

An Integrating Weissenberg Camera for Low and High Temperature Studies

A. K. SINGH AND S. RAMASESHAN

*Department of Physics, Indian Institute of Science,
Bangalore 12, India*

ABSTRACT

The paper describes the construction of an integrating Weissenberg Camera. The mechanism of the angular integration is very similar to that used in the original Wiebenga design. The linear integration is effected by a rack and pinion arrangement. Both these are made repetitive, so that one complete integration takes place in about one hour. A Fankuchen-Lipscomb cooling arrangement has been incorporated into this camera for study of crystals at low temperatures. The problem of removing the cassette for loading and developing without disturbing the cooling arrangement has been solved by making the jacketed cold and warm air leads thin enough to pass through the slot of a conventional cassette. A high temperature attachment for study of crystals up to 500°C has been made using a nickel foil both as a heater element and an X-ray filter.

1. INTRODUCTION

It is now accepted that for extremely accurate determinations of crystal structures in which photographic techniques are used, the recording of intensity data on integrating cameras of the Wiebenga type is perhaps the best. Many eminent crystallographers have advocated the precise determination of atomic and thermal parameters of crystals at low temperatures. There is no doubt that the Fankuchen-Lipscomb (Kaufman and Fankuchen, 1949; Abraham *et al.*, 1950) arrangement for cooling the crystal has many advantages over other methods, particularly when one is interested in the study of the crystal structures of substances which are liquids at room temperature.

As present writers had no access to any camera that combined the essential features of these two techniques it was decided to design and construct such a camera. As this camera has some novel features, its constructional details have been described in this paper. The problem of removing the cassette for developing the X-ray film without interfering with the cooling arrangement has been solved in a simple manner.

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Crystallography and Crystal Perfection. Ed. G N Ramachandran. p309. Academic Press, New York, 1963

2. DETAILS OF CONSTRUCTION

A. *The integrating process*

The process of integration consists of imparting two types of movements to the cassette during the exposure. At the end of every to-and-fro traverse of the carriage a cylindrical cassette rotates through a small angle ($\approx 0.1^\circ$) about its axis and at the same time it is translated parallel to its axis by about 0.05 mm. In the present camera the mechanism for angular movement is very similar to the one used by Wiebenga (Wiebenga and Smits, 1950). However a rack and pinion arrangement is used for the linear integration. Unlike Wiebenga's original design both the movements are made repetitive and one complete integration takes about an hour.

B. *The carriage design*

A carriage consisting of two parallel plates of brass AA ($12 \text{ cm} \times 8 \text{ cm} \times 0.6 \text{ cm}$) separated by another brass plate B ($12 \text{ cm} \times 3.8 \text{ cm} \times 0.6 \text{ cm}$) is capable of moving freely on rollers R, R_1 supported on parallel cylindrical rails (R,R), one being placed vertically above the other (Fig. 1).

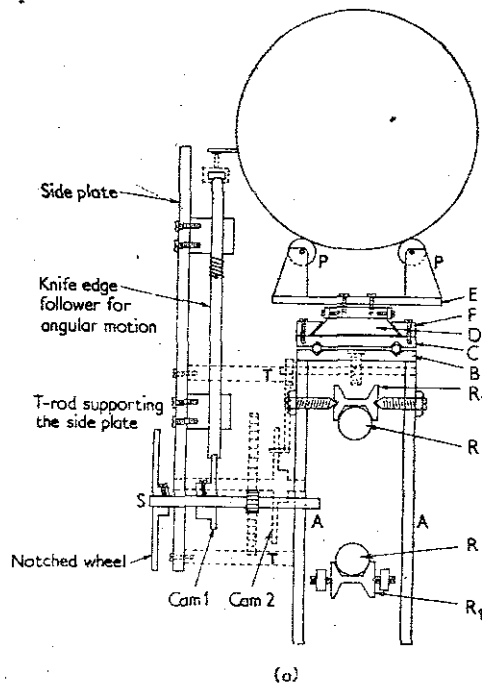


FIG. 1 (a) The end-on view of the integrating Weissenberg camera.

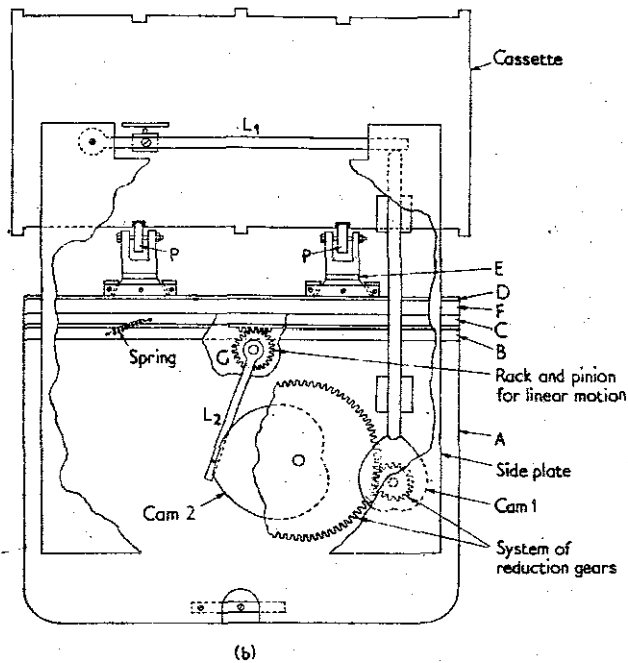


FIG. 1 (b) Diagram showing the arrangement for rotational and translational integration.

On the upper side of the plate B there are two V-grooves parallel to the length and 1.5 cm apart. A similar plate C having identical V-grooves on the lower side is kept on plate B. Four balls kept in the grooves minimize the friction so that plate C becomes a platform capable of sliding parallel to the length of the carriage. On this platform are mounted two pairs of ball-bearings for supporting the cassette (only one pair is shown in sectional diagram). The assembly of ball-bearings is capable of a movement parallel to the length of the carriage (by D sliding over C). This movement is necessary when taking equi-inclination Weissenberg photographs and it also makes it possible to remove the cassette without disturbing the cooling arrangement which is described later. Each set of ball-bearings can move, independent of each other, in the transverse direction—a feature essential for fine adjustment while properly aligning the camera.

C. The mechanism for angular rotation

As in the Wiebenga design, a notched wheel having fourteen teeth is mounted on the side of the carriage by means of a horizontal shaft, S.

At the end of every oscillation of the carriage the teeth of the notched wheel are engaged in a pin which causes a rotation of the notched wheel. A spring and roller arrangement makes the rotation of the notched wheel uni-directional and in equal steps. Thus at the end of every oscillation of the carriage, the notched wheel rotates through $(360/14)$ degrees causing the rotation of a cam mounted on the common shaft. A vertical knife-edge follower moves through a distance (0.4 mm) for every $(360/14)$ degrees rotation of the cam. This movement is further reduced by a lever arrangement and it is imparted by an adjustable pin to the cassette sitting on the four ball-bearings described earlier. Total rotation of the cassette can be adjusted by changing the position of the pin on the lever arm.

D. *The mechanism of linear integration*

The rotation of the notched wheel is transmitted to cam 2 by a system of reducing gears (ratio 5:1). A lever L_2 (Fig. 1 (b)) kept pressed against cam 2 follows the motion of the cam. As cam 2 rotates the lever oscillates with an amplitude of 20° . This oscillating lever rotates a pinion G which is fixed just below C between the plates AA. On the lower side of plate C is attached a spur rack which through a slot in plate B is engaged to pinion G. A spring pulls the plate C so that the lever L_2 is always pressed against the cam 2 and any possible backlash is avoided. The slightest movement of cam 2 and thus of lever 2 results in a linear movement of the plate C. In this particular arrangement the total movement of C is 1.5 mm. Different amplitudes can be obtained by using cams of different sizes which are mounted on the common shaft. However, a continuous variation in the amplitude is not possible.

It is possible to take with this camera, Weissenberg photographs, without any integration, with either angular integration or linear integration only or with both simultaneously.

In this camera both movements complete an integral number of oscillations in about 70 min, the angular movement completing five oscillations while the linear movement completes one. Thus the exposure time chosen must always be an integral multiple of 70 min.

3. LOW TEMPERATURE ATTACHMENT

The Fankuchen-Lipscomb arrangement of cooling consists of blowing a jet of dry cold air on to the crystal. The cold air jet is surrounded by a sheath of dehydrated air at room temperature and this prevents any condensation of moisture on the crystal. This method is simple and extensively used in the X-ray investigation of crystals at low temperature. However one has to face the difficulty of removing the cassette (for loading and developing purposes) without disturbing the cooling arrange-

ment—a feature essential when one wishes to study crystals of substances which are liquids at room temperature. This difficulty is often overcome by using a split cassette. The use of a split cassette in an integrating

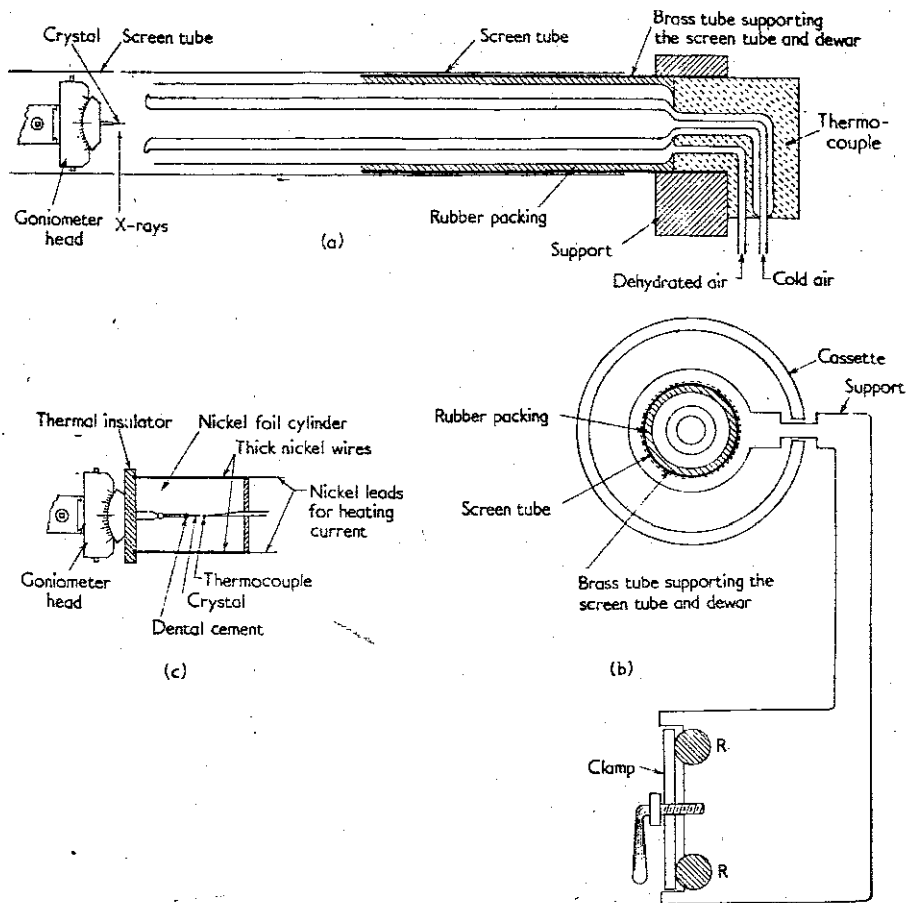


FIG. 2 (a) The cooling attachment. (b) End-on view of the screen tube holder which permits the removal of the cassette. (c) Heater attachment to the goniometer.

camera is, though not impossible, yet sufficiently complicated to make one look for some simpler arrangement. Recently Viswamitra (1962) has designed a special camera which permits the removal of the cassette from the goniometer end of the camera. The camera employs a large number of bevel and spur gears wherein the possibility of backlash cannot be so easily avoided.

The low temperature attachment described here permits the removal of an ordinary cassette without disturbing the cooling arrangement. This is achieved by making the screen tube holder support and cold-air lead thin enough to pass through the slot of the conventional cassette.

As shown in Fig. 2(a), cold air is passed through the Dewar tube which is surrounded by another glass jacket. Dehydrated air at room temperature is passed through the gap between the Dewar tube and outer jacket. The whole assembly is mounted inside a brass tube which also serves as the screen tube holder. This screen tube holder is held in position by a bracket-like brass support bent twice at right angles (Fig. 2(b)). The lower part of this support is clamped on to the camera. From Fig. 2(b) it is clear that cassette can be removed without disturbing the cooling system.

Some of the important features of the new design of the cooling attachment are:

- (i) It permits the removal of a conventional cassette without disturbing the cooling arrangement, so that crystals of the substances which are liquids at room temperature can be studied.
- (ii) Since the Dewar tube is enclosed by the screen tubes, the removal of the cassette does not change the condition of cooling so the temperature does not rise.
- (iii) The mounting of the Dewar tube inside the screen tube makes it quite safe against mechanical shock.
- (iv) This low temperature attachment can be used in any other commercially available Weissenberg camera (integrating or ordinary) with equal facility provided the screen tube holder (Fig. 2(b)) is made.

4. HIGH TEMPERATURE ARRANGEMENT

Steward (1949) suggested a heating arrangement using a nickel foil both as heater element and as a filter for $\text{Cu K}\beta$ radiation. This heater-cum-filter arrangement has been slightly modified to suit the present camera, and it is illustrated in Fig. 2 (c). The base of the furnace is an insulating material which forms a part of the goniometer head and it holds a cylinder (diameter 2 cm) of this nickel foil. This foil is heated up by passing current at low voltage. The cylinder is closed by another insulator and this effectively prevents any fluctuations in temperature due to convection currents. A thermocouple placed near the crystal records the temperature.

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