Laboratory Notes

J. Appl. Cryst. (1981). **14**, 75

A simple method for obtaining a bent LiF X-ray monochromator

In order to use efficiently a powerful X-ray generator with a rotating anode without loss of intensity in studies of weak diffuse scattering, such as those due to short-range order in alloys, point defects in irradiated metals and so on, utilization of a bent-crystal monochromator is required, by which a divergent X-ray beam from the source is focused at the specimen position or at the window of the detector. The best monochromator would be a doubly bent pyrolytic graphite (PG) crystal. However, it is difficult in our laboratory to bend a PG crystal so as to fit the radius of curvature to the distance between the monochromator and counter as required. In this circumstance, it would be useful to employ a LiF single crystal instead because of its fairly high reflectivity and narrow mosaic breadth of reflection compared with that of the PG monochromator. Furthermore, it is easy to bend a LiF crystal by a process at high temperatures.

A cleaved LiF single crystal of about 20 $\times 20 \times 1$ mm was sandwiched between concave and convex moulds made of brass and then annealed at 770 K in vacuum. It is, however, not easy to bend a crystal because of the fractures introduced in the course of bending. We found that the number of such fractures was less if the cleaved LiF single crystal in the mould was heated in a silicon-oil bath up to 520–550 K, and pressed gradually up to 3×10^{-4} kg m^{-2} and annealed for 5 h. By cooling the crystal slowly to room temperature, a fairly good bent LiF monochromator was obtained. In order to see the convergence of an X-ray beam, a series of X-ray photographs was taken at various distances from the monochromator with Cu $K\alpha$ radiation. Fig. 1 shows the convergence of a monochromator with a radius of curvature $R = 350$ mm obtained in the way mentioned above. It is seen that the doublet of Cu $K\alpha$ radiation, $K\alpha_1$ and $K\alpha_2$, is separated clearly in these photographs. It was found that the intensity reflected from this monochromator is 6·5 times that of a flat LiF crystal at a peak position.

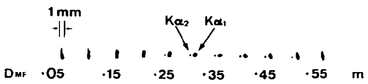

Fig. 1. A series of X-ray photographs taken at various distances from the monochromator D_{MF} with Cu $K\alpha$ radiation. The X-ray source is a fine-focus Cu tube operated at 50 kV, 10 mA. Exposure time is 15 s.

The authors would like to thank Professors J. Harada and N. Sakabe for helpful discussions.

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(*Received* 21 *July* 1980;
accepted 3 *September* 1980)

J. Appl. Cryst. (1981). **14**, 75

A device which improves the cooling of protein crystals during X-ray data collection

In measuring X-ray diffraction data, the cooling of protein crystals often causes great problems. The capillary in which the protein crystal is placed must be kept at constant temperature. If the temperature is different at different parts of the capillary, liquid will condense onto the cooler parts, thus changing the concentration of the buffer inside the crystal and disrupting the order in the crystal. This is often the case if the cold air stream cannot be blown perfectly parallel to the capillary. Even when the cold air can be blown parallel to the capillary, temperature fluctuations may occur. If there is a draught in the room, for example if the door is opened or a person moves close to the X-ray camera, the cold air stream will be deflected for a few seconds. Such effects were observed when collecting data on a nucleotide derivative of the protein disk of tobacco mosaic virus. The temperature just outside the capillary was measured using a thermocouple. When the door to the room was opened the temperature could rise from 277 K to 283 K or even more for a few seconds. The effects of this could be seen in the microscope of the X-ray camera – water droplets were formed on the cooler parts of the capillary wall, as water evaporated away from the two mother liquor plugs onto the crystal. A perfect crystal, diffracting to better than 2·8 Å could lose its order in a matter of minutes, as was seen in still pictures.

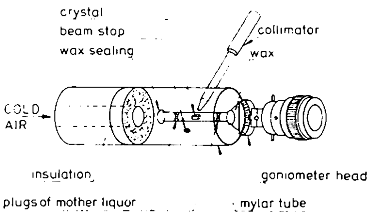

Fig. 1. Schematic drawing of a device for keeping the temperature constant around a capillary.

All these problems were solved by extending the cold air tube by a tube made out of thin mylar plastic, to enclose the whole length of the capillary, as shown in Fig. 1. It was found essential that the mylar extended past the crystal as well as both mother liquor plugs, since, if this was not the case, water from the plug outside the mylar tube would condense onto the crystal, and cause the diffraction pattern to fade out in a matter of hours. With this device every crystal diffracted perfectly after several days, whereas without it most crystals, although perfect when the first stills were taken, were disordered within less than 24 h, although room-temperature data collection is possible. Nevertheless, in cases like this, the value of the device is that it provides a constant temperature during data collection.

The mylar used was so thin that less than about 1% of the diffracted X-rays were absorbed. A hole was made for the collimator so that the direct beam did not hit the plastic. The beam stop was placed immediately outside the mylar tube in order to prevent any background scattering from the direct beam hitting the mylar.

A problem which might arise if the relative humidity of the air is very high is condensation of water onto the outside of the mylar tube. A limitation of the device is that it is only suitable for such data collection strategies where the capillary is more or less stationary relative to the X-ray source, as is the case with rotation cameras and perhaps precession cameras but not with diffractometers.

This study was carried out while S. Hovmöller held an EMBO fellowship.

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(*Received* 9 *June* 1980;
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0021-8898/81/010075-01$01.00