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Tracking geochemical reactions and going deep into the Earth with high intensity neutron powder diffraction at D20, ILL

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D20 at ILL provides the highest available intensity in constant wavelength neutron powder and liquid diffraction. The incident flux on the sample reaches up to 10^8 n/s/cm^2 at 1.3 Å wavelength. A stationary, curved linear position sensitive detector covers continuously 153.6° in 2θ with parallel 1536 cells. This makes D20 an ideal tool for in-situ diffraction studies with time constants even below a second and encourages the use of difficult sample environments. Four vertically focusing monochromators, 15 take-off angles, and optional Soller collimators in the primary beam provide a large choice in Q-space, resolution, wavelength (0.8 to 2.4 Å), and flux, adapting D20 to various levels of crystallographic complexity and rapidity of an observed phenomenon.

The continuous and simultaneous detection of series of complete diffraction patterns is necessary for the investigation of phase transitions during variation of a parameter like pressure or temperature (thermodiffractometry). One-shot experiments study the structural evolution of solids in situ during a chemical reaction with single diagrams of down to 400 ms in highest intensity configuration, allowing quantifying short-living intermediate phases and elucidate subtle structural changes. High-resolution powder diffraction patterns can be obtained in a few minutes only at the highest take-off angle. Faster, but cyclic phenomena are observable in a stroboscopic data acquisition mode. Time resolution is only limited by the travel time of neutrons through sample and detection gap, ≈ 10 μs for thermal neutrons.

Very high pressures can be obtained with the Paris-Edinburgh cells. A test experiment on the ice-VI/VII phase transition showed the possibility of using this type of pres-

sure cell to obtain pressures above 10 GPa in neutron powder diffraction. Only D20 provides the intensity to obtain statistically satisfying, clean and complete powder diffraction patterns in about 10 minutes from squeezed samples of 5 mm in diameter and 0.7 mm in height inside a TiZr gasket and between anvils of WC or BN.

Cryogenic medium pressure equipment allows the investigation of the kinetics of CO_2- and CH_4-gashydrate formation and decomposition at given gaseous pressures and temperatures. A dedicated furnace with vanadium heating element allows investigations on powders and liquids from room temperature up to 1300 C without significant background contributions from the sample environment. For even higher temperatures a mirror furnace or a furnace with niobium heating elements, both available from ILL's sample environment pool, have been used, as well as an user-provided electromagnetic levitation furnace for metallic melts. An oscillating radial collimator with a focus aperture of 22 mm and an angular coverage of 156° in 2θ will allow the use of most types of sample environment without any background contributions from, e.g., aluminium calorimeters or niobium heating elements.