CHEMICAL IMAGING OF CATHODE MATERIALS

Automating the analysis of XRD-CT data to facilitate quicker understanding of the structure and life cycle of a $LiNiO_2$ cathode

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ABSTRACT

Lithium-ion batteries (LIBs) provide a combination of high energy and power density that is incomparable to other battery materials. The most heavily researched and implemented LIB uses $LiCoO_2$ (LCO) as the cathode material. However, the use of cobalt is greatly problematic due to the environmental and human costs of its extraction.[1] Therefore, research into alternative cathode materials is vital.

This study aims to develop programs in Python which automate the analysis of X-ray Diffraction Computed Tomography (XRD-CT) data. The programs are able to identify regions of certain crystallographic orientations and extract physical information such as lattice parameters and crystallite size.

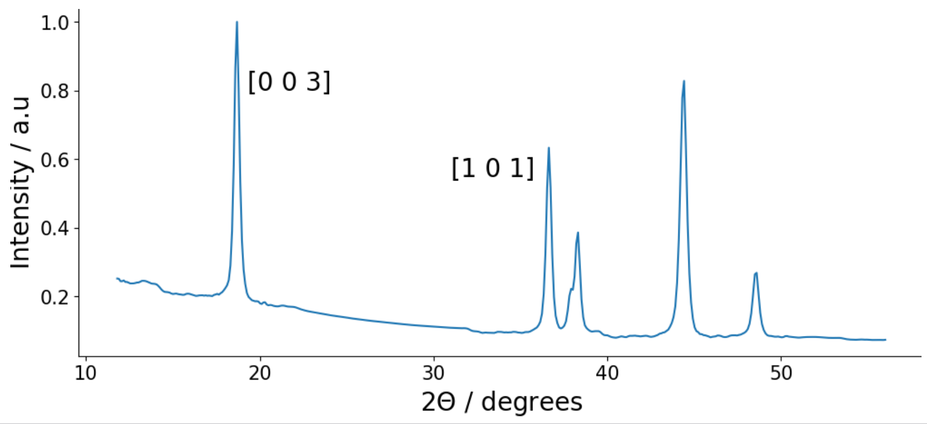
The programs were applied to samples of a $LiNiO_2$ (LNO) cathode in its pristine state and after 100 cycles to observe changes in the crystal structure.

MOTIVATION

- Facilitating **quicker and easier data analysis** to accelerate the research of more environmentally-friendly cathode materials than LCO.
- Researching the structure of the LNO cathode so that it may **exceed the performance and usage of unethical LCO** in the future.
- **Understanding degradation mechanisms** of LNO allows improvement of the cathode material.

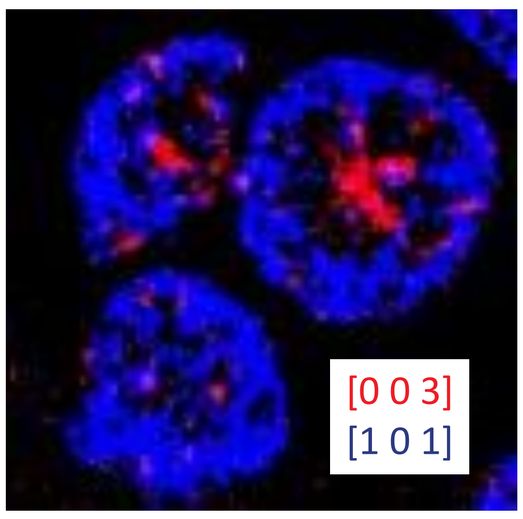
METHODS

- **XRD-CT** scans of LNO cathode samples with 150 nm resolution.
- Finden Ltd **reconstruction** program to produce 3D models of the sample.
- **Segmentation** methods to determine different phases in the cathode.
- **Peak fitting** of diffraction patterns to allow crystallite size and lattice parameters to be calculated.



ORIENTATION ANALYSIS

- Both the crystallites around the edge and the crystallites in the centre are highly ordered, but they differ in orientation.
- The crystallites around the edge are **radially aligned**, which **aids the intercalation process**. The different orientation in the centre will impede Li transport at high charge states.



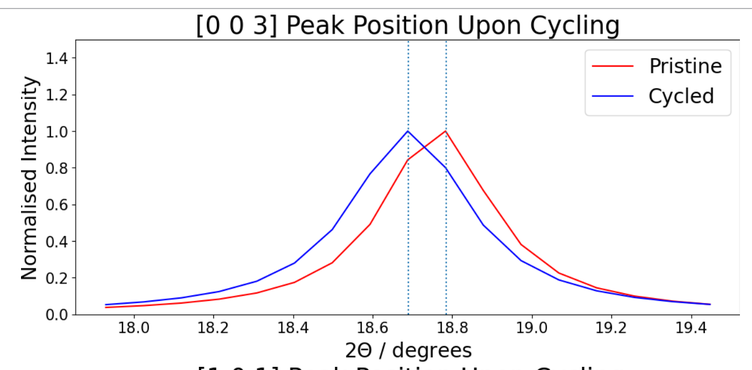
CHANGES IN D-SPACING

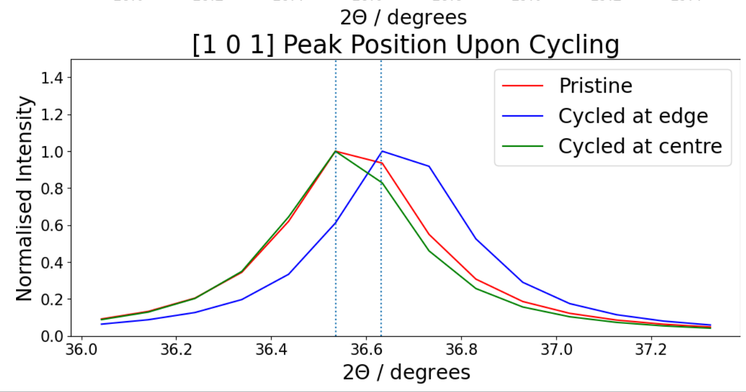
Decrease in peak position of [0 0 3] upon cycling.

- Increase in d-spacing consistent with shift from H1 to H2 phase.[2]

Increase in peak position of [1 0 1] upon cycling at edges of secondary particle only.

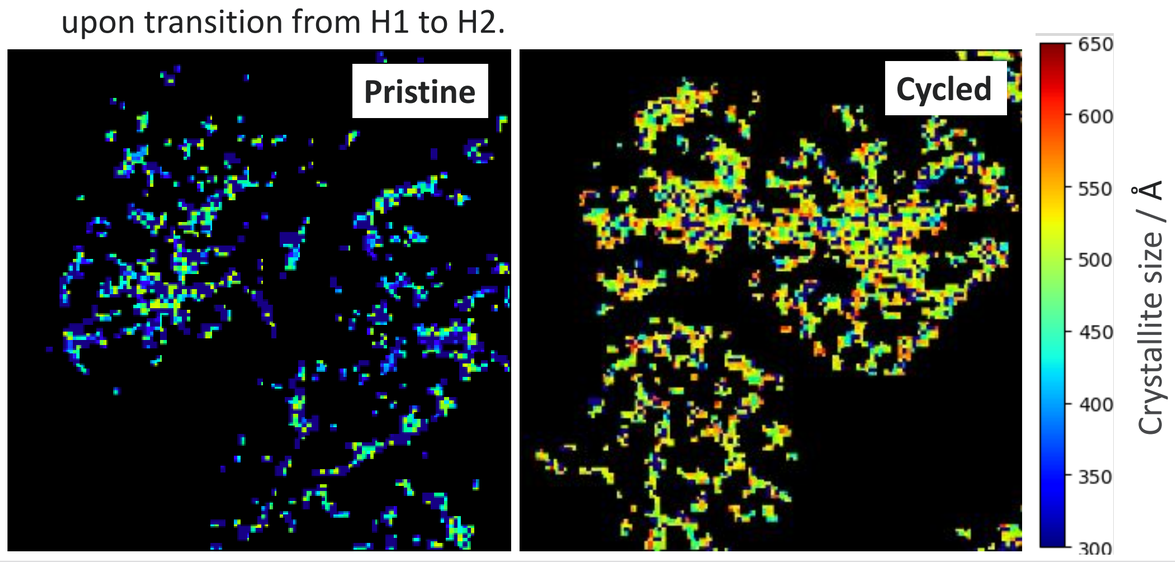
- Decreased d-spacing therefore smaller unit cell volume at edges.
- Could be linked to **increased stress** in the core.





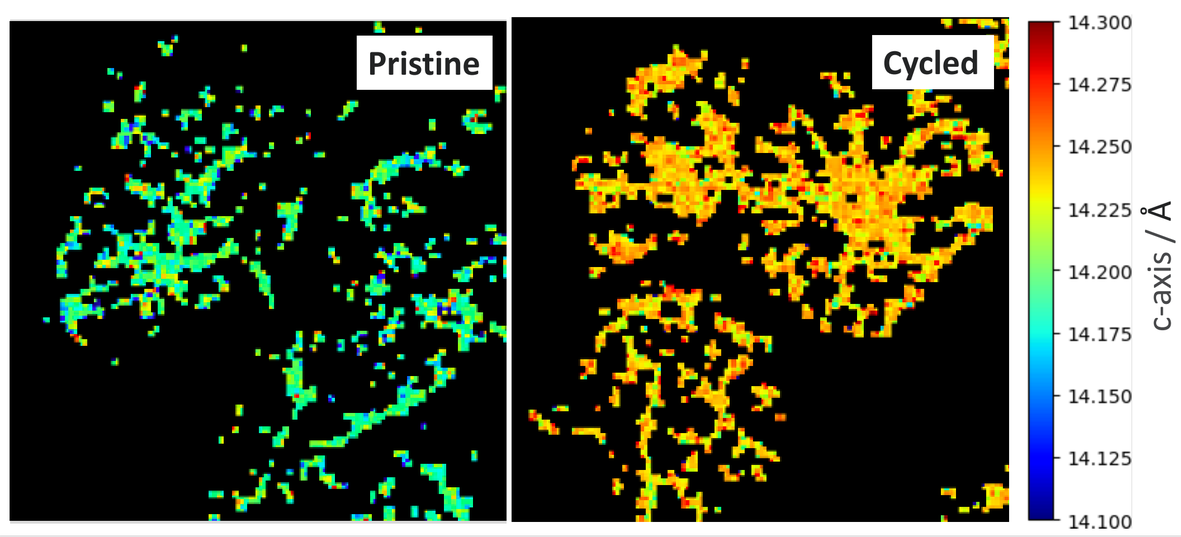
CRYSTALLITE SIZE

- **Increase in crystallite size upon cycling** due to increased ordering/layering upon transition from H1 to H2.



C-AXIS PARAMETER

- **Increase in c-axis upon cycling** is consistent with transition from H1 to H2.



IMPACT / NEXT STEPS

- The data analysis procedures developed in this project have been shown to work reliably and efficiently for the LNO cathode material- the structural changes observed are consistent with those cited in literature.
- Valuable insights into the LNO structure have been obtained in this project which could be used to improve the cathode material. Engineering the cathode particles to have no differing orientation in the centre could be attempted to see if there is increased performance.
- Possible next steps would be to analyse the material after one or two cycles, or to monitor the effects of doping or heat-testing.
- XRD-CT can be applied to many materials in the battery field and beyond, so the procedures developed in this project have a wide scope.

REFERENCES

[1] S. Lee, A. Manthiram, *ACS Energy Letters,* 2022, **7**, 3058-3063

[2] J. Xu, E. Hu, D. Nordlund, A. Mehta, S. Ehrlich. X. Yang, W. Tong, *ASC Appl. Mater. Interfaces,* 2016, **8**, 31677-31683

INTERN BIO

Abby is studying Chemistry at The University of Birmingham. She is interested in computational chemistry and characterization methods, specifically UV-Vis spectroscopy and XRD. Having gained invaluable experience in programming and XRD-CT analysis through her internship, she aspires to apply the skills and knowledge she has learnt to a future career in scientific software development.

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