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A simple attachment to Debye–Scherrer X-ray powder diffraction cameras to obtain powder patterns from single crystals

MANY methods of simulating X-ray powder patterns from single crystals have been published (e.g. Gordon, 1947; Switzer and Holmes, 1947; Matthews and McIntosh, 1949; Hiemstra, 1956; Gandolfi, 1964, 1967; Graeber and Jelinek, 1966; Corbett, 1972; Jenkins and Haas, 1973; Stern, 1977; Moss *et al.*, 1979; Koto *et al.*, 1984). They all provide for a random, or near random rotation of the investigated crystal. Notwithstanding the possibility of a non-random rotation, with as a consequence diffractograms that are not precise duplicates of powder patterns, the results are quite adequate for measurement and identification. In most designs the randomization of the crystal is effected by simultaneous rotation of the crystal around two inclined axes. Generally the primary axis is that of the camera itself and the secondary axis is that of the instrument. All these designs necessitate the purchase or fabrication of complicated and expensive special-purpose equipment. Moreover, these designs often require a careful and time-consuming centring procedure under a telescope which may be cumbersome in practice because it involves a simultaneous alignment of the crystal with respect to the two rotation axes.

Description. In our instrument (Figs. 1, 2, and 3) the positioning of the sample is straightforward because the rotation around the primary axis can be adjusted by means of the original centring device on the powder diffraction camera. The unique features of this instrument are that (1) it is uncomplicated; (2) it is inexpensive to manufacture; (3) it can be used with only a minor adaptation in most commercially available Debye–Scherrer powder diffraction cameras; and (4) it allows for a very simple positioning of the sample in the X-ray beam.

The apparatus is machined out of aluminium in the shape of a saucer. In the centre of the saucer a steel cylinder has been inserted with a central channel drilled lengthwise through it. The diameter of the cylinder is chosen in such a way that it fits closely in the hollow axle of the powder diffraction camera. This rod forms in fact the primary rotation axis of the instrument. In the wall of the saucer a steel secondary rotation axle provided with ball bearings has been constructed. This axle makes an angle of 45° with the primary axis of the instrument and an angle of 90° with the side-wall of the saucer. One end of the secondary axle protruding outside the saucer is fitted with a supple rubber tip that can be brought into contact with the flat wall of the cylindrical powder diffraction camera. The other

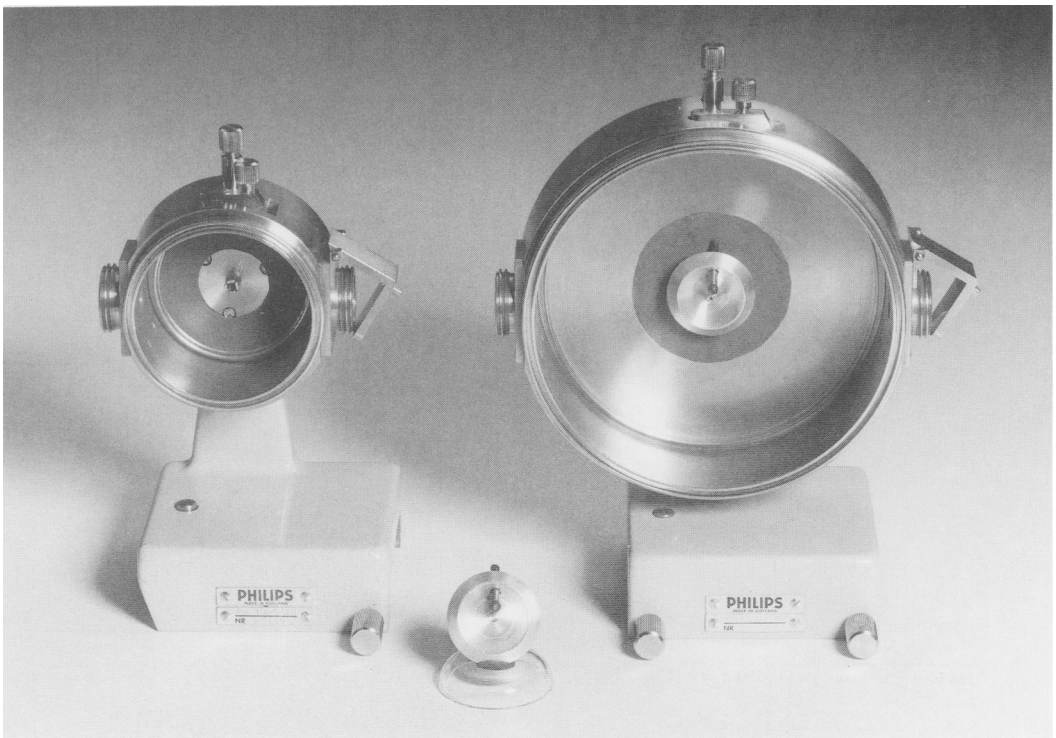

FIG. 1. Left: 57.3 mm diameter Debye–Scherrer X-ray powder diffraction camera with paper friction ring showing the counter sunk screws replacing the original cylinder head screws in the centring device of the camera. Centre: camera attachment to effect simulated powder diffraction patterns from single crystals. Right: 114.6 mm diameter camera with friction ring and camera attachment.

end of the axle is threaded in such a way that a hollow brass cylinder with internal thread can be screwed on it. One end of this cylinder can be provided with a piece of modelling clay in which a thin (50–100 μm) glass-fibre can be stuck. The tip of the fibre has to be centred permanently by trial and error in such a way that upon rotation of the secondary axle the tip coincides exactly with the primary axis of rotation. To facilitate the centring, the rod forming the primary rotation axis of the instrument has been provided with a small 'viewing-channel', so that the centring can be performed with the help of a microscope. With a thin 'one-haired' brush or a needle, the sample, a single crystal or crystal fragment, or even a cluster of fragments, may be attached to the tip of the glass fibre wetted beforehand with glycerine. The sample is held on the fibre by surface-tension of the glycerine and can be recovered by dipping it into water, or else, by removing it with the brush. The rotation of the camera axle that is directly connected with the instrument also drives the secondary axle of the instrument by the friction between the rubber end of the secondary axle and the flat wall of the camera. To bring the tip of the glass-fibre in the middle of the X-ray beam, the same centring procedure can be followed that is customary in obtaining ordinary

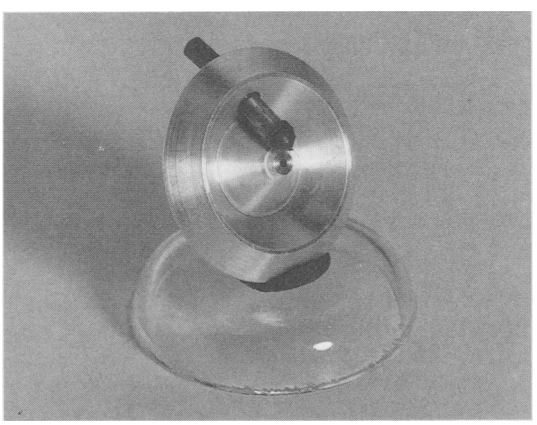

FIG. 2. Detail of camera attachment.

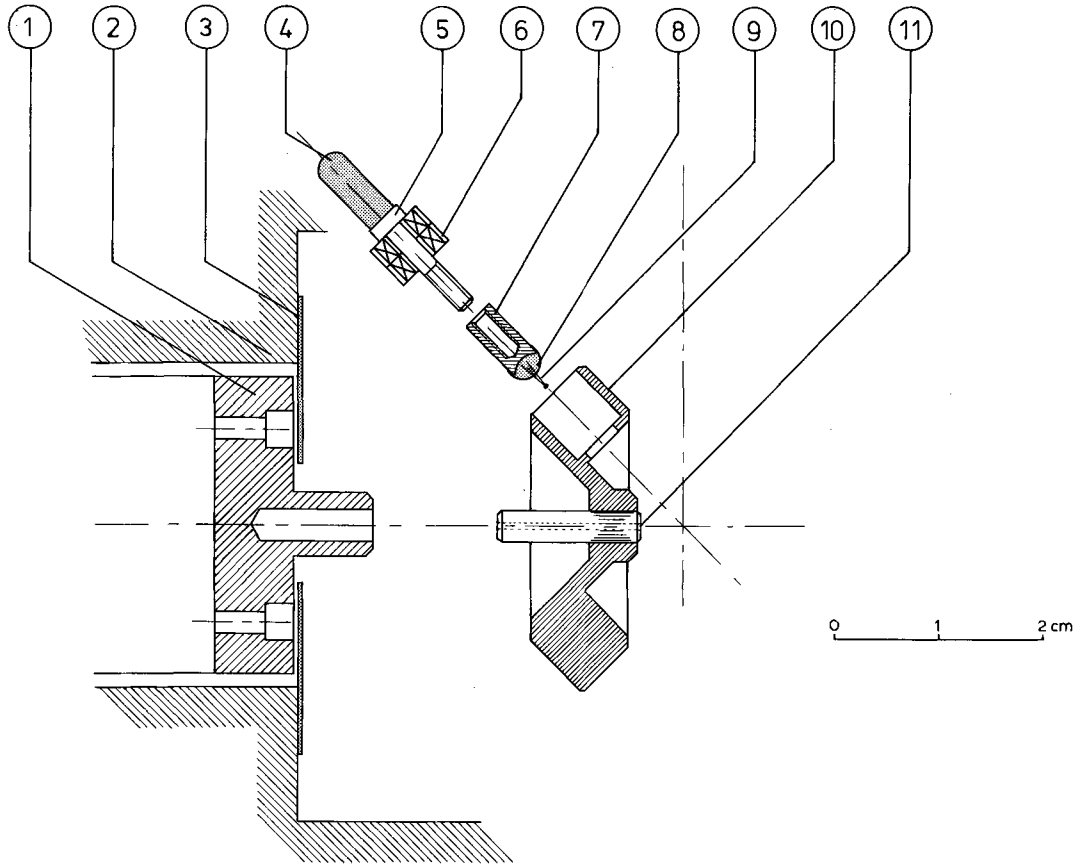

FIG. 3. Dimension sketch of the camera attachment. 1. Counter sunk screws replacing the original cylinder head screws in the camera centring device. 2. Camera house. 3. Ring on flat wall of the Debye-Scherrer camera; paper. 4. Elastic tip glued on secondary axle; rubber. 5. Secondary axle, threaded at one end; steel. 6. Ball bearings. 7. Internally threaded cylinder; brass. 8. Modelling clay. 9. Fibre; glass, with crystal fragment. 10. Saucer-shaped body; aluminium. 11. Primary axle with central 'viewing channel'; steel.

powder diffractograms with Debye-Scherrer equipment. The only necessary adaptation of the camera is to replace the original screws with cylinder heads inside the camera by counter-sunk screws, to ensure unhampered rolling of the rubber tip over the flat wall of the camera. In case of insufficient friction this can be remedied by a paper ring of appropriate thickness glued inside the camera. With the centring properly carried out, the diffracted rays will all emanate from approximately the same point and the resulting arcs should be sharp and accurately positioned.

The crystals or crystal fragments may be in the size range 10–100 μm. Larger ones cause excess line-broadening and accentuate undesirable absorption effects in some cases. Generally, a sample of a few micrograms is sufficient to produce enough observable diffraction lines on the X-ray film to enable identification (applied exposure times: up to about 20 hours using the 114.6 mm diameter camera and 10 hours for the 57.3 mm diameter camera). From thin sections, crystals may be drilled out with a micro-drill (e.g. Verschure, 1978) or prised out with a needle using a micromanipulator.

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Merlinoite in manganese nodules from the Indian Ocean

A GROUP of silicate minerals including quartz, feldspar, montomorillonite, illite, phillipsite, mordenite, etc. has been found associated with manganese nodules from the Pacific Ocean, either as a nucleus or as disseminations within sub-layers (Cronan and Tooms, 1968; Burns and Burns, 1977). These are either detrital or derivatives of pumice, basalt, glass, tuff, etc. Detailed studies of individual minerals are reported by Piper and Williamson (1981) and Bischoff *et al.* (1981). While carrying out mineralogical characterisation of core materials of manganese nodules collected from different localities in the Central Indian basin, Indian Ocean, the authors recorded the occurrence of the mineral merlinoite, details of which are presented here. To our knowledge, this mineral has not been reported previously in manganese nodules.

The nodule samples were collected during September 1982 between latitude 6° and 8° S and longitude 81° to 87° E by the first author (in cruise nos. 111 and 112) in the research vessel 'Gaveshani' by the free-fall grab technique.

Merlinoite was identified by X-ray powder diffraction (XRD) technique using a Philips diffractometer, with Mo-$K\alpha$ radiation and zirconium filter, operating at 40 kV and 20 mA. The XRD pattern is almost identical to that of phillipsite, with the exception of the 10.02 and 4.48 Å lines (Table 1) which are characteristic of merlinoite (Passaglia *et al.*, 1977). Subordinate amounts of plagioclase feldspar (P), stilbite or chabazite (S) and quartz (Q) have been recorded in several samples. In thin section, merlinoite invariably appears as aggregates showing interpenetrant complex twins. Under a JEOL scanning electron microscope it appears as thin overlapping euhedral lamellae of fibrous crystals (Fig. 1). An EDAX spectrum shows that the grains contain Si, Al, K, Ca in decreasing order