Improved Camera for Better X-Ray Powder Photographs

Designers and manufacturers of X-ray cameras and photography support equipment and research groups that utilize X-ray diffraction analysis will be interested in learning about an improved camera for better X-ray powder photographs. This camera is used for obtaining powder-type photographs of single crystals or polycrystalline powder specimens. The X-ray diffraction photographs of a powder specimen are characterized by improved resolution and greater intensity. A reasonably good powder pattern of small samples can be produced for identification purposes.

Three drawings are used in describing in detail the camera shown mounted on a guide which in turn is fastened on a camera track (not shown). This track has a screw adjustment to allow aligning the camera with the X-ray tube. The camera slide is fitted onto the track and can be moved horizontally with an adjusting screw which also serves for alignment of the camera with the X-ray tube. This alignment is facilitated by mounting a counter tube on the track behind the camera and adjusting the track and camera to obtain the highest intensity.

The film holder is a cylindrical-shaped surface which holds the lighttight film envelope. When analyzing small specimens a 20 mm radius is a satisfactory compromise between resolution and intensity, although other radii may be used. In order to allow the specimen holder at least 90°, 2θ scanning movement normal to the center line of the cylinder, the upper portion of the cylinder wall is cut away. The film holder has four notched pins, of which two are stationary and two are mounted onto a sliding curved segment with an inner diameter the same as the outer diameter of the cylinder, which has two elongated holes that allow the pins to pass through.

When the envelope is placed in the cylinder and clamped between the four notched pins, it takes the shape of the inner diameter of the cylinder. Then the curved segment is clamped to the cylinder with a knurled head screw.

The incident beam collimator and exit port are precisely aligned with the center line of the film holder. Tips of the collimator and port are shaped to allow a maximum diffraction angle of front and back reflections. The front end of the collimator has a ray proof fitting with the X-ray tube tower. At the end of the film holder the film is sandwiched between this and a cylindrical metal mask which blocks out radiation from the film of exactly known circumference length. This shadow is used as a scale to measure the change

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of film length during processing. Serving as radiation protection in front of the film holder is a plug. It is removed when centering the crystal.

The film holder is mounted with three clamps onto a flanged shaft and is marked to show the angle of view of the X-ray tube target. In case illumination is required from the back to align the specimen, the shaft is hollow. It is mounted onto a vertical plate and attached to the horizontal slide.

For scanning, a motor is mounted onto a vertical plate with a worm gear reduction to a drive shaft and handle and also a slip clutch for manual drive. On the end of this shaft another worm engages a worm gear fastened to an L-shaped motor bracket. The motor bracket rotates on a pair of preloaded ball bearings on the flanged shaft. On the far end of the bracket a reduction gear motor with shaft extension is clamped. By accurately machining the collimator mount in the camera and by providing adjustments of the specimen position in three orthogonal directions, the specimen is accurately centered so that it remains in the center of the camera during rotation and scanning over the entire angular range.

An X-Y cross slide is mounted on the shaft extension. The cross slide has an extension with a hole in the center. The end on the outside is threaded and has two elongated slots $180^\circ$ from each other. A compression spring is inside of the extension. On the end of the hole a receptacle is fitted with a guide pin which fits the slots of the extension. The guide pin rests on a nut which elevates or lowers the receptacle and provides the Z motion in order to align the mounted specimen in the center of the rotations and the X-ray beam.

The crystal or crystals are glued to a fiber such as a glass fiber, which in turn is glued to a nipple fastened to the receptacle. In order to align the mounted specimen, a microscope with a cross hair marker is mounted on a plate fastened on the front end of the horizontal slide. In centering, the specimen is aligned while rotating on its own axis. Then it is pivoted in the plane which intersects the axis of rotation. The cross hair marker of the microscope assists the alignment of the specimen, which has to remain centered at one point while rotating and scanning.

The worm gear has a segment of a dial and two adjustment stop buttons. A flange is attached to the stationary shaft and has an extension on which a dial indicator is placed. This dial indicator has two adjustment screws which alternately engage and disengage a microswitch at one end of the scanning period and the other. This makes the bracket oscillate between the two stop buttons.

In conclusion, a brief description of the improved X-ray powder camera is offered. In this camera, the film is bent into a semicircle; a collimated X-ray beam strikes the specimen which is simultaneously rotated around two axes; and the diffracted X-rays fall upon the film as a series of concentric rings. The camera employs a semicircular cassette around the inner wall of which the film is arranged in a lighttight envelope. Because the specimen rotates about the axis of the cassette, only diffraction patterns of polycrystalline powder specimens can be recorded by using the camera. It requires random distribution of the powder grains to insure that there will be enough particles oriented at the correct angles to the incident beam to allow reflections of characteristic X-rays from all possible reflecting planes.

**Patent status:**

Title to this invention has been waived under the provisions of the National Aeronautics and Space Act (42 U.S.C. 2457 (f), to the North American Philips Company, Inc., Norelco Division, Briarcliff Manor, New York, 10510.