International Tables for Crystallography (2018). Vol. H, Section 2.3.4.4, pp. 91–92.

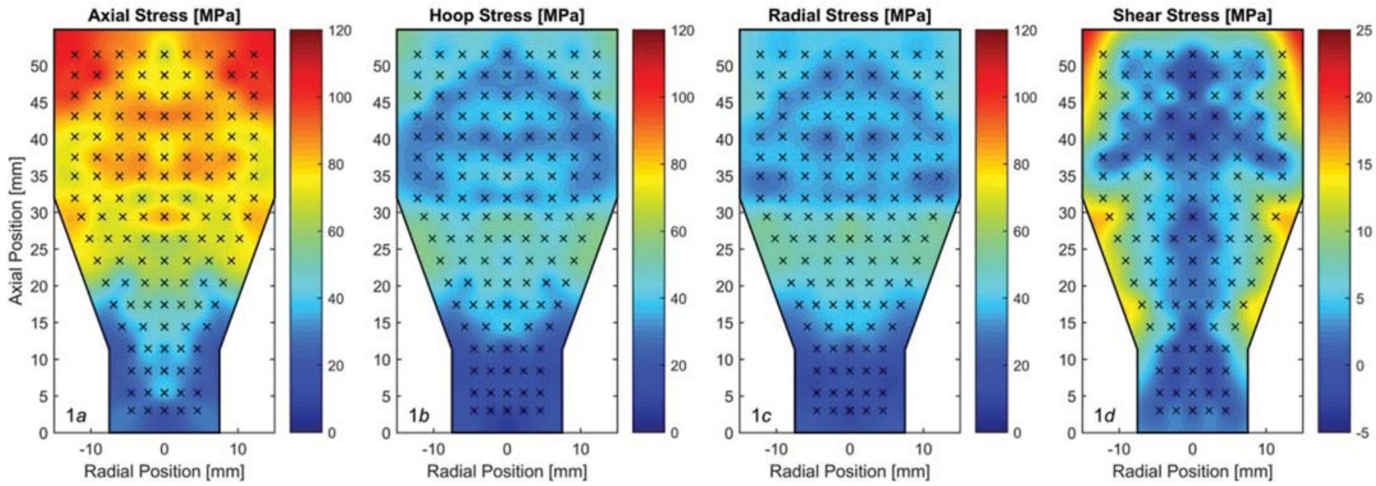

Figure 2.3.23
Stress distribution for four stress components in an iron powder compacted within a convergent die (see also Zhang *et al.*, 2016).

neutrons of different wavelength, each is recorded under different conditions for attenuation and extinction. In addition, to make full use of all the data, whole pattern or Rietveld analysis using a preferred-orientation (texture) model has to be conducted for each of the multitude of diffraction patterns recorded. As well as being time consuming, the reliability of the resultant pole figures and orientation density function is governed by the quality of all the individual models (for background, peak shape, peak width, sample centring, attenuation *etc.*) within the Rietveld refinement as well as the ability of the preferred-orientation model in the Rietveld program to accurately fit the real texture. A pure model-independent texture measurement can only be obtained using CW or TOF single-peak methods.

The instrument WISH at ISIS represents a departure from the normal TOF diffractometer design in that it receives *long wavelength* neutrons (1.5–15 Å) from a cold neutron source at Target Station 2. Ballistic supermirror neutron guides and three choppers deliver neutrons in an active bandwidth of 8 Å for a given chopper setting (https://www.isis.stfc.ac.uk/Pages/Wish.aspx). The pixelated ^{3}He detectors cover Bragg angles in the very wide range 10–170°. WISH is designed for the study of complex magnetic structures and large-unit-cell structures in chemistry and biology. Polarization analysis is available to assist the former.

The concept of long-wavelength neutron powder diffraction will be taken a step further in the DREAM instrument planned for the European Spallation Source (ESS, https://europeanspallationsource.se/realizing-dream-versatile-powder-diffractometer). This instrument will receive neutrons simultaneously from *thermal* and *cold* neutron moderators. It will have a complex array of choppers to shape the incident pulse prior to arrival at the sample. Modelling has indicated that intensity gains of a factor of 10–30 are to be expected and that the instrument may be able to deliver $\Delta d/d$ as low as 4×10^{-5}, albeit at very long wavelengths. More typically the projection is that $\Delta d/d$ as low as 1×10^{-4} could be achieved with more conventional wavelengths. Perhaps the major advantage of the instrument will not be its absolute resolution but the ability to change resolution over the full range during the experiment by simply altering the chopper settings. Therefore unexpected phenomena (phase transitions *etc.*) can be tracked during the initial experiment with no time lost by having to prepare a proposal for a different higher-resolution instrument.

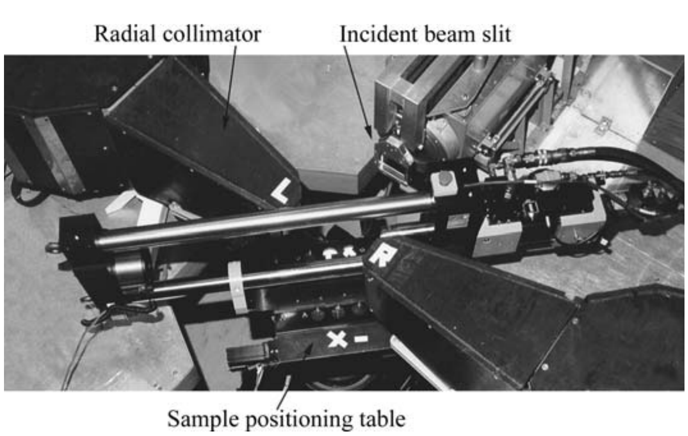

Figure 2.3.24
The engineering diffractometer ENGIN-X at ISIS. The incident beam enters through the flight tube at the top and the left (L) and right (R) 90° detector banks simultaneously record patterns with the scattering vector perpendicular and parallel to the sample axis, respectively. A mechanical testing machine used for *in situ* application of loads is also shown (https://www.isis.stfc.ac.uk). (Credit: STFC.)

2.3.4.4. *Comparison of CW and TOF diffractometers*

The preceding discussion has demonstrated that, although not necessarily the case for other types of neutron scattering, powder diffraction can be very successfully conducted on either CW or TOF instruments. Their relative advantages for the various types of powder-diffraction experiment are embedded in the discussion above and summarized in Table 2.3.5.

Plotting and summarizing the approximate intensity and resolution of different types of neutron diffractometer may be of assistance in assessing the options (Fig. 2.3.25). In the figure, resolution is shown as the inverse of the FWHM ($\Delta d/d$) and intensity is shown as the inverse of the time in seconds taken to record a single diffraction pattern, so that improvements follow the positive x and y axes.

There are two particular cases where the distinction between CW and TOF instruments can determine the success or failure of a neutron powder-diffraction experiment. The first is where crystal structures or phase transitions involving extreme pseudosymmetry are being studied. In this case, the very high resolution available over the entire Q-range (d-spacing range) using high-resolution TOF instruments such as HRPD at the ISIS

Table 2.3.5
Advantages of CW and TOF instruments (modified from Kisi & Howard, 2008)

CW	TOF
(1) Incident beam may be essentially monochromatic, in which case the spectrum is well characterized	(1) The whole incident spectrum is utilized, but it needs to be carefully characterized if intensity data are to be used
(2) Large d-spacings are easily accessible for study of complex magnetic and large-unit-cell structures	(2) Data are collected to very large Q values (small d-spacings)
	(3) Few cold neutron instruments are available for study of complex magnetic and large-unit-cell structures
(3) Can fine tune the resolution during an experiment	(4) Resolution is constant across the whole pattern
	(5) Very high resolution is readily attained by using long flight paths
(4) More common	(6) Complex sample environments are very readily used if 90° detector banks are available
(5) Peak shapes are simpler to model	
(6) Absorption and extinction corrections are relatively straightforward	(7) Simpler to intersect a large proportion of the Debye–Scherrer cones with large detector banks
(7) Data storage and reduction is simpler	
(8) Extremely rapid data collection and stroboscopic measurements are feasible	(8) Very fast data collection is feasible
(9) Engineering diffractometers are very well suited for strain scanning in complex objects	(9) Engineering diffractometers use an extended diffraction pattern, ideal for *in situ* loading and/or heating
(10) Texture is straightforward to measure on engineering diffractometers	(10) Texture can be measured on universal instruments

facility (UK) or SuperHRPD at J-PARC confers a particular advantage. The CW equivalent high-resolution powder diffractometers such as D2B at ILL and ECHIDNA at ANSTO can almost match the absolute resolution of the TOF instruments, D2B achieving $\Delta d/d$ of 5.6×10^{-4}; however, the resolution function for a CW diffractometer [equation (2.3.18)] has a strong minimum and so this resolution can only be achieved over a restricted range of d-spacing. The reflections appearing in the highest-resolution zone can be shifted by wavelength changes, which of necessity require re-recording of the pattern.

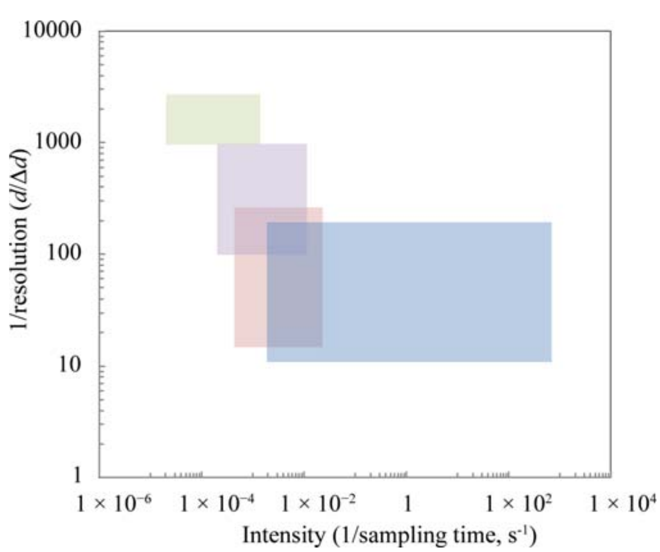

Figure 2.3.25
Schematic showing regions of intensity–resolution space in which different diffractometer types typically operate. High-resolution TOF diffractometers operate in the green area, engineering diffractometers (TOF or CW) in the purple area, multi-purpose TOF diffractometers such as POLARIS in the orange area and very high intensity CW diffractometers in the blue area.

The second extreme case is when rapid kinetic behaviours are to be studied. In this case, a small number of CW diffractometers (*e.g.* D20 at the Institut Laue–Langevin or WOMBAT at ANSTO) have a distinct advantage. Therefore at this time, processes that occur reproducibly and uniformly over a large sample on sub-1 s timescales are best suited to stroboscopic studies using one of the very rapid CW diffractometers available. There are nonetheless a great number of processes that can be studied on the timescales accessible using TOF, where near-constant resolution across the entire diffraction pattern lends considerable advantage.

If unaffected by extremes of resolution, intensity or highly specialized data types (stress, texture *etc.*), the choice between a CW or TOF instrument can be made based more casually on proximity to neutron sources and the access arrangements for national or regional neutron users.

2.3.5. Experimental considerations

2.3.5.1. *Preliminary considerations*

Neutron-diffraction studies are motivated by a desire to exploit the unique properties of neutrons as listed in Sections 2.3.1 and 2.3.2. As access to neutron diffraction is carefully regulated through an experiment proposal system, considerable planning is required in order to write a successful proposal. Owing to the expense of operating a neutron source and pressure on instrument time, there is an onus on the experimental team to make the best use of neutron beam time. Consideration should be given to the type of instrument required, the resolution that is needed, the d-spacing range of interest, how long each pattern will take to record, the requirement (or not) for standard samples and whether a special sample environment is needed.

references