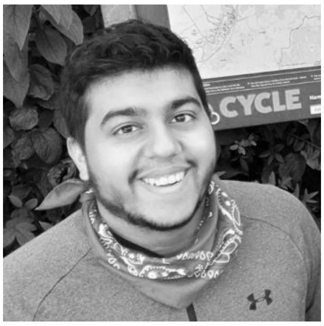
Durham X-ray Absorption Facility (DXAF) – Newsletter January 2023

Welcome to the first-ever newsletter for Durham X-ray Absorption Facility (DXAF)! Our facility aims to bring XAFS to the labs, making it capable of understanding the structure and electronics of your system without the need for a Synchrotron Radiation source. This newsletter series should give you an update about the facility, including what new and exciting things we have in store!

Introduction



My name is Monik Panchal. I am the current Technical Manager for the facility. Before the role, I was a PhD Student at University College London, based at the UK Catalysis Hub, Harwell. Under supervision from Dr Emma Gibson and in partnership with Johnson Matthey, my project consisted of multiscale imaging and *in situ* studies of gasoline particulate filters with a focus on using *ex situ* and *operando* XAFS studies at elevated temperatures (1000 °C). Hopefully, I will spend the next few months learning the instrument and its capabilities, commissioning *in situ* cells for the system, and interacting with the XAFS community!

The academic lead for the project is Simon Beaumont, an Associate Professor working in the area of heterogeneous catalysis here at Durham. Simon has worked with x-ray spectroscopy since his PhD and led the bid to EPSRC to get this exciting new equipment.

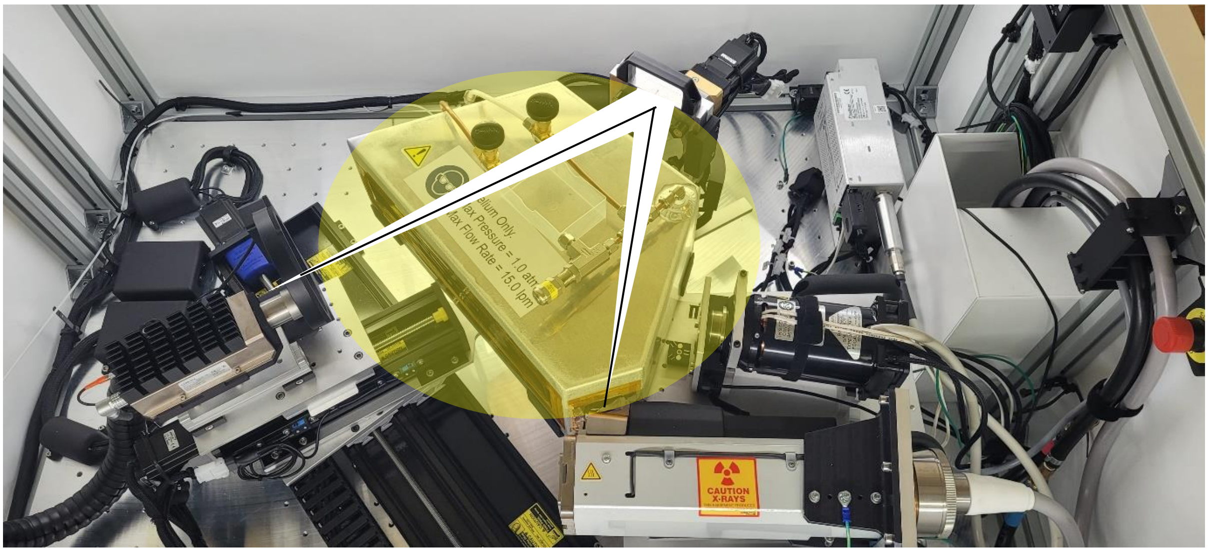
About the Facility

DXAF main attraction is the EasyXAFS300+. This instrument performs XAFS (XANES and EXAFS) and XES measurement between 5 to 18 keV. In transmission mode, we can collect XANES data from a catalyst with around 1 wt% metal (although samples would ideally have more, and many of those we have tried so far do, this was done to test the limits of the capability)!

Unlike typical Synchrotron-based XAFS experiments, the optics of the EasyXAFS300+ is based upon a Rowland circle geometry, using a “fixed-source” drive system to change the energy. The facility has available 15 different spherically-bent Bragg crystal analysers (SBCA) made from Si or Ge, as well as different x-ray sources (Ag and Mo for XAFS, W and Pd for XES)

We also have a Hiden Catlab microreactor, with the capabilities of flowing multiple non-corrosive gases (8 mfc connections), as well as volatile liquids through a vapouriser and a mass spectrometry on the outlet gas feed (HPR 20-EGA).

A separate inlet line is available connect to custom cells used within the EasyXAFS300+, so desired *operando* and *in situ* XAFS experiments can be performed. For example, *operando* studies following CO oxidation and biomass conversion or *in situ* operations such as temperature programmed reductions (H_2-TPR). The facility is also interested in branching into other fields, which includes electrochemistry and biocatalysis.



Ex situ pellet experiments are currently being performed at the facility. Commissioning of other *in situ* cells are in the pipeline, which includes an inert environment sample holder and pellet/capillary reactors capable of being heated in a gaseous environment. If you are interested in the design of a particular reactor cell, please get in touch. We will also be looking to arrange some focus groups so please let us know if you would be interested in this or watch out for them.

Example of Spectra

So far samples have been run on various elements and edges such as La, Ni and Zn. While lab source instruments are not as fast as high flux beamlines, we have also been experimenting with what information we can get from even quite rapid scanning that might be used for instance during temperature programmed experiment. As highlighted in fig. 1, low metal weight loaded Ni samples (1 wt % Ni), the collection time for an *ex situ* Ni K-edge XANES spectra can be as little as 2 mins to obtain an albeit noise signal!

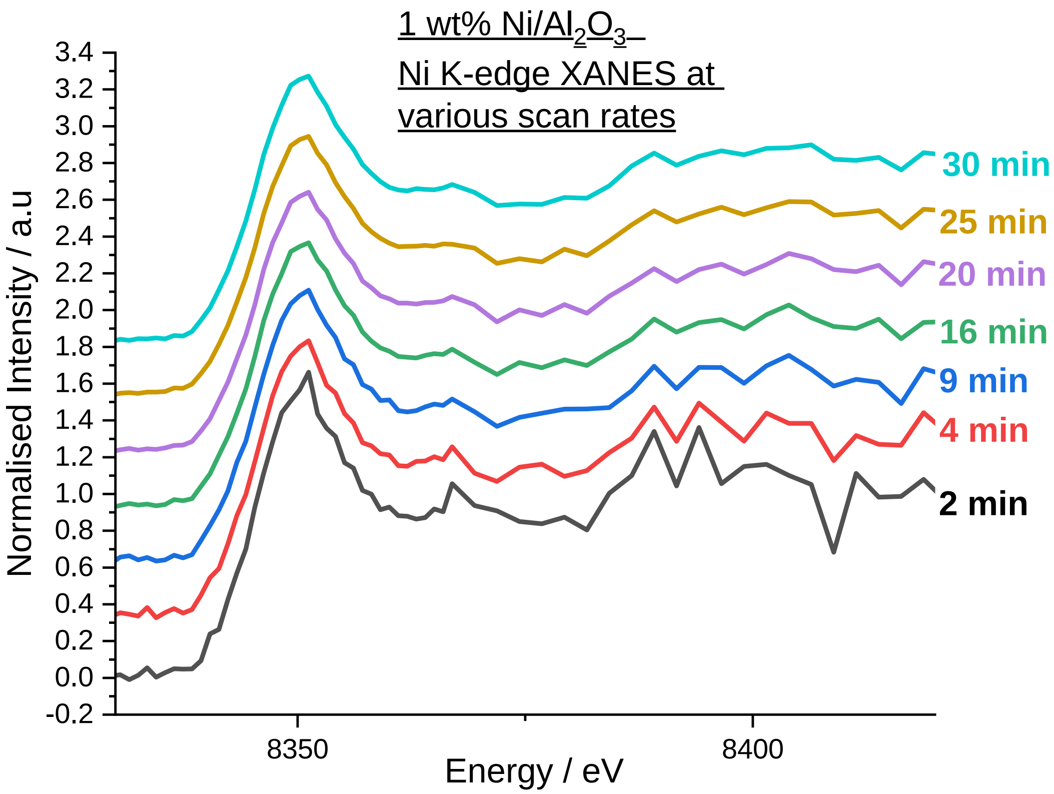


Fig. 1: Ni K-edge XANES on 1 wt% Ni /Al_2O_3 at various scan rates.

What is Next?

6 Months

- Intitial invitiation for experienced users to conduct *ex situ* samples
- Production of cells capable of *in situ* cells

12 months

- Establish a wide XAFS/XES user base of the lab equipemnt, both academic and industrial partners.
- Capability of performing *in situ* and *operando* experiments

18 months

- Have an established facility to complement synchtoron based experiments and enable different types of experiments using lab-based environment at DU chemistry.

We currently invite experienced users to run suitable "light-touch" *ex situ* pellet experiments to get an idea of the instrument's capabilities, so please get in touch if you are interested. Similarly, if you think lab source XAS measurements could be useful in longer term grant applications, please discuss costings with us / obtain letters of support – in the EPSRC funded period of DXAF (until January 2025) we will not charge for reasonable facility use which has been included in successful UKRI grants via a letter of support and can provide costs at submission stage if your grant extends beyond this.

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Useful links:

https://www.easyxafs.com/

Durham Uni DXAF website coming soon!