International Tables for Crystallography (2018). Vol. H, Figure 2.6.6, p. 153.

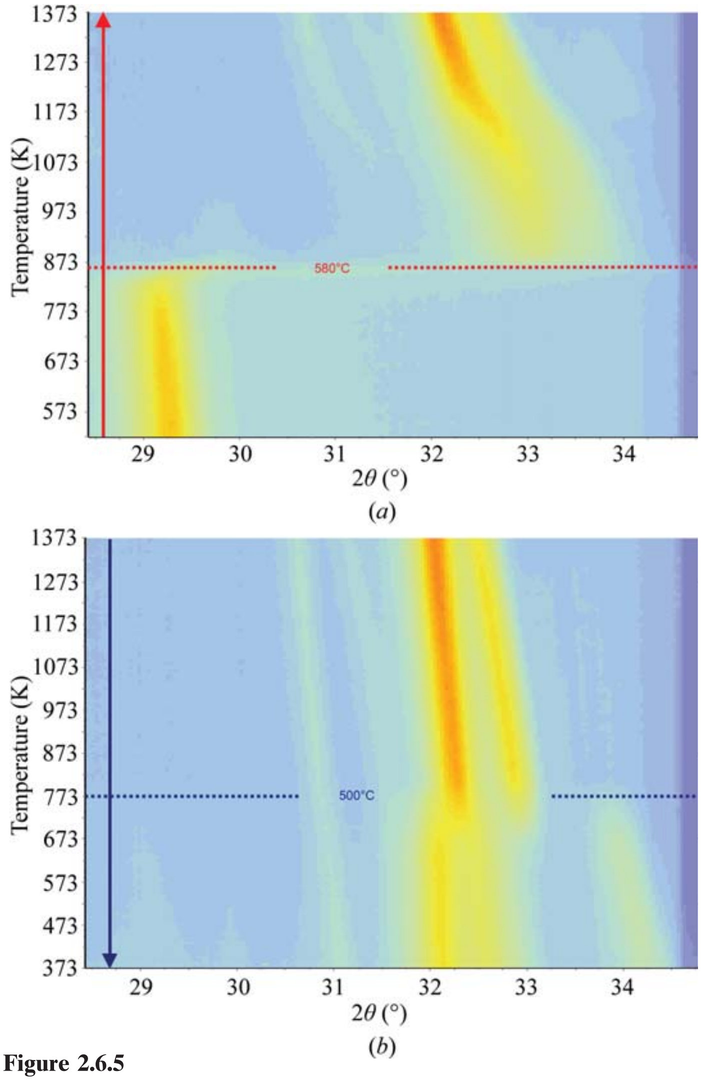

Figure 2.6.5
(*a*) Upon heating, $CaCO_3$ (the peak at about 29.3° in 2θ) reacts with SiO_2 (amorphous); at 853 K the new phase α'_L-Ca_2SiO_4 is formed (the peak between 33 and 32° in 2θ). (*b*) During cooling α'_L-Ca_2SiO_4 (the two peaks between 33 and 32° in 2θ), a different dicalcium silicate polymorph is formed at 773 K; this is β-Ca_2SiO_4.

et al., 2006). Such a furnace can heat a specimen to >2000 K in air. A recent application of this furnace is the characterization of high-temperature phase transitions in $Zr_2P_2O_9$ (Angelkort *et al.*, 2013).

2.6.6.4. *Domed hot stage*

Sample stages with an X-ray transparent dome, such as the DHS 1100 domed hot stage manufactured by Anton Paar (Fig. 2.6.6), give another dimension to polycrystalline diffraction. The dome is made of highly transparent graphite. The transmission of the primary and diffracted beams depends on the wavelength used, and for Cu *Kα* radiation 65% is transmitted. The dome can be used on most of the commercially available modern multipurpose X-ray diffractometers with linear or two-dimensional detectors. Mounted on an *XYZ* table or a cradle, these sample stages can be used to study texture, stress/strain and other phase-induced changes in (for example) thin-film layers under non-ambient conditions.

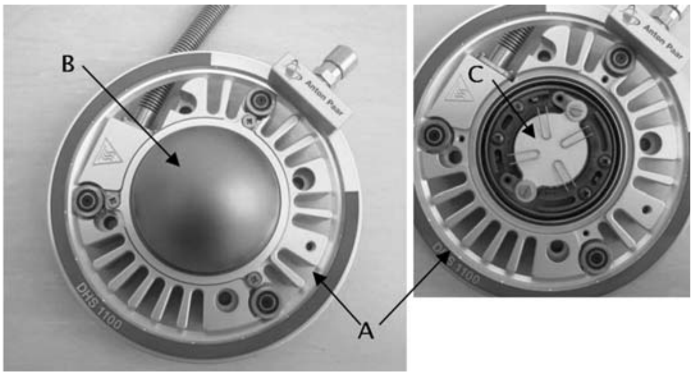

Figure 2.6.6
Sample-heating stage (Anton Paar DHS 1100) with lightweight, air-cooled housing (A), dome-shaped X-ray window (B) and heating plate with sample fixation (C).

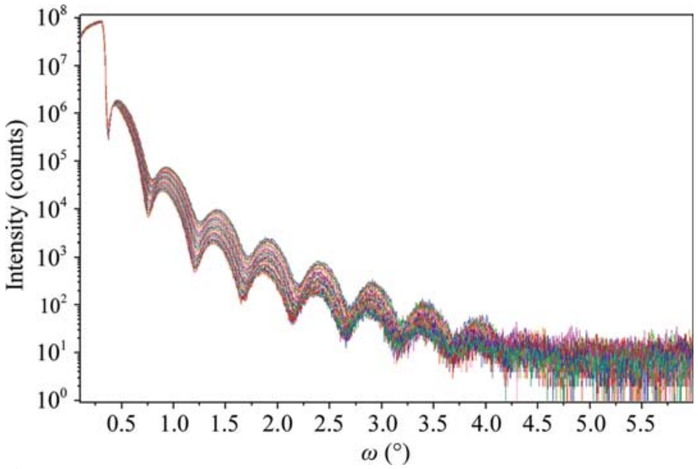

Figure 2.6.7
Monitoring of layer thickness and roughness by X-ray reflectivity measurements during annealing at 823 K.

Example: thin films. A great deal of research has been devoted to the development of gallium nitride (GaN)-based high-electron-mobility transistor (HEMT) structures (Kelekci *et al.*, 2012; Butté *et al.*, 2007). The structural quality of the layers and their interfaces is critical for the performance of the device (Teke *et al.*, 2009). Detailed knowledge of the effects of further process steps, such as thermal annealing, on these parameters is crucial. X-ray reflectivity can be used for monitoring, among other things, the layer thickness and (interface) roughness (Daillant & Gibaud, 2009). To monitor the annealing process, a wurtzite-type AlInN/AlN/GaN/ heterostructure was mounted on a DHS 1100 domed hot stage; 26 scans were made, each of which lasted 1 h and 59 min at a temperature of 823 K (Fig. 2.6.7). From these reflectivity measurements the activation energy could be calculated and compared with the results from X-ray diffraction data from a nominally identical structure (Grieger *et al.*, 2013). The same value was found for both experiments within 5%, giving valuable information about heterostructure layer and interface stability.

2.6.7. Low-temperature sample stages

2.6.7.1. *Cryogenic cooling stages/cryostat*

For cryogenic experiments, liquid nitrogen (boiling point 77.4 K at 1 atm, where 1 atm = 101 325 Pa) or liquid helium (boiling point 4.3 K at 1 atm) is required (Weast, 1980). The most common types of chambers for medium-to-low temperatures are chambers with continuous-flow cooling. Here, a continuous flow of liquid nitrogen is provided from a Dewar storage vessel and the cooling process is controlled by a liquid-nitrogen controller. For lower temperatures helium is used. Helium is an expensive gas, and therefore a more economic method is to use a closed-