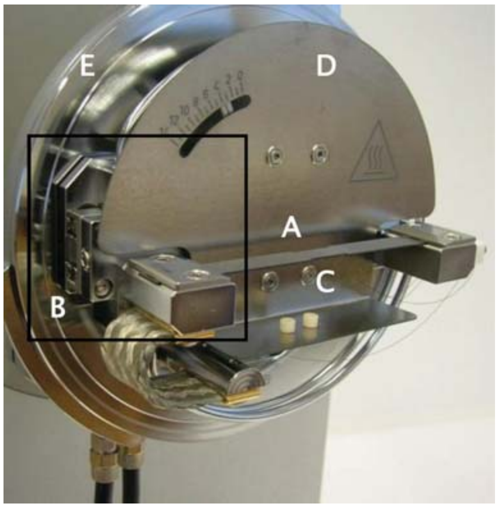

Figure 2.6.3
The interior of a typical strip-heater sample stage (Anton Paar HTK 2000N) with heating strip (A), mechanics to compensate strip expansion (B), thermocouple wires (C), heat shield (D) and water-cooled base plate (E).

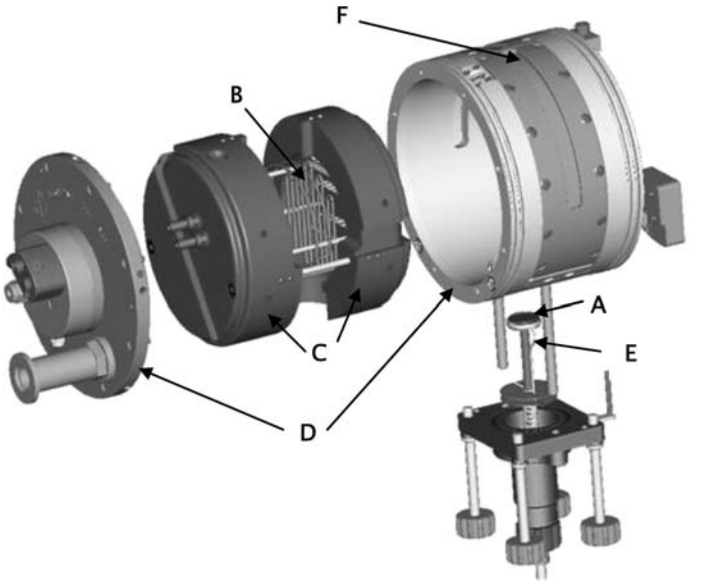

Figure 2.6.4
A typical furnace heater (Anton Paar HTK 1200N) consisting of sample holder (A), heater (B), thermal insulation (C), water-cooled housing (D), thermocouple (E) and X-ray window (F).

2.6.6.1. *Direct heating: strip heaters*

The highest temperatures can be reached with so-called strip heaters (Fig. 2.6.3). Commercial stages that can heat to up to 2573 K are available. Sample heating is performed with a high-current resistance heater. The specimen is placed directly on the strip or in a crucible on the strip. Typical strip materials are platinum (which can be heated in air to up to 1873 K) and tungsten (maximum temperature 2673 K), which requires a vacuum or an inert-gas atmosphere. Less common strip materials which have to be operated in vacuum or in an inert-gas atmosphere are graphite (maximum 1773 K), molybdenum (maximum 2173 K) and tantalum (maximum 2873 K). In addition to very high temperatures, these heaters offer very fast heating and cooling. The HTK 2000N from Anton Paar, for example, can reach up to 2573 K in 3 min. The temperature is measured with a thermocouple, which is usually welded to the heating strip. The main disadvantages of strip heaters are possible chemical reactions between the heating strip and sample, difficulties in measuring the sample temperature accurately and difficult sample preparation. Often, it is not the starting material that reacts but the products that form during heating. Another strip material can be chosen if reactions are known to occur. Inaccurate temperature measurements can be minimized by placing a second temperature sensor on top of the sample.

2.6.6.2. *Environmental heating: the oven*

The second common type of sample stage for high temperatures are oven heaters, also called environmental heaters (Fig. 2.6.4). An electrically heated wire is formed into a cage, which is surrounded with thermal insulation. The heater and insulation form a furnace which almost completely surrounds the sample, creating a very uniform temperature distribution on the inside and minimizing the heat transfer to the housing of the sample stage. Heat is transferred *via* radiation and convection to the sample. The sample is placed on a sample holder in the centre of the furnace, without direct contact with the heater. The sample temperature is measured with a thermocouple located close to the sample, providing accurate measurement of the sample temperature. In addition, it is possible to oscillate the sample to improve the data quality (by reducing granularity), and the user can measure (polycrystalline) solid samples as well as powder samples. In most cases, a long sample holder must be used to place the sample in the centre of the furnace. The thermal expansion of the sample holder while heating must be compensated for by z adjustment to avoid sample displacement (see Section 2.6.4.5). Windows for letting the X-rays enter and leave the chamber should preferably have no influence on the diffraction process. Different materials are available depending on the requirements of the non-ambient measurements. Kapton is the most commonly used window material, followed by graphite, aluminum and beryllium. Environmental heating is also one of two methods used to heat capillaries for X-ray diffraction with transmission geometry. The other option is heating the capillary with a gas flow.

Example: Cement. Cement consists of different calcium silicates (see Chapter 7.12). The exact phases that are present and their abundances determine important physical properties of a cement such as its strength. One of the phases in cement, belite (Ca_2SiO_4), exhibits rapid phase transitions. Fast transitions require good time resolution to detect short-lived intermediate phases and to follow the kinetics of fast phase transformations. An Anton Paar HTK 1200N oven was used for this experiment together with a PIXcel3D detector in static mode using a radius-reduction interface to allow snapshots to be taken over a 2θ range of 6° within a time frame of less than 1 min. Bragg–Brentano geometry was used to achieve a good resolution in 2θ and, to compensate for thermal expansion of the sample holder, an automatic height compensation was applied. On heating $CaCO_3$ with amorphous SiO_2 at 10 K min^{-1}, a solid-state reaction was seen at 853 K; α'_L-Ca_2SiO_4 is formed together with CO_2 (Fig. 2.6.5*a*). Dicalcium silicate exists in five polymorphic forms (Odler, 2000). During cooling, one of the other polymorphs of dicalcium silicate, β-Ca_2SiO_4, is formed, which has a different crystal structure and optical properties (Fig. 2.6.5*b*).

2.6.6.3. *Environmental heating: lamp furnace*

Another approach to designing an environmental chamber is the quadrupole lamp furnace developed by W. M. Kriven (Sarin