allow bulk properties to be investigated. The cell had a dumbbell configuration, assembled from commercial stainless-steel components, that held most of the hydrothermal liquid, and a zero-scattering Ti–Zr alloy sample compartment with a 0.4 mm wall thickness. This choice of material and wall thickness for the sample cell dramatically reduced the background scattering from the container, but limited the operational range to temperatures up to 573 K and pressures up to 9 MPa. The cell was commissioned at the Wombat neutron powder diffractometer at ANSTO, using the *in situ* kinetic study of the hydrothermal phase transformations from leucite ($KAlSi_2O_4$) to analcime ($NaAlSi_2O_6{\cdot}H_2O$) as a demonstration of the capabilities of the equipment.

2.9.4. Complementary methods and future developments

Standard crystallographic powder-diffraction methods can provide information about component phases and particle size, and can also be used to determine crystal structure, but the data quality required means that long-range crystalline order needs to be present. However, many real systems have amorphous components or demonstrate various degrees of disorder. In order to provide complementary information on the disordered components, alternative techniques are needed. In the early 1990s, Couves and co-workers started to combine XRD with XAFS in one setup in order to complement the XRD data (Couves *et al.*, 1991); they were quickly followed by Clausen and co-workers (Clausen *et al.*, 1993). Shortly afterwards, small-angle scattering (Dokter *et al.*, 1994) and vibrational spectroscopic techniques such as infrared and Raman were also added to complement the diffraction information (Newton & van Beek, 2010). More recently, a very old technique (Tarasov & Warren, 1936) based on the pair distribution function (PDF) has become immensely popular with the advent of high X-ray energies and efficient detectors (Chupas *et al.*, 2003). The PDF technique does not depend upon any assumptions about long-range crystalline order and can therefore be used to extract information on amorphous materials, defect structures and the structures of nanoparticles (see Chapter 5.7). It has the same huge variety of applications as traditional diffraction methods, and provides complementary information. Several of the *in situ* cells described above can also be used for combined techniques and PDF experiments at synchrotrons. One of the many reasons behind the rapid success of the PDF method is the availability of well developed software for data analysis (Juhás *et al.*, 2013) and modelling (Neder & Proffen, 2008).

We have seen in some of the examples above that acquisition times are reaching down into the millisecond range and the quantity of data being delivered by modern systems is becoming increasingly difficult to analyse. There is progress in automated sequential and parallel parametric refinements with traditional data-analysis software. However, we believe new strategies are necessary in order to make better, more efficient use of modern detectors. There are efforts in this direction in automated chemometric methods (Burley *et al.*, 2011) stemming from spectroscopy. However, these algorithms are not always well adapted to analyse data derived from powder-diffraction measurements. Chernyshov *et al.* (2011) have performed theoretical and experimental work taking the interference nature of diffraction into account in their method, which is based on modulation. Nevertheless, improvements in data analysis are still trailing far behind experimental progress and much effort will be necessary in this area. Choe *et al.* (2015) have even performed stroboscopic high-resolution powder diffraction on piezoelectric ceramics, detecting sub-millidegree shifts with microsecond time resolution.

In contrast to the pursuit of speed, the Diamond Light Source have decided to extend their powder-diffraction beamline and make it suitable for experiments lasting several months or even more, by moving slowly aging samples automatically into the measurement position at regular intervals (hours, days or even weeks) in a long-duration experiment (LDE) facility. Relevant applications are in batteries, fuel cells, crystallization, gas storage, mineral evolution, seasonal effects, thermal and electrical power cycling, and corrosion science.

In addition to the developments in instrumentation presented here, the availability of new radiation sources is opening up many interesting possibilities for studying chemical reactions. Not only are more, and better equipped, synchrotron beamlines becoming operational, but there are new facilities in planning or under construction that will dramatically change the way in which chemical processes can be investigated. New spallation sources and free-electron lasers (FELs) open up new possibilities in the time and space domains. In particular, FELs will facilitate the study of reactions on sub-picosecond timescales. Preliminary experiments using picosecond to nanosecond time resolution have already been carried out on synchrotron beamlines to investigate transient structural changes in organic powders (Techert *et al.*, 2001). It is evident that the huge increase in flux per pulse and the much shorter pulse length available from FELs will open up completely new dimensions in the field of *in situ* experiments.

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