# APPARATUS FOR OBTAINING POWDER-TYPE *X*-RAY DIFFRACTION PATTERNS FROM SINGLE CRYSTALS

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Abstract

An *x*-ray diffraction camera has been designed by means of which powder-type diffraction patterns are obtained from single crystals. Such patterns are obtained by imparting to the specimen a combination oscillatory and rotatory motion.

## Introduction

There is a definite need for an instrument that will yield *x*-ray diffraction patterns useful for identification purposes (preferably, powder-type patterns) from large single crystals, or coarse crystalline aggregates. In cases where it is undesirable to remove and powder a fragment of the material to be tested, such an instrument would prove valuable. This need is felt especially in the field of gem identification, but also in many other types of work.

The instrument herein described was developed in the laboratories of the Gemological Institute of America, to be used in gem identification, especially for use with opaque materials or those having no plane surfaces, such as double cabochons or carved or irregular pieces. The instrument's design is based upon a suggestion made by Dr. Samuel G. Gordon, Associate Curator of the Academy of Natural Sciences of Philadelphia, and a member of the Educational Advisory Board of the Gemological Institute of America.

## Instrument

To obtain a powder-type *x*-ray diffraction pattern of a crystalline substance, it is necessary to be able to bring a large group of atomic planes into a reflecting position. This is ordinarily accomplished by finely powdering the material, and by rotating the sample to bring about completely random orientation of the grains.

A pattern of this type from a single crystal may be obtained by imparting to the specimen a motion which will, during the course of the exposure, bring a much larger number of atomic planes into reflecting position than can be obtained by any simple rotary motion. When this is done, a powder-type pattern is obtained. The orientation of the single crystal at the time the sample is mounted in the camera is of no consequence.

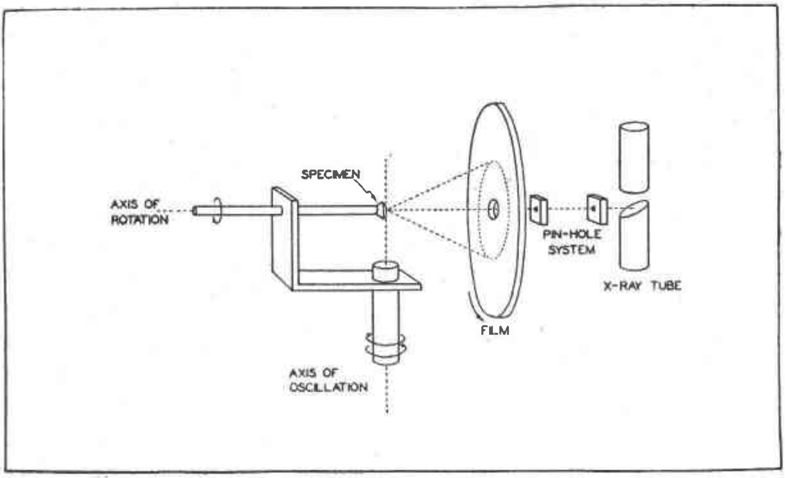


FIG. 1. Schematic representation of a camera designed to give powder-type *x*-ray diffraction patterns from single crystals.

The type of motion necessary to approximate the random-orientation condition of a powdered sample is produced by simultaneous rotation and oscillation of the specimen. The apparatus used to accomplish this is shown schematically in Fig. 1. The first model of the camera, now in use, employs an 80 degree oscillation of the sample at the rate of one degree per minute, and simultaneous rotation of the sample at one revolution per minute.

Because of the size of the samples ordinarily encountered, it was most desirable to utilize the back-reflection method. These are recorded on a flat film. A back-reflection type camera is also more feasible from the standpoint of mechanical design. In this respect, the camera resembles the standard back-reflection type commonly used in metallurgical work. In some cases, due to lack of completely random orientation, incomplete circles were recorded. This difficulty is easily overcome by rotating the film about the *x*-ray beam as an axis during exposure.

## RESULTS

Typical results obtained with the instrument described are shown in Figs. 2 and 3. All of these patterns were obtained from single cut gems (single crystals) using filtered copper radiation, and a specimen to film distance of four centimeters. It will be noticed that there is a sharp separation of the $K_{\alpha_1}$ and $K_{\alpha_2}$ lines, as is to be expected from high angle reflections of this type.

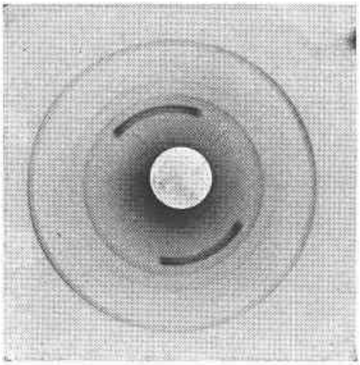

FIG. 2. Diffraction pattern of single crystal of corundum. Filtered copper radiation. Exposure 6 hours.

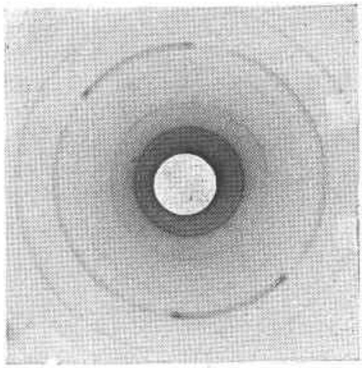

FIG. 3. Diffraction pattern of spinel. Filtered copper radiation. Exposure 6 hours.

## CONCLUSIONS

The results herein described are the first obtained with an experimental model camera. An improved camera design is being worked on at the present time. The method could probably be adapted to an apparatus employing automatic recording of the $x$-ray spectra by means of a Geiger counter.

In practice, the advantages of a camera of this type for many kinds of work are manifold. The specimen may be a single crystal or a crystalline aggregate. In size it may vary from one millimeter or less up to any size that will be accommodated by the instrument. Specimens 10 centimeters in diameter can be handled in the present camera. A catalog can be readily built up similar to that published by the American Society of Testing Materials, using the spacings of the three strongest lines as a key. Such a catalog is now being prepared for gem materials, and will probably be expanded to other substances. Modification of the camera design to utilize forward reflections would make it possible to use the present A.S.T.M. cards.