

AN X-RAY POWDER CAMERA

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The writer has used a battery of *x*-ray powder cameras of novel design for several years in conjunction with a Hadding gas tube. The cameras have given such satisfactory operation in this and several other laboratories that it seems desirable to publish a brief note of the design, which departs considerably from that of the usual powder camera, and offers a number of conveniences.

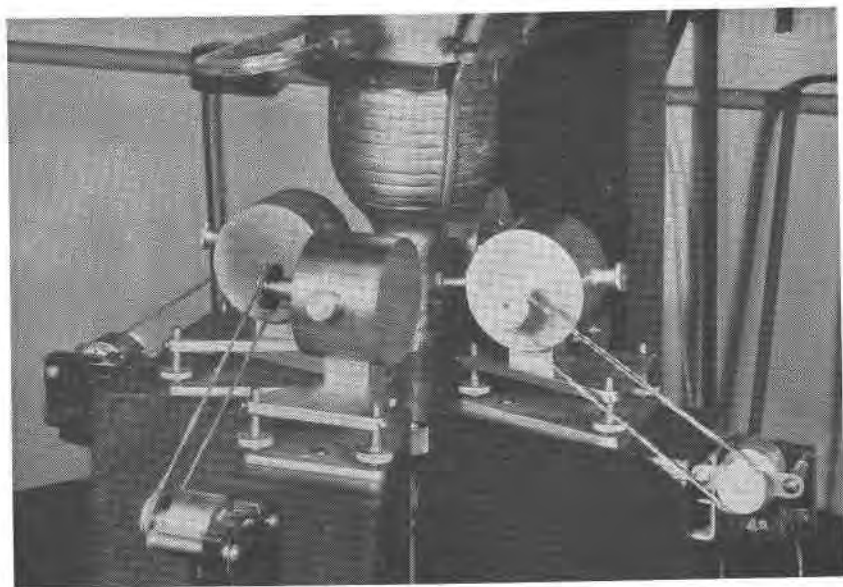


FIG. 1. Hadding *x*-ray tube with battery of four powder cameras. The support for each camera assembly is a wooden wedge which rests against cleats set at the wedge angle on the table top. Other apparatus may be used in conjunction with the *x*-ray tube by removing the wedges whose positions may later be exactly recovered by pushing them against the cleats.

GENERAL DESIGN.—The general appearance of the camera is indicated in figure 1 and the construction is illustrated in some detail in figure 2. The bare film is wrapped around the inside of the cylinder and held in place by two wedge rings, the inside one being soldered in place, the outside one loose. The film is protected from light by a screw-on cover.

The powder specimen, mounted as described beyond and attached to a $3/32$ inch brass rod, is held in a rotating, centering holder, illustrated in the lower part of figure 3 and described beyond. The inside of the

camera, against which the film is wrapped, and the bearing for the specimen holder spindle are turned and bored with the same lathe setting from a solid bronze casting, so there can be no eccentricity of the specimen as in most powder cameras.

The pin-hole system, illustrated in detail in the upper half of figure 3 and described further beyond, is arranged so that the pin-hole which

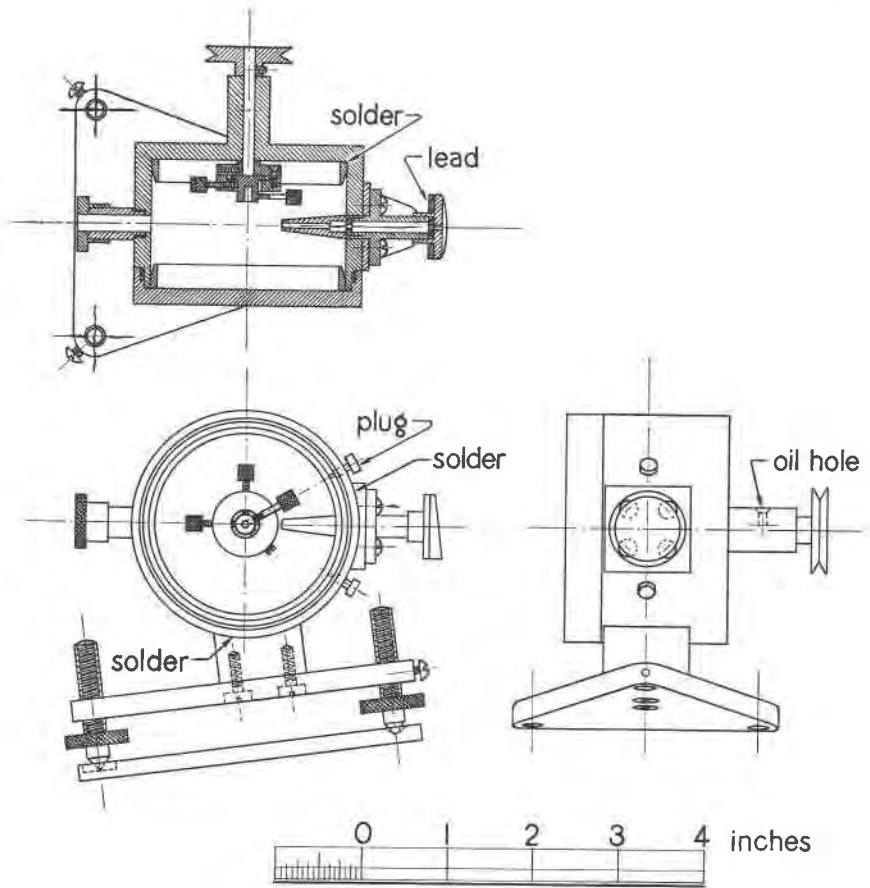


FIG. 2. Construction of powder camera. Levelling screws and lower base have been omitted from front view for clearness.

limits the rays is at the circumference of the camera to satisfy focussing requirements. The direct beam leaves the camera through a tube at the rear. In order to facilitate adjusting the pin-hole system to the best part of the *x*-ray beam, this exit port is fitted with a disk of fluorescent screen, held against the back of the tube by a friction cap. Patterson type B

fluorescent screen, facing outward and covered with cellophane for dust protection, has been found excellent for this purpose. The screen is backed with a disk of black paper to prevent the entrance of light into the camera.

The cylindrical body of the camera, soldered to a brass block, is adjustable on a triangular base by means of two loose-fitting screws. The base is supported by means of three pointed levelling screws which rest in three indentations of a second permanent, triangular base. The forward indentation is a cone, the others are a wedge groove and a flat groove respectively, giving a point-slot-plane support, so that the position of the camera is recoverable after removal for loading.

The sample is rotated by a type B3, 1 R.P.M., Telechron motor driving through a pair of pulleys connected by a looped rubber-band belt. The motor has no metallic contact with the camera. This prevents possible burning out of the motor windings in case of any sparking from the tube during times of temporarily erratic operation such as the breaking-in period.

DIMENSIONS.—The powder cameras illustrated are used chiefly for the identification of crystal phases by comparison of a film of the unknown phase with one of the suspected phase. For this purpose as well as many others, cameras of large diameter are not necessary. Indeed it is desirable to use cameras of as small a diameter as possible to cut down time of exposure. A convenient camera diameter is 57.26 mm., which makes one millimeter on the film correspond to a camera arc of 2° and to a glancing angle, θ , of 1° . The illustrations are for cameras of this size, but of course, the general design may be applied to cameras of any size. Films for this camera are $6\frac{3}{4} \times 1$ inches. They are conveniently prepared by cutting a standard 5×7 inch x-ray film into five 1 inch strips with the aid of a print trimmer and then finishing the strips as described beyond under *Film Size Uniformity*.

For the camera illustrated, the film ordinarily becomes over-exposed with unfiltered radiation if the exposure time exceeds 4 hours, using a sharply focussed Hadding tube running on half wave rectification, and passing 10 milliamperes at about 45 kilovolts. Shorter exposures are more usual, and considerable reductions may be made by using full wave rectification, and, at the expense of fine lines, by increasing the size of the limiting pin-hole in the pin-hole system.

MATERIAL.—The cylindrical body of the camera together with the spindle bearing are turned out of a single bronze, cup-shaped casting. The rest of the camera parts are made of brass, except as follows: The spindle, levelling screws and other large screws are of steel; the pin-hole system diaphragms and front cap backing are of 1/16 inch sheet

lead. All parts except the interior of the cylinder, and the adjusting sample holder, are nickel plated.

DETAILS OF CONSTRUCTION.—A detailed illustration of the centering specimen holder is given in the lower half of figure 3. The brass rod to which the sample is attached is held in the holder by means of the set screw operated by the outer knurled knob. The two inner knurled knobs operate two screws which translate the holder along two component directions at right angles to one another. The holder is held against these

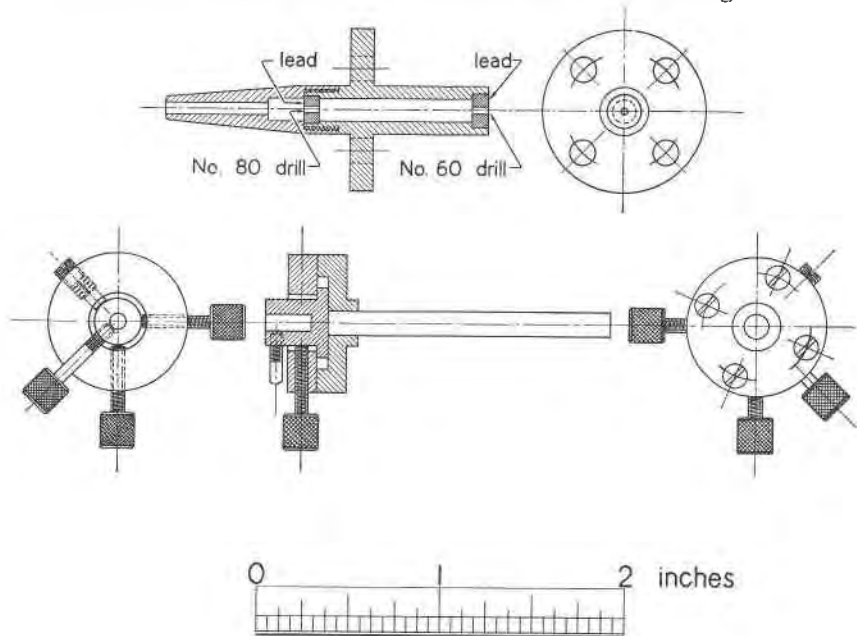


FIG. 3. Details of construction of pinhole system (above), and of centering specimen holder (below).

adjusting screws by a spring-actuated plug. This centering device enables one to make the axis of the powder sample accurately coincident with the axis of rotation.

The pin-hole system is illustrated in detail in the upper half of figure 2. It consists of two turnings which screw together. The right member bears the pin-hole system proper, consisting of two perforated sheet lead disk plugs. The 80-drill hole in the inner disk limits the beam and fits into a position at the circumference of the camera. The 60-drill hole in the outer disk merely limits the cone so as to prevent the *x*-ray beam from touching the sides of the exit port. This arrangement gives fine lines and excellent doublet resolution. The rays scattered by the inner pin-

hole are prevented from entering the camera by the conical cap which screws on to the pin-hole tube. The entire assembly is held by four screws to a block soldered to the outside of the camera cylinder. The holes in the pin-hole assembly, through which these screws pass, are purposely loose so as to allow a translation adjustment for making the pin-hole system axis intersect the specimen rotation axis. (See beyond, under *Primary Adjustments*.)

The cap on the end of the pin-hole system, illustrated in figure 2, serves three purposes: Its primary function is to make the camera light tight by holding a disk of black paper against the front pin-hole. When desirable, a piece of filter may be similarly held in the same place. In addition, the broad surface of the cap prevents accidental puncturing of the *x*-ray tube window if the camera should be carelessly placed against the tube, and its lead backing screens the camera against the direct beam when hard radiation is used.

A pair of plugs cover two 80-drill holes in the cylindrical part of the camera. Removal of these for a moment in an artificially lighted room exposes the film beneath these two tiny openings, so that when the film is developed, two small calibration dots appear. Their distance apart is a measure of the film shrinkage and permits calibration of the camera.

PRIMARY ADJUSTMENTS.—Before using the camera, the pin-hole system should be adjusted as follows:

1. A short section of $3/32$ inch rod, having one end turned down to a thin needle, is clamped in the specimen holder. The caps are removed from the pin-hole system and exit port, allowing one to sight through the pin-hole system. When the spindle is rotated, the needle appears to execute a small oscillation as seen through the pin-hole system, because its axis is not in the axis of rotation. The two translation screws are adjusted until, on rotating the spindle, the needle remains fixed.

2. With the needle axis coincident with the spindle axis as described above, the four screws holding the pin-hole system to the camera are loosened. While looking at a well illuminated white surface through the pin-hole system it is translated up or down until the needle appears in the center of the field. The screws are then permanently tightened and the axis of the pin-hole system remains intersecting the axis of the sample rotation.

PREPARATION OF POWDER MOUNT.—The powder mount is easily and quickly made as follows (figure 4): a quarter inch length of $3/32$ inch brass rod, held in pliers, is heated in a flame and a small lump of picein wax melted on to the tip of one end. A $5/16$ inch section of fine glass capillary, made by drawing out pyrex tubing, is inserted into this wax after the latter is softened by reheating. After a short cooling period, the

axis of the glass capillary can be made accurately parallel to the axis of the brass pin by pressing the thumb nail into the pisein on the side toward which the capillary leans. Only the parallelism of axes need be adjusted by this means for the centering adjustment of the holder takes care of making the axis of the capillary coincide with the rotation axis. The mount for holding the powder is now complete. It is convenient to make up a large number of these at once, ready for application of any powder which it becomes desirable to mount.

After trying a number of procedures for mounting the powder, the following was found to be most convenient and give best results: The substance to be examined is ground in an agate mortar and heaped in a small pile at the edge of a small sheet of paper. A drop of mucilage from a Le Page's "grip spreader" is then placed on the edge of the thumb nail of the left hand, the mount is picked up in the right hand and the capillary rolled around in the mucilage to spread it evenly over the surface

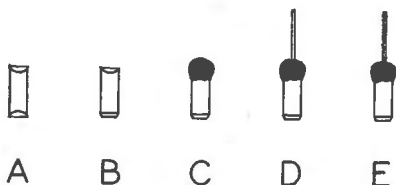


FIG. 4. Stages in preparation of powder mount. A. Rough $\frac{5}{16}$ inch section of $\frac{1}{8}$ inch brass rod as clipped from stock. B. Brass pin with lower clipping burr removed by filing. C. Brass pin with pisein lump melted on burr end. D. Pyrex glass capillary inserted in pisein. E. Completed mount with specimen powder stuck to capillary.

of the glass; then after picking up with the left hand the paper with the powder, the capillary is rolled around in the powder until an even coating adheres to it. Both the bare capillary mounts and the completed powder mounts may be stored by plugging the brass rods into holes in a wooden block in the same manner that machine drills are stored. The holes in the block should be shallow enough so that the brass part of the mount may be gripped in forceps for mounting and other handling.

LOADING.—The brass pin of the powder mount is clamped in the specimen holder by tightening the set screw knob, and the camera is set on a table with both the pin-hole system cap and exit port cap removed. The powder sample is transferred to the specimen holder of the camera by means of forceps, and the capillary centered in exactly the same way as the needle during the adjustment of the pin-hole system. The caps are replaced and the camera taken into the darkroom for loading. With the screw cover and outer film ring removed, the film is fitted against the inside of the cylinder and slipped down behind the permanent back ring, taking care that the edge of the film is not turned over

in wedging it home. The outer film ring is then wedged over the outside of the film, using the same precautions against deforming the film. The cover is then screwed on and the camera is ready for use. Note that the screw cover is fitted with threads for only half of its overlap with the camera cylinder. This arrangement permits seating the screw before tightening it and makes it virtually impossible to start the two parts of the screw in disregistry, a great convenience in the dark.

FILM SIZE UNIFORMITY.—Maximum information may be obtained from a film if reflections of both lowest and highest glancing angles are recorded. This camera is designed to accommodate a film which records

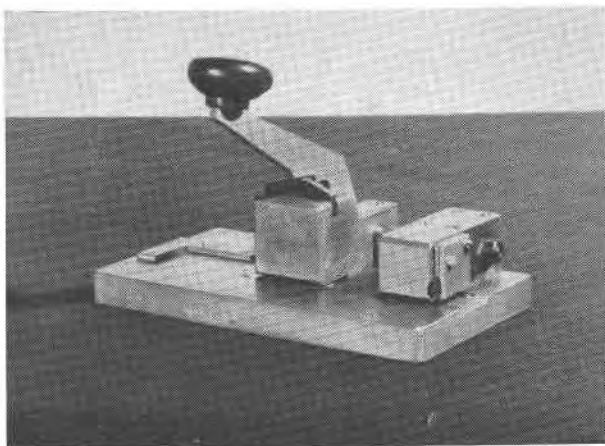


FIG. 5. Film trimmer for x-ray powder camera. The film is laid on the flat surface and pushed back and left against the cleats. When the knob is depressed, a central hole is punched in the film by a die in the central block and at the same time the right edge is trimmed to size by the knife.

reflections of high glancing angles limited only by the pin-hole system cone. In order to utilize the low glancing angle region most effectively, the hole in the film over the exit port of the camera should be clean-cut, accurately located, and of minimum dimensions. This is best taken care of by a punching and trimming outfit of the kind illustrated in figure 5. This takes a one inch film strip, cuts it to exact length ($6\frac{3}{4}$ inches) and at the same time punches a minimum size hole ($\frac{1}{4}$ inch) in the exact center of the film. In placing the film in the camera, the hole is automatically centered over the exit port because the free ends of the film just touch the pin-hole system cone.

ACKNOWLEDGMENT.—The writer is indebted to Professor J. T. Norton for a number of ideas used in the design of this camera. A comparatively crude prototype of similar proportions in use in his laboratory was used as a starting point in the design described.