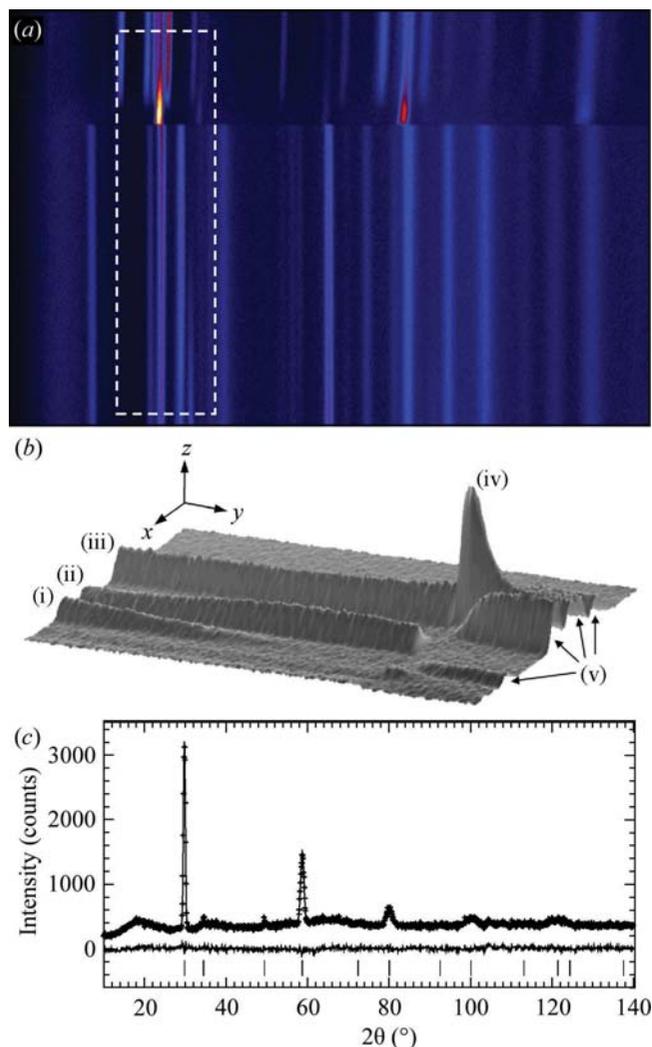


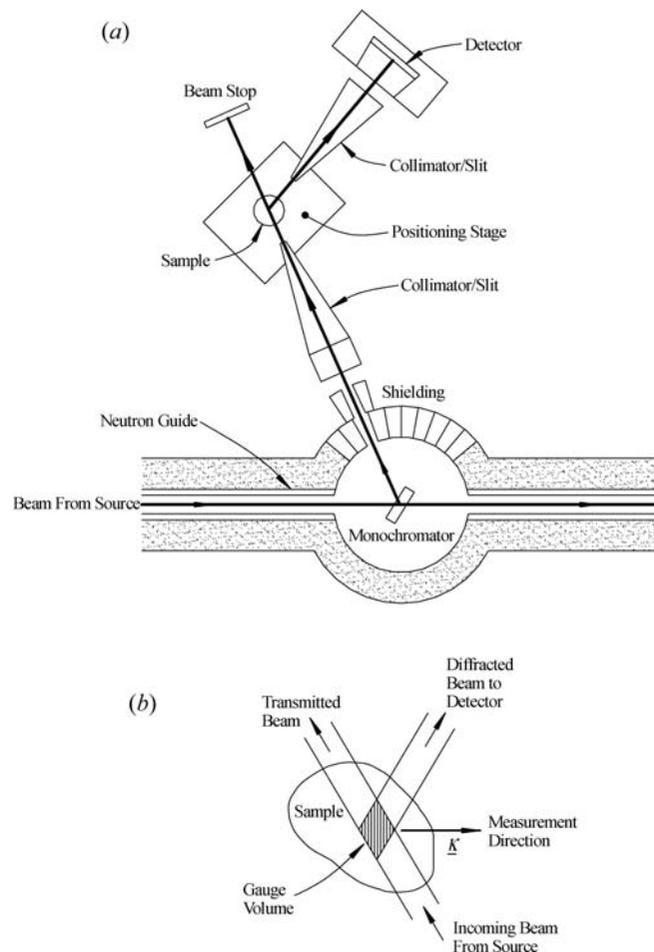
2. INSTRUMENTATION AND SAMPLE PREPARATION

**Figure 2.3.21**

Neutron powder-diffraction patterns during combustion synthesis of Ti_3SiC_2 recorded in 400 ms each on the diffractometer D20 at ILL (Riley *et al.*, 2002). Panel (a) shows an overview of the reaction process with time vertical, diffraction angle horizontal and intensity as colour/brightness. Panel (b) is a three-dimensional view of the portion enclosed by dashed lines in (a), representing 140 s of reaction, wherein the numbered reflections show (i), (ii) a phase change in Ti, (iii) SiC, (iv) formation of an intermediate phase $\text{Ti}(\text{Si},\text{C})$ and (v) growth of the Ti_3SiC_2 product. Panel (c) illustrates *via* Rietveld refinement the high quality of diffraction patterns even on this short timescale.

TOF engineering diffractometers record a full diffraction pattern at each position. Localization of the gauge volume is achieved using symmetric detector banks and radial collimators on either side of the sample position (Fig. 2.3.24). All other instrument-design criteria are generally secondary to this, as a parallelepiped-shaped gauge volume allows a seamless strain (stress) map to be obtained. These instruments are usually 40–50 m long and have moderately high resolution, which allows peak positions and hence strains to be measured to a precision of 5×10^{-5} in favourable circumstances. In common engineering materials (steels, aluminium alloys *etc.*) this equates to an absolute minimum stress uncertainty of 4–10 MPa. The extreme resolution that would be available using very high resolution designs like HRPD and Super-HRPD (above) is sacrificed in order to obtain data on a reasonable timescale given the generally small gauge volume (0.5–30 mm³) and the need to map the strain field piecewise over an extended region of the sample.

Although it is not usual for instruments to be specifically designed for the purpose, neutron diffraction is also particularly

**Figure 2.3.22**

Illustrating (a) a CW engineering diffractometer and (b) the formation of a gauge volume at the intersection of the incident and diffracted beams.

useful for studying crystallographic texture in materials, as the neutron-diffraction pattern is not distorted by surface coatings or preparation methods. In principle, any diffractometer can be used for measuring texture simply by recording a large number of diffraction patterns with the sample rastered in small angular intervals (5° is common) about two mutually perpendicular axes to form a grid over all orientations. This is extremely time consuming on a conventional CW diffractometer, although the whole pattern is captured each time, as the intensity recorded for the different reflections is subject to different corrections. This can be greatly sped up by using a CW engineering diffractometer (SALSA, KOWARI) with an intense, well collimated incident beam and fitted with an area detector. For example, on KOWARI, the detector spans 15° in both horizontal and vertical directions and so the sample needs to be re-positioned far fewer times. An added advantage is that the diffraction geometry is identical for each sample position and almost so for each reflection studied, and so a pure (*i.e.* model-independent) texture measurement is obtained. Texture measurements on modern TOF diffractometers (*e.g.* GEM, POLARIS, POWGEN, NOMAD and iMATERIA) are in principle quite straightforward. Because there are detectors in many positions all around the sample, the scattering vector and hence orientation of diffracting planes (crystal orientation) is sampled in many orientations all in one data collection. If data from the individual detectors are not ‘focused’ into composite diffraction patterns as for crystal-structure studies, then very few re-orientations are required to record data representing the full texture. However, since each reflection in each detector bank is sampled using