RECIPROCAL LATTICE PHOTOGRAPHY

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1. Introduction

Of the single-crystal X-ray photographic methods, the method of reciprocal lattice photography yields undistorted records of the reciprocal lattice. The photographs obtained are not only symmetry-true but also enable one to index the spots by visual inspection.

The well-known De-Jong Bouman camera was the first of its kind. Improved types of cameras were later designed and constructed by M. J. Buerger. A publication bearing the title "The Photography of the Reciprocal Lattice" by M. J. Buerger published as a A.S.X.R.E.D. Monograph, No. 1, 1944 gives in detail the principles involved in reciprocal lattice photography and describes a reciprocal camera, its uses, and advantages over other methods. The principle involved can be stated very briefly as follows: The Ewald construction for the reflection of X-rays by a set of planes indicates that an X-ray reflection occurs whenever a reciprocal lattice point intersects the sphere of reflection. The direction of the reflected beam is got by joining the centre of the reflecting circle and the reciprocal lattice point. If one can always keep the film plane parallel to the reciprocal lattice plane that is being investigated, the record obtained under such conditions represents a scaled replica of the reciprocal lattice plane. This can be shown to be the case with a simple geometric construction shown in Fig. 1. In order to cover as many points of the reciprocal plane as is convenient, a very ingenious kind of rotation has been introduced, in which the normals to the reciprocal lattice plane and the film plane follow a motion, similar to that of a precessing top about an axis which is the direction of the incident X-ray beam. Hence the instrument was given the name "X-ray precession camera". An arrangement is provided in the camera for this type of precessional motion wherein it is possible to set different angles of precession. In order that the film and the reciprocal lattice should remain parallel to each other during the motion, a link system is incorporated in the design of the camera. Further, a layer-line screen whose distance can be altered with

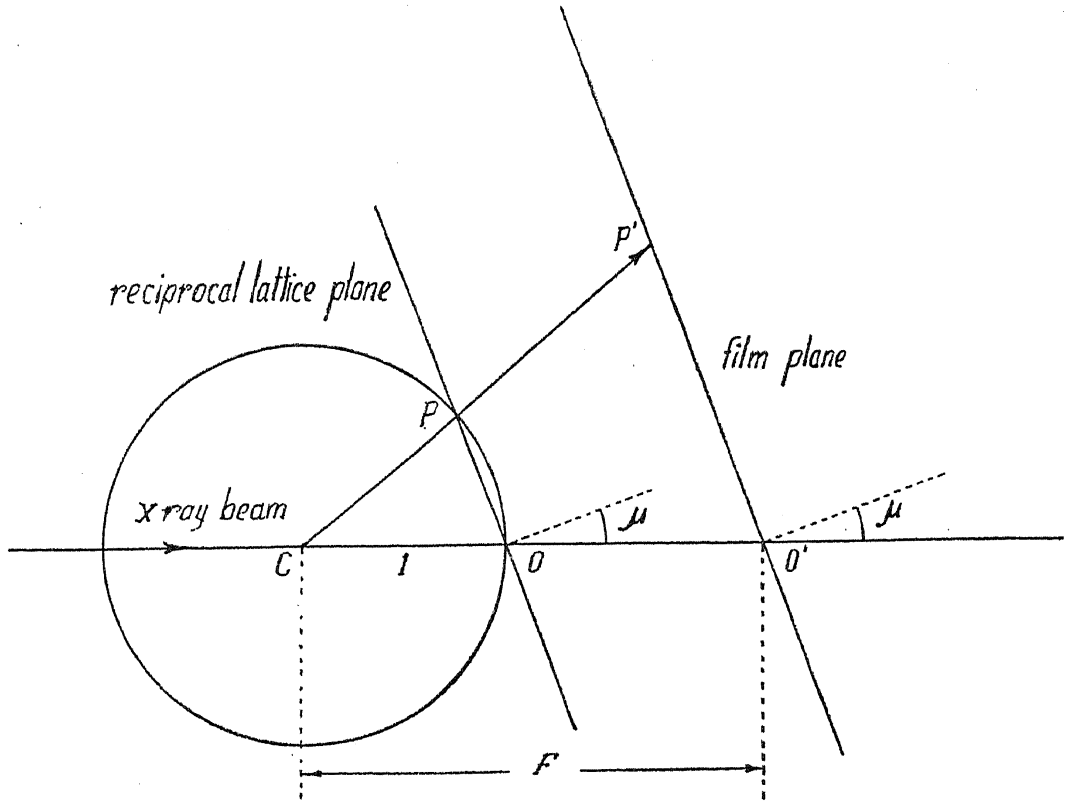


FIG. 1

$$\frac{O'P'}{OP} = \frac{CO'}{CO} = \frac{F}{1} \therefore O'P' = F.OP$$

respect to the crystal is provided, with a view to isolate reflections of a particular layer. A movement for translating the film towards the crystal is also provided so that different reciprocal planes can be recorded without distortion. The geometric conditions necessary for obtaining these results are described and discussed in detail in the book referred to above.

The construction of such a camera was undertaken at this Institute and the present communication describes the same. It is not claimed that the instrument incorporates any new features. It appeared worthwhile however to describe our experience in making and using such a camera for the benefit of those interested in such work.

2. THE CONSTRUCTIONAL FEATURES

A photograph of the reciprocal camera is reproduced in Fig. 2. The different parts of the camera described in this section can be clearly seen on an inspection of the same. The arc-shaped arm which is the principal part of the driving mechanism of the camera can be seen. The arc was cut out

of $\frac{1}{4}''$ brass plate and represents a part of a circle of radius 16·5 cm., the segment subtending an angle of about 40° at the centre. It is mounted on a bush bearing which is supported on a pillar fixed to the base. The arm can be rotated by a motor about the X-ray beam as its axis. A sliding block on the arm carries the driving axis which goes and fits into a cylindrical brass bush attached to the back of the film holder carrier. The sliding piece at its lowest setting represents a precession angle of 0°, while at the maximum it represents a precession angle of 30°. A square frame which carries the film holder is mounted on a two-axis universal joint. The unmoved point of the universal joint is precisely set in the centre of the circle mentioned above. A sliding arrangement is provided for translating the film holder towards the crystal. The crystal holder assembly is mounted on another two-axis universal joint. The distance between the film and the crystal universal joint centres represents the specimen to film distance, which has been set at 6 cm. in the instrument. The crystal holder assembly supports an L-shaped arm, and this carries a movable layer-line screen. The layer-line screen is made out of brass sheet, 10 cm. square, with an annular opening of 15 mm. in radius. A layer-line screen with an annular opening of 30 mm. in radius will also be found useful. Provision has therefore been made to be able to use the desired layer-line screen. A parellelogram link motion with ball and socket joints at the upper two corners serves as the link between the reciprocal lattice plane and the film plane. The precessional motion executed by the latter is transmitted by this link to the former, maintaining at the same time both the planes strictly parallel. The link is attached at its two lower ends to the projecting horizontal, film and crystal axes. For facilitating initial setting of the crystal, the link at the crystal axis end can be decoupled, leaving the crystal axis free. Further, the platforms which support the film holder carrier and the crystal holder are coupled by two brass links. This serves as a parallelogram link in the horizontal plane. The former link has to be of the ball and socket type, since it has to take care of parallelogram motions in two mutually perpendicular planes. The latter link is absolutely necessary, but is of a very simple type as it has to take care of a parallelogram motion in one plane only, namely, the horizontal plane. The X-ray collimator is carried by the pillar which is seen at the extreme left of the photograph. There is a lead stop which serves to screen off the incident beam after it has emerged from the crystal.

The universal joint carrying the film holder is capable of being moved along the axis of the instrument, while the universal joint carrying the crystal is capable of being moved at right angles to this axis. The X-ray collimator can similarly be moved at right angles to the axis of the instrument. After

proper alignment, these can be firmly clamped. The geometry of the instrument strictly demands that the incident X-ray beam should pass through the two universal joint centres which remain unmoved during the motion. The beam should be set parallel to the axis of the instrument.

In making these universal joints, a tapered cone assembly was used for the vertical axis. Very smooth and satisfactory running is ensured by putting a steel ball on top of the male cone, such that the hollow cone part rests on the steel ball, at the same time remaining just in contact with the male cone. This arrangement helps in reducing the grip between the two members which would otherwise result, and has been found to be very satisfactory. For the horizontal axis two ball bearings were used. A major portion of the instrument was made by machining $\frac{1}{2}''$ thick perspex sheet. It is very easy to shape this material and stick pieces of these together with trichloroethylene. Wherever necessary, pieces were stuck and strengthened by screws and nuts. Since the instrument was at the outset started as a model, the above materials were used, but to our surprise, the materials have been found to be very satisfactory.

Smooth running of the instrument depends on accurate alignment and accurate balancing of the different parts. It may not be out of place to mention here that the camera can be made at a cost much less than one has to pay for the commercially available ones.

3. Photography by the Precession Method

A preliminary step in recording reciprocal lattice photographs is to set the chosen axis of the crystal, parallel to the X-ray beam. If the axis of the crystal is roughly set by trial photographs, or from its known optical properties, accurate setting can be made by taking a series of precession photographs at small precession angles. Such photographs can be taken with short exposure times. The procedure for setting is explained in the book referred to. Once the axis is set parallel to the X-ray beam, the instrument is ready for recording the reciprocal lattice. For recording the zero layer, the film is kept such that its centre coincides with the unmoved point of the universal joint through which the X-ray beam also passes and intersects the film normally. The desired precession angle is then set. The appropriate layer-line screen is chosen and its distance from the crystal is set according to the equation

$$r_S/S = \tan \cos^{-1} (\cos \mu - d^*)$$

where r_S is the radius of the annular opening, S is the screen to crystal distance, μ is the precession angle and d^* is the reciprocal lattice spacing. For

zero layer, d^* is zero and the equation reduces to $r_s/S = \tan \mu$. For recording of higher planes, the appropriate value of d^* should be substituted in the equation.

For recording n levels without distortion, it is necessary to displace the film away from the universal joint centre towards the crystal, by a distance:

$$d' = Fd^*$$

where F is the crystal to film distance, and d^* the reciprocal lattice plane spacing.

Three X-ray precession photographs are reproduced in the plate accompanying this paper. Fig. 3 is a zero layer reciprocal lattice photograph of quartz recorded with the trigonal axis as the precession axis. Mo white radiation was employed. Fig. 4 is again a zero layer photograph of quartz taken with MoKα radiation. Fig. 5 represents the zero layer reciprocal lattice photograph recorded with the b-axis, of the monoclinic crystal cobalt nitrate hexahydrate, as the precession axis. In this case MoKα radiation was used.

The chief advantages of the method are, that it gives a scaled replica of the reciprocal lattice and indexing can be carried out by visual inspection. With crystals of large spacing, the determination of the space-group is comparatively quicker. Another advantage is that if a crystal is set with one crystallographic axis parallel to the goniometer axis, information regarding two reciprocal cell-axes can be obtained without having to remount the crystal, as in the case of other methods. After obtaining photographs about one axis, one has to rotate the crystal through the appropriate angle to bring the second reciprocal cell-axis along the X-ray beam to obtain the records. This is decidedly a great advantage over other methods and especially so when one is dealing with crystals of small dimensions and in low temperature X-ray crystallography.

The author wishes to express his grateful thanks to Professor Sir C. V. Raman, F.R.S., N.L., for his interest in the work. It is a pleasure to thank Dr. S. Ramaseshan for the many illuminating discussions the author had with him.

SUMMARY

A precession X-ray camera constructed at this Institute is described. Reciprocal lattice photographs of quartz and $CO(NO_3)_2 6H_2O$ recorded with the camera are reproduced and the general principles involved in X-ray reciprocal lattice photography are discussed.

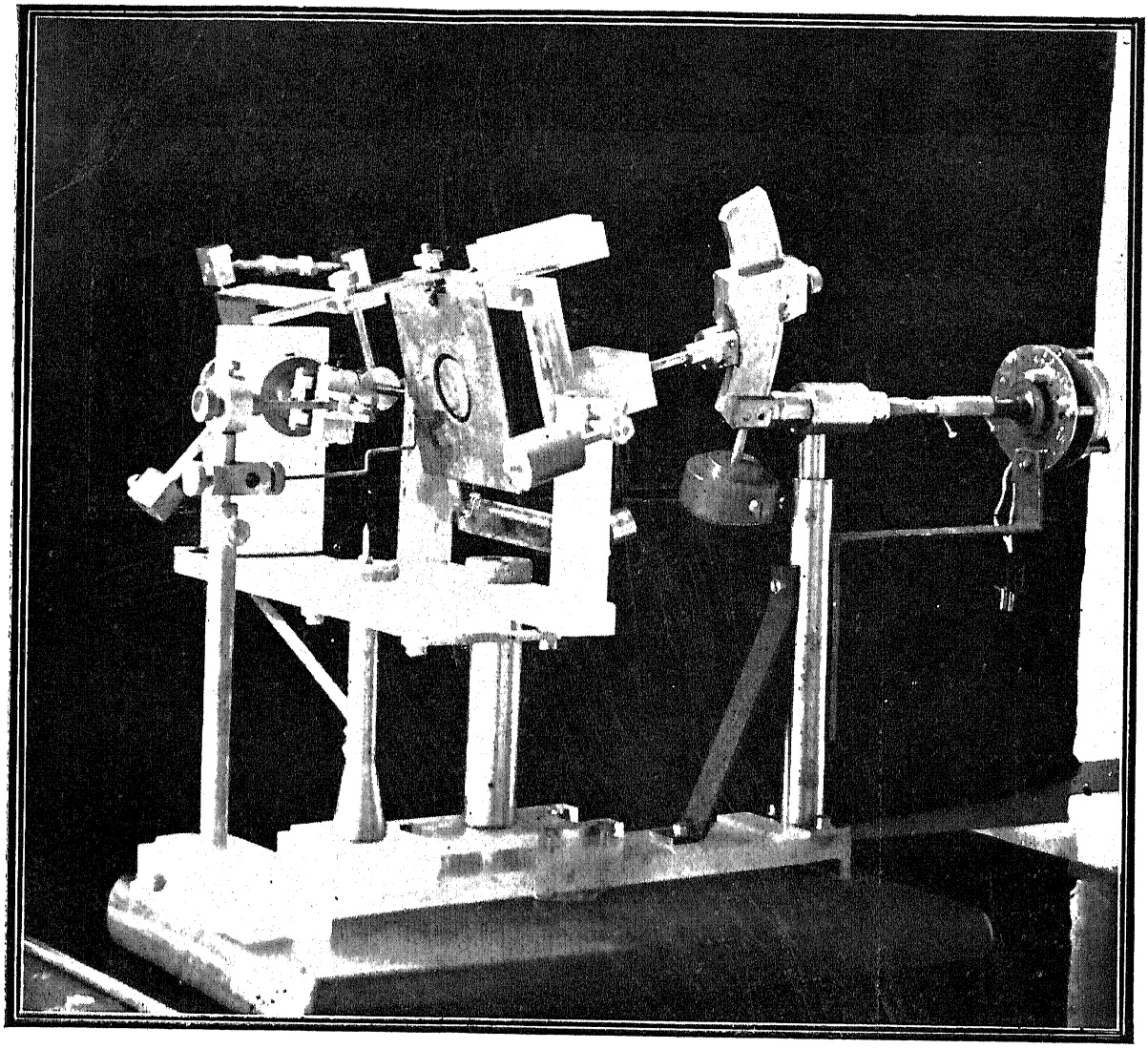


FIG. 2

FIG. 3

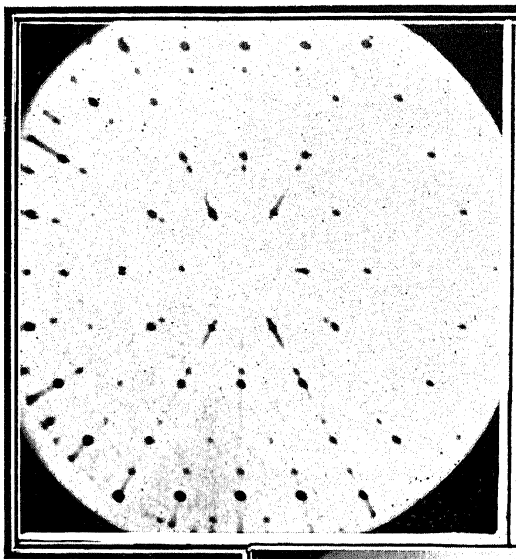
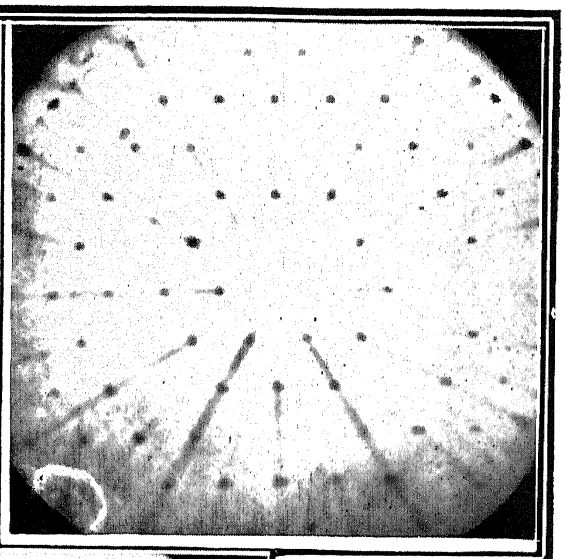


FIG. 4



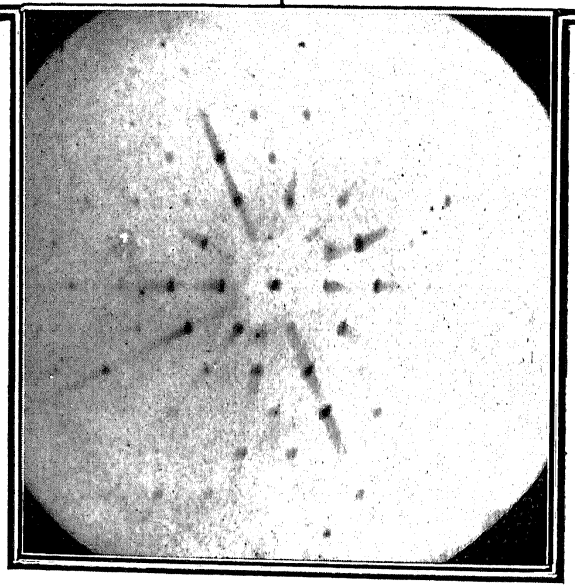


FIG. 5