Durham X-ray Absorption Facility (DXAF) – Newsletter October 2023

Welcome to the DXAF newsletter for October 2023. As this is the spookiest month of the year, we are hoping to treat you to some updates on the facility including the new high temperature *in situ* pellet cell and the spotlight section!

What has been going on?

In situ reactor cell is here!

From our previous newsletters, our community has been aware about DXAF's aim to conduct lab-based *in situ* XAS studies. We have recently produced an *in situ* pellet cell, which has been seeing success when using in tandem with the EasyXAFS300+ and the Hiden Catlab set up.

Initial benchmarking of the cell are as follows:

- Typical temperature range of between 20 °C to 400 °C.
- Achievable maximum temperature of 420 °C.
- Little temperature variation of cell vs a model pellet (made from macor ceramic) of 8 ± 4°C on average.
- Kapton film windows used which provide a suitable seal without attenuating X-ray radiation significantly.
- Viton O-ring used to maintain gas environment within the body of the cell.

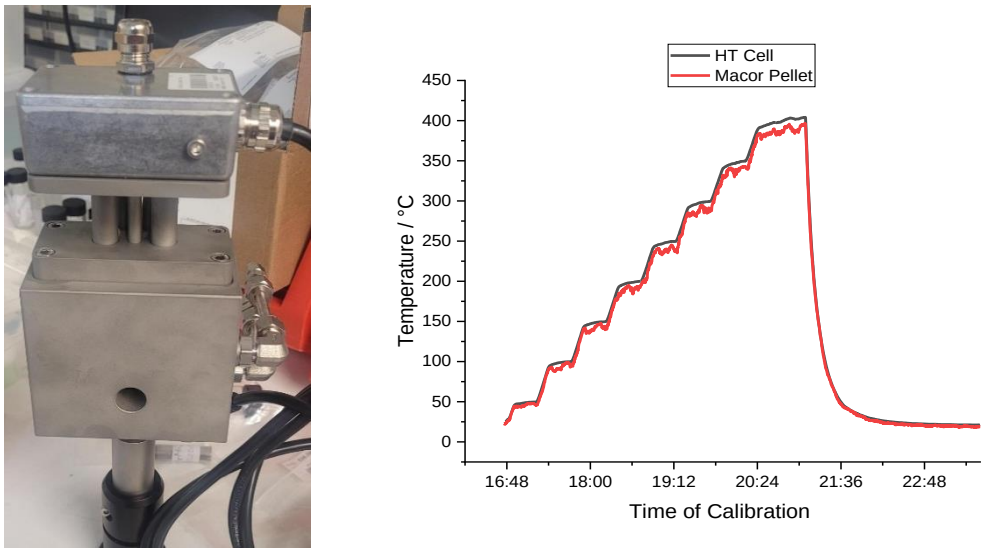

Fig. 1: Image of 'Mjolnir' the high temperature *in situ* cell (left) and a temperature calibration curve showing the variation in the temperature between the cell and a model pellet.

Our *in situ* cell, nicknamed Mjolnir for its similar appearance to the famous hammer has found use in the field already. Unlike said mythological weapon, it has been used to perform routine H_2-TPR studies on CuO to show that reductivity can be observed! (Also it does not discharge electricity like said hammer and electrically safe!).

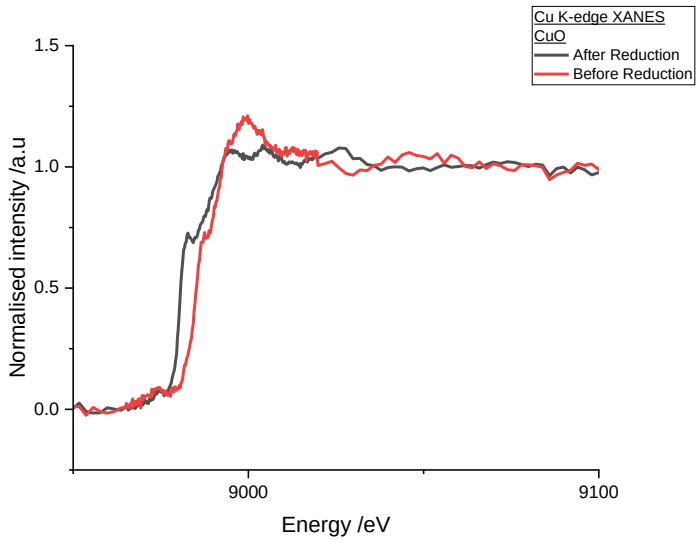

Fig 2: Cu K-edge XANES of CuO, before and after reduction. The scan duration of 6 mins per XANES scan. 10 consecutive scans before and after reduction were merged. Using 5% H_2/Ar at 50 ml min^{-1} to 420 °C at a ramp rate of 5 °C min^{-1}. Background and Cu foil were taken post reaction.

The facility is looking at how to optimise the data collection of the *in situ* setup to improve signal-to-noise whilst still maintaining suitable time resolutions for experiments. DXAF would like to thank the team within the Mechanical Workshop and Electrical Workshop for their consultation and making the cell into a reality!

Spotlight! *Operando* is in the air! Recent work from the Weckhuysen Group

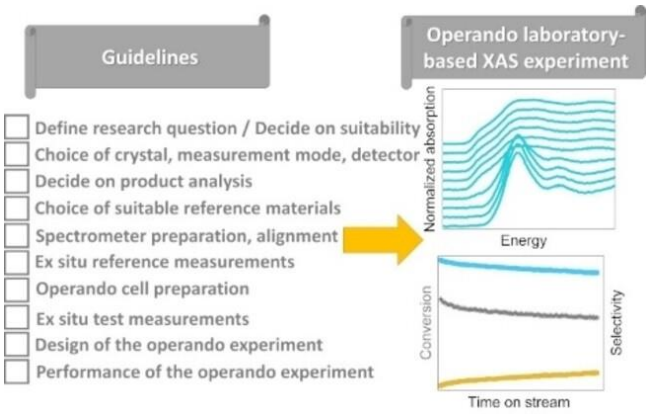

Fig. 3: Graphical abstract from paper of Genz *et al* described in the spotlight.

A very intersting read from the Weckhuysen group which focuses on the understanding required when performing *operando* laboratory based XAS studies. This article looks at the groups work with their own *operando* set up as well as looking more broadly at the application of lab-based XAS studies and the considerations during experiment. If you are interested in finding out more, please use the doi link below:

https://doi.org/10.1002/cmtd.202300027

If you would like to spotlight some of your XAS work in the upcoming newsletters , please get in touch with the facility!

What is coming up?

DXAF will now be looking to perform more i*n situ* and *ex situ* samples so please get in touch! We are still looking at the development of new *in situ* and *operando* cells so please get in touch if there is any particular set up which may be of interest to you! Please go to our website or contact us using the details below.

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Useful links:

EasyXAFS Website: **https://www.easyxafs.com/**

CONEXS Website: **https://research.ncl.ac.uk/conexs/about/**

DXAF Website: **https://www.durham.ac.uk/research/institutes-and-centres/dxaf/**

Genz *et al* paper: **https://doi.org/10.1002/cmtd.202300027**