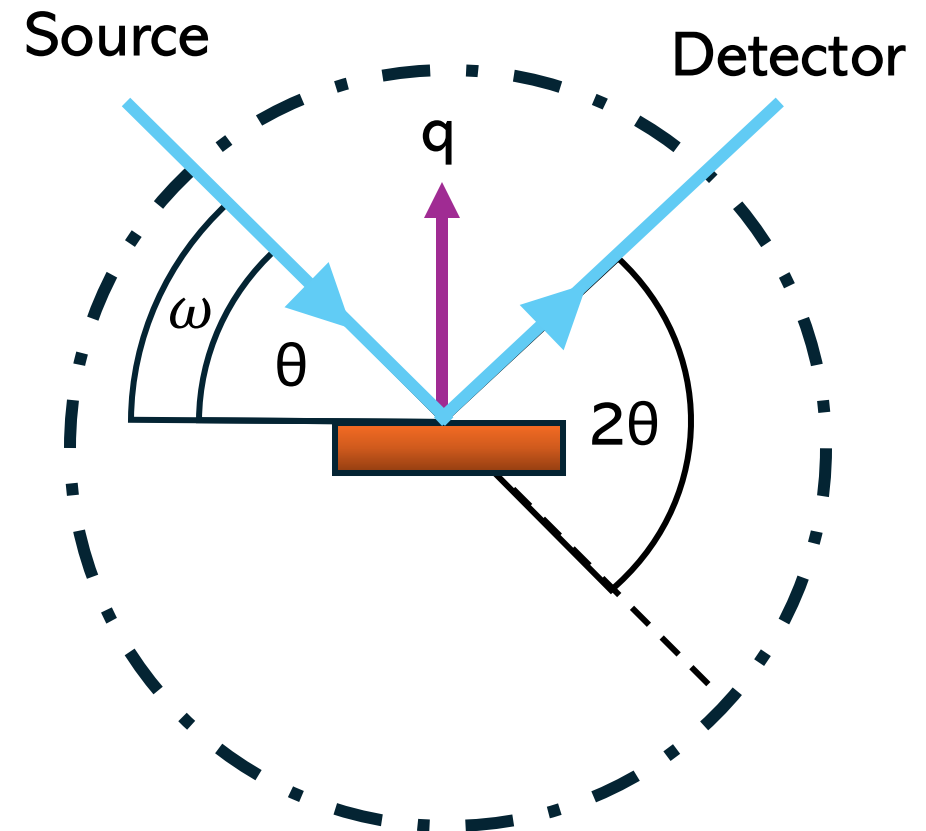


Talk structure

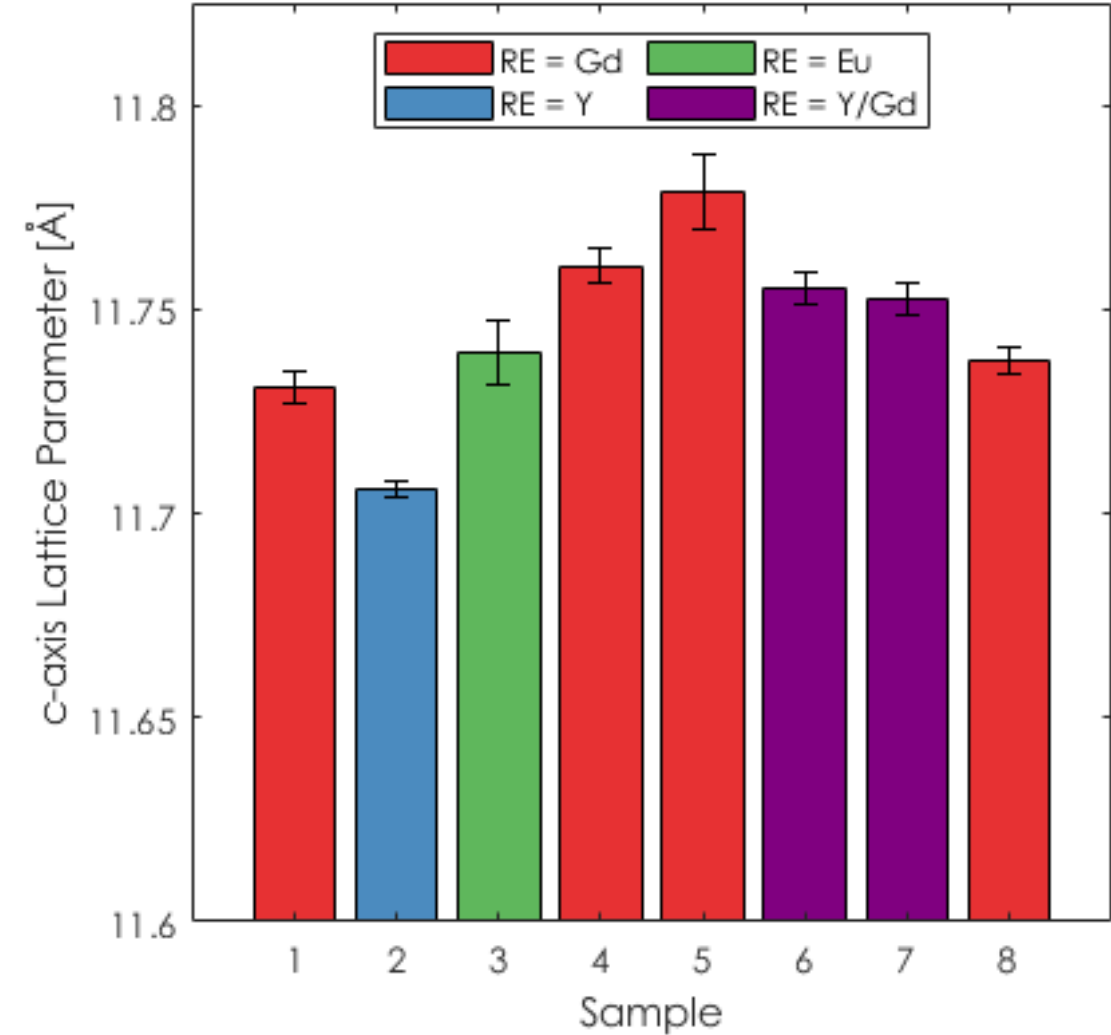
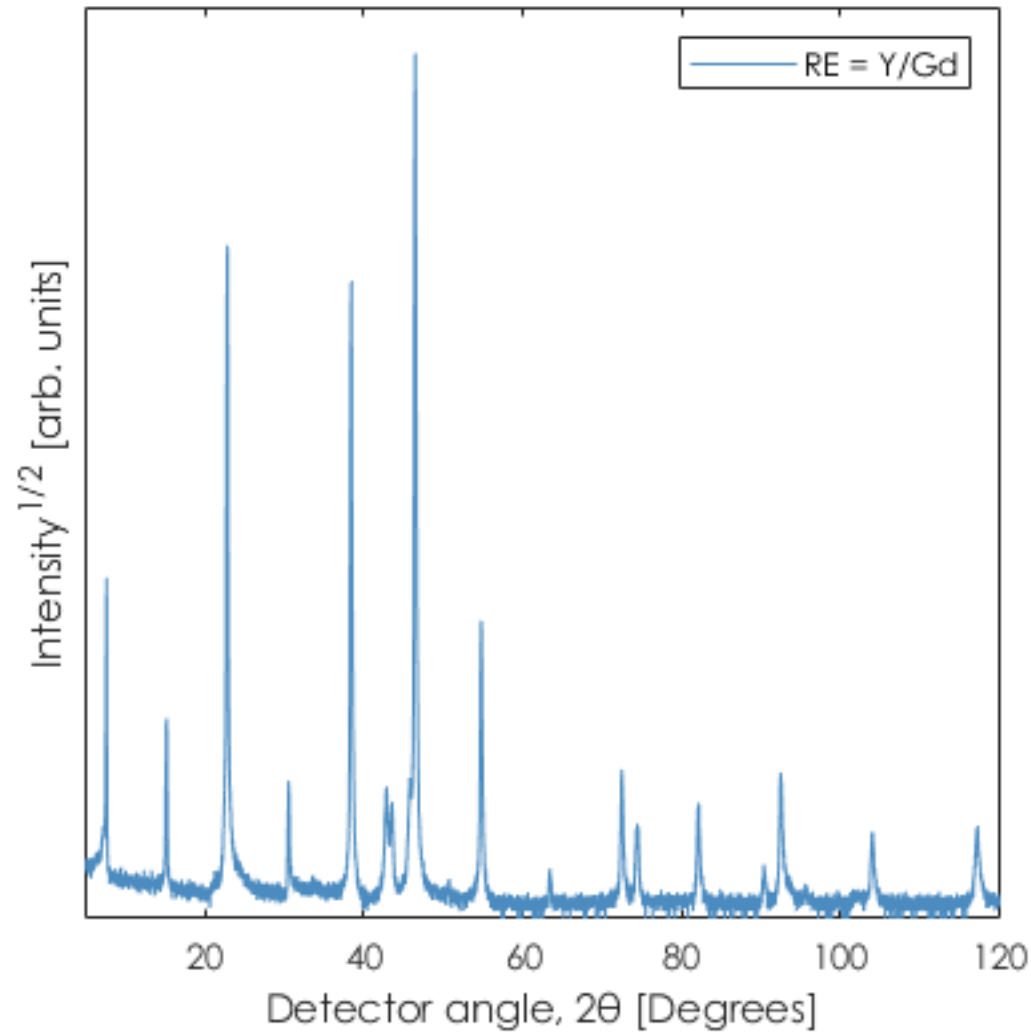
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X-ray Diffraction Characterisation (XRD)

- XRD collected using a Malvern Panalytical Empyrean system.
- Scans utilise a 3-axis stage and a 1D detector.
- Uses a Cu tube ($\lambda_{\alpha_1} = 1.5406 \text{ \AA}$) source, with Cu K_{β} removed with a Ni filter.

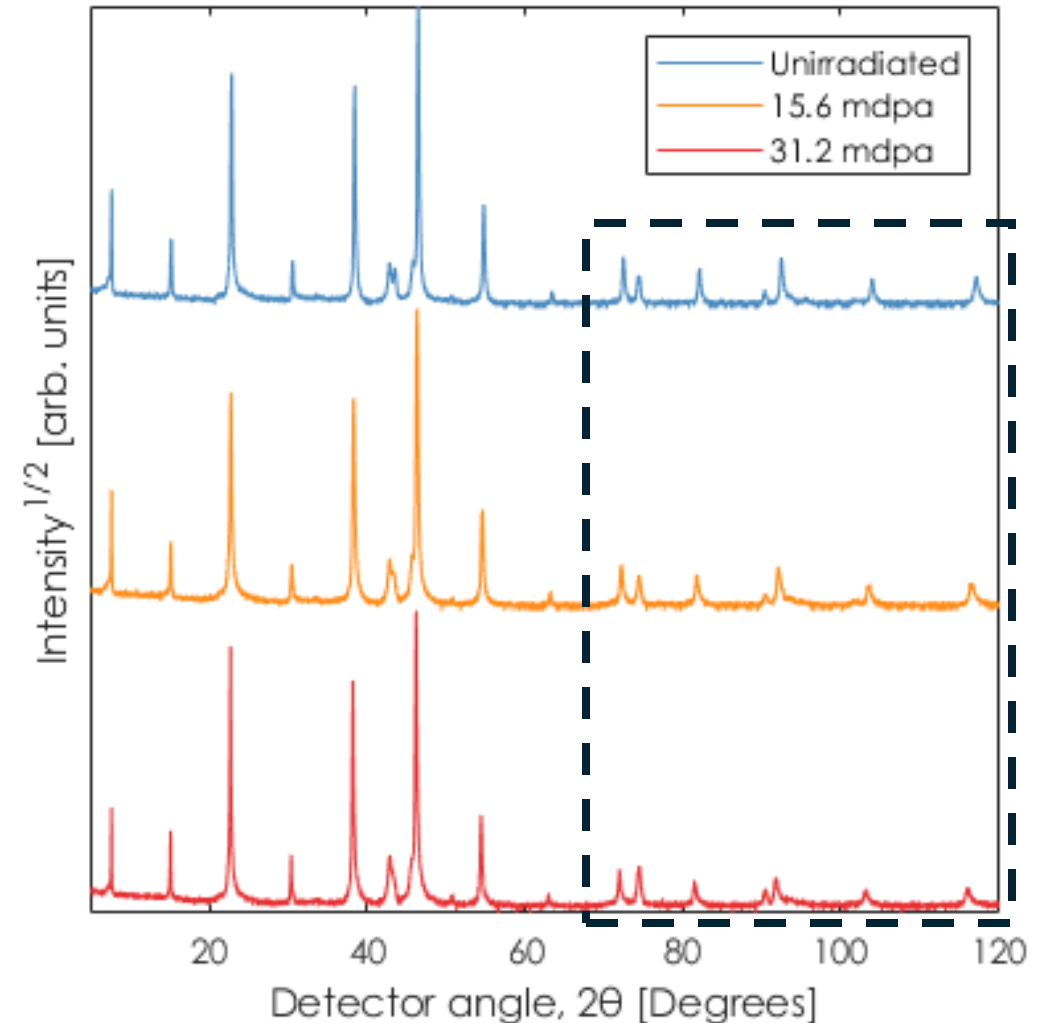


XRD: Pristine Material



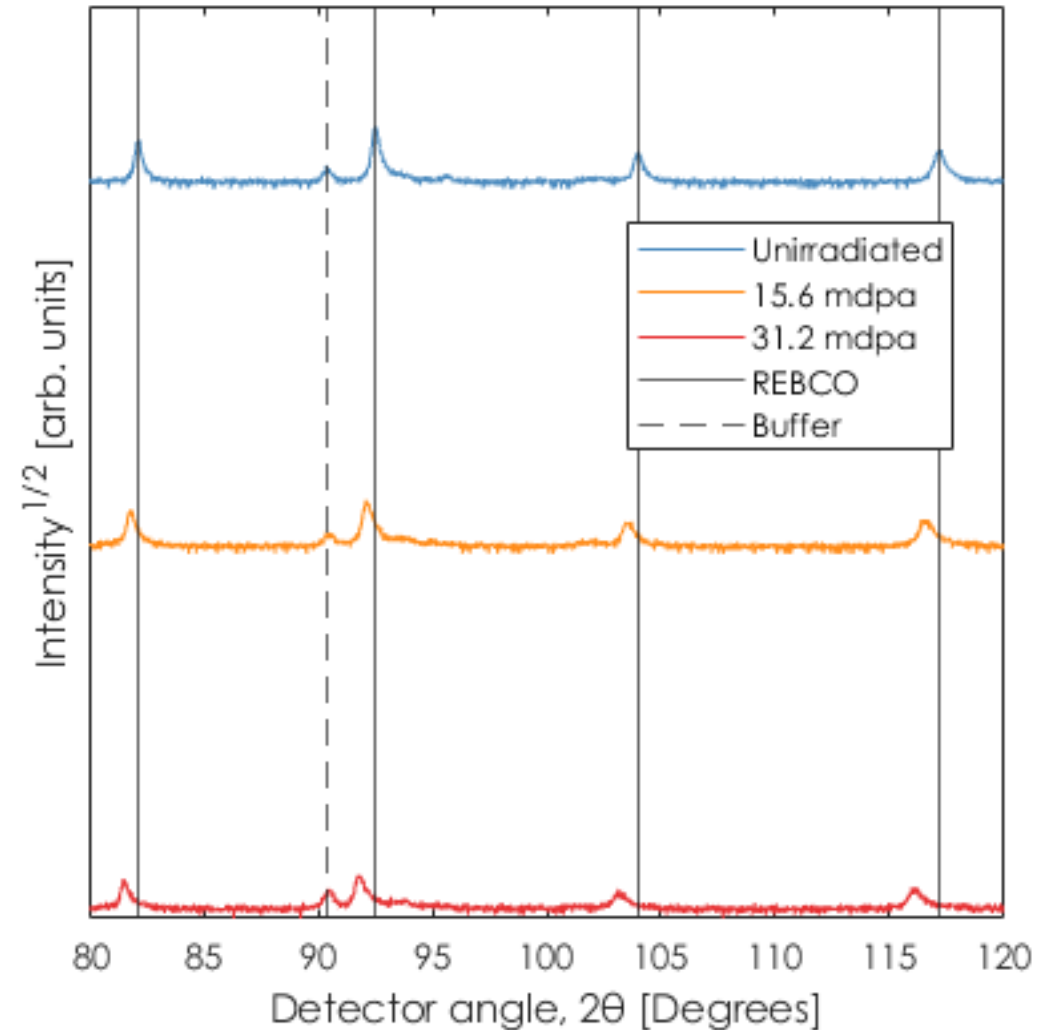
XRD: 2 MeV He⁺ Characterisation

- All samples had an increase in c-axis following ion irradiation, proportional to mdpa.



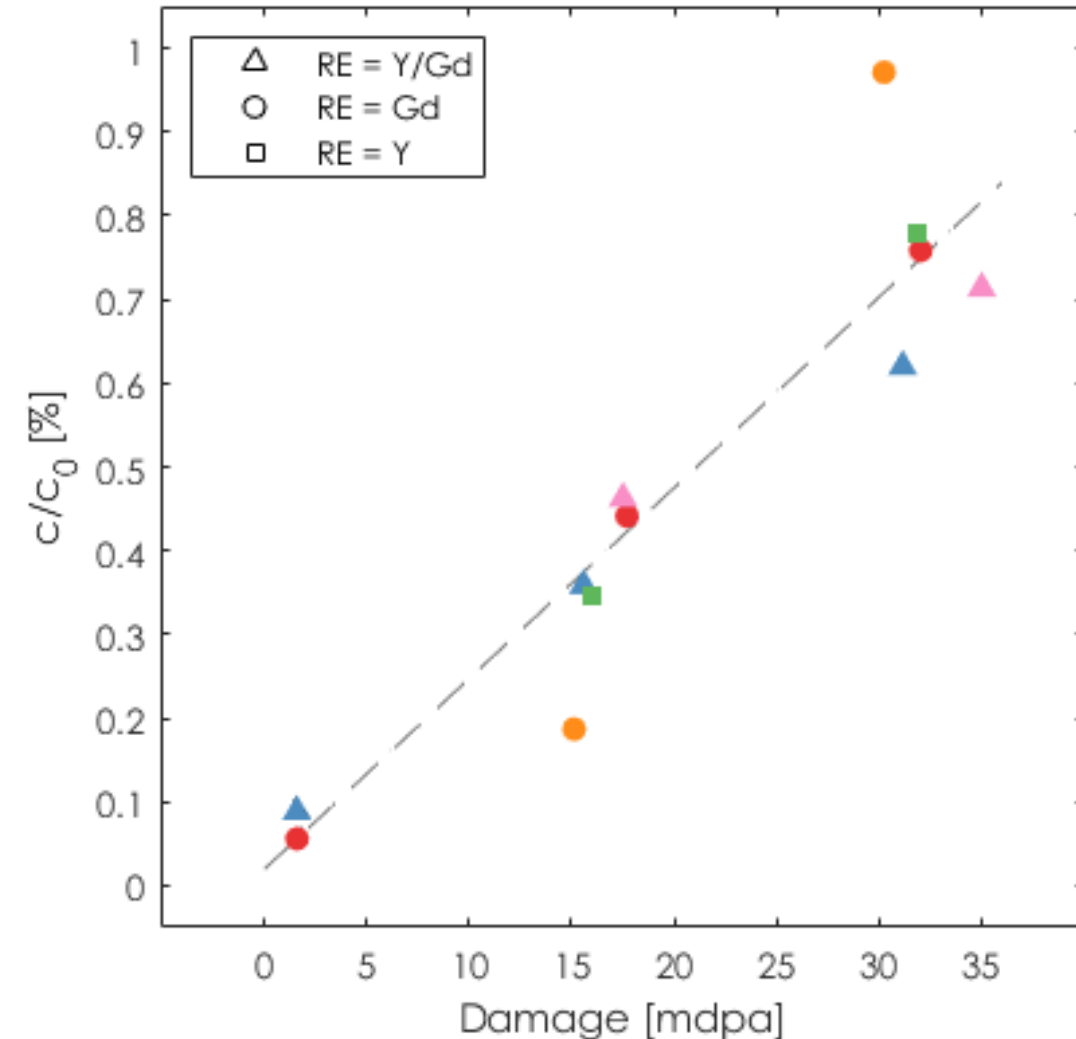
XRD: 2 MeV He⁺ Characterisation

- All samples had an increase in c-axis following ion irradiation, proportional to mdpa.
- Notably no evidence of change to the buffer layer.



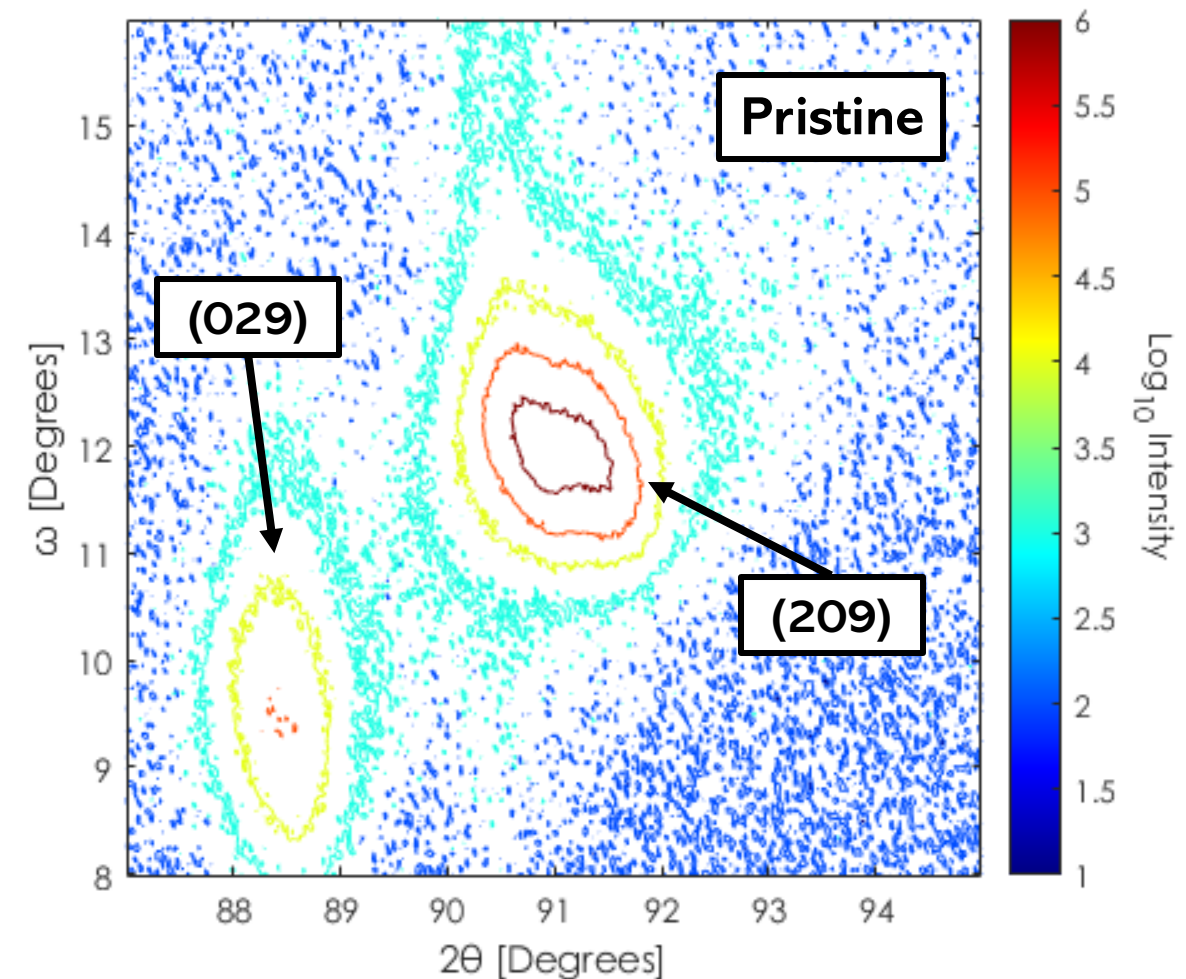
XRD: 2 MeV He⁺ Characterisation

- Increase in c-axis following ion irradiation largely invariant with RE, manufacturer, or thickness.
- ~ 1% increase in 40-45 mdpa



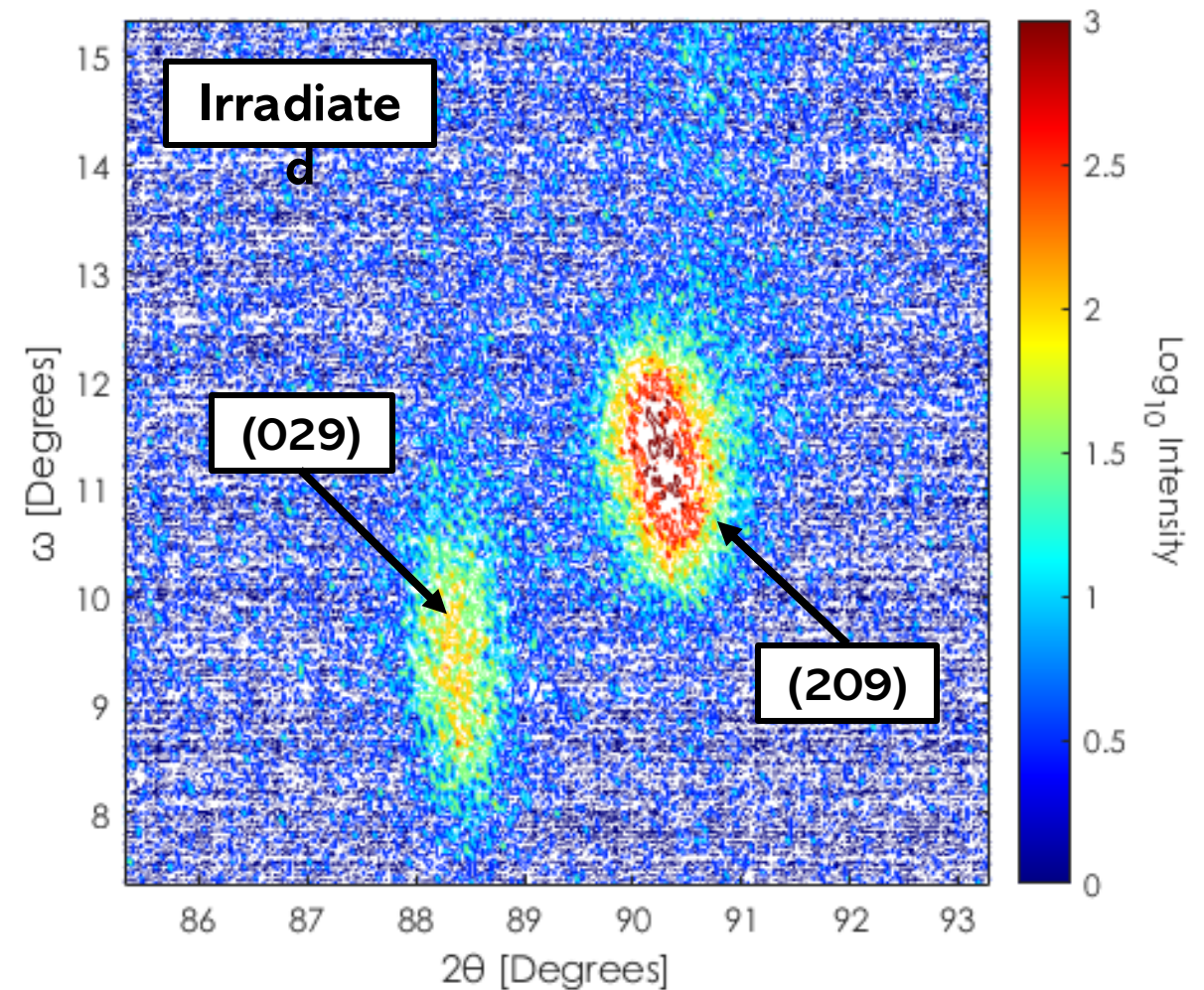
XRD: 2 MeV He⁺ Characterisation

- ω - 2θ scans measure the a and b axis lattice parameters.
- Variation between tapes more pronounced and complicated by different buffer peaks.



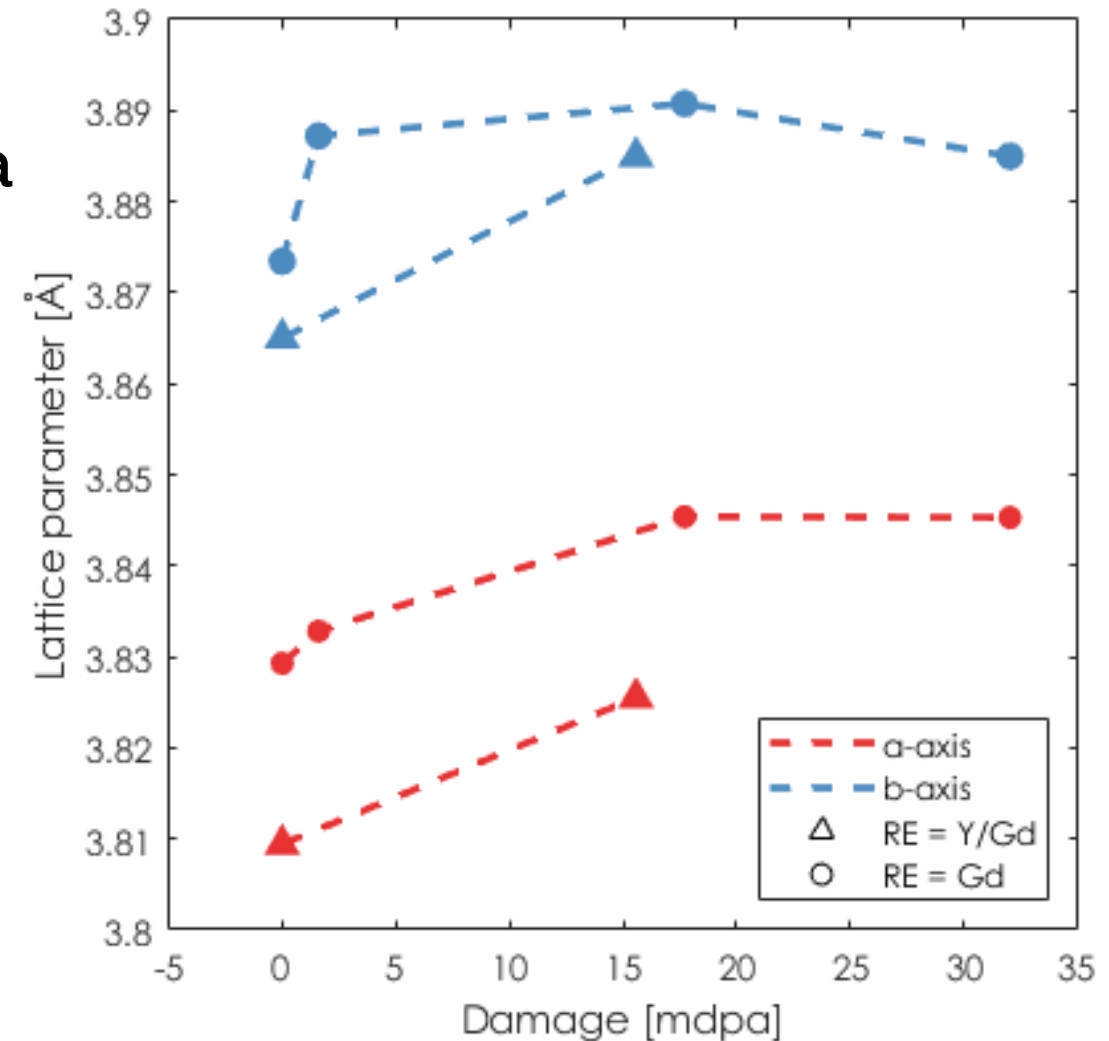
XRD: 2 MeV He⁺ Characterisation

- Characterisation is complicated after He⁺ irradiation, with a/b peaks less well defined and less intense.



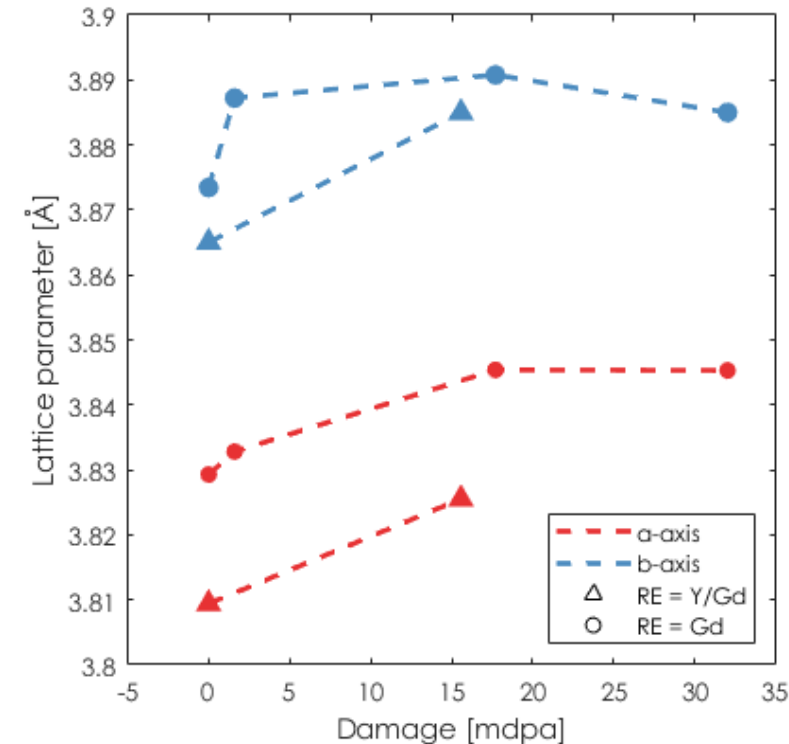
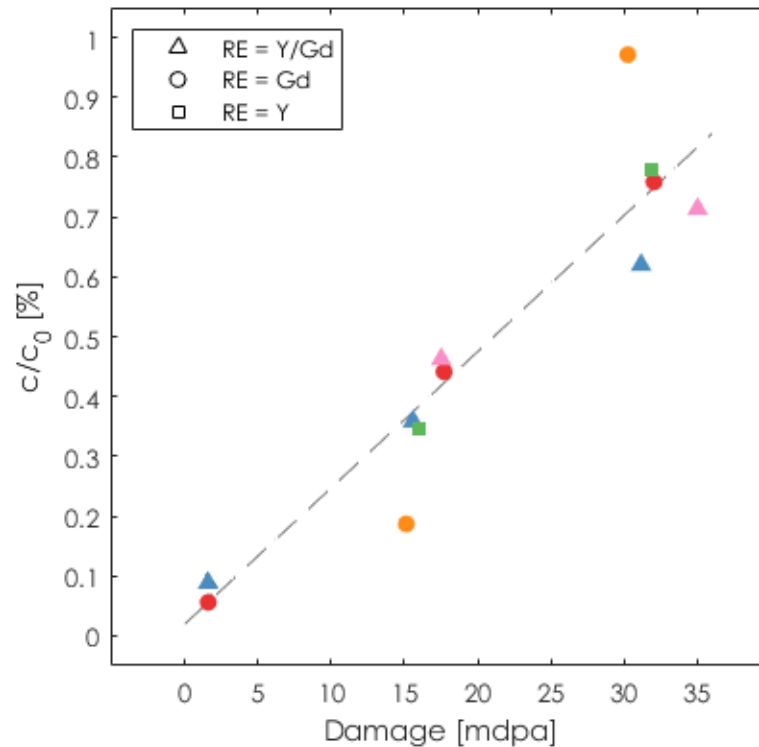
XRD: 2 MeV He⁺ Characterisation

- Preliminary data set suggests a plateau following irradiation, unlike the c parameter.



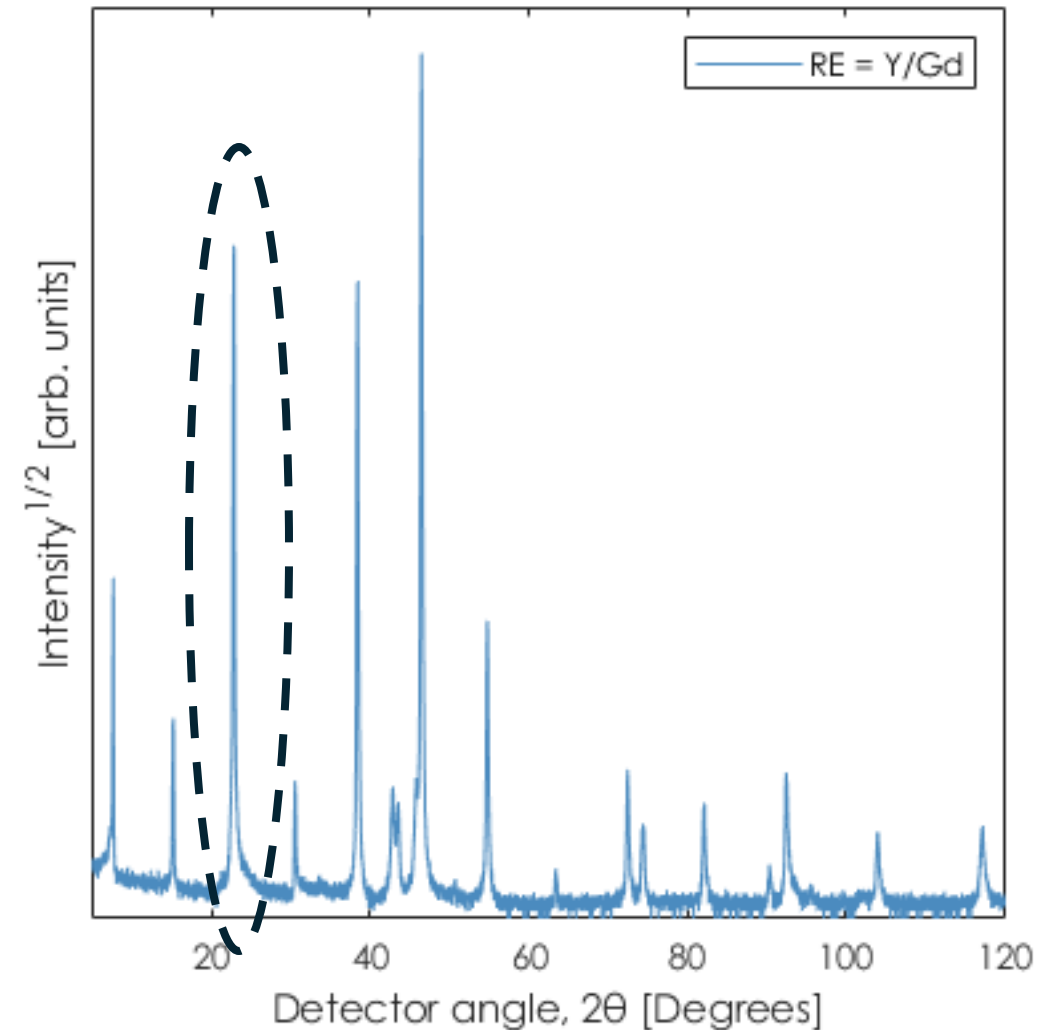
XRD: 2 MeV He⁺ Characterisation

- Working towards a larger sample of RE, manufacturers, and mdpa.



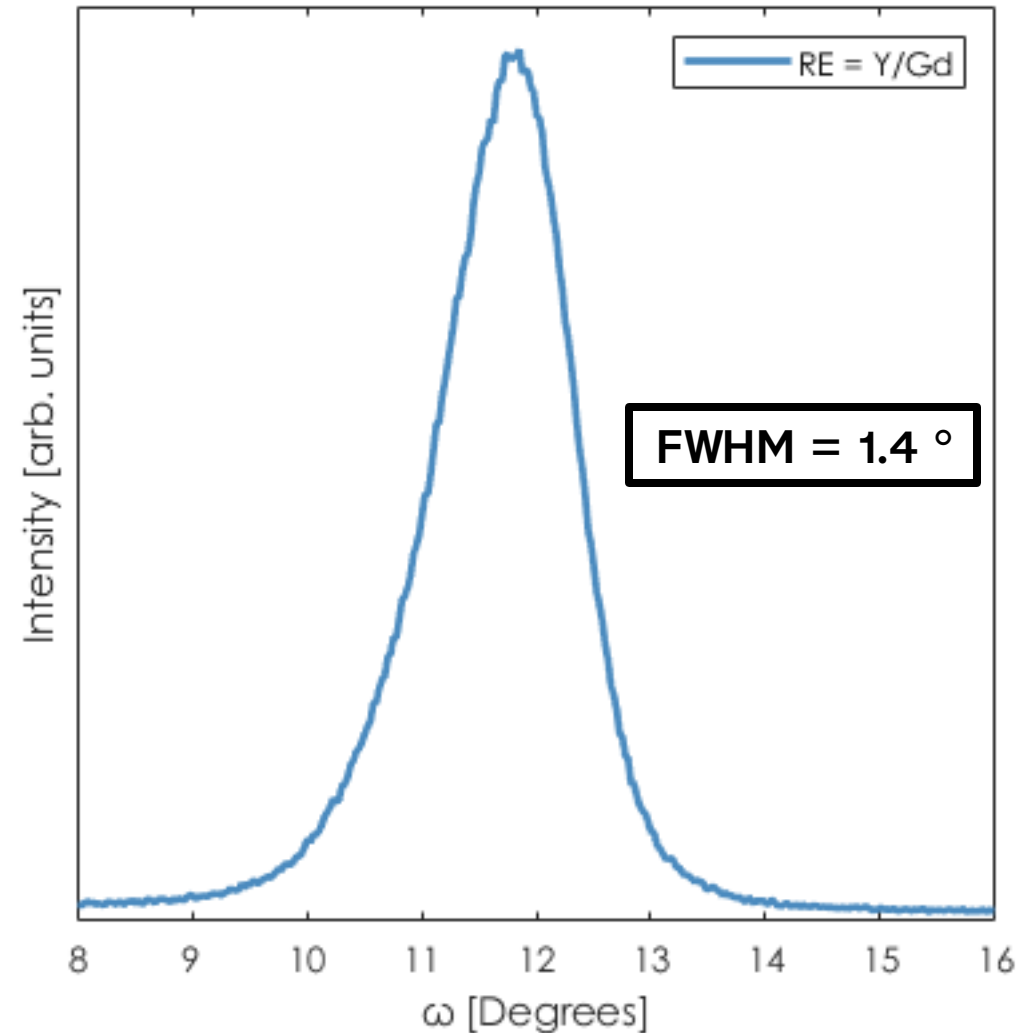
XRD: Pristine Material

- ω scans are performed by changing the incident angle offset (ω) away from Θ .
- The (003) Bragg reflection was chosen due to its intensity and distance from buffer peaks.



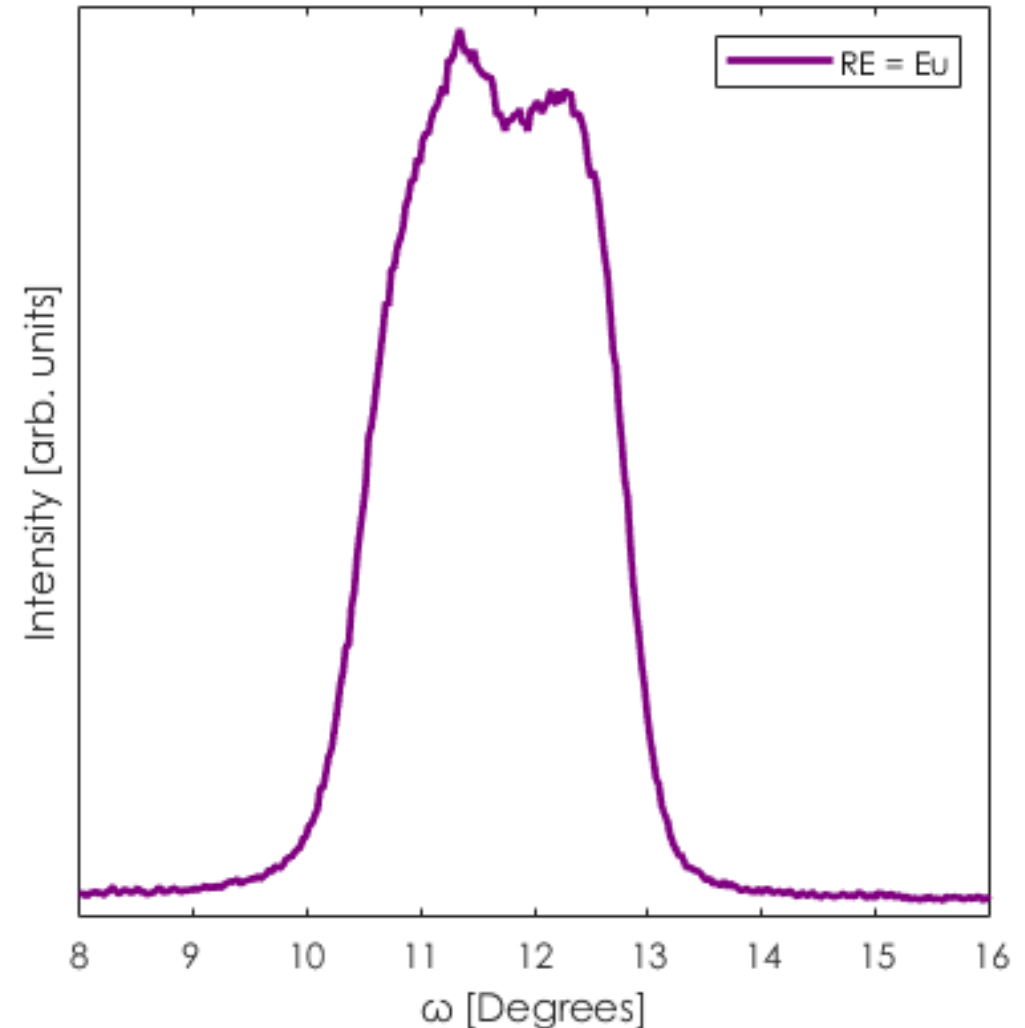
XRD: Pristine Material

- ω scans probe the distribution of crystallites within a layer, and thus crystal layer quality.
- Significant variation seen in ω scan FWHM between reels.



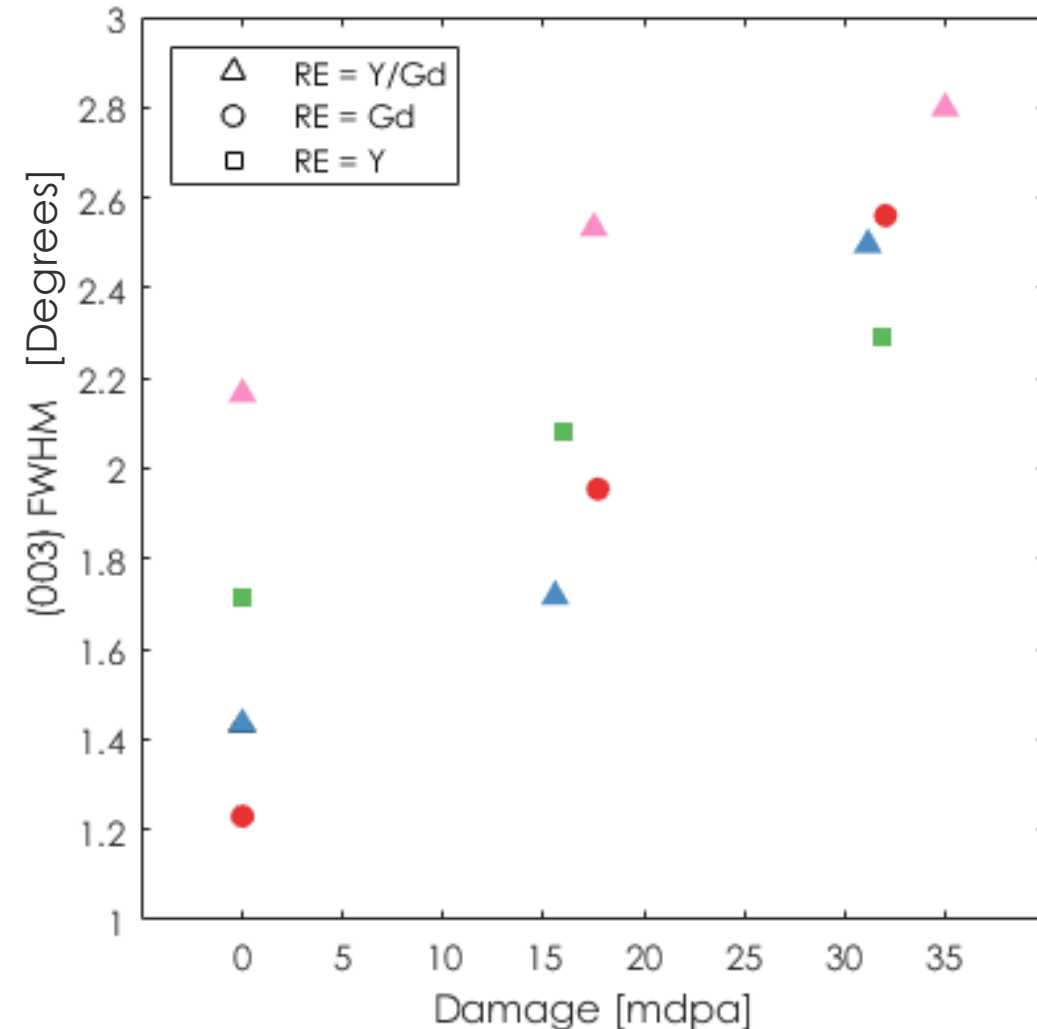
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XRD: 2 MeV He⁺ Characterisation

- ω scan FWHM much more sensitive to irradiation.
- Tapes with sharper initial FWHM seem less resilient to damage from 2 MeV He⁺.



Talk structure

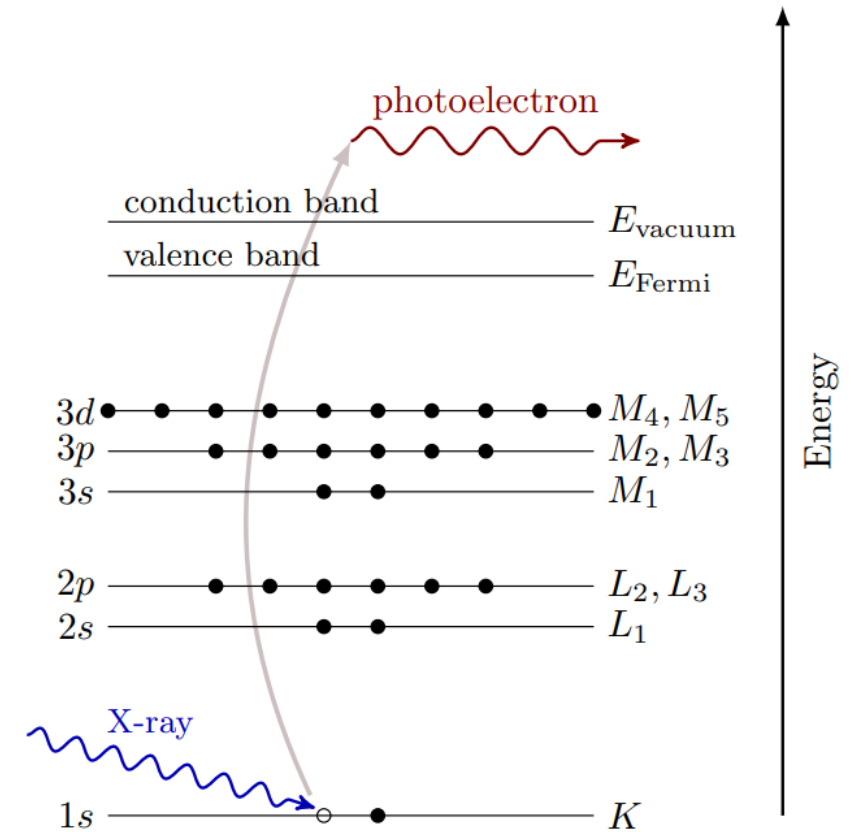
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X-ray Absorption Spectroscopy (XAS)



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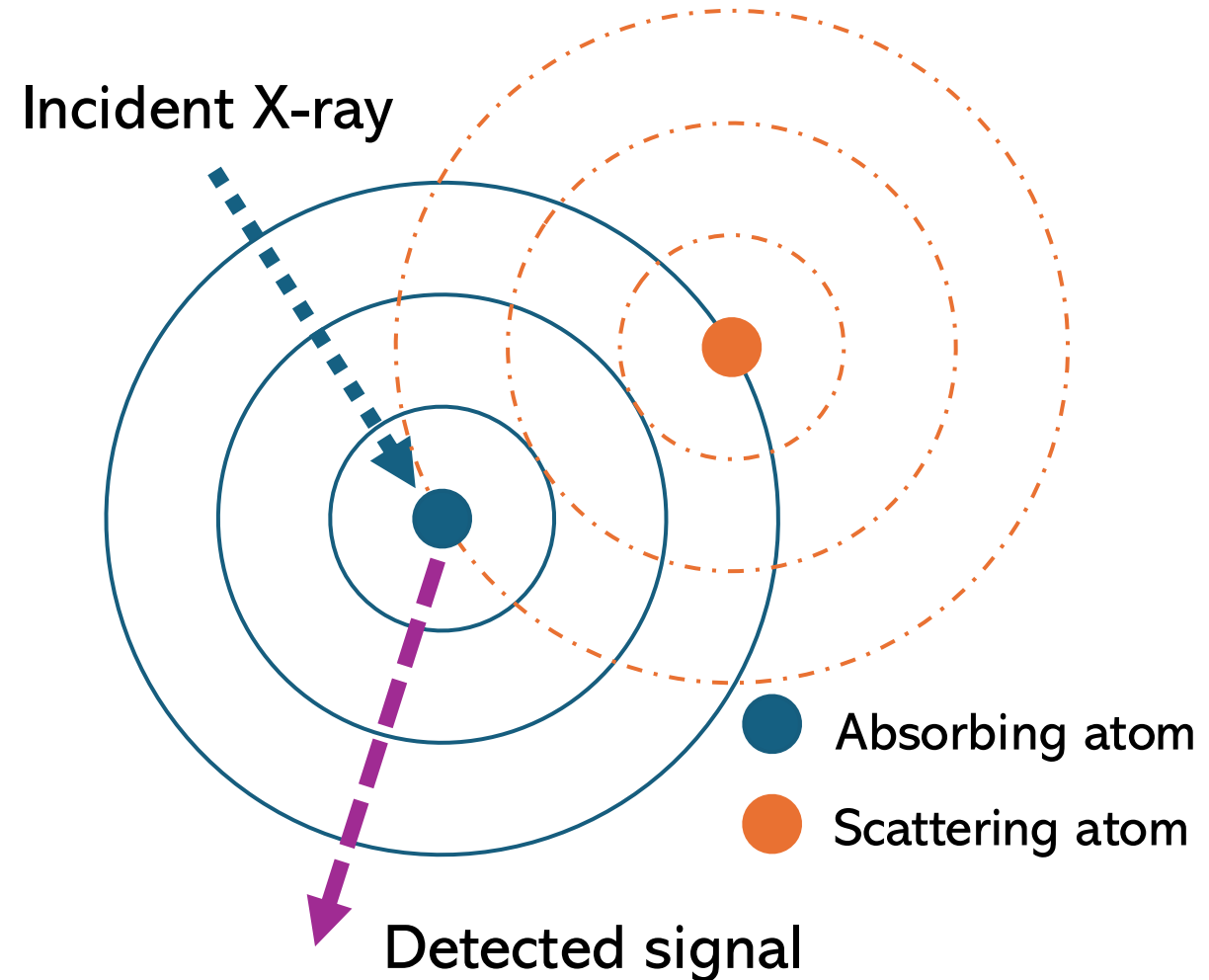
- Volume averaged technique.
- Provides an elementally specific probe via electronic excitation.
- Contains *localised* information independent of diffraction based (crystal structure) probes.



[3] – Newville, M., *Reviews in Mineralogy and Geochemistry*, 2014, Vol.78 (1), p.33-74.

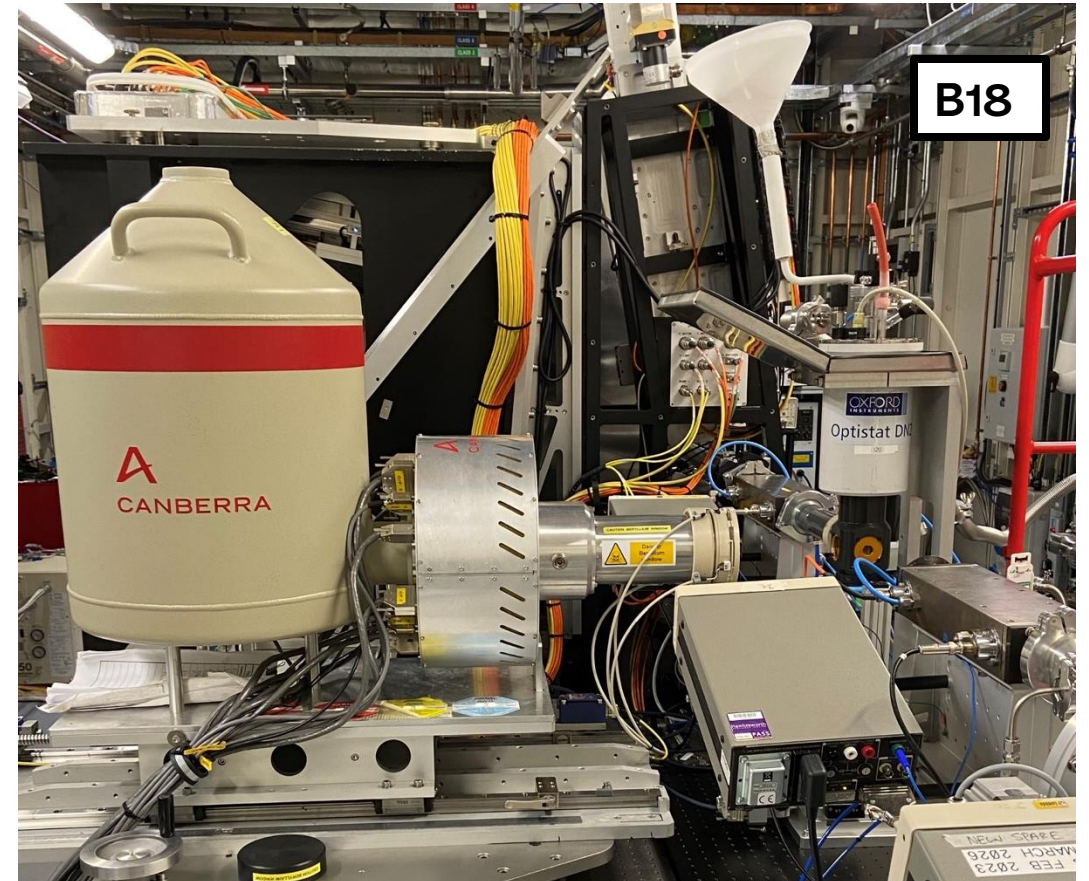
X-ray Absorption Spectroscopy (XAS)

- Excited photoelectrons scatter off neighbouring atoms.
- Scattering encodes the local structure into the detected X-ray fluorescence signal.



X-ray Absorption Spectroscopy (XAS)

- Fluorescence geometry used to avoid attenuating signal via the buffer and substrate layers.



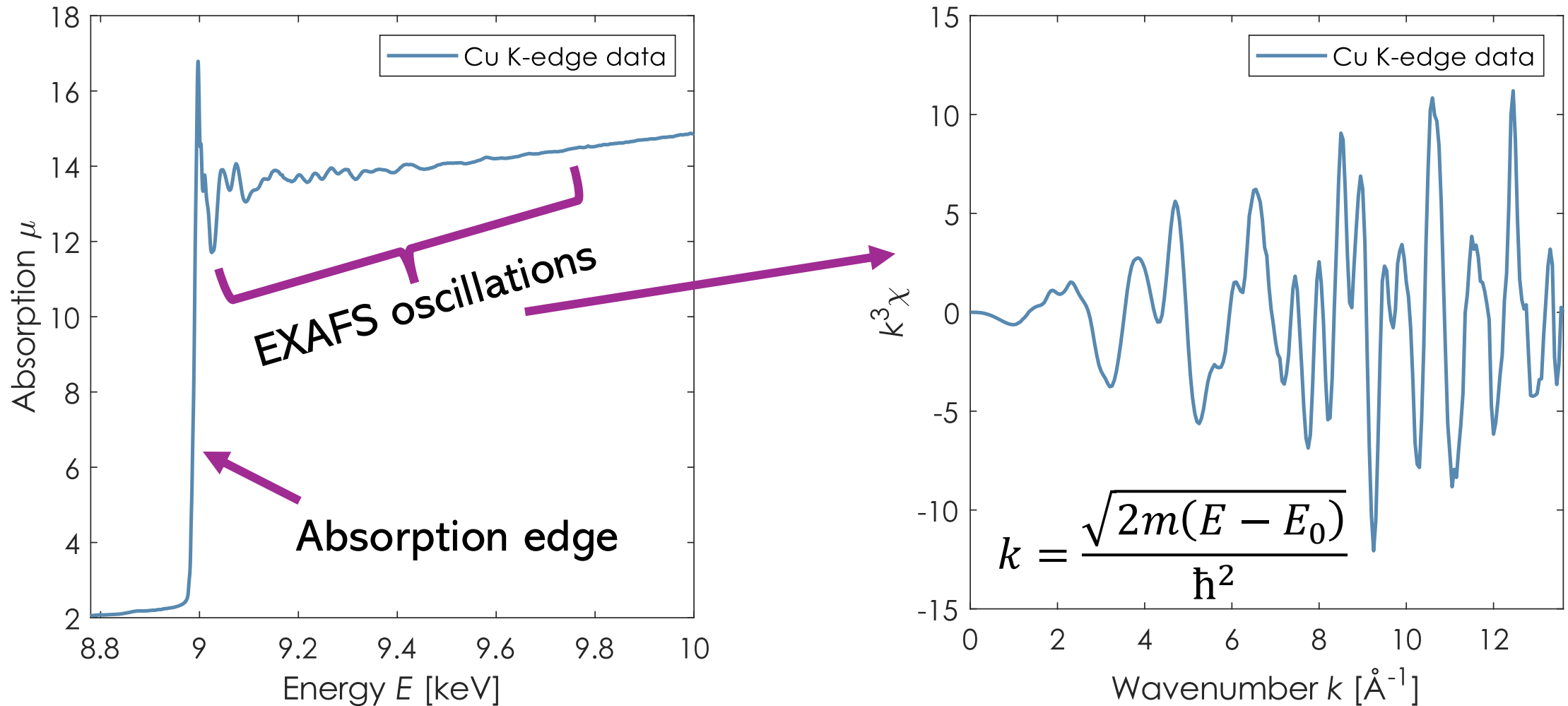
X-ray Absorption Spectroscopy (XAS)

Depending on experimental set-up X-ray absorption can provide:

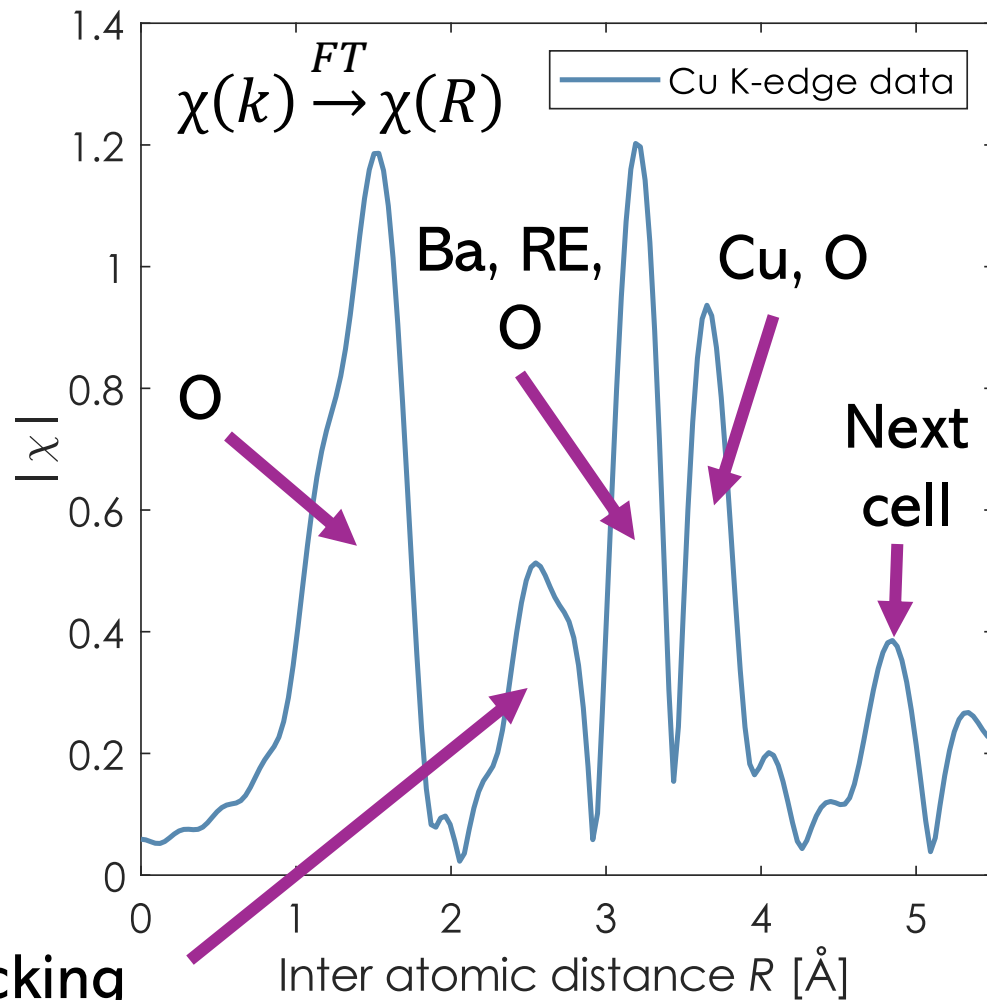
- Chemical information (HERFD-XANES)
- Structural information (EXAFS)

Both are inherently localized probes ($< 6 \text{ \AA}$), constrained by the finite core-hole recombination lifetime.

X-ray Absorption Spectroscopy (XAS)



X-ray Absorption Spectroscopy (XAS)



Stacking
Faults

- EXAFS analysis is sensitive to defects, vacancies, and interstitials.
- As a scattering technique there is no requirement for long range order and so is sensitive to diffuse phenomena.
- Motivates high mdpa levels.

X-ray Absorption Spectroscopy (XAS)

- EXAFS data is analysed with an empirical equation, with each of the j scattering paths modelled using a REBCO CIF input.

S_0^2 – Amplitude reduction factor

f – Scattering amplitude

N – Coordination number

λ – Photoelectron mean free path

R – Inter-atomic distance

σ^2 – Debye-Waller factor (disorder)

δ – Phase difference

$$\chi(k) = \sum_j \frac{S_0^2 N_j f_j(k) e^{-2R_j/\lambda(k)} e^{-2k\sigma_j^2}}{kR_j^2} \sin[2kR_j + \delta_j(k)]$$

[3] – Newville, M., *Reviews in Mineralogy and Geochemistry*, 2014, Vol.78 (1), p.33-74.

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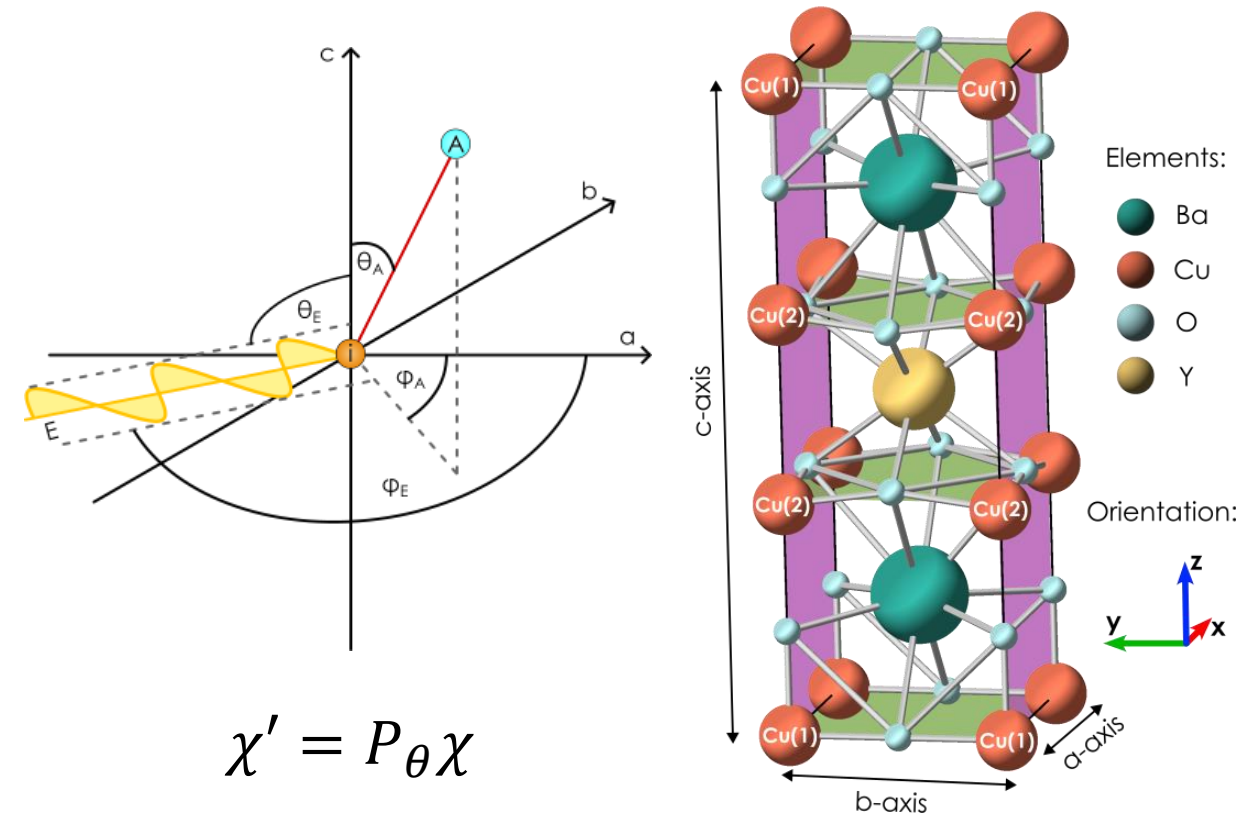
DFT

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X-ray Absorption Spectroscopy (XAS)

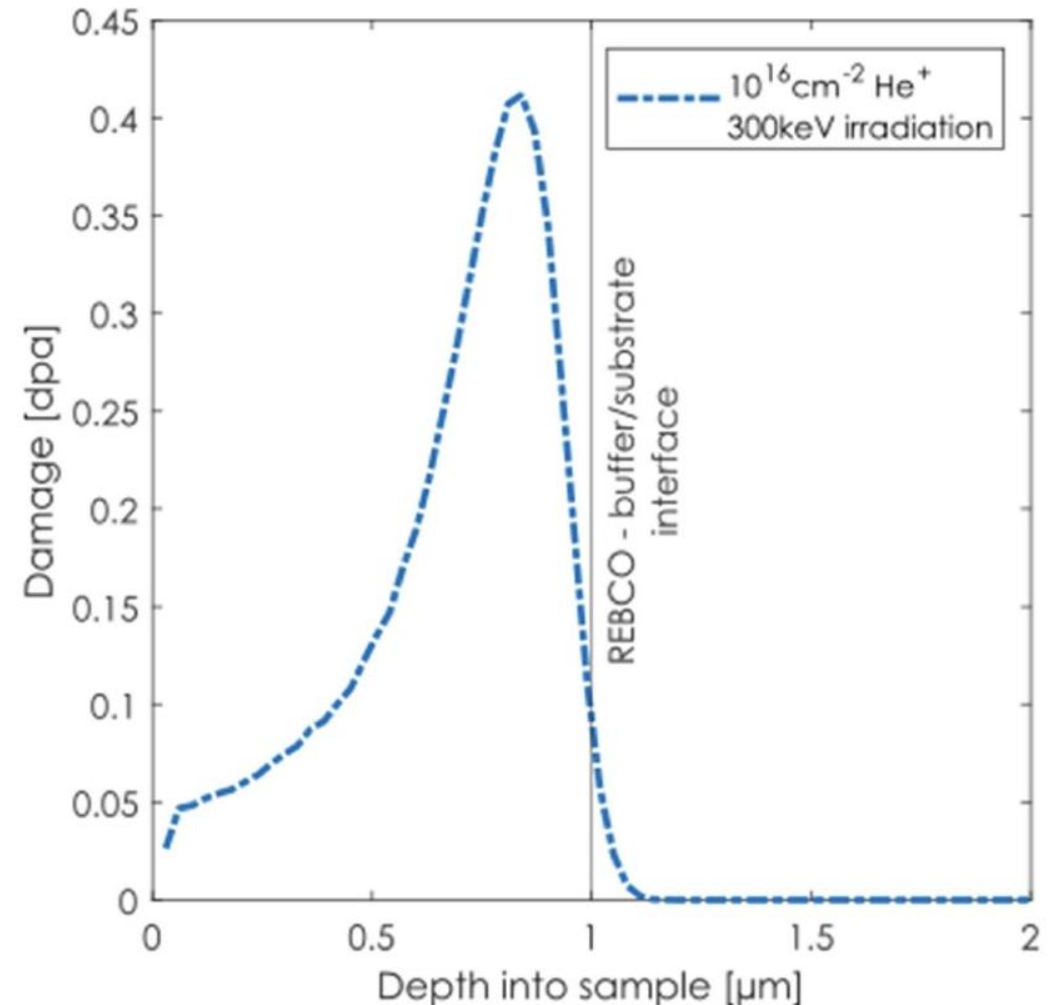
- Rotating the sample enables preferential scattering in the a, b, or c direction.
- In structurally anisotropic HTS, this allows polarised XAS studies.
- Note twinning convolutes a/b.



[4] – Lewis, J.C. *et al.*, *Superconductor Science & Technology*, 2025, Vol.38, 015022.

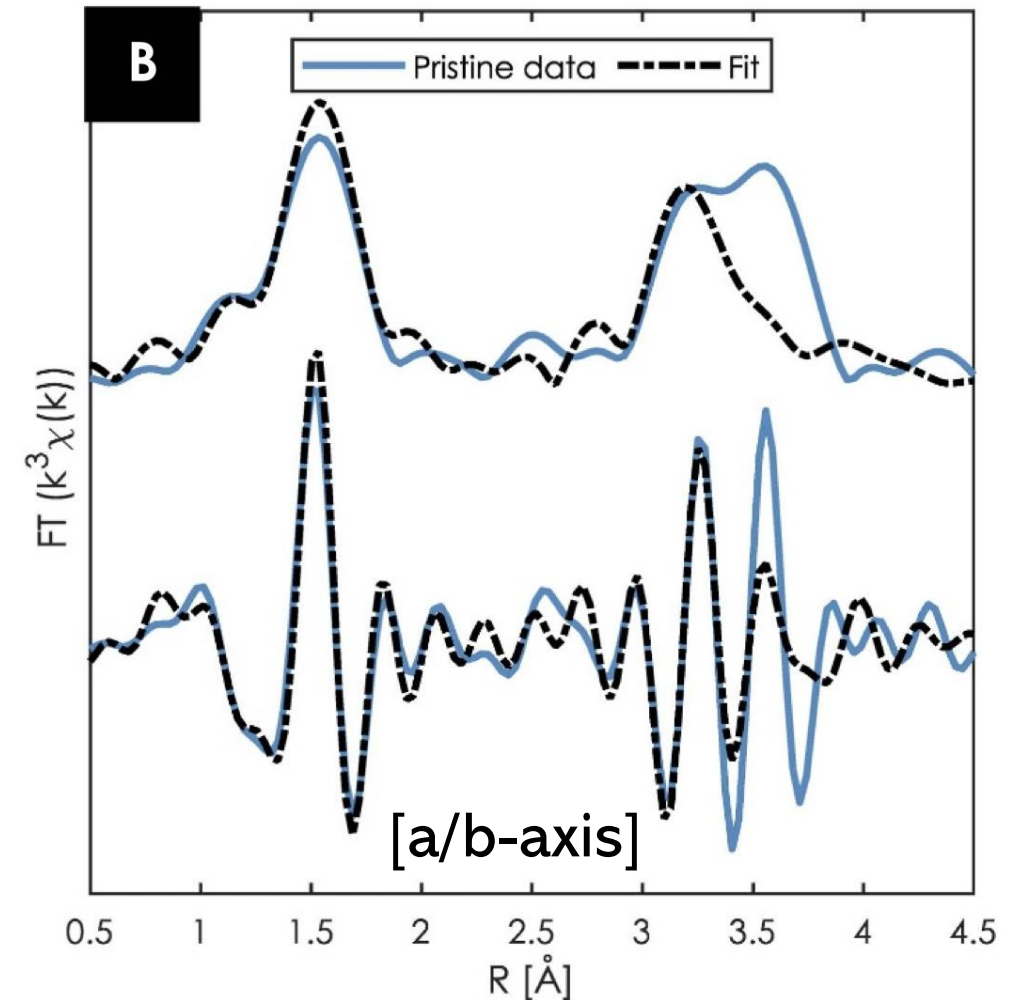
Cu K EXAFS: 300 keV He⁺ Study

- 300 keV He⁺ places the Bragg peak in the REBCO layer.
- Expect a continuous variation in damage and thus in the Cu local environments.



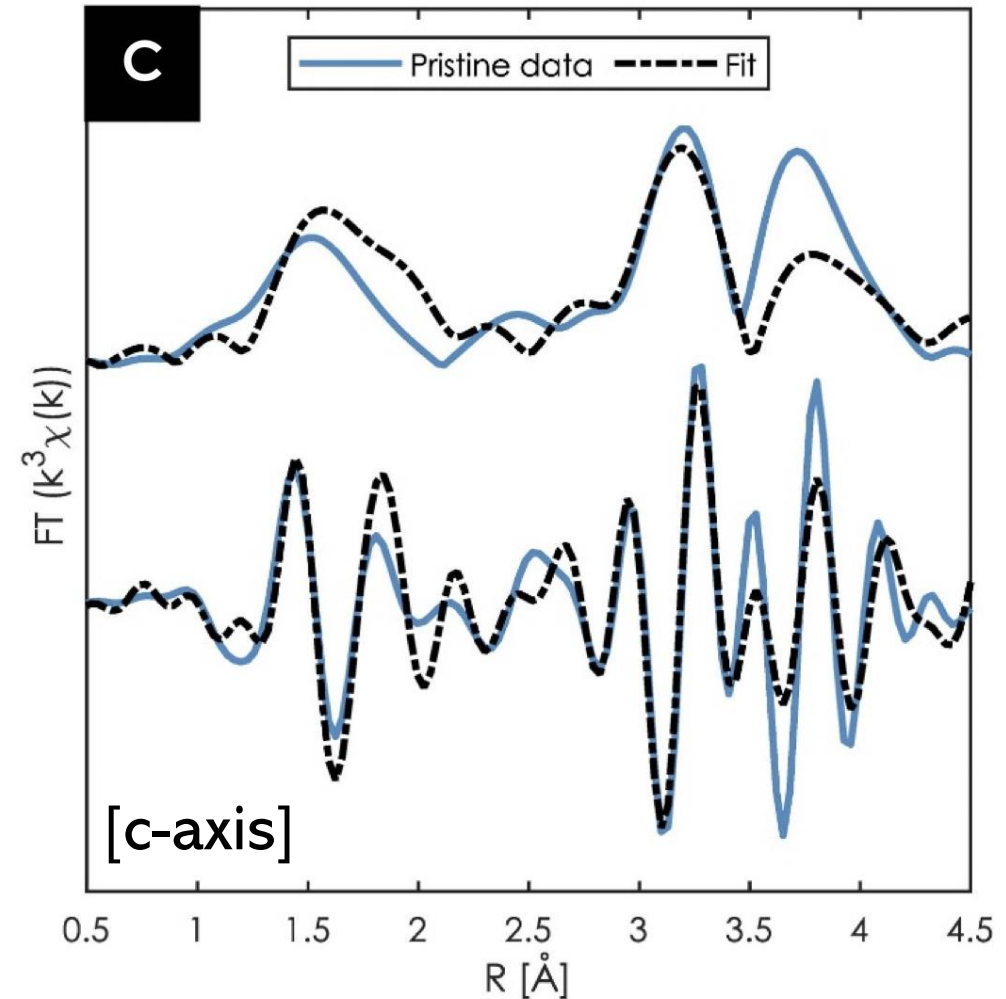
Cu K EXAFS: 300 keV He⁺ Study

- Using SuperPower tape with RE = Gd/Y, the pristine local structure was well modelled by REBCO-123 along a/b.



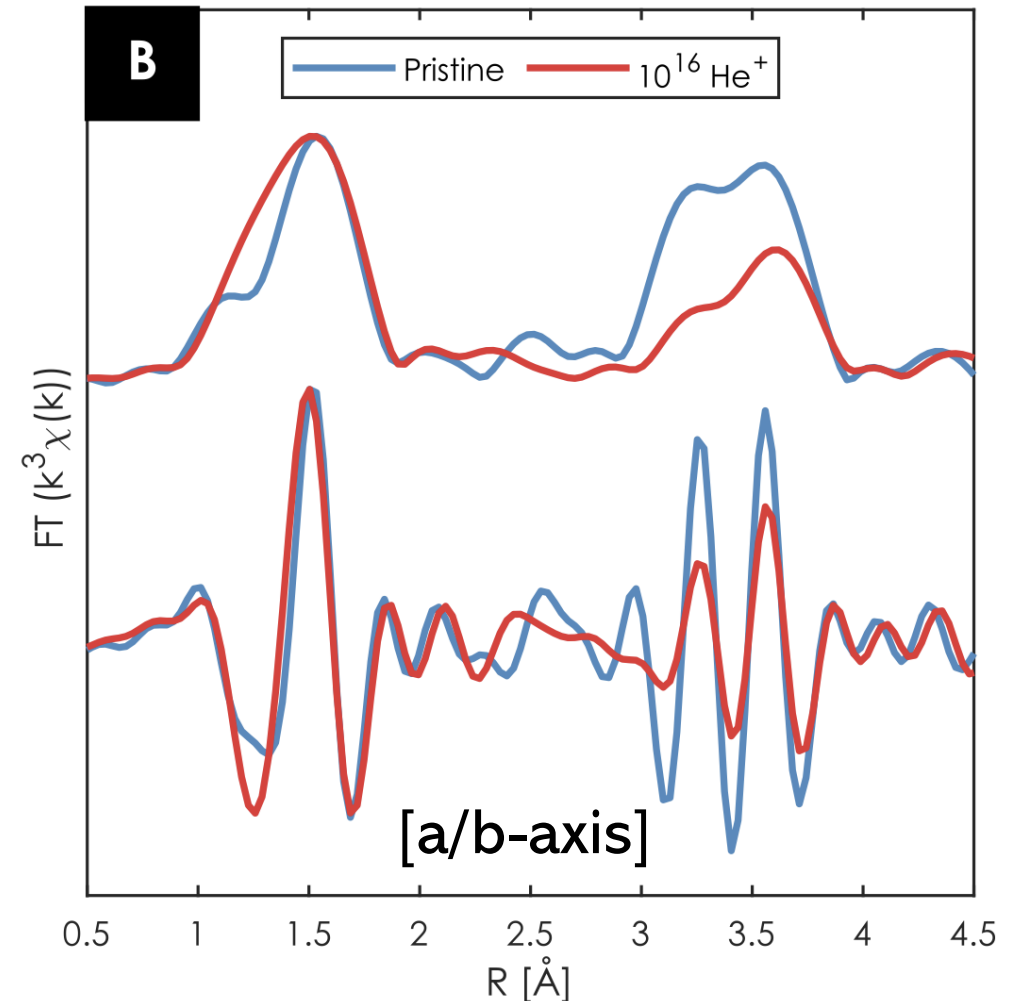
Cu K EXAFS: 300 keV He⁺ Study

- The c-axis pristine local structure was less well modelled.
- Disagreement noted around the Cu1-O4 distance.



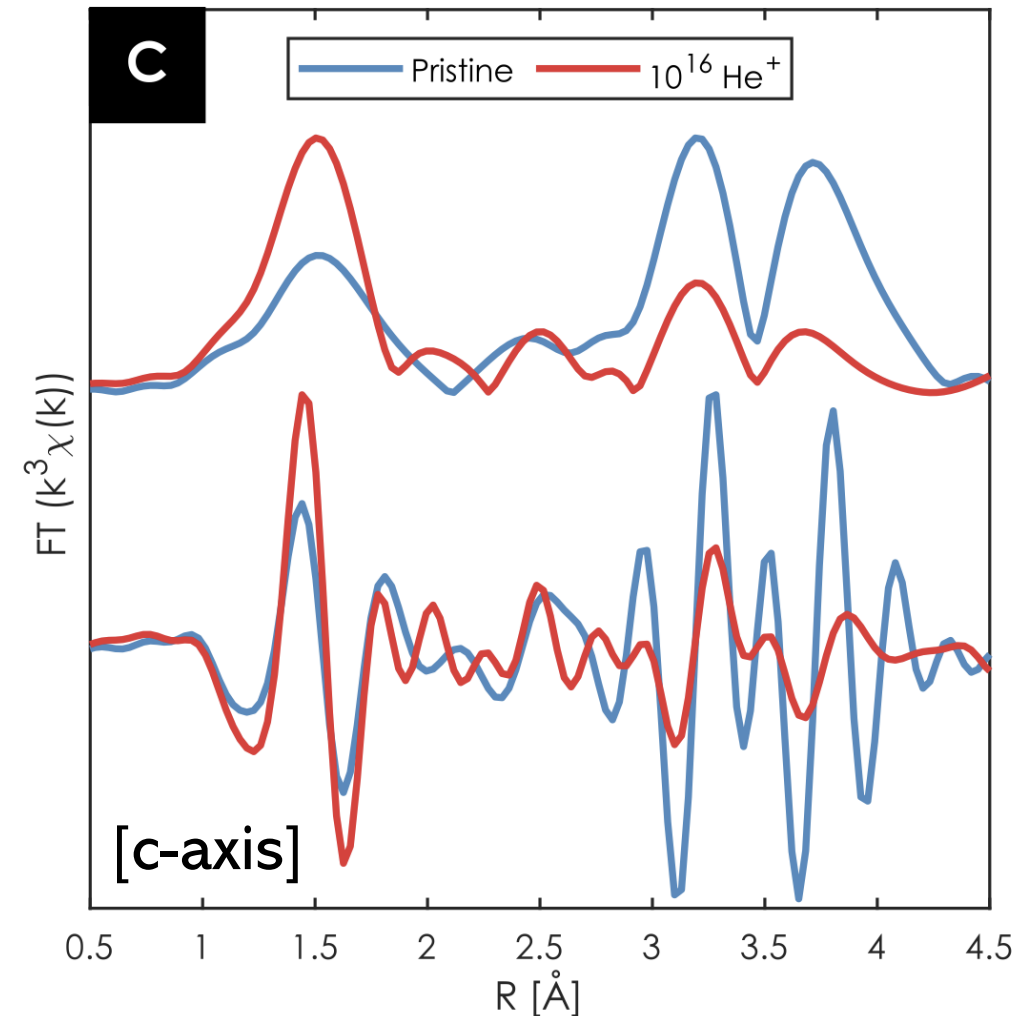
Cu K EXAFS: 300 keV He⁺ Study

- With irradiation, the a/b lattice changed predominantly above 3 Å.
- Indicates the first Cu-O shell is largely unaffected.



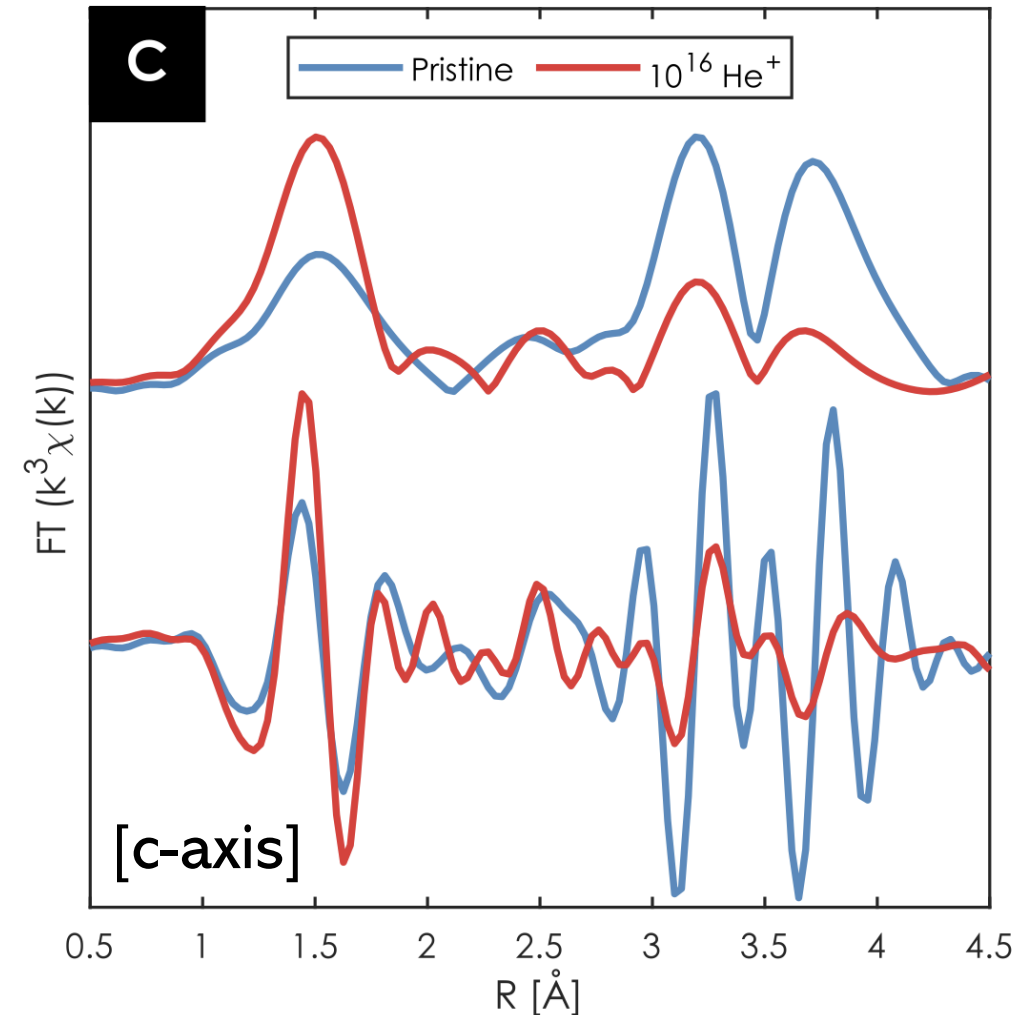
Cu K EXAFS: 300 keV He⁺ Study

- The c-direction is much more sensitive.
- Changes to the nearest Cu-O position and further scattering paths implied.



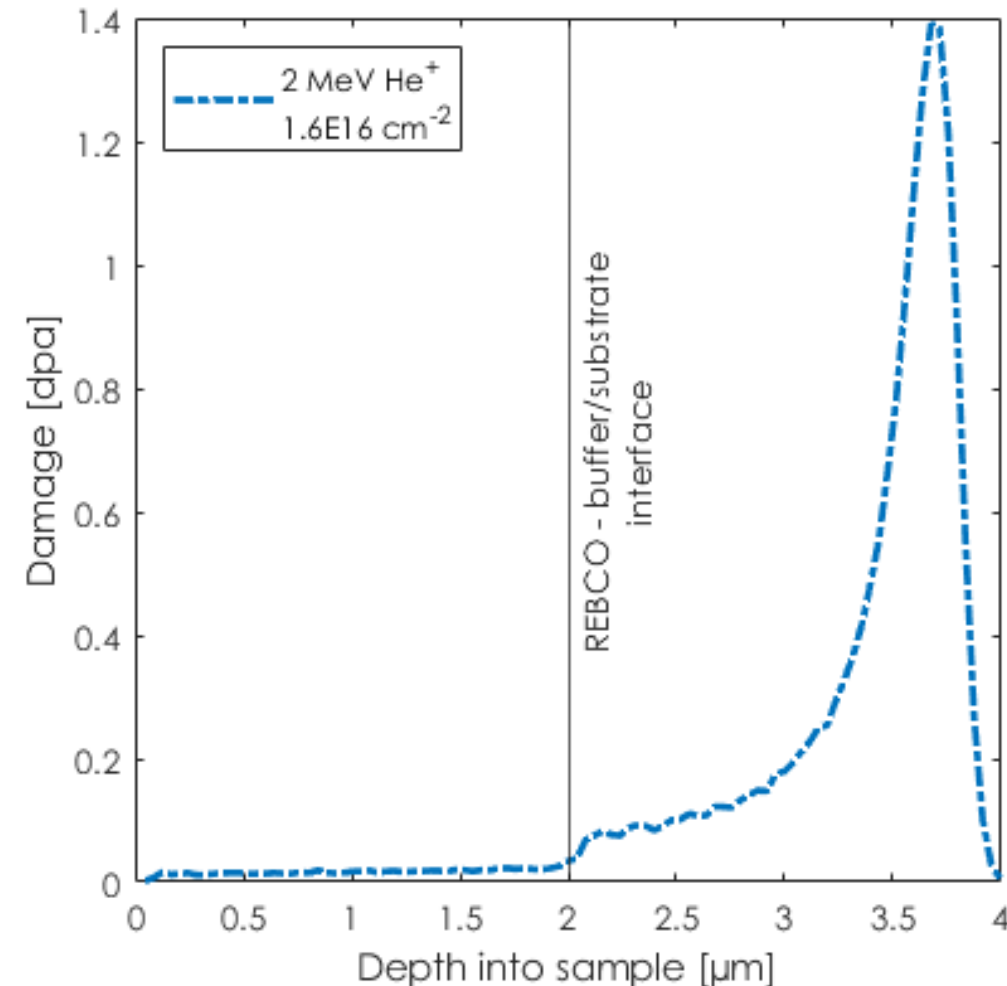
Cu K EXAFS: 300 keV He⁺ Study

- 300 keV study limited by the inhomogeneity of the damage profile to a qualitative analysis of spectral features.
- Shifts do not disagree with FP formation, but not conclusive.



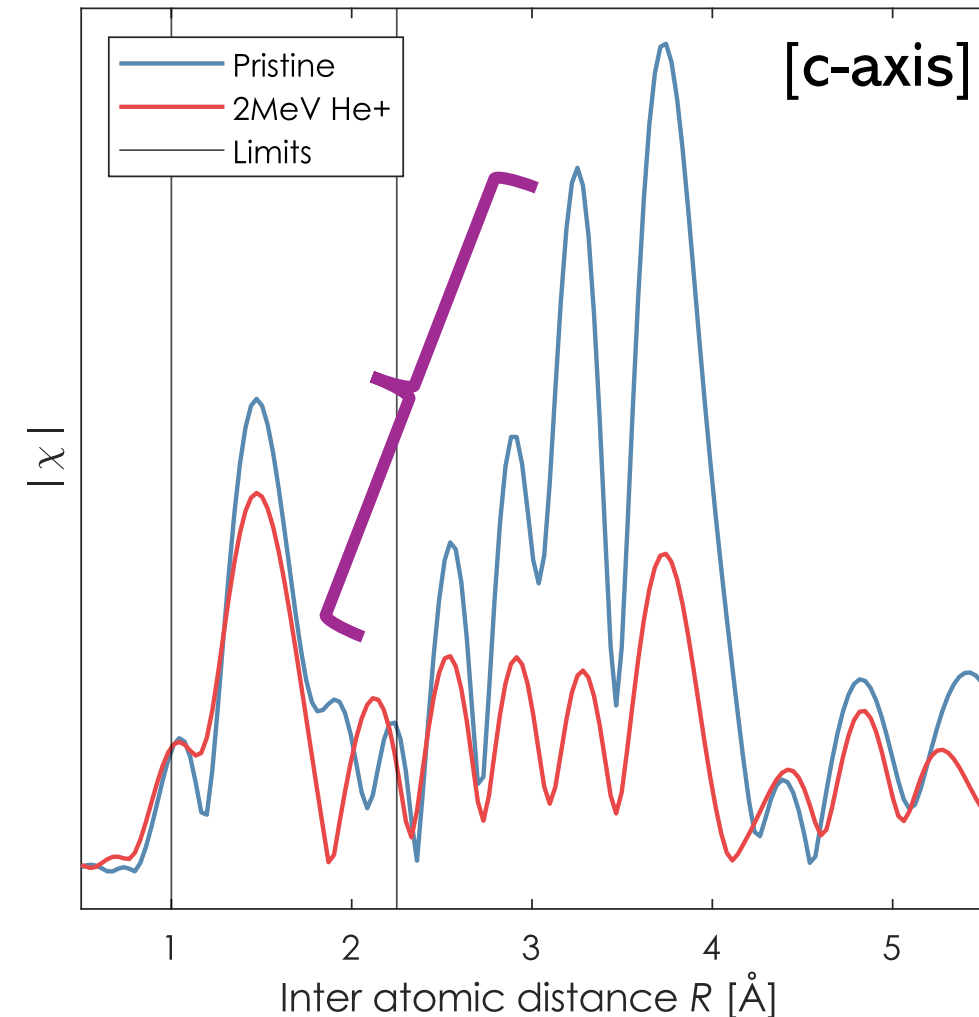
Cu K EXAFS: 2 MeV He⁺ Study

- To isolate defects for fitting and analysis, a more uniform damage profile was chosen.
- 2 MeV He⁺ was used to irradiate a GdBCO R&D tape.



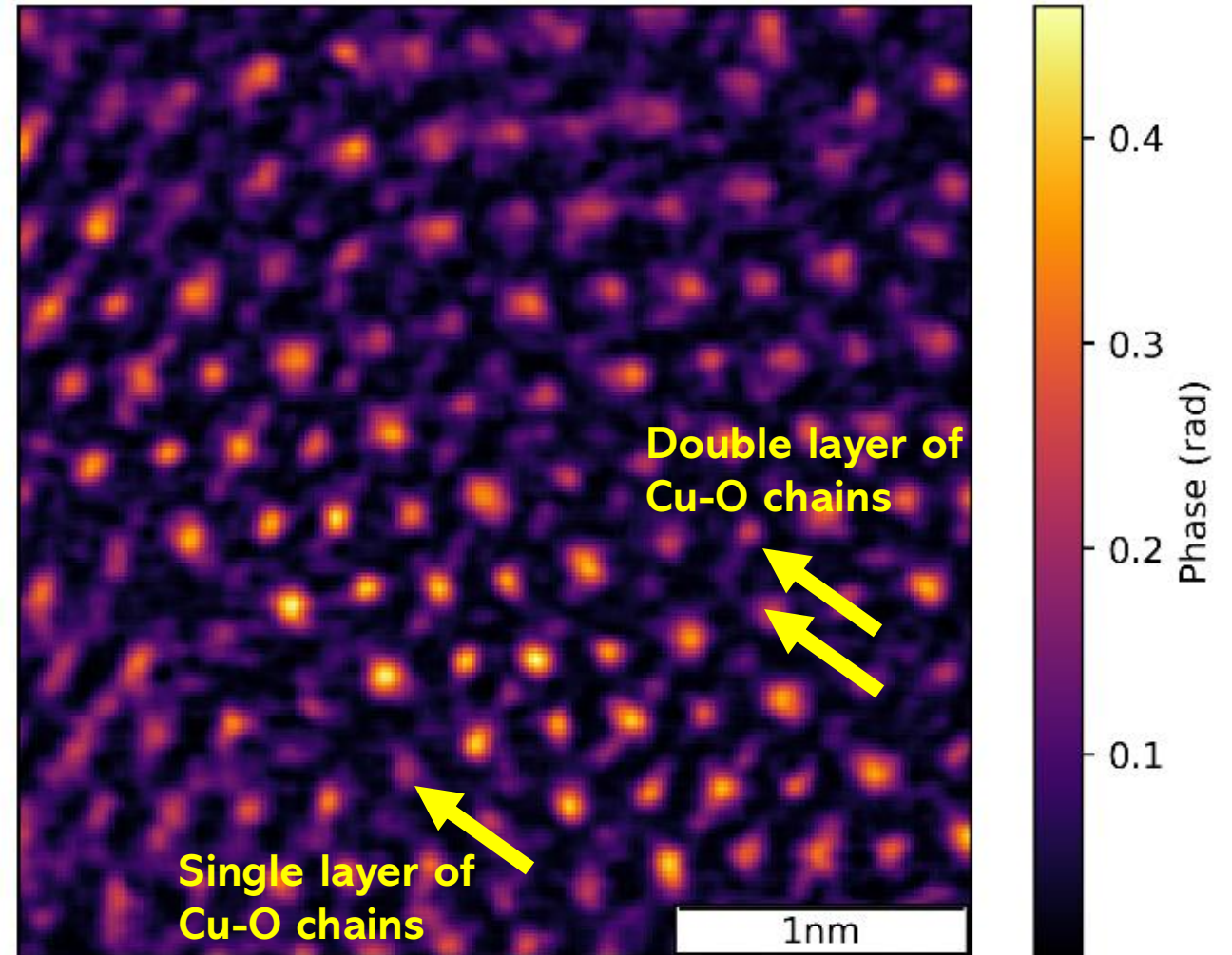
Cu K EXAFS: 2 MeV He⁺ Study

- Impact of irradiation most evident along the c-axis.
- Spectral shift suggests change in local structure with irradiation.



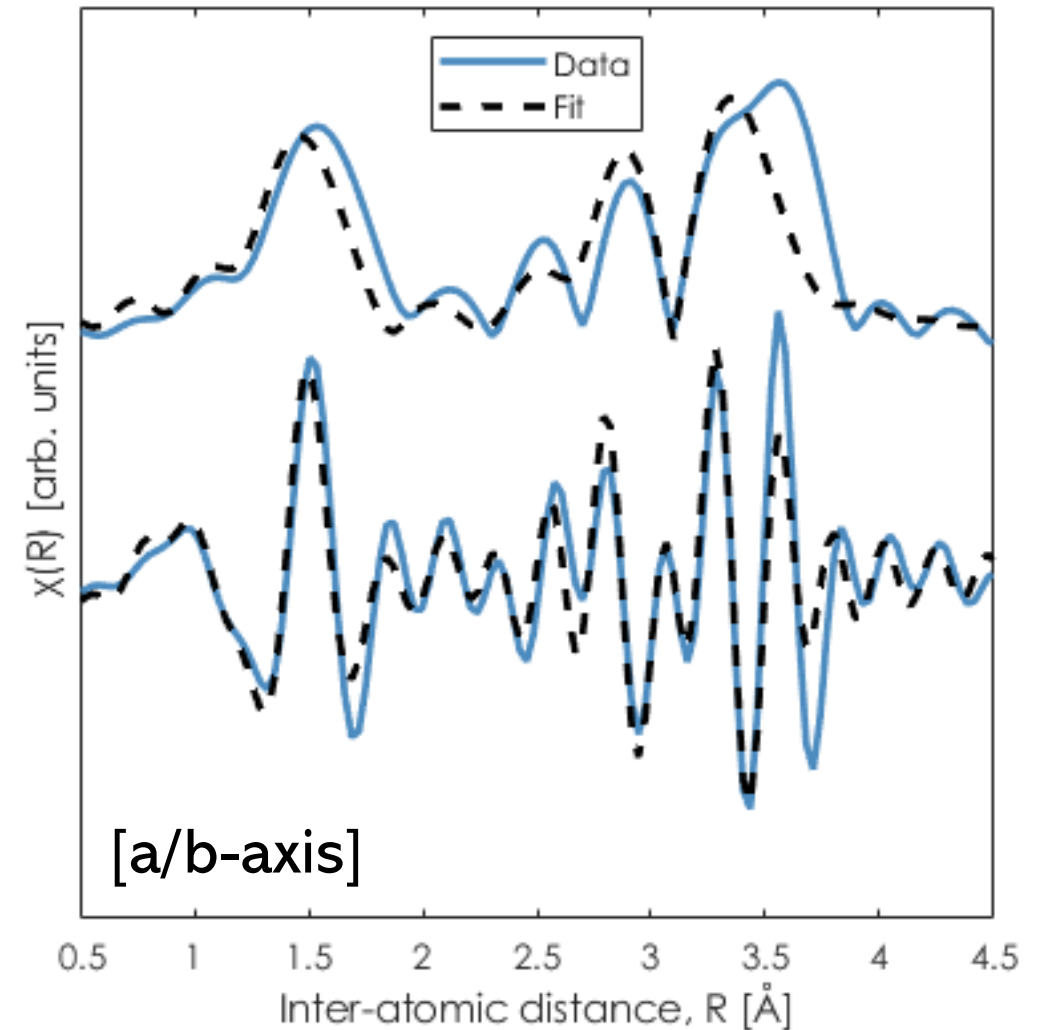
Cu K EXAFS: 2 MeV He⁺ Study

- Additional $\chi(R)$ peaks correspond well to RE-124 stacking faults (SF) seen in ptychography lift-out from the same sample.



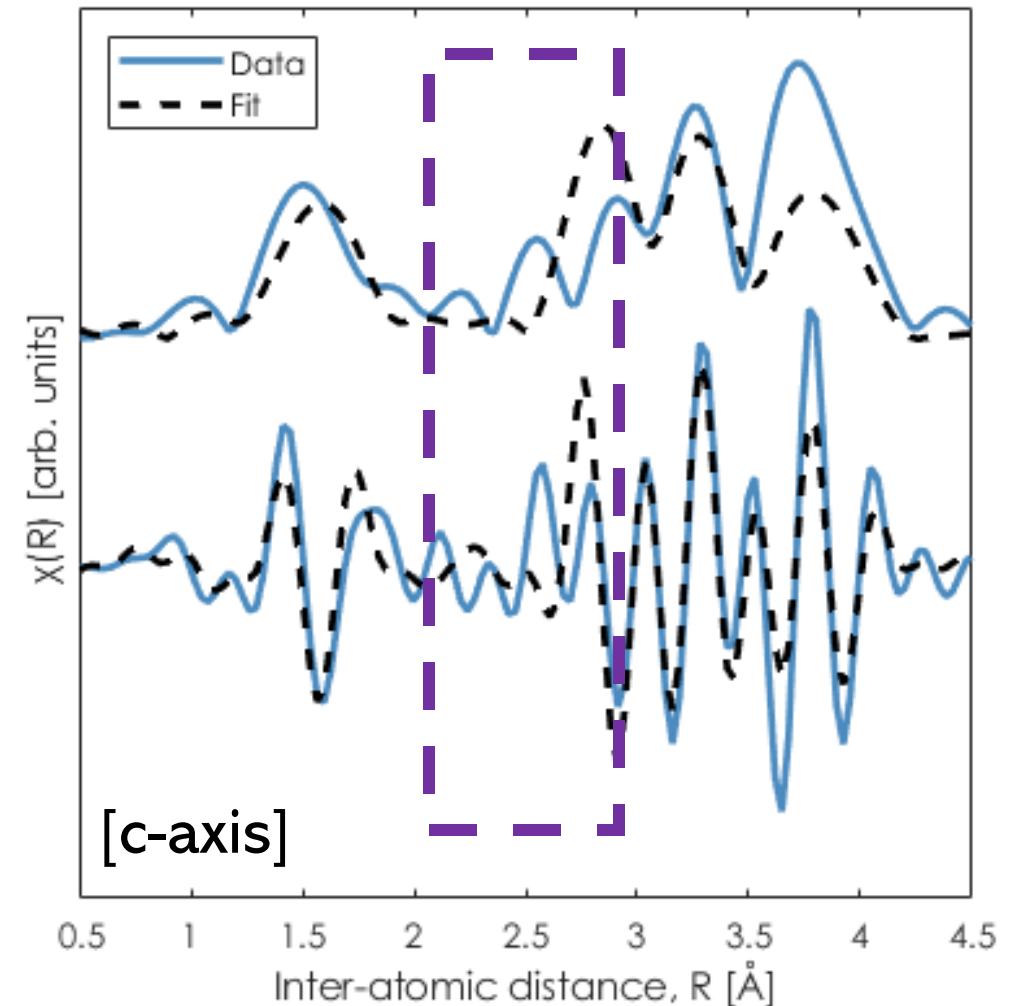
Cu K EXAFS: 2 MeV He⁺ Study

- Pristine material again captured well with a RE-123 structure in the a/b plane.



Cu K EXAFS: 2 MeV He⁺ Study

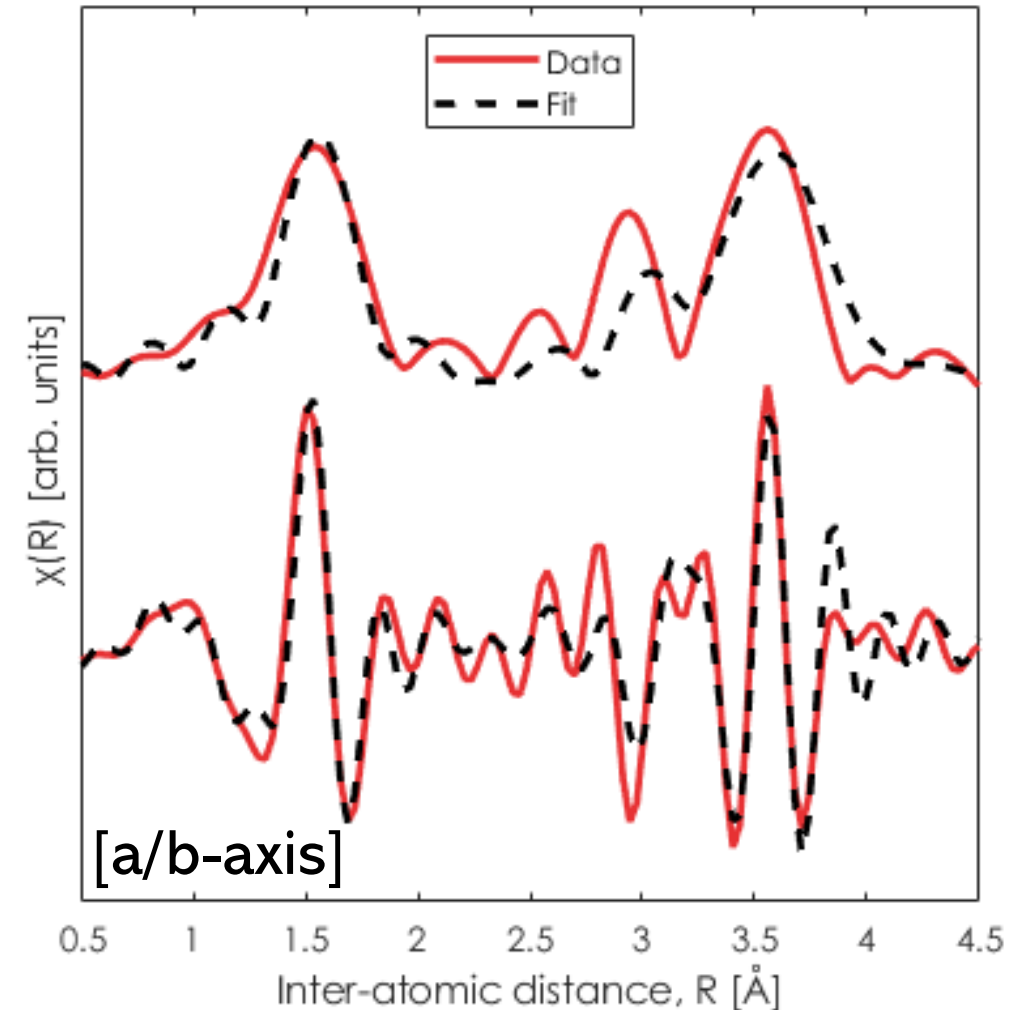
- Less agreement in the c-axis direction, even when including a Cu-O path for the RE-124 structure [7].
- Currently looking into possible interstitial paths.



[7] – Nicholls, R. *et al.*, Presented at: RADSUM Workshop (2025)

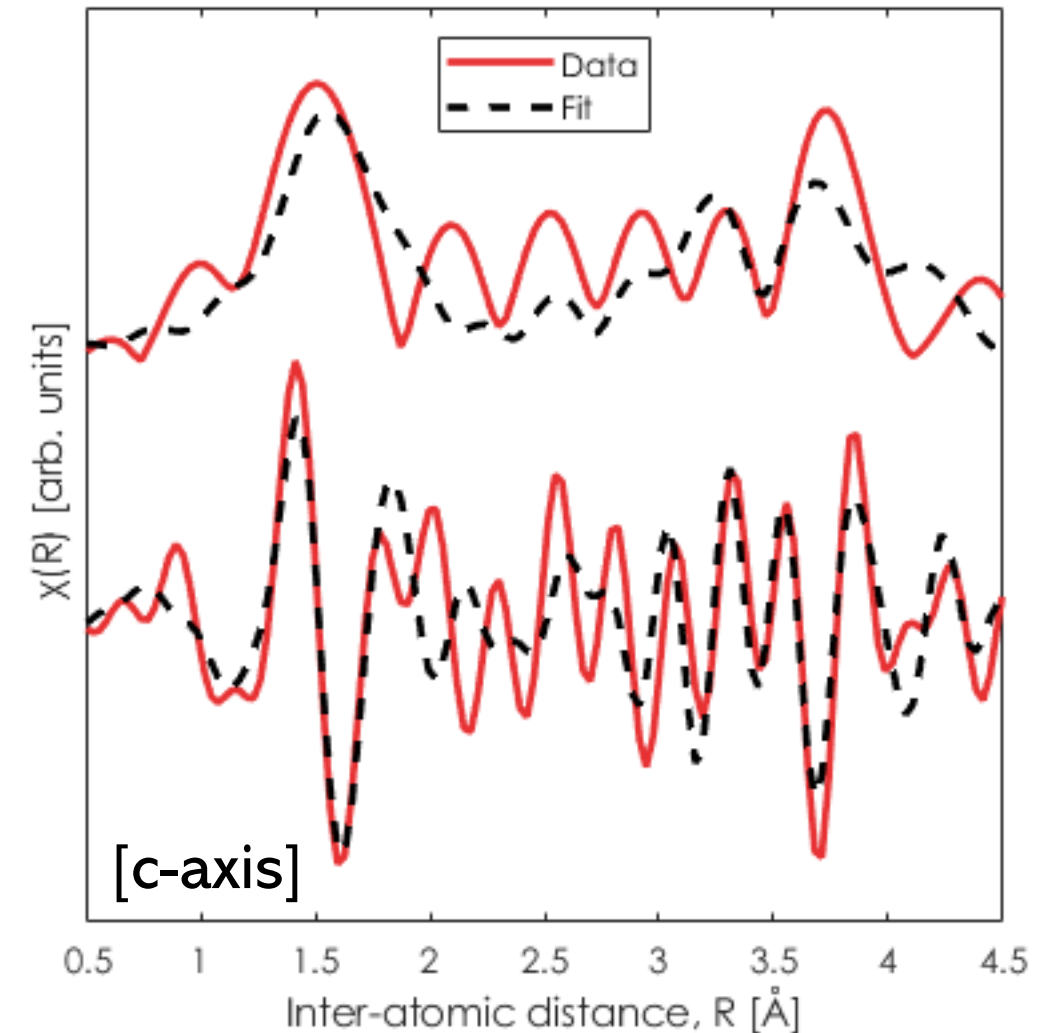
Cu K EXAFS: 2 MeV He⁺ Study

- Fit to irradiated data shows less agreement, as expected.
- The RE-123 model captures the spectral features along the a/b plane well.



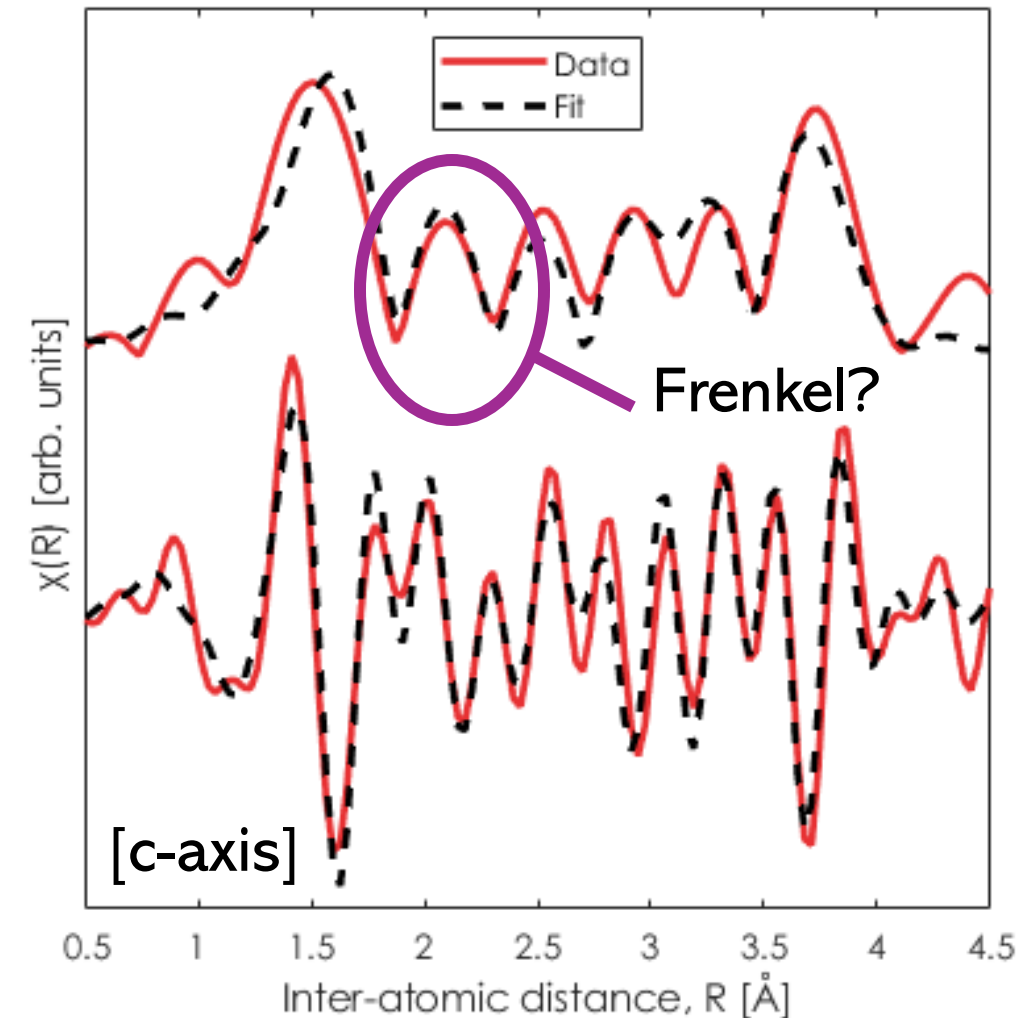
Cu K EXAFS: 2 MeV He⁺ Study

- Irradiated c-axis spectra is more complex.
- Significant spectral features and weight missed by the RE-123/RE-124 model.

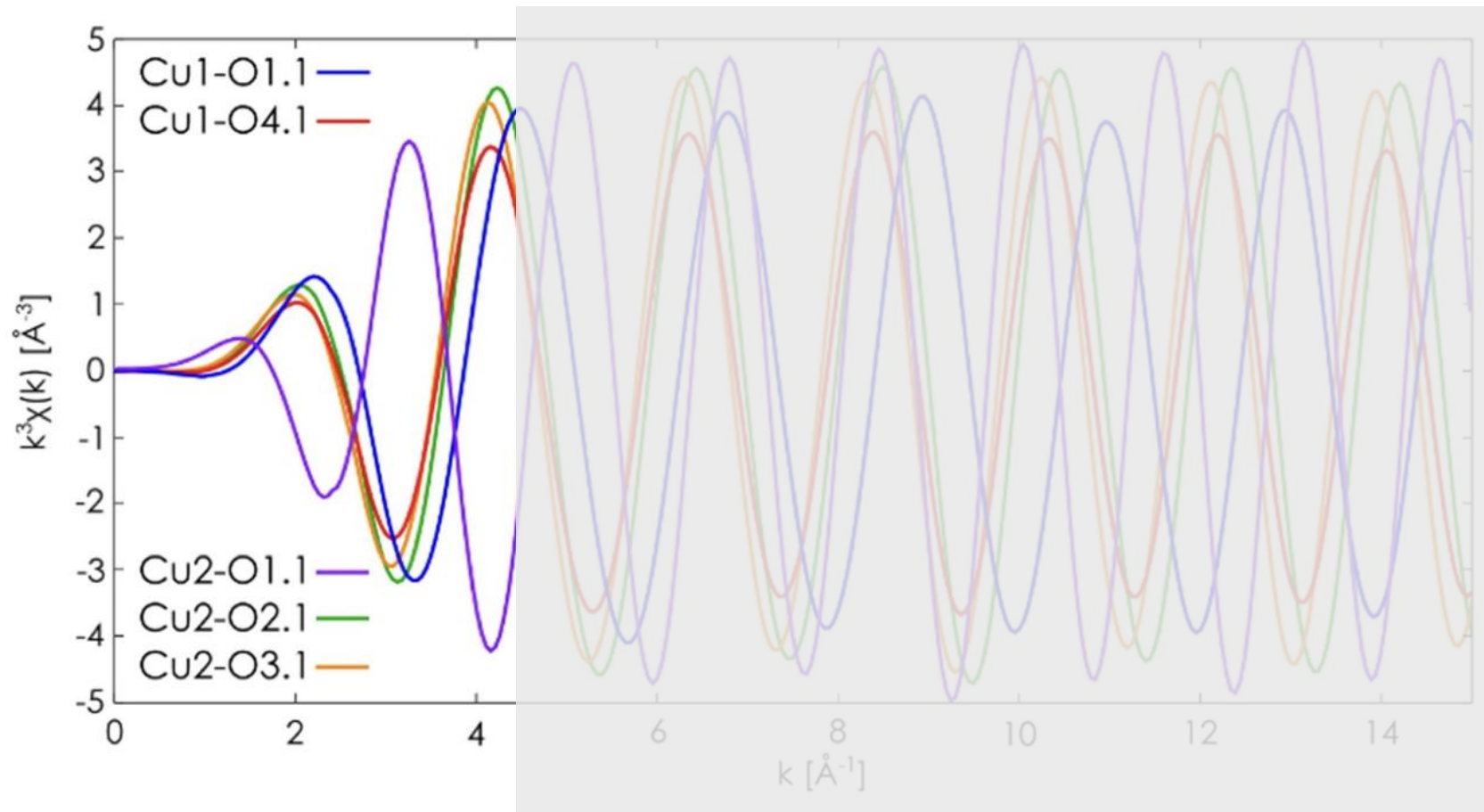


Cu K EXAFS: 2 MeV He⁺ Study

- Including an additional Cu-O interstitial path along with the Cu-O from the RE-124 SF brings model into alignment.
- Validity needs to be confirmed with further tests, along with the pristine c-axis model.

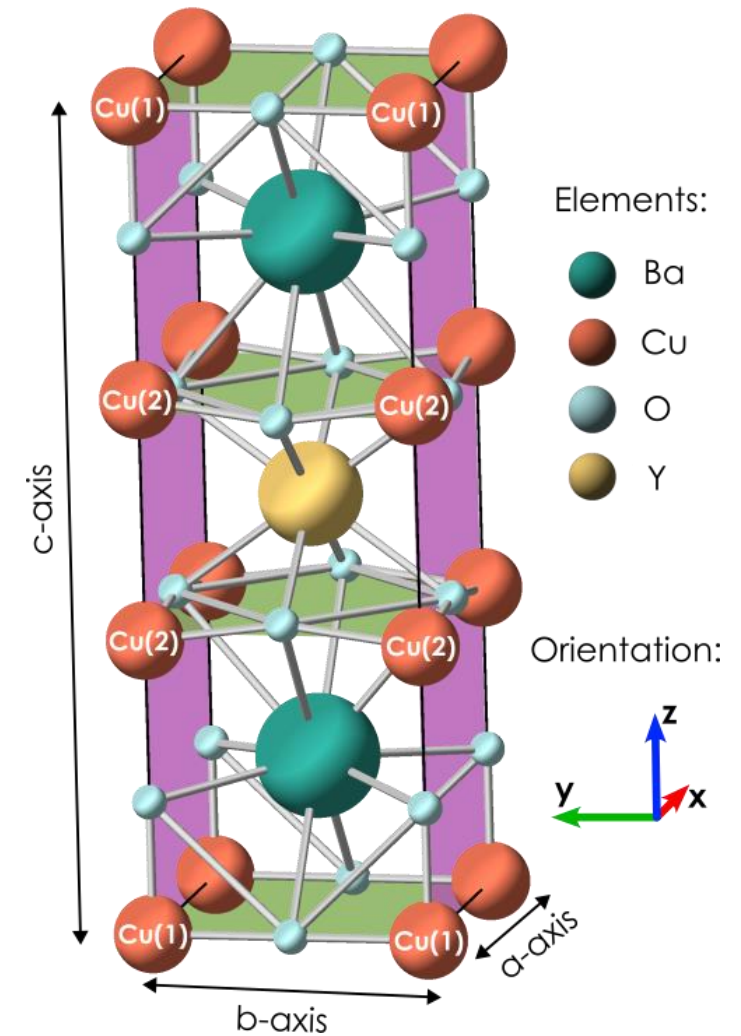


Cu K EXAFS: 2 MeV He⁺ Study



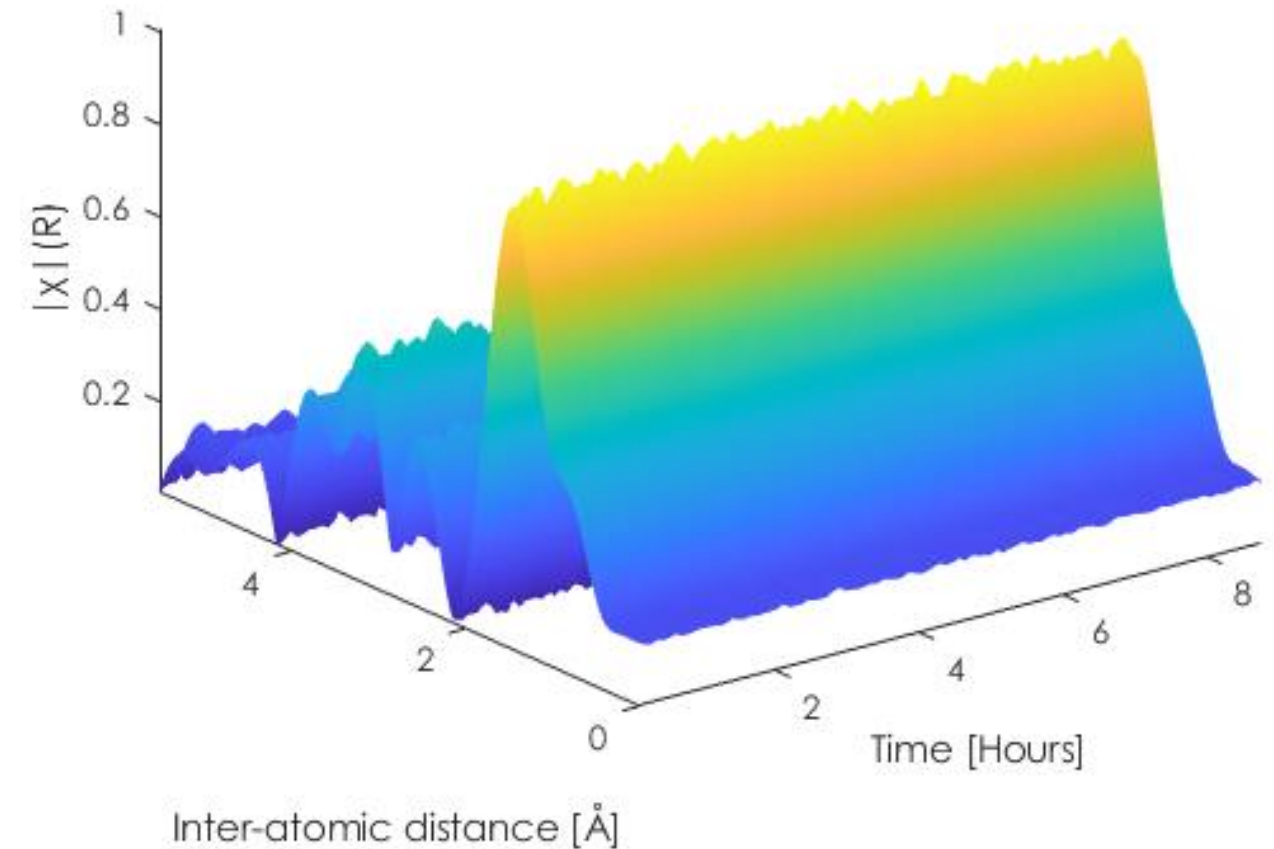
Cu K, Ba L₃, Gd L₃: 2 MeV He⁺ Study

- Three absorption edges were collected for the same sample with polarisation.
- Aim is to develop a multi-edge self consistent local structure model.



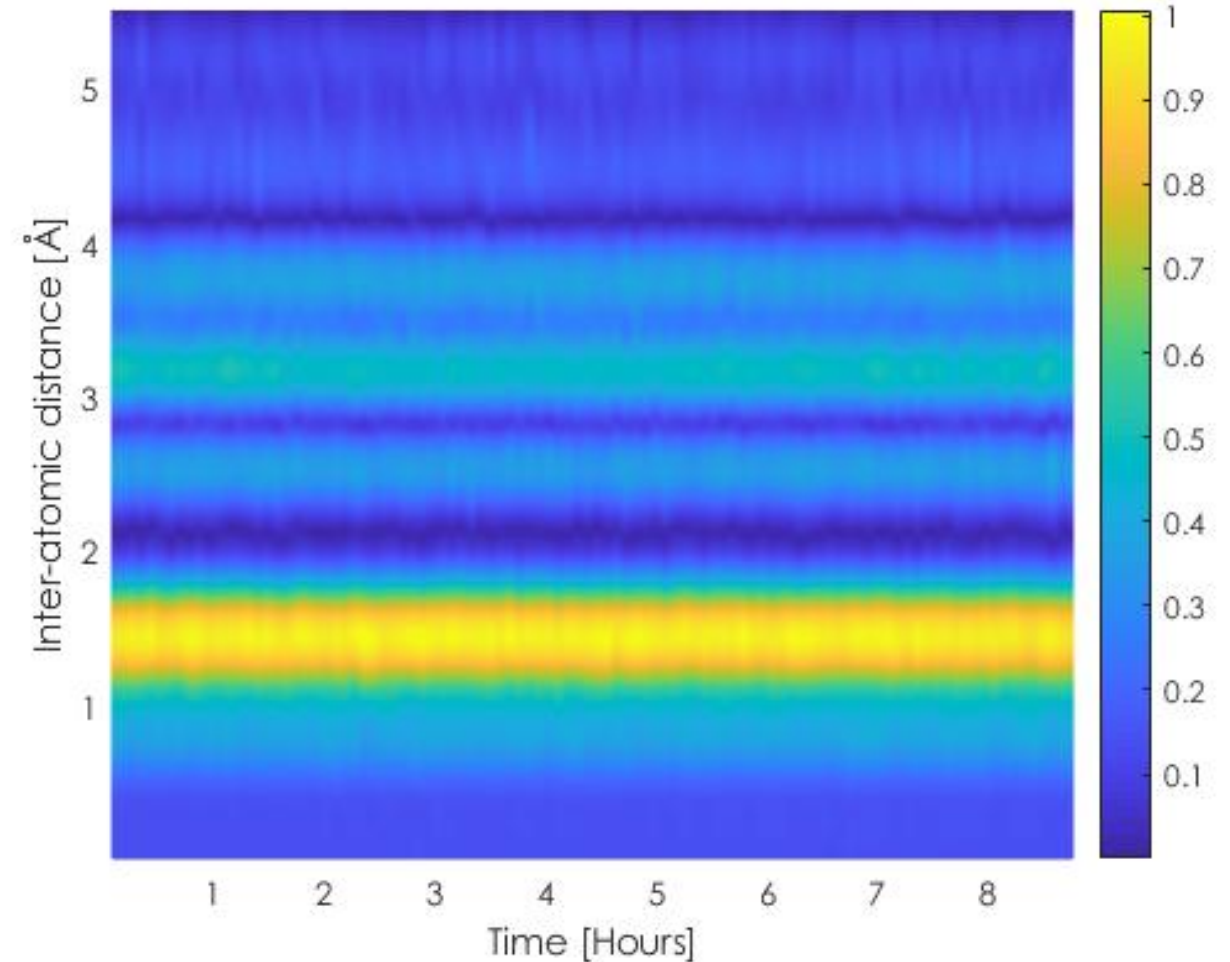
Cu K EXAFS: Beam damage?

- A common concern in XAS is beam damage altering.
- We investigated this using SuperPower tape over 8 hours of continuous measurement.



Cu K EXAFS: Beam damage?

- No evidence of local structure shift from the prolonged beam exposure (8hr).
- (Flux at sample $\sim 1\text{E}11 \text{ } \gamma\text{s}^{-1}$)



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Conclusions & Future Work

- Preliminary XRD analysis shows differing sensitivity of crystal lattice (a,b,c) and crystallite distribution (ω FWHM) to 2 MeV He⁺.
- Polarised EXAFS sensitive to SFs and movement of atoms.
- Multi-edge study analysis nearing completion with Cu K-edge fitting progress, but pristine c-axis spectra remains to be solved.

Conclusions & Future Work

- To analyse:
 - Polarised Cu K-edge EXAFS vs Temperature (77-300 K)
 - Polarised Cu K-edge HERFD-XANES vs Temperature (77-300 K)
- Next experiments:
 - EXAFS + HERFD-XANES of neutron irradiated material (August)
 - Cold transfer LN2 irradiated material to Diamond Light Source for in-situ annealing studies with Cu K-edge HERFD-XANES (September)

Thank you for your attention!

Quick advert:

CfAS Irradiation of HTS at EUCAS 2025 - Porto

Talks

- **K Adams**, (1-MO-IE.2) – Monday 22nd 17:15
- **M Coulson**, (3-MO-FP.5) – Wednesday 24th 15:30
- **Prof S Speller**, (P4) – Thursday 25th 11:00

Posters

Monday 22nd (12:00-13:15)

- **J Tufnail** (1-MP-IE-1)
- **J Lewis** (1-MP-IE-2)
- **J Fihosy** (1-MP-IE-3)

Tuesday 23rd (12:00-13:15)

- **A Gupta** (2-MP-FE1.5)
- **J Lewis** (2-MP-FM.6)