ESRF	**Experiment title:** *Operando* X-ray absorption spectroscopy on individual nanoparticles	**Experiment number**: HC2622
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Report:

The ability to obtain spectroscopic information at a nanometer scale could lead to a breakthrough in the understanding of catalysis. Of particular interest is the local chemical state of catalysts *during* operation. Synchrotron X-ray scanning tunneling microscopy (SXSTM) may be able to provide such information. In a SXSTM measurement, the enhancement of the tunneling current induced by X-rays is measured. In vacuum, it was shown that this signal depends so strongly on the local X-ray absorption coefficient that nanometer resolution chemical maps can be obtained[1]. Using our experience in HC1754 and HC2291, we were able to show the first proof-of-principle chemical contrast maps on a nanoparticle catalyst in a millibar gas environment during HC2622. Essential to this development was the use of new miniature reactor platform and vibrational isolation stage (see Figure 1). A double shielding scheme provided electronic noise reduction.

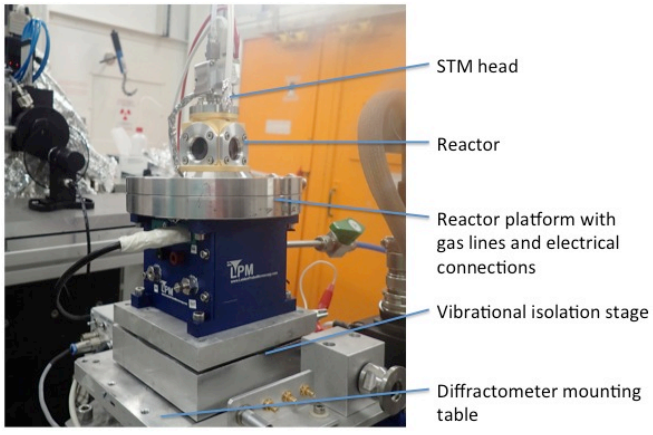

Figure 1: Miniature high-pressure SXSTM reactor, mounted on diffractometer

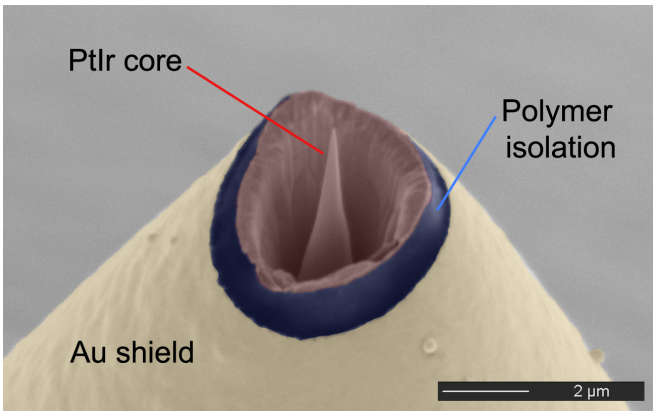

Figure 2: False color SEM image of a coaxially coated STM tip used to reduce the background signal from ion current.

Thus, we were able to provide good STM characteristics, while maintaining the ability to mount and tilt our reactor chamber on the diffractometer. To suppress the collection of the ion currents that are generated in the gas phase by photo-electrons, coaxially coated STM tips were used (see figure 2). The conductive outer shield of the coaxial tips can be biased to deflect ions away from the tip core. Special care was taken in the production process to obtain good tip sharpness, resulting in adequate resolution in the STM images.
The origin of the signal in SXSTM measurements is not well characterized. Therefore, we measured the X-ray induced increase in the tunneling current as a function of sample bias and tip-sample distance.
Finally, we have measured the local X-ray induced current on MoS_2 nanoparticles supported on Au(111) in approximately 1 mbar air. Figure 3 shows that the MoS_2 nanoparticles clearly stand out in the local X-ray induced signal, providing a proof-of-principle that chemical contrast can be obtained with nanometer spatial resolution using our methodology, even at elevated pressures.

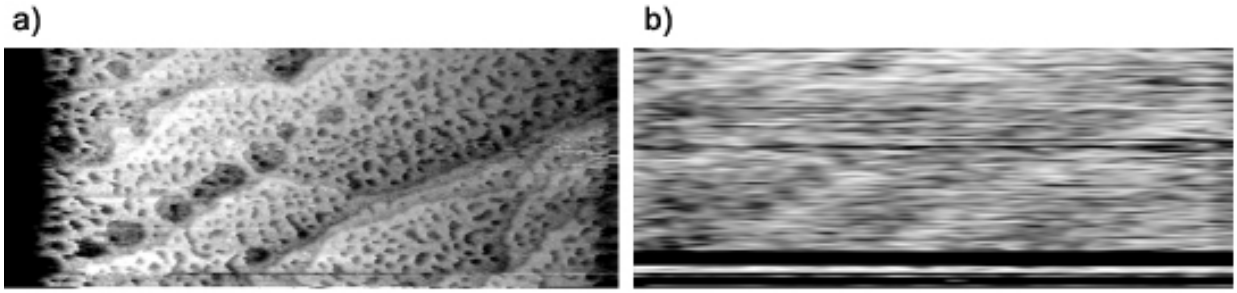

Figure 3: Proof-of-principle SXSTM data showing chemical contrast on MoS_2 nanoparticles on Au(111) in ~1 mbar air. Image size: 250 nm x 640 nm. a) Topographical image. b) chemical contrast image

[1] N. Shirato *et al., Nano Letters* 14, 11, 6499-6504, **2014**
[2] S.L.M Schroeder *et al., Surface Science Letters* 324, L371-L377, **1995**