MS27-2-8 Crystallography under extreme conditions on the CRISTAL beamline @ SOLEIL
#MS27-2-8

P. Fertey [1], V. Baledent [2], G. Guillier [2], F. Legrand [1], P. Foury [2]
[1]Synchrotron SOLEIL - Gif-sur-Yvette (France), [2]Université Paris-Saclay, CNRS, Laboratoire de Physique des Solides - Orsay (France)

Abstract
Pressure as a thermodynamic potential has long been a subject of a large interest since it can drives main interesting phenomena like structure–property relations, conformational and structural transformations of molecules, polymerization, structural and electronic phase transitions, polymorphism or triggering new chemical reactions.
Understanding the crystal structure under compression is a key step to elucidate the underlying mechanisms. Structure determination of single crystals under pressure is commonly performed at room temperature, both in the laboratory and at synchrotron radiation facilities [1]. The sample is placed in a Diamond Anvil Cell which strongly constrains the completeness of the acquired data sets for structural model resolution/refinement. Devices that allow measurements under pressure and at low temperature are much less common. Most of them are based on cryostats that limit the number of degrees of freedom of the sample orientation to a single degree of rotation. If this limitation is not constraining for powder samples, it however drastically reduces the completeness of the datasets in the case of a single crystal, often involving the need to introduce more or less strong constraints to reach a satisfying structural model.
A new cryostat specifically designed for the acquisition of the most possible complete data sets will be presented. Thanks to an additional degree of freedom allowing the rotation of the DAC around its axis, the completeness of datasets can be significantly increased allowing the release of constraints in the refinement stage. Furthermore, the pressure can be adjusted in situ at any temperature between 8K and the room temperature, avoiding warming the sample to change the pressure, which is mandatory if, for example, the pressure induces an irreversible phase transition.
The performance and relevance of this new sample environment for the study of the structural properties of single crystals in the (P ,T) phase diagram will be illustrated through different examples as well as the possibility of high pressure diffraction at the CRISTAL beamline at synchrotron SOLEIL [2].

References
[1] McMahon, Top Curr Chem 315, 69–110 (2012)
[2] https://www.synchrotron-soleil.fr/en/beamlines/cristal