Quantitative work with this apparatus to date has been concerned with measuring intracrystal lattice-parameter differences, in which case crystals are studied one at a time, and only one goniometer head is carried in the frame. In a major study, which recorded lattice-parameter differences between growth sectors in a large synthetic diamond (Lang, Moore, Makepeace, Wierzchowski & Welbourn, 1991), a fortunate configuration of growth sectors plus little overall warping of the specimen combined to ensure that only two $\chi$ settings, $180^\circ$ apart, were needed, after a correct initial $\chi$ setting on the goniometer head. The present apparatus then fulfilled all needs. At Daresbury, laser-beam reflection from a glass plate held against the steel rods was used again, this time to establish the crystal orientation on the camera, and was a great help towards quick attainment of the double-crystal reflecting orientation in both $\chi$ settings.

A heating device for four-circle diffractometers. By H. BOHM, Institut für Geowissenschaften der Universität, D-55099 Mainz, Germany

Abstract

A heating device for diffractometers is proposed that has the advantage of not impeding the movements of the circles nor the crystal. A stream of hot gas that coincides with the primary beam is used to heat the crystal.

Many existing heating devices for diffractometers cause problems by impeding the movement of the circles or the detector or by absorbing the diffracted beam. A new simple device that avoids this problem is described. The essential feature of the device is that the crystal is heated by a stream of hot gas in the same direction as the primary beam. This is achieved by sliding the device over the collimator so that the primary beam coincides with the axis of the heating coil of the device. Therefore, the crystal can be as close as 2–3 mm to the outlet of the gas stream within an area of constant temperature. Since the collimator is protected against collision with other parts of the instrument, the device does not impede the measurement.

The function of the heating stage is depicted in detail in Fig. 1. A metal cone (e) is slid over the end of the collimator and fixed by a small screw. The device is slipped over the cone and it is fixed by three adjustment screws (d in Fig. 1). These screws allow adjustment of the axis of the heating coil parallel to the beam. The proper adjustment of the device on the collimator can be verified by taking a photograph of a centered steel tip in the primary beam. Nitrogen gas from a steel tank is reduced to 5 kPa, and the gas flow is adjusted to approximately 0.51 min$^{-1}$ by a needle valve. The gas hose is connected to the vessel of the device (a in Fig. 1). Part of the gas is heated in an inner SiO$_2$-glass tube whereas the rest flows as a cold gas stream between the inner and outer tubes (c in Fig. 1). A beryllium foil (b) allows the beam to pass along the axis of the device. The heating coil consists of Pt–30%Rh wire. Nitrogen is used instead of air to prevent the oxidation and gradual transport of platinum to the crystal; the deposition of platinum on the surface of the crystal would cause unwanted absorption effects. The coil is heated by a stabilized DC power supply. This guarantees a temperature stability of $\pm 1$ K (at 1070 K). However, in this case, the diffractometer should be operated within an enclosure to prevent draught from outside. The device has been used in long-term experiments (approximately 3 d) at 1120 K. However, an upper limit for the temperature has not been determined.

The temperature is set to a fixed value before collecting the data. It is measured by an Ni/NiCr thermocouple with a wire 0.01 mm thick. The head of the thermocouple can be allowed to touch the crystal when the temperature is set before the measurement. During the experiment, the thermocouple is moved away but it may stay close to the crystal to monitor the temperature. Instead of using a DC power supply, a controller may be connected to the device to control the required temperature.

References


Fig. 1. Schematic drawing of the heating device: (a) gas inlet; (b) beryllium foil; (c) SiO$_2$-glass tube with heating coil; (d) adjustment screws; (e) adjustment cone; (f) collimator.

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