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University of Strathclyde Glasgow

Material Development to Enhance Reversible Solid Oxide Cells for Hydrogen Production and Power Generation

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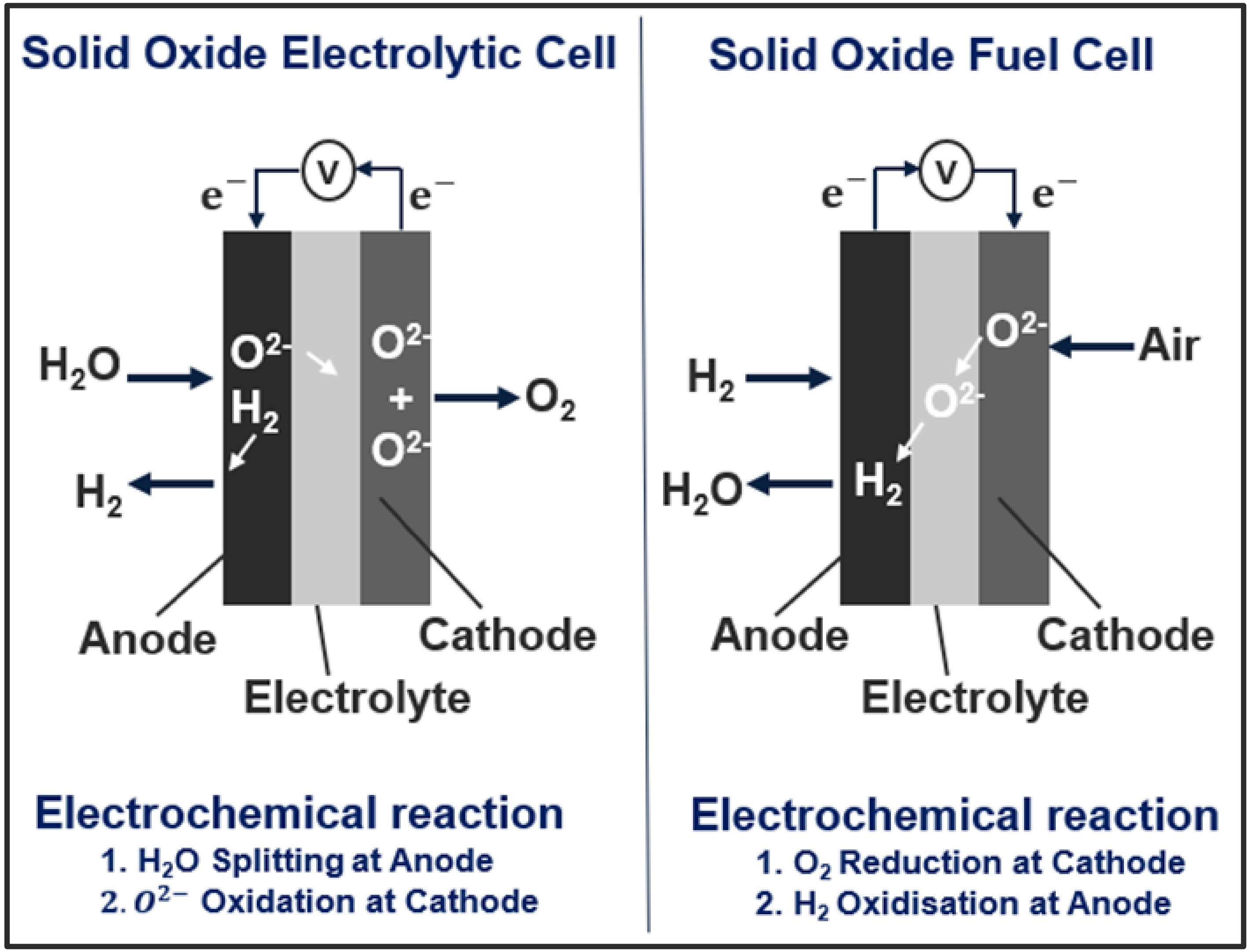
THE PLACE OF USEFUL LEARNING

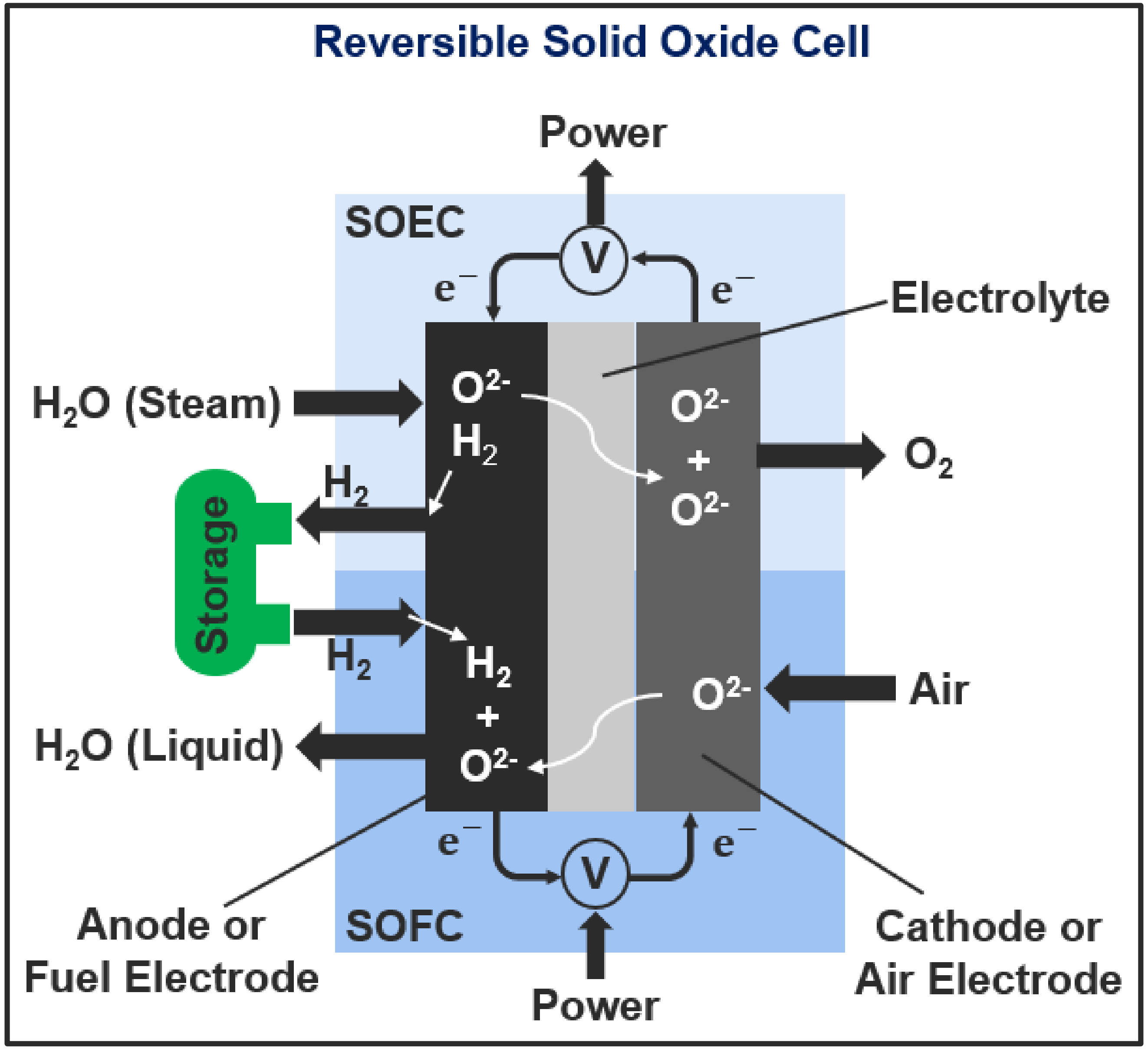
Introduction

❖ Solid Oxide Cells



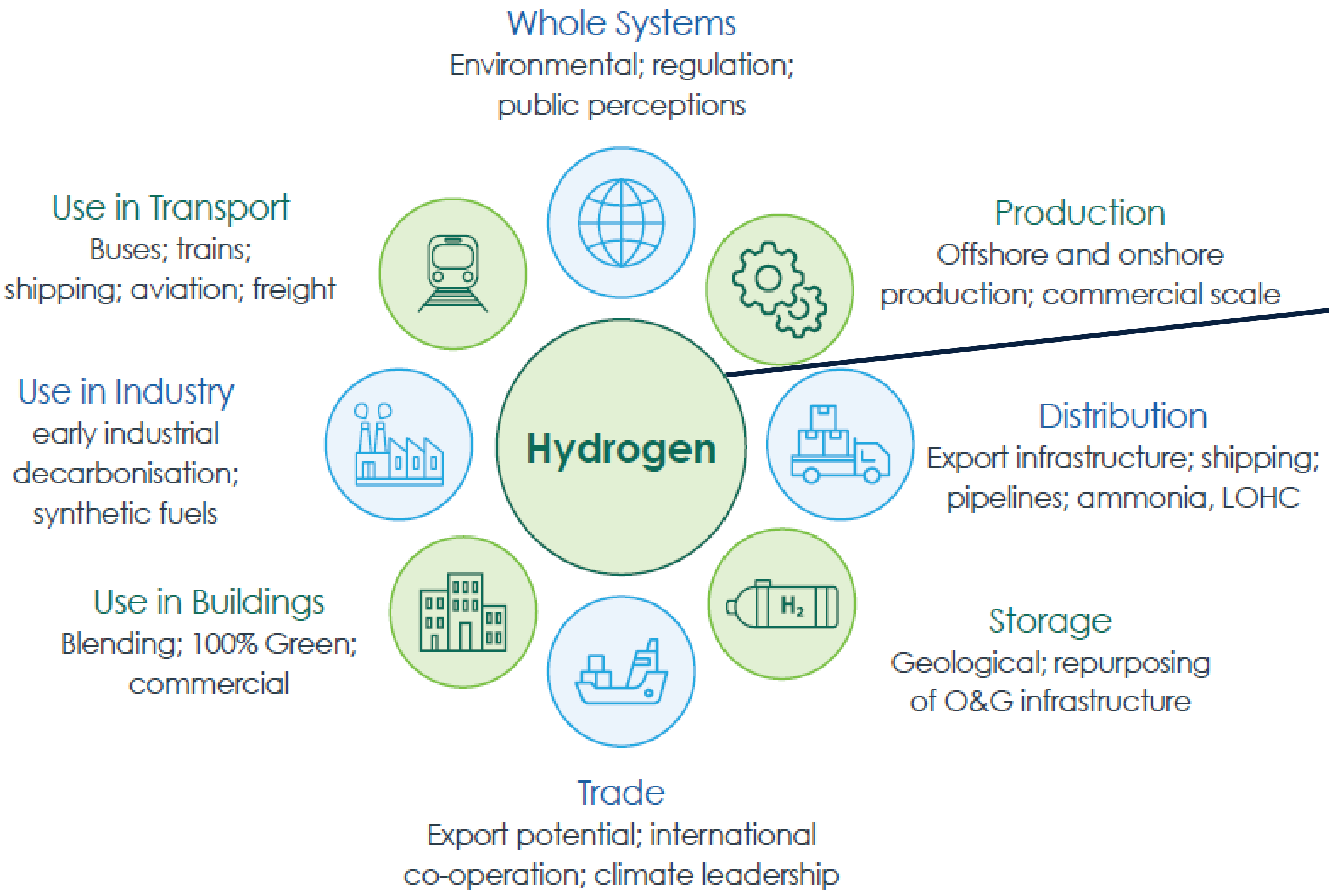
Introduction

- The Reversible Solid Oxide Cell (RSOC)



Introduction

❖ A Net-zero or Hydrogen Economy



A 100% renewable energy economy
+
Zero carbon

Efficient production of green hydrogen from renewable sources

Electrolysis of water
Solid oxide cells

Scottish Government. Draft Hydrogen Action Plan. (2021)

Background of the Study

❖ The Need for a Novel Perovskite Material

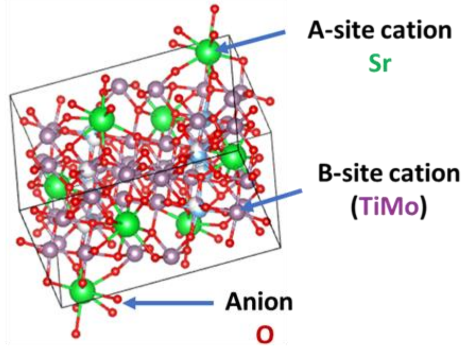
Problem

The Challenges of RSOCs

- The Multiple Electrochemical Requirements of RSOCs:
  - ❑ High ionic and electronic conductivity,
  - ❑ High catalytic activity.
- Cell components' stability

Solution

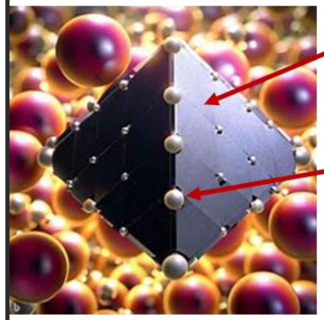
Perovskite Material



A Crystal structure of $Sr(TiMo)O_3$

⬇

Innovate Material



Perovskite Material
- Properties tuning?
- Exsolution rate?
- ...?

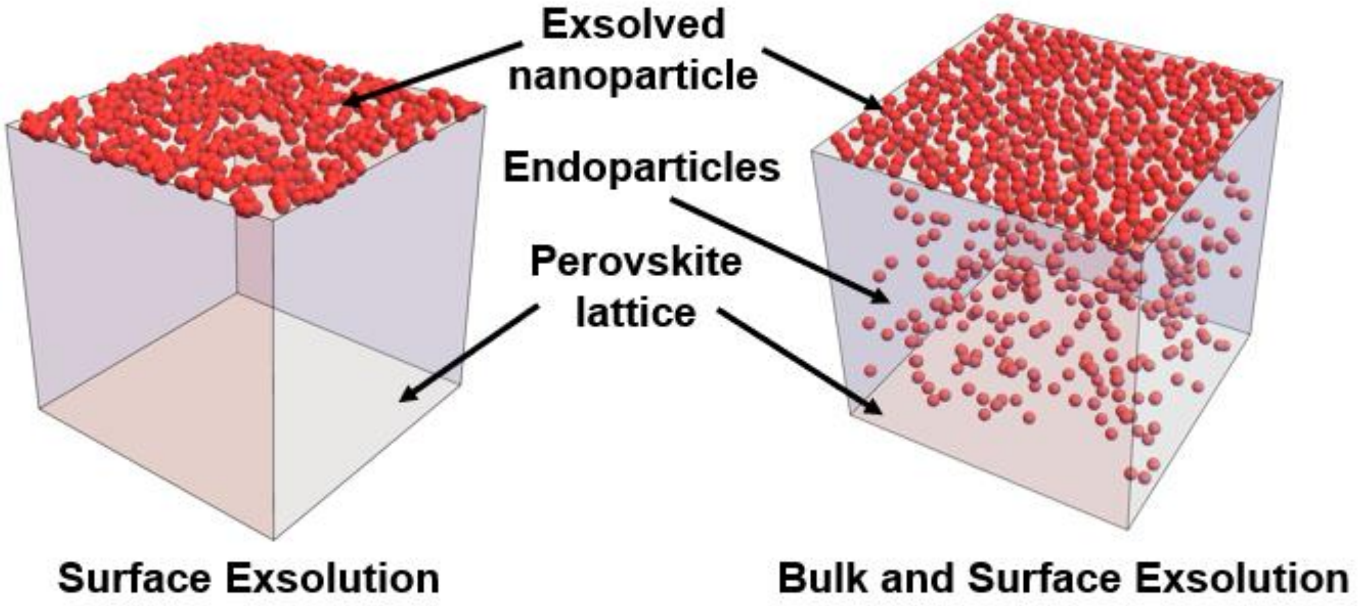
Exsolved nanoparticle
- How many?
- Where?
- What size and shape?
- Catalytic activity
- ...?

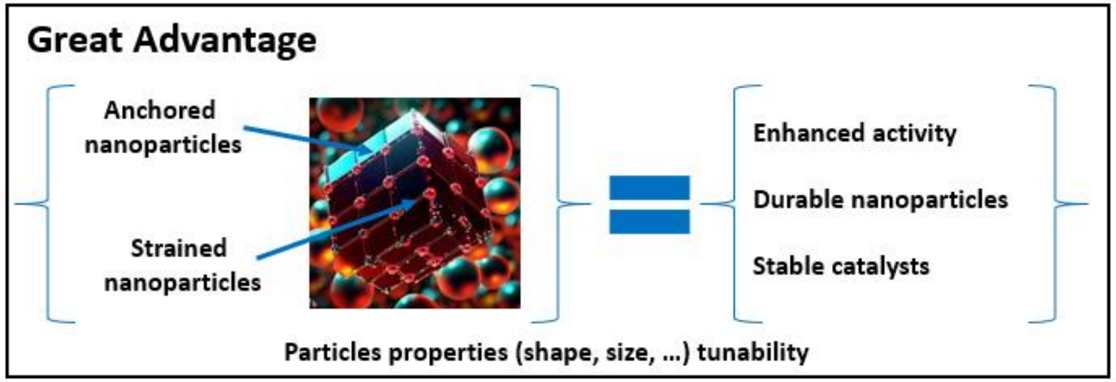
Goal

Highly efficient RSOCs

Background of the Study

❖The Exsolution Process





Neagu, D. *et al*. *Nat. Chem.* **2013**, 5**,** 916–923.

Research Focus

This research aims to develop a novel perovskite material capable of surface and bulk exsolution to fulfil the multiple electrochemical requirements of RSOCs.

- Target Material

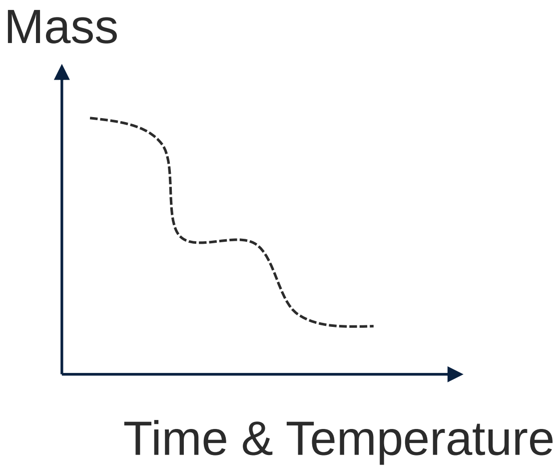
An A-site deficient perovskite of the family with a $(Sr,Ca)_{1-\alpha}(Ti,Fe,Ni)O_3$ stoichiometry.

Materials and Method

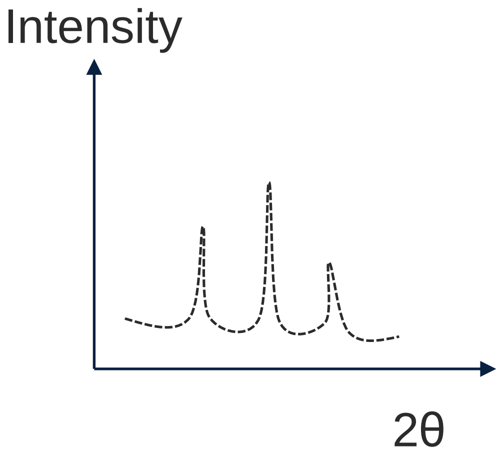
- **Materials selection and study of parameters related to the new perovskite material synthesis**

  ✓ Characterization of potential precursor materials:
  $Fe(NO_3)_3.9H_2O$, $Ni(NO_3)2.6H_2O$, $CaCO_3$, $SrCO_3$, TiO_2, $CuNO_3$

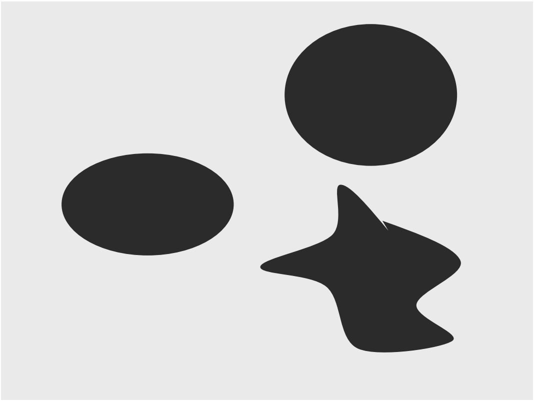
Thermogravimetry analysis (TGA)



X-Ray diffraction analysis (XRD)

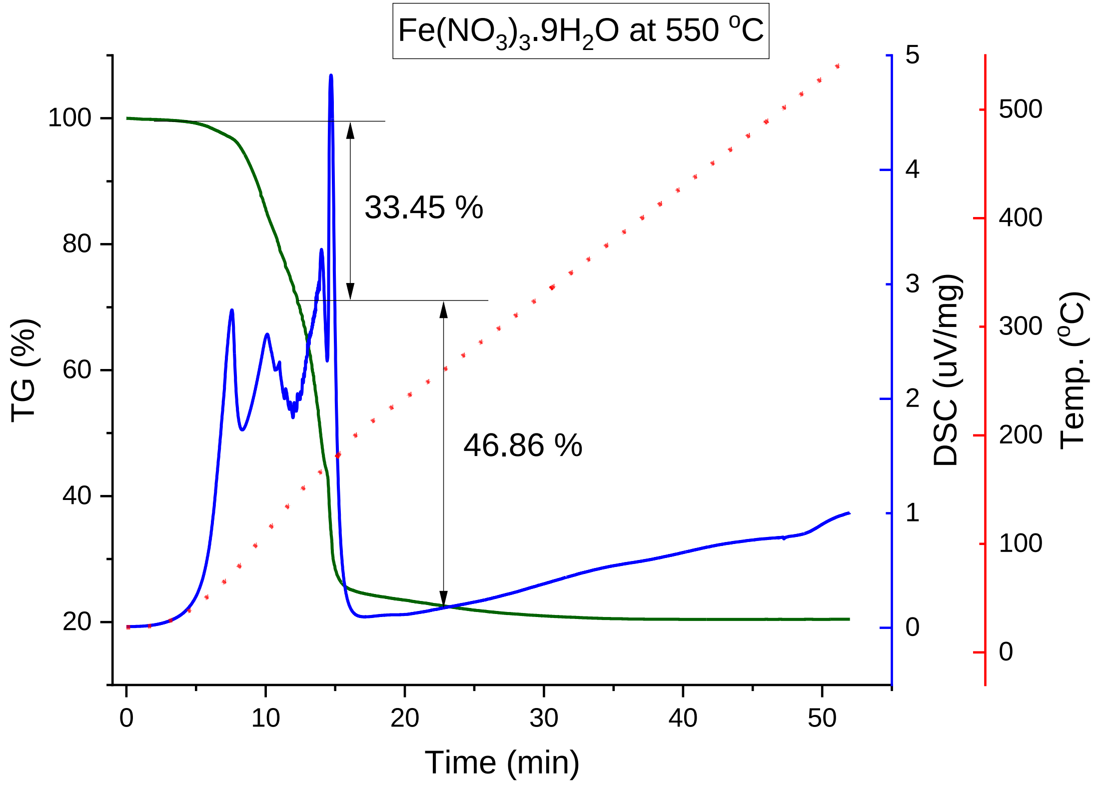


Scanning electron microscopy (SEM),

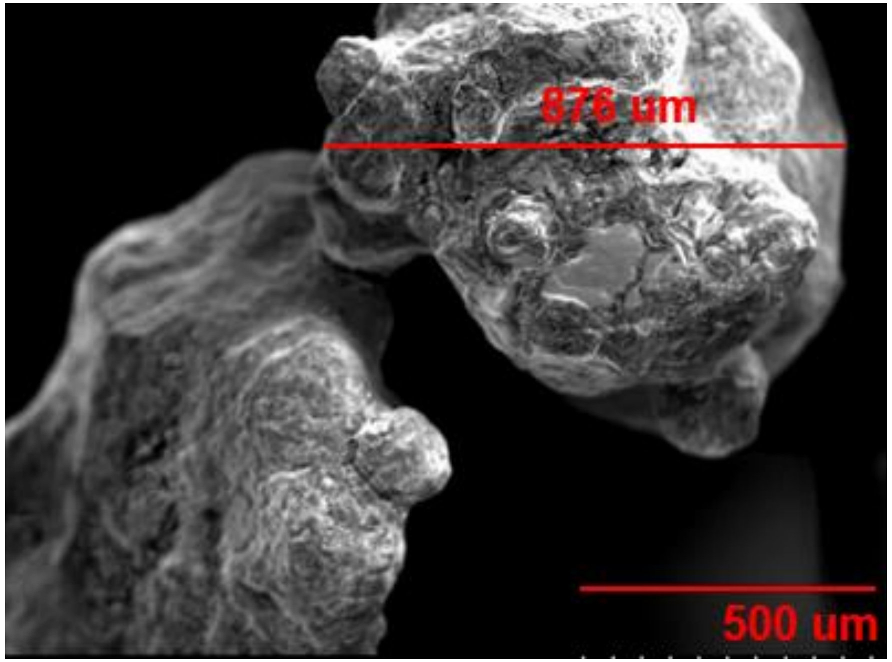


Materials and Method

❖ **Selection of synthesis route from the preliminary study on the precursor materials**



(a)

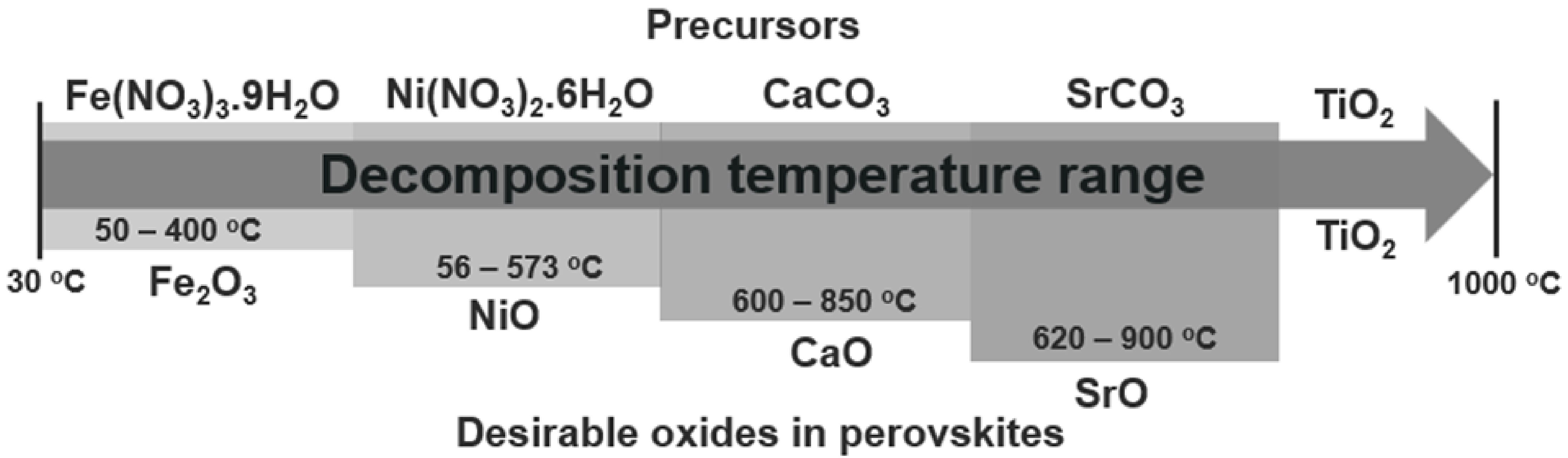


(b)

A combined TG curve, DSC curve, and temperature response for (a) $Fe(NO_3)_3.9H_2O$ and (b) SEM image of $Fe(NO_3)_3.9H_2O$

Materials and Method

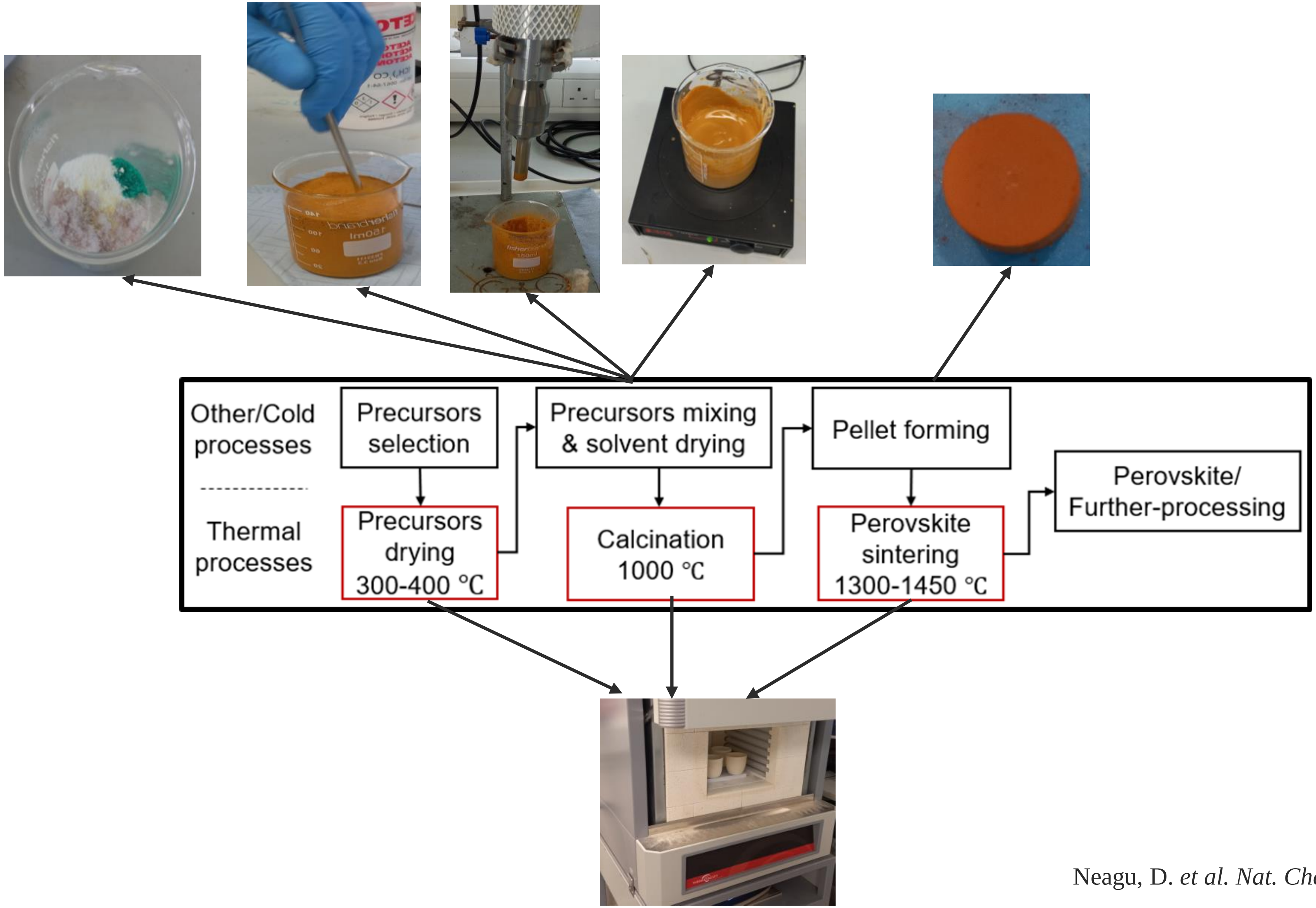
- **Selection of synthesis route from the preliminary study on the precursor materials**



Key findings from TGA of the precursor materials, predicting 1000 ^oC as the calcination temperature for the novel perovskite

Materials and Method

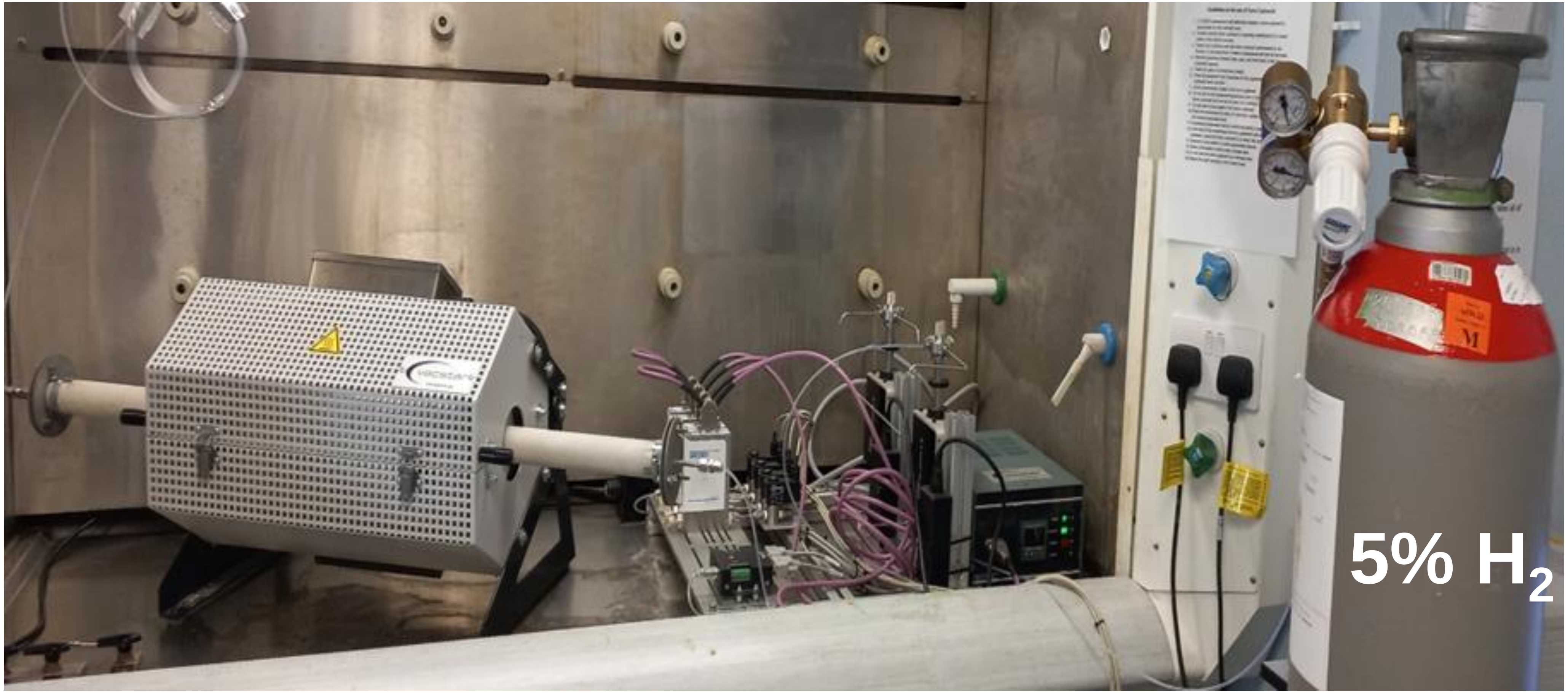
- **Selected synthesis route:** a modified solid-state synthesis method



Neagu, D. *et al. Nat. Chem.* **2013**, 5, 916–923.

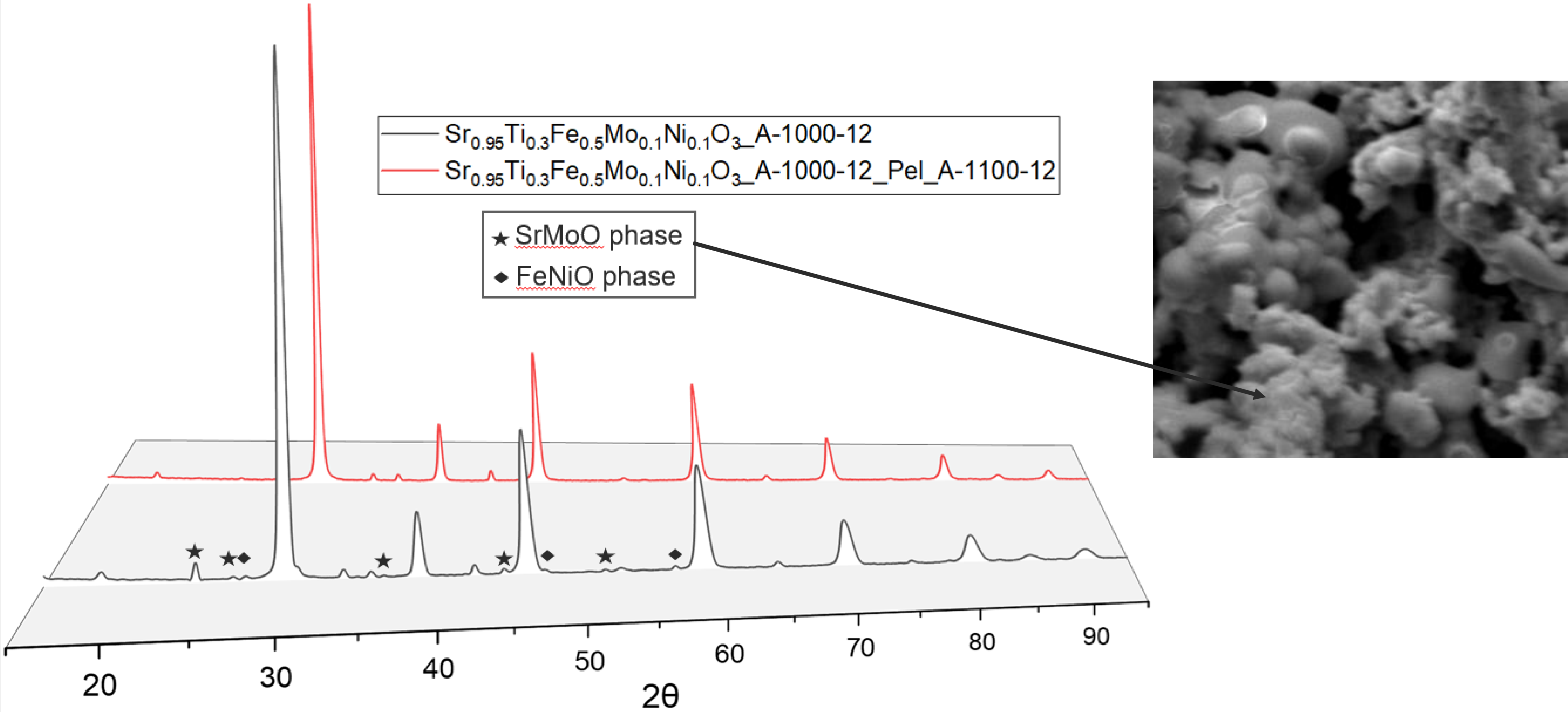
Materials and Method

❖ Reduction of Perovskite Samples.



A reduction furnace set up for exsolution analysis, recently setup by our research group

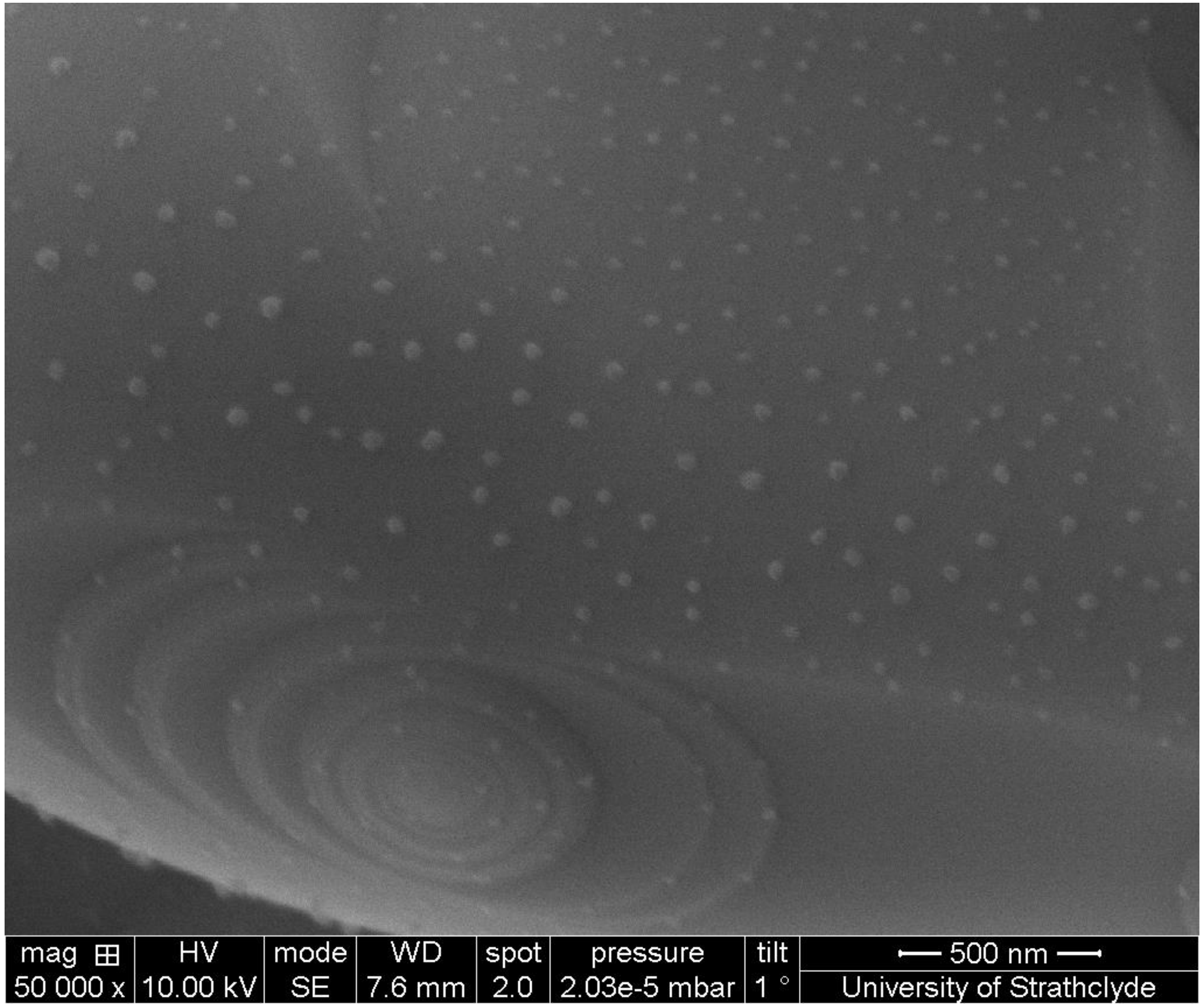
Result



Pelleting and sintering at 1100 °C enabled trace phases like SrMoO and FeNiO to diffuse into the desired perovskite crystal structure.

Result

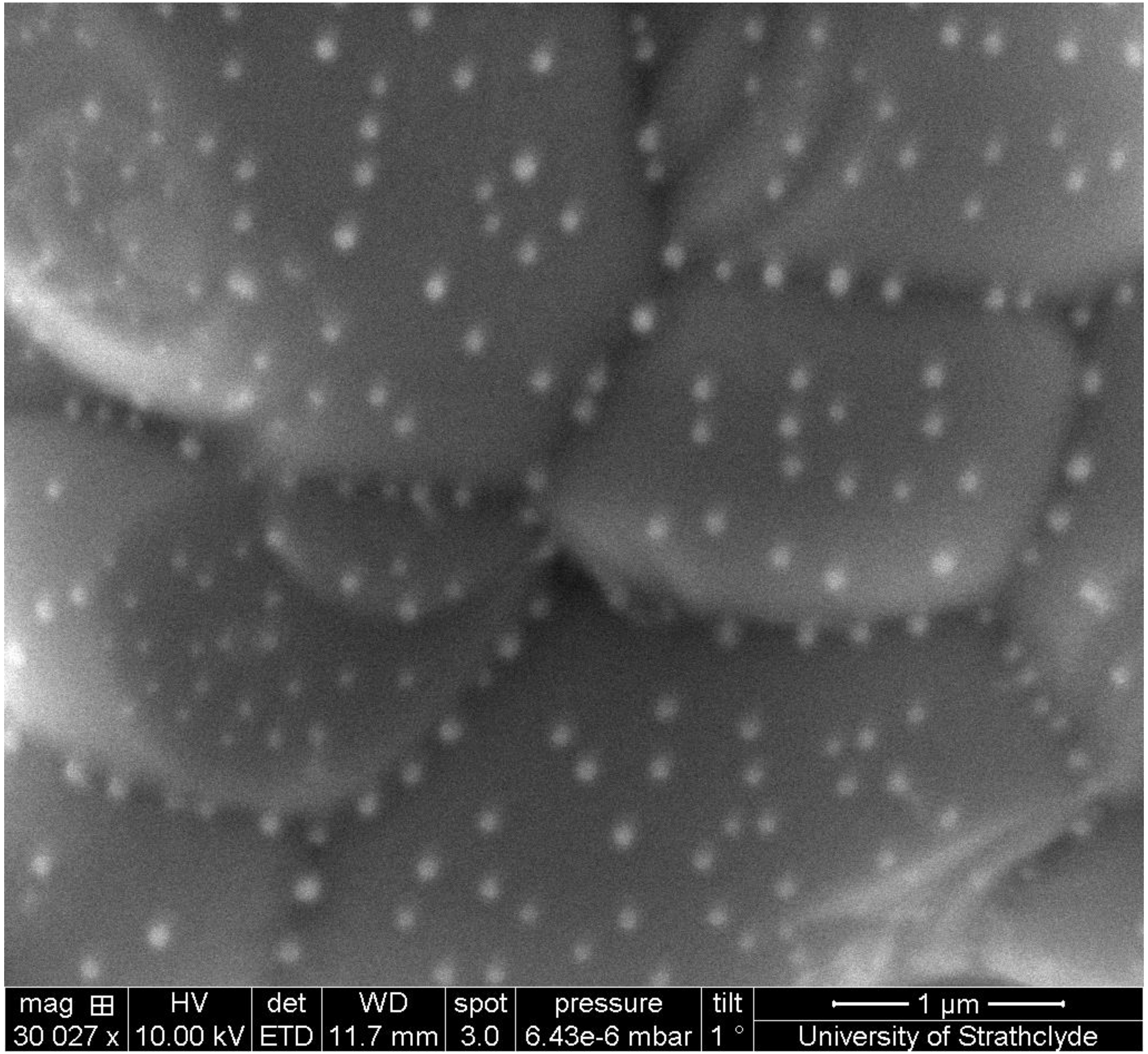
❖ SEM Image of Sample after Reduction



$Sr_{0.95}Ti_{0.3}Fe_{0.5}Mo_{0.1}Ni_{0.1}O_3$

- Reduction **at 600 ^oC** for **1 hr**.
- Exsolution was observed
- Particle size analysis revealed an **average exsolved particles size**: **33 nm**
- Some particles emerged fully to the surface and look much bigger than those partially emerging.

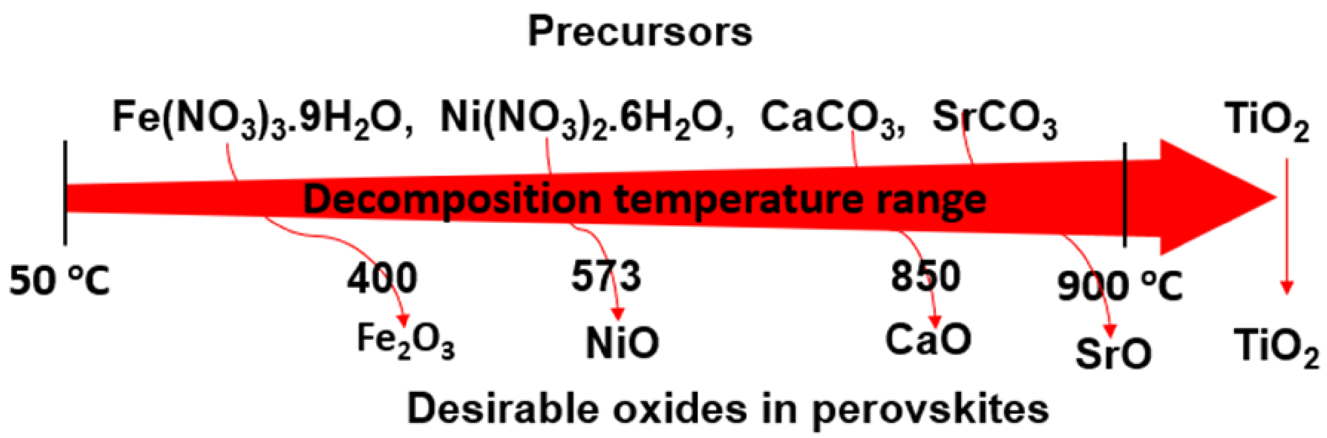
Result



$Sr_{0.95}Ti_{0.3}Fe_{0.5}Mo_{0.1}Ni_{0.1}O_3$

- Reduced at **800 ^{o}C** for **1 hr**.
- Particle size analysis revealed an **average exsolved particles size**: **68 nm**
- More particles fully emerged on the surface
- The high exsolution rate at 800 ^{o}C can be linked to the high-temperature effect.

Conclusion



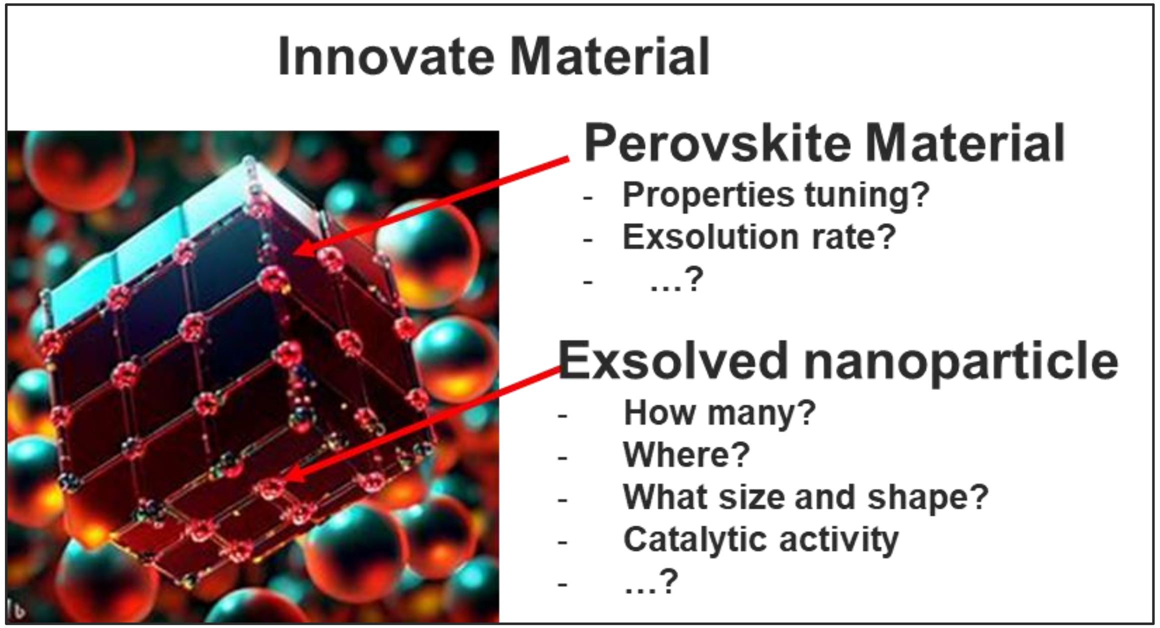
❖ The preliminary study on the precursor materials revealed their decomposition products as valuable oxides in the novel perovskite material.

❖ The modified solid-state synthesis method
- achieves homogeneous mixing of the precursors,
- minimising phase separation in the perovskite, and
- enabled further optimisation of the synthesised perovskite structure.

❖ SEM Image of $Sr_{0.95}Ti_{0.3}Fe_{0.5}Mo_{0.1}Ni_{0.1}O_3$ after reduction has shown that engineering A-site deficiency in SrTiFeMoNi perovskite can significantly improve exsolution in the materials.

Future Work

- ❖ Synthesizing other compositions of the perovskite for further studies and comparison;
- ❖ Characterisation of all the synthesized perovskites to understand the composition of the exsolved particles;
- ❖ Fabricate SOC electrodes from the synthesized perovskites for electrochemical tests;
- ❖ Modelling the exsolution process in the developed perovskite for properties tuning and performance optimisation.



Acknowledgement

I acknowledge

Dr Dragos Neagu

DiTo-H2

RSE

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Thank you for listening

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