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# nmRC
# CASE STUDY

## Investigating the internal structure of Mg ion batteries

nmRC_CS_16

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# Preparing and imaging thin sections of Mg battery deposits

## FIB-SEM lift out and TEM case study

# INTRO TO Mg BATTERIES



Li-ion batteries are used in a range of technologies from **laptops to electric vehicles**.

However, we are fast approaching a theoretical energy limit for this type of cell chemistry.

They also use unsustainable materials such as cobalt, and therefore next generation battery compositions are being studied.

These include **magnesium** based technologies.

**Pros**

High volumetric capacity:
(3833 mAh $cm^{-3}$ vs 2046 mAh $cm^{-3}$ )

Substantially more abundant than lithium

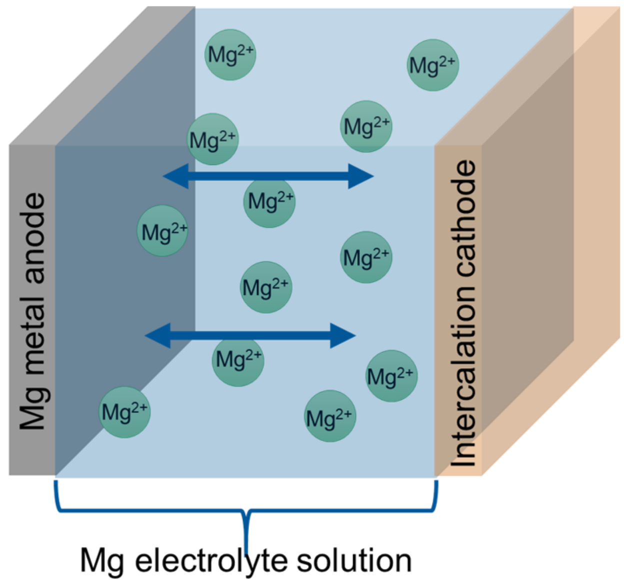
Cheaper than Li: Li (13$/kg), Mg (2.5$/kg)

Cell cycling doesn't incur dendrite growth

**Cons**

Development of cathodes is limited by poor ion insertion kinetics

Poor compatibility with current electrolytes and electrodes

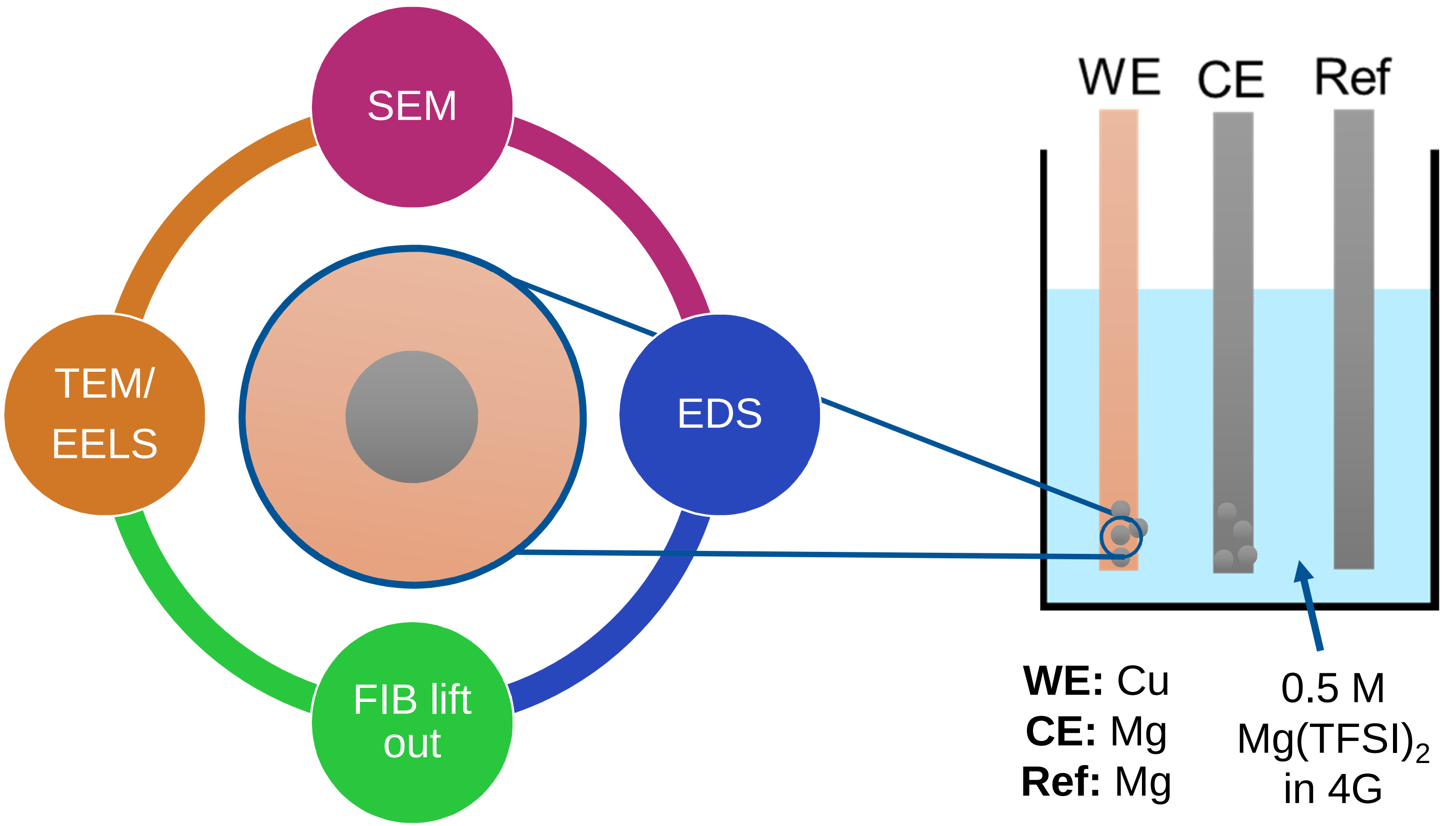


# CHARACTERISATION



The aim of this study is to characterise the structure and composition of the **deposits on the working electrode (WE)** at the nanoscale after multiple charging/discharging cycles. This is important to understand why performance may be affected over time. To achieve this, the following techniques will be used.

- **Scanning Electron Microscopy**
- **Energy-dispersive X-ray Spectroscopy**
- **Focussed Ion Beam lift out**
- **Transmission Electron Microscopy and Electron Energy Loss Spectroscopy**



# CHEMICAL AND STRUCTURAL ANALYSIS OF DEPOSIT SURFACE

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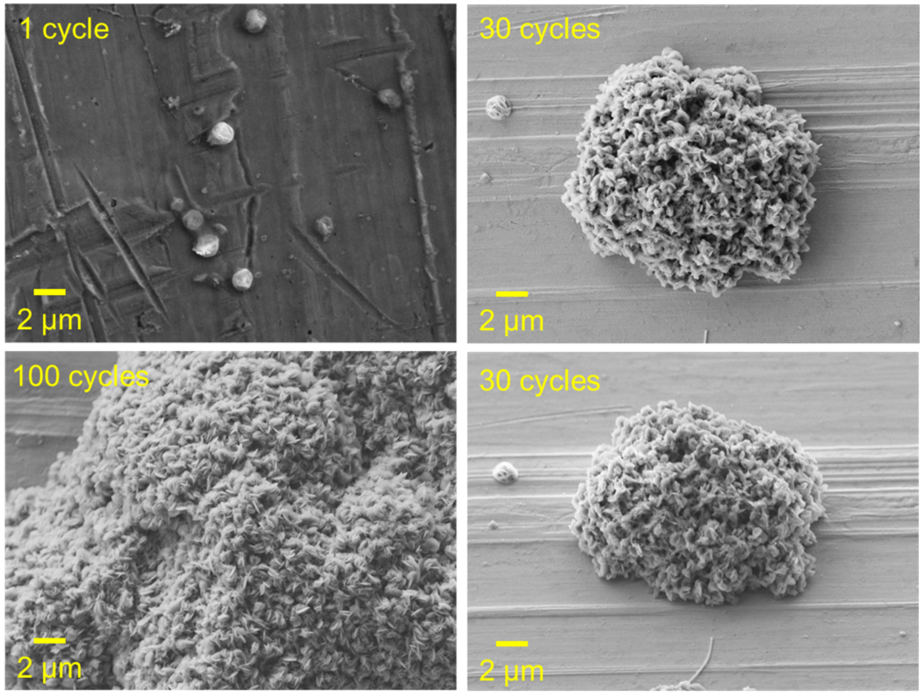
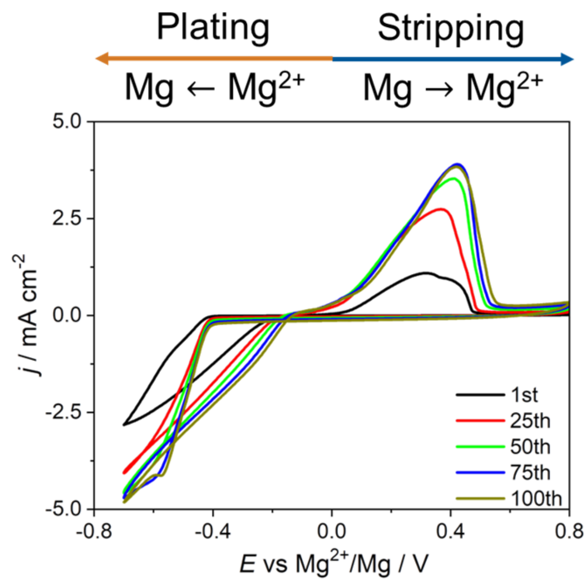
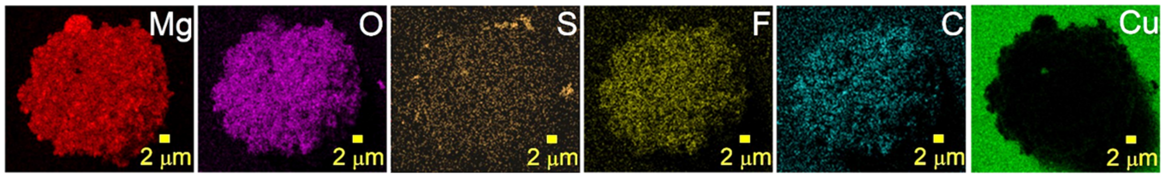
The deposits on the working electrode were imaged using SEM. After an increasing number of charging/discharging cycles, it was found that the **deposits grew during repeated plating and stripping**.




*SEM images of the deposit after 1, 30, and 100 cycles showing aggregation upon increasing cycles.*



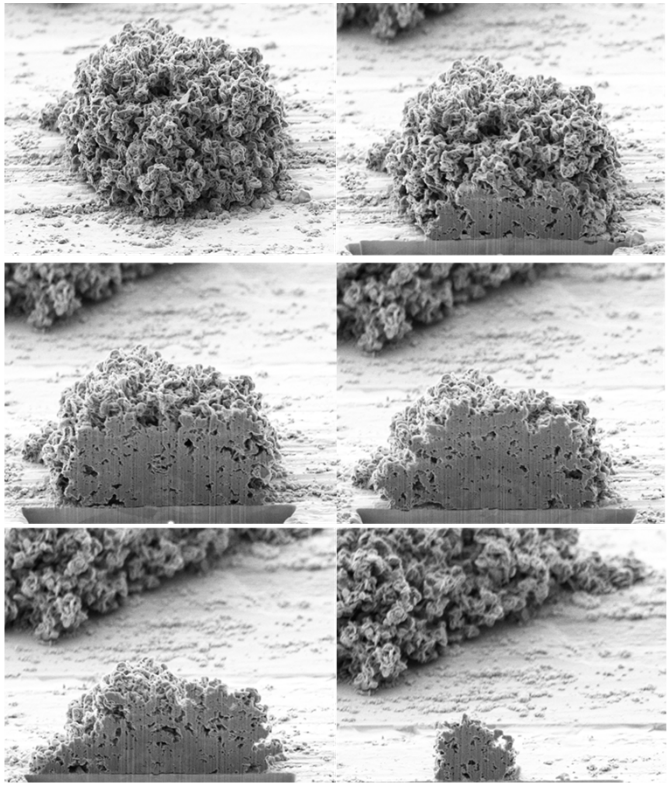

*EDS mapping of the deposit after 30 cycles.*

# CHEMICAL AND STRUCTURAL ANALYSIS OF DEPOSIT CROSS SECTION

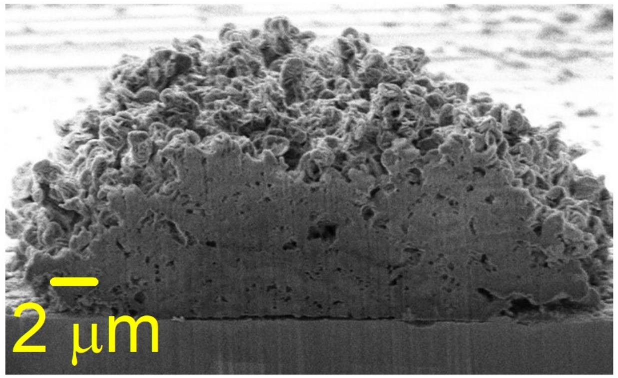


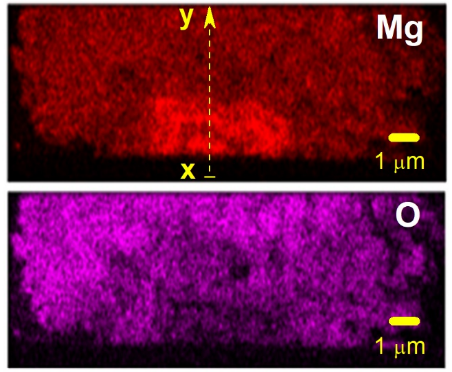
To examine the internal structure and composition of the deposit, FIB milling was employed coupled with in-situ EDS mapping.

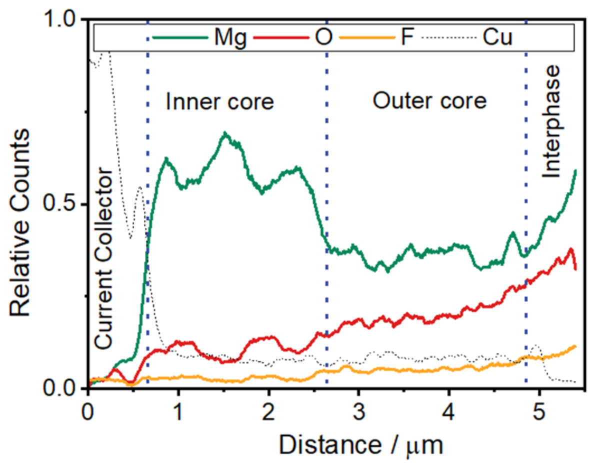
This revealed **three distinct regions**: a Mg core, a MgO-rich inner shell, and the outer shell.

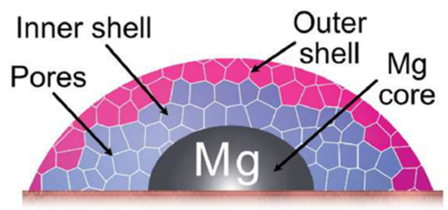


*Sequential FIB milling through the deposit.*



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# FIB LIFT OUT FOR TEM ANALYSIS

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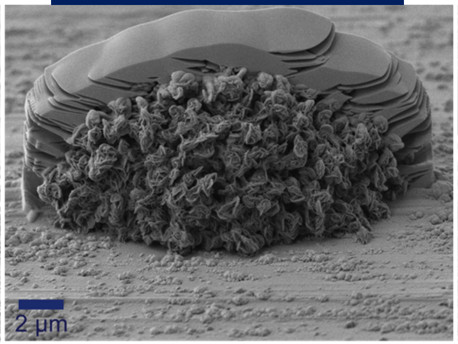
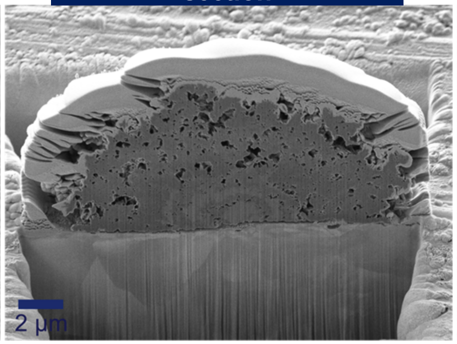
To gain a more **detailed understanding of the pores** within the deposits, a FIB lift out was performed to produce and extract a thin section (50 µm) suitable for further TEM analysis. The steps below show the process to lift out the **thinned section** onto a support grid.

**1. Mg deposit**

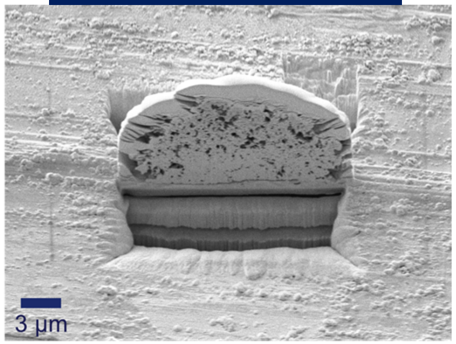

**2. Pt deposited**




**3. Deposit milled to a thin section**

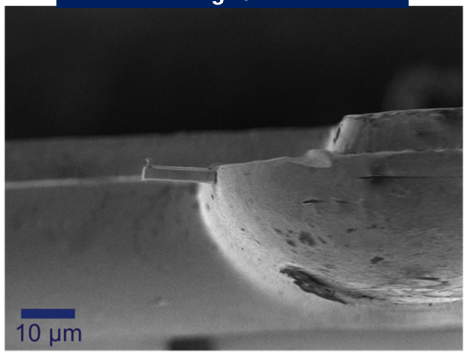



**4. Section cut away**

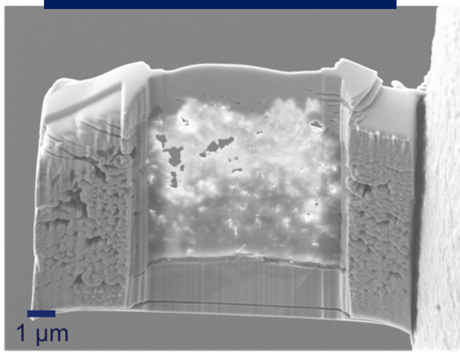



**5. Micro manipulator attached**

**6. Loaded onto Cu support grid**




**7. Finished section on grid**



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# TEM IMAGING OF THIN SECTION

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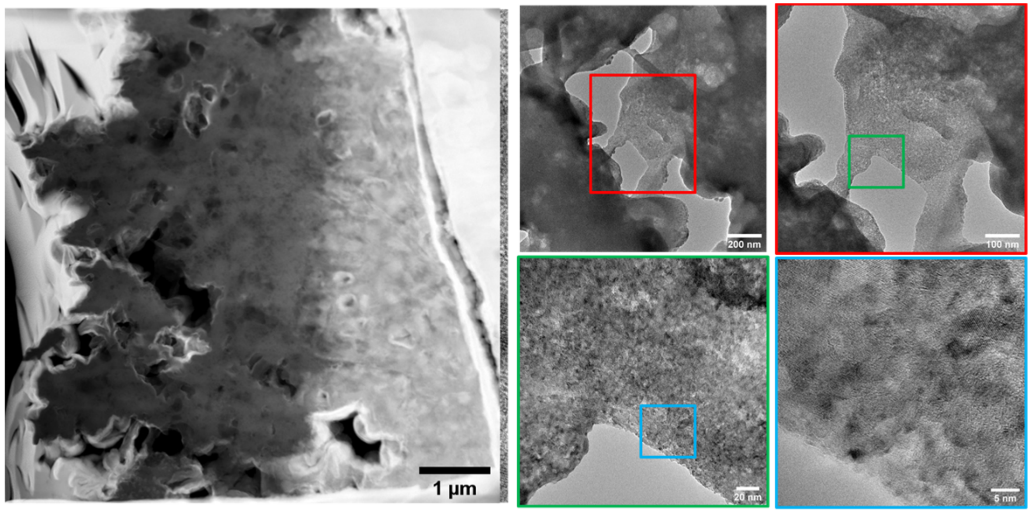
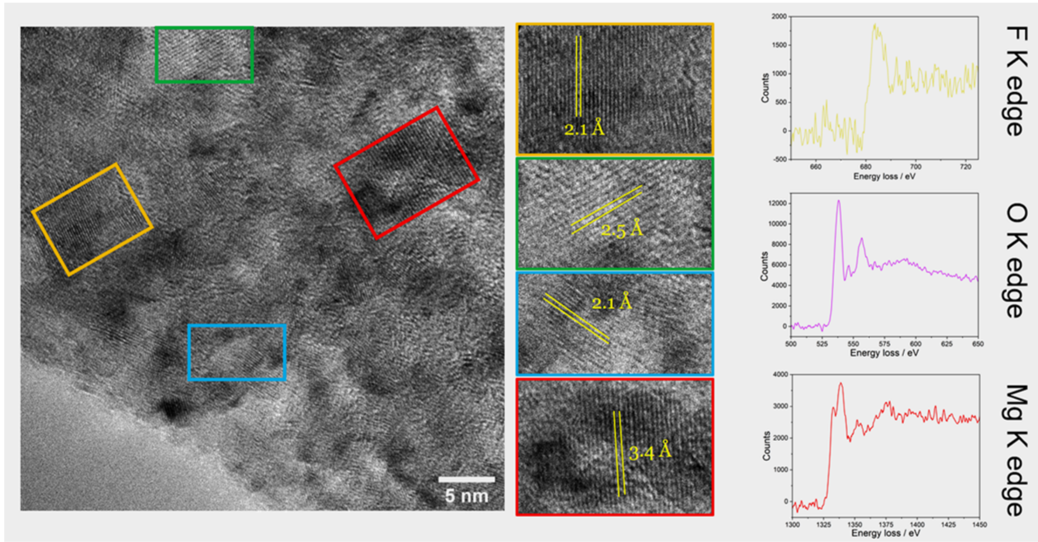
The thin section was transferred into the TEM under a **controlled atmosphere** using a glovebox and an air-free TEM holder.

*Air free transfer using a glovebox*

TEM analysis showed the structure was a **crystalline-aggregate** with d-spacings observed corresponding to MgO (2 - 2.5 Å) and MgF (3.4 Å), which were also confirmed by in-situ EELS analysis.



# SUMMARY



- Initial SEM imaging of the deposits showed that they grew over time due to repeated plating and stripping.
- FIB-SEM coupled with in-situ EDS allowed the internal structure and composition of the deposits to be examined and found that there were three distinct regions: a Mg core, a MgO-rich inner shell, and the outer shell.
- Using FIB lift out combined with air free transfer into a TEM provided thin section imaging which showed crystalline structure consisting of areas of MgO and MgF.

# REFERENCES / ACKNOWLEDGEMENTS

The FIB-SEM-EDS and TEM-EELS analysis documented here were performed at the Nanoscale and Microscale Research Centre (nmRC) at the University of Nottingham. www.nottingham.ac.uk/nmrc

**This work was published in Energy Storage Materials:**

Dimogiannis, K., Sankowski, A., et al. Structure and chemical composition of the Mg electrode during cycling in a simple glyme electrolyte. Energy Storage Mater. 67, 103280 (2024).

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# ADDITIONAL INFORMATION

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- If you wish to get in touch with us to discuss the information provided, raise a query/concern or provide feedback then please feel free to get in touch via any of the methods listed below:


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