Oral presentation

In situ high pressure powder diffraction studies in the home laboratory

Martin Ward

The University of Strathclyde

martin.ward@strath.ac.uk

High pressure techniques have proven instrumental in the discovery of new solid forms of materials, including agrochemicals and pharmaceuticals. Traditionally, these discoveries have been supported by high pressure single crystal diffraction studies. However, this method's reliance on relatively large, high-quality crystals (~200 microns) limits its applicability, as many materials of interest only occur in powder form or as small particles. This presents a significant challenge for high pressure diffraction studies, particularly in home laboratory settings, away from synchrotron facilities.

In response to this challenge, our team at CMAC has explored methods to collect in situ high pressure powder diffraction data within a standard home laboratory setting. This technique facilitates the analysis of materials previously unsuitable for high pressure single crystal diffraction studies. We have demonstrated that this method can collect high-quality diffraction data robust enough for phase identification through fingerprinting techniques and for performing Pawley refinement to extract unit cell parameters.

The development of HP-PXRD as a routine technique in the home lab, supported by a comprehensive workflow, significantly expands the range of materials that can be studied. This method offers a rapid compression-decompression study cycle, with data collection times as short as 60 seconds per frame, compared to the hours typically required for traditional single crystal X-ray diffraction datasets. Additionally, powder X-ray diffraction is especially beneficial in cases of reconstructive phase transitions, where the material undergoes significant structural changes. These improvements not only enhance the scope of material science research but also increase the efficiency and applicability of high pressure studies in diverse laboratory settings.