Gandolfi attachment to a Weissenberg camera

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Abstract

Attachment of Gandolfi mode is designed to a Weissenberg camera. The attachment is mountable to a universally used single crystal goniometer head and demountable from it. One can take either oscillation and powder pattern photographs from a single crystal mounted on the attachment. To facilitate centering of a specimen, a simple centering stage is designed. Powder patterns have been successfully obtained by using the attachment.

Introduction

For identification of crystalline materials, the X-ray powder diffraction method is one of the most useful and convenient methods and therefore universally used. For this purpose, powder diffractometer and Debye-Scherrer powder cameras are popular. On the other hand, several methods have been designed to obtain the powder profile directly from a single crystal or assemblage of a few single crystals without grinding the specimens. Among them, a Gandolfi camera is practical and purchasable (Gandolfi, 1967). Simple Gandolfi attachments were designed for a Debye-Scherrer camera (Moss *et al.*, 1979; Rendle, 1983) and a device for the easy centering of a specimen was described (Bruyn *et al.*, 1984).

This paper describes a Gandolfi attachment designed for a Weissenberg camera.

Gandolfi mode

Basic principle of Gandolfi mode is described as follows. To obtain a powder profile from a single crystal, a reciprocal lattice point r_{hkl} must come to every point of the circle c_{hkl} by subsequent driving of the point r_{hkl} . Here c_{hkl} is an intersection of the diffraction cone with Ewald's sphere (Fig. 1). Principle of a Gandolfi camera is briefly described by two combined rotations according to two axes, one of which is perpendicular to the X-ray beam (A' -axis) and the other (A'' -axis) inclined by an angle α on A' -axis, and driven by this (Gandolfi, 1967). By these combined rotations about the A' - and A'' -axes, a reciprocal point r_{hkl} partly intersects Ewald's sphere on the circle c_{hkl} .

FIG. 1. Principle of obtaining powder pattern from a single crystal by Gandolfi mode. A reciprocal lattice point r_{hkl} intersects Ewald's sphere on the circle c_{hkl} by two combined rotations about the A- and A'-axes. A spherical surface s_{hkl} has a center at the origin of the reciprocal space and radius r_{hkl} . θ is Bragg angle (Gandolfi, 1967).

The zones to which two reciprocal lattice points, r_{hkl} and its Friedel pair $\bar{r}_{\bar{h}\bar{k}\bar{l}}$, come across the sphere s_{hkl} , depend on the angle β . Whole circle c_{hkl} is covered only in case of $\beta=45^\circ$ or 90° if $\alpha=45^\circ$ (Gandolfi, 1967). However in case of higher symmetry than triclinic symmetry, the crystallographically equivalent reflections are more than two according to the Laue symmetry and therefore they have more chance to come to the circle c_{hkl} even in case of $\beta \neq 45^\circ$ and 90° . On the other hand, the whole circle c_{hkl} can be also covered in compensation by using a few fragments of single crystals mounted on the attachment.

Attachment to a Weissenberg camera

The attachment shown in this paper is of basically the same structure as that by Gandolfi (1967) and its design and advantages are as follows. (1) Brass is used for all the components. (2) An attachment is designed so as to be mounted to a universally used single crystal goniometer head (Fig. 2). The angle α is 45° . The Gandolfi mode is obtained by applying the rotation mechanism of the Weissenberg camera and by holding the A'-axis component by a rod to the X-ray beam stopper (Figs. 2 and 3). (3) A distance from the bottom of the specimen holder to the intersection of the A'-

FIG. 2. Gandolfi attachment to a Weissenberg camera, mounted to a single crystal goniometer head. Component part A is held by a rod to the X-ray beam stopper.

FIG. 3. A Gandolfi attachment mounted to a Weissenberg camera.

and A'' -axes is 10 mm (Fig. 4a). The distance is adjusted by changing the length of a piece of wire. (4) By structure of the goniometer head, strict centering is only necessary about the A'' -axis concerning with the attachment. Centering about the A' -axis can be performed by structure of the goniometer head. Therefore centering of the specimen can be made easily. (5) To facilitate centering of the specimen about the A'' -axis, a simple centering stage is designed (Fig. 4). (6) Either oscillation and powder

(a)

(b)

FIG. 4. (a) Schematic picture of Gandolfi attachment (GA) mounted to a centering stage about the A'' -axis (CS). The A' - and A'' -axes make an angle 45° . A specimen mounted on the end of a glass fiber which is fixed to Gandolfi attachment by a piece of wire, is viewed along the A'' -axis through microscope. By trial and error, adjustment of centering is made. (b) Gandolfi attachment and a centering stage about the A'' -axis.

pattern photographs can be taken from the specimen mounted on the single crystal goniometer head.

Powder patterns of several inorganic compounds have been successfully obtained by using the attachment. The oscillation and powder pattern photographs of a single crystal NaCl are shown in figure 5, for example.

(a)

(b)

FIG. 5. (a) 25° oscillation photograph of a single crystal NaCl. (b) Powder pattern of the same specimen by Gandolfi mode, taken with Ni-filtered Cu $K\alpha$ radiation.

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