International Tables for Crystallography (2018). Vol. H, Figure 2.8.13, p. 183.

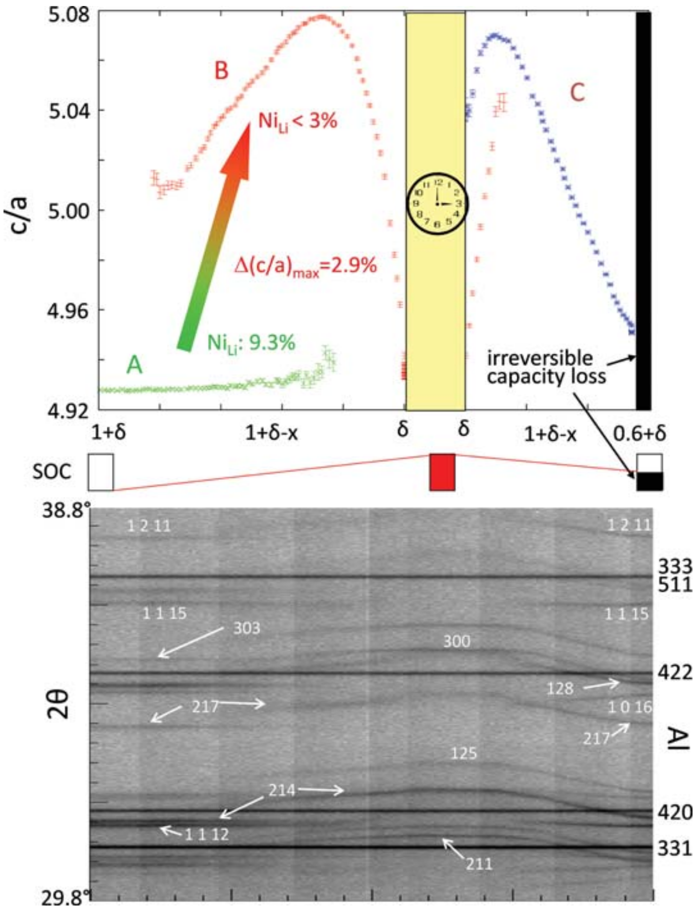

Figure 2.8.12
$Li_{1+\delta}NiO_2$ during charge and discharge. From about 210 complete diffraction patterns [a small section is shown below (λ = 0.499366 Å)], the structural response to Li extraction and insertion was monitored. In addition to changes in the unit-cell metric, the distribution of Li and Ni onto layers becomes more ordered during the first highly charged state. A pronounced capacity loss is observed during discharge after a holding time of 3 h in this overcharged state. A, B and C are three successively appearing phases. SOC = state of charge.

Such *in situ* studies are very important for elucidating the working mechanism and degradation processes for intercalation electrodes (Senyshyn *et al.*, 2012). Nevertheless, complementary methods are also essential for providing all the necessary information, especially about the surface near-interface region between the electrode and electrolyte, which has to be studied with surface-sensitive and local methods.

2.8.3.3. *Diffraction under a magnetic field*

2.8.3.3.1. *General remarks*

The majority of synchrotron and neutron experiments are currently limited to superconducting magnets with fields of 5–16 T. When higher fields are required there are essentially two possible solutions: pulsed resistive or steady-field resistive (or hybrid: resistive inner coil, superconducting outer coil) magnets. Pulsed fields are often used when signals are strong and the signal-to-noise ratio is good, whereas steady fields are primarily used for techniques with relatively long counting times and many data points. The relatively short pulse duration (from microseconds to milliseconds) along with the rather large sample volume required severely limit the use of pulsed magnetic fields in powder diffraction applications as well as more exotic methods of achieving high magnetic fields, *e.g.* magnetic flux compression or single-turn coils (Schneider-Muntau *et al.*, 2006). Furthermore,

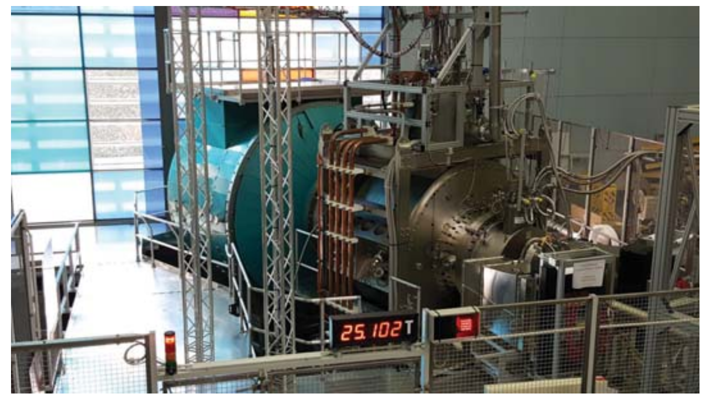

Figure 2.8.13
The High Magnetic Field Facility for Neutron Scattering at the Helmholtz-Zentrum Berlin has two main components: the High Field Magnet (HFM) and the Extreme Environment Diffractometer (EXED). Courtesy of Dr O. Prokhnenko.

because of the large stresses, the lifetime of a resistive/pulsed magnet is finite: pulsed magnets have typical lifetimes of 500 shots at 95% of the design field, and their lifetimes are virtually independent of pulse duration.

At present, the maximum field for technical superconductors is 23 T and modern developments in superconductor design along with robust magnet manufacturing techniques have made possible high-flux-density magnets for neutron scattering studies up to 16 T. For resistive magnets, there are in principle no limitations to the generation of the highest continuous fields apart from economics, as approximately 1 MW of electric power is consumed per 1 T field strength. Therefore, in order to reduce the running costs (*i.e.* the required power per unit magnetic field), hybrid magnets are becoming increasingly popular. In this context we mention the joint developments between the National High Magnetic Field Laboratory (Tallahassee, FL, USA), the Spallation Neutron Source (Oak Ridge, TN, USA) and Helmholtz-Zentrum Berlin (Germany) in the development of high-steady-field (25 T with a 4 MW resistive insert, 30 T with an 8 MW resistive insert) hybrid magnets for neutron scattering (Bird *et al.*, 2009). A high-field magnet has been installed and is in routine user operation (Fig. 2.8.13) at the Extreme Environment Diffractometer (HFM-EXED) of Helmholtz-Zentrum Berlin (Prokhnenko *et al.*, 2015).

On the other hand, it is quite simple to produce magnetic flux density uniformity or homogeneity over the required sample volume down to the p.p.m. level with a solenoid magnet. However, the majority take the form of split-pair solenoids. With this setup, the access to the sample environment (along the magnetic axis) can be orthogonal to the beam-access plane. The split-coil setup is the most popular in neutron scattering, but the geometry constraints imposed by the neutron aperture make the creation of very uniform flux density much more difficult. In general, for a typical neutron-scattering sample volume of 1 cm^3, the magnetic field homogeneity is normally limited to the range 0.1 to 5%. As the available current density for a given conductor decreases with increasing flux density, the flux density seen by the superconductor inside the magnet windings is greater than the 'nominal' central value. This is particularly the case for split-pair magnets, where the ratio of the two can be large, *e.g.* for a central value of 9 T, a ratio of 1.6 gives a maximum flux density of 14.3 T (Brown, 2010).

As already pointed out, diffraction studies under a magnetic field are almost always performed with neutrons. However,

references