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In Situ Photo-Electrochemical Investigation Using Surface X-ray Diffraction: From Ultra-High Vacuum to Solid/Electrolyte Interfaces

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A custom apparatus was developed at I07 Surface and Interface Diffraction beamline, Diamond Light Source (DLS) to perform operando measurements of solid/electrolyte interfaces under external potential and UV illumination using surface X-ray diffraction (SXRD).

Samples can be prepared under ultra-high vacuum (UHV) using standard methods in DLS Surface and Interface Laboratory. Pre-characterisation such as low energy electron diffraction, scanning tunnelling microscopy and X-ray photoelectron spectroscopy (LEED, STM, XPS) can be performed prior to transferring the sample under UHV to the Photo-ElectroChemical DrOplet Cell (PEC-DOC). The PEC-DOC is then transferred and mounted on I07's EH1 diffractometer for SXRD structure determination under UHV. A custom liquid delivery device comprising a working and reference electrodes is then introduced in the chamber under controlled atmosphere. A droplet of electrolyte is contacted to the surface using an endoscope, also utilised to monitor the shape of the droplet during acquisition.

Using the PEC-DOC, the electrical double layer of the $\text{TiO}_2(110)$ interface with 0.1 M KCl(aq) has been elucidated. This represents an insight into the behaviour of the semiconductor in a neutral interface to define the structure of the electrical double layer present. The electrochemical capabilities of the PEC-DOC enabled cyclic voltammograms of the surface interface to be recorded. It also allowed a bias voltage to be applied for SXRD measurements, providing a tentative insight into the electrochemical desorption taking place as a result of applied positive potential.

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I plan to submit also conference proceedings

Yes

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