An X-ray Diffraction Camera for the Alignment of Large Single Crystals*

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Abstract-- A back-reflection Laue camera incorporating three rotation axes and three orthogonal translation axes is described. This camera allows for the alignment of large single crystals with a precision of $\pm$ 0.25°. The degree of single crystallinity of a specimen may be easily examined. In addition it is possible to accurately mark a crystal for subsequent utilization.

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DISCLAIMER

INTRODUCTION

Many research investigations necessitate the growth and orientation of large single crystalline specimens. In this paper an x-ray diffraction camera is described which is specifically designed for use in such work. This camera has several desirable features. First, it will accommodate large specimens of up to an inch in diameter and three or more inches in length. Second, it is possible to determine in a straightforward manner whether or not a given specimen is a single crystal. To do this it is not necessary to determine the crystal orientation. Third, it is possible, with relatively little training on the part of the operator, to determine a precise orientation for a single crystal. Finally, it is possible to scribe a specimen while mounted in the camera so that the specimen may be subsequently cut to some desired orientation or otherwise utilized.

DESCRIPTION OF THE CAMERA

The camera is an adaptation of a back-reflection Laue camera. Figures 1 and 2 show, respectively, line drawings of side and top views of the camera. Figure 3 shows an oblique photograph of the camera. A brass plate mounted on three adjustable legs serves as the camera base. A Unicam S.31 film cassette and collimator assembly is attached to the base with an adjustable support. The leg and support adjustments are for adaptation of the camera to the x-ray source unit and are not relevant to further operation of the camera.

A goniometer head which incorporates three orthogonal translation axes is attached to the base and may be rotated in the plane of the base through an angle of 180^{o} about the point of attachment. The goniometer head incorporates a sample holder which may be rotated through 360^{o} about an axis coincident with the axis of a one inch diameter hole, 5/8 inches deep, in which the sample may be secured by means of a set screw. The assembly to which the sample holder is attached may be rotated through 100^{o} about an axis normal to the two rotation axes previously described. The latitude in rotational and translational freedom allows exposure to the x-ray beam of any spot on the surface of the test specimen with the exception of that limited portion which is recessed in the sample holder.

OPERATION OF THE CAMERA

Determination of single crystallinity. To determine whether a specimen is a single crystal, several Laue photographs are taken at translational intervals along a face of the sample. The sample-to-film distance and the angular orientation are held constant during this process. If the sample is a single crystal, all of the photographs will be identical. An additional check may be made by rotating the sample 180° and repeating the procedure. Again, if the sample is a single crystal, the Laue photographs will be identical except for the mirroring introduced by the 180° rotation.

Orientation of single crystals. For the initial exposures in the orientation process it is most convenient to use a short sample-to-film distance of three centimeters or less. This reduces the necessary exposure time and compresses the data on the film so that the symmetry about points of interest may be more easily recognized. In addition a greater angular area of diffracted information is intercepted, and fewer pictures are required to find an axis of interest.

The alignment procedure is to examine the initial exposures for symmetry elements. The most straightforward method is to look for points which mark the intersection of several zone lines. These points define directions in which symmetry axes may be expected to occur. The undistorted symmetry about such a point of interest may be examined by rotating the crystal so that the point lies at the center of the film. The required rotational adjustments may be determined with the aid of a Greninger chart[1] scaled for the appropriate sample-to-film distance. Alternatively, the distance from the center of the film to the point of interest may be measured. This distance divided by the sample-to-film distance provides a direct measure of the Bragg angle. In addition a rotation angle from either a horizontal or vertical reference line may be determined. These two angles may be readily interpreted to provide the appropriate rotational adjustments for centering the point of interest on the film. During the course of the rotational adjustments the translational adjustments should be used to maintain the desired sample-to-film distance and to locate the sample so that the incident beam strikes the desired area of the sample. When a point of interest is centered on the film the symmetry of the crystallographic axis coincident with the incident beam can be observed. The observed symmetry limits, and in some cases uniquely defines, the crystallographic direction[2]. For all crystals with orthorhombic or higher symmetry, the observed symmetries for a few simply related crystallographic directions define the crystal orientation.

When a precise orientation of any crystallographic direction is desired, the foregoing procedure may be modified so that in the terminal stages of the rotational adjustments the sample-to-film distance is increased. An increase in sample-to-film distance enhances the linear distance on film subtended by any rotational correction angle. The maximum sample-to-film distance for the camera is about eight centimeters, and at this distance a precision of alignment of $\pm$ 0.25° may be attained.

Scribing the crystal. After a crystal has been aligned with a desired axis normal to the plane of the film, it may be readily marked to facilitate subsequent cutting. A simple device to do this consists of a small brass cylinder with a scribe attached near one end. The marking is accomplished by placing one end of the cylinder against the film holder and scribing a line around the sample as shown in Figure 3. In this way one may be sure that the scribed line is parallel to the film and hence perpendicular to the desired axis. In some instances a coating on the crystal, such as a colored wax, may facilitate the scribing operation.

DISCUSSION

The technique for aligning single crystals described in this paper requires only a very limited knowledge of crystallography in that one need only recognize the symmetries of the axes of interest. The conventional technique employing stereographic projections requires a more extensive knowledge of crystallography plus a knowledge of stereographic projections and of the interplanar angles for the crystal being investigated. Tables of such angles are available for the cubic system but must be calculated from the cell constants for other systems. While the method described here requires the taking of several pictures to orient a single crystal, it is generally faster and easier than the method using stereographic projections even though the latter method requires only a single photograph. This is particularly true for non-cubic crystals. In addition, once a crystal is oriented, the method herein described allows the sample to be readily and accurately marked for subsequent utilization.

ACKNOWLEDGMENT

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REFERENCES

1. A. B. Greninger, Trans. Am. Inst. Mining Met. Engrs. 117, 61 (1935); see also A.S.T.M. Standards 1949 part 2, (ASTM, Philadelphia 1950) pp 1105-1117.
2. M. J. Buerger, X-ray Crystallography, (Wiley, New York, 1942), pp 468 and 475.

FIGURE CAPTIONS

Fig. 1. Side view of camera.

Fig. 2. Top view of camera.

Fig. 3. Oblique view of camera showing a sample being scribed.

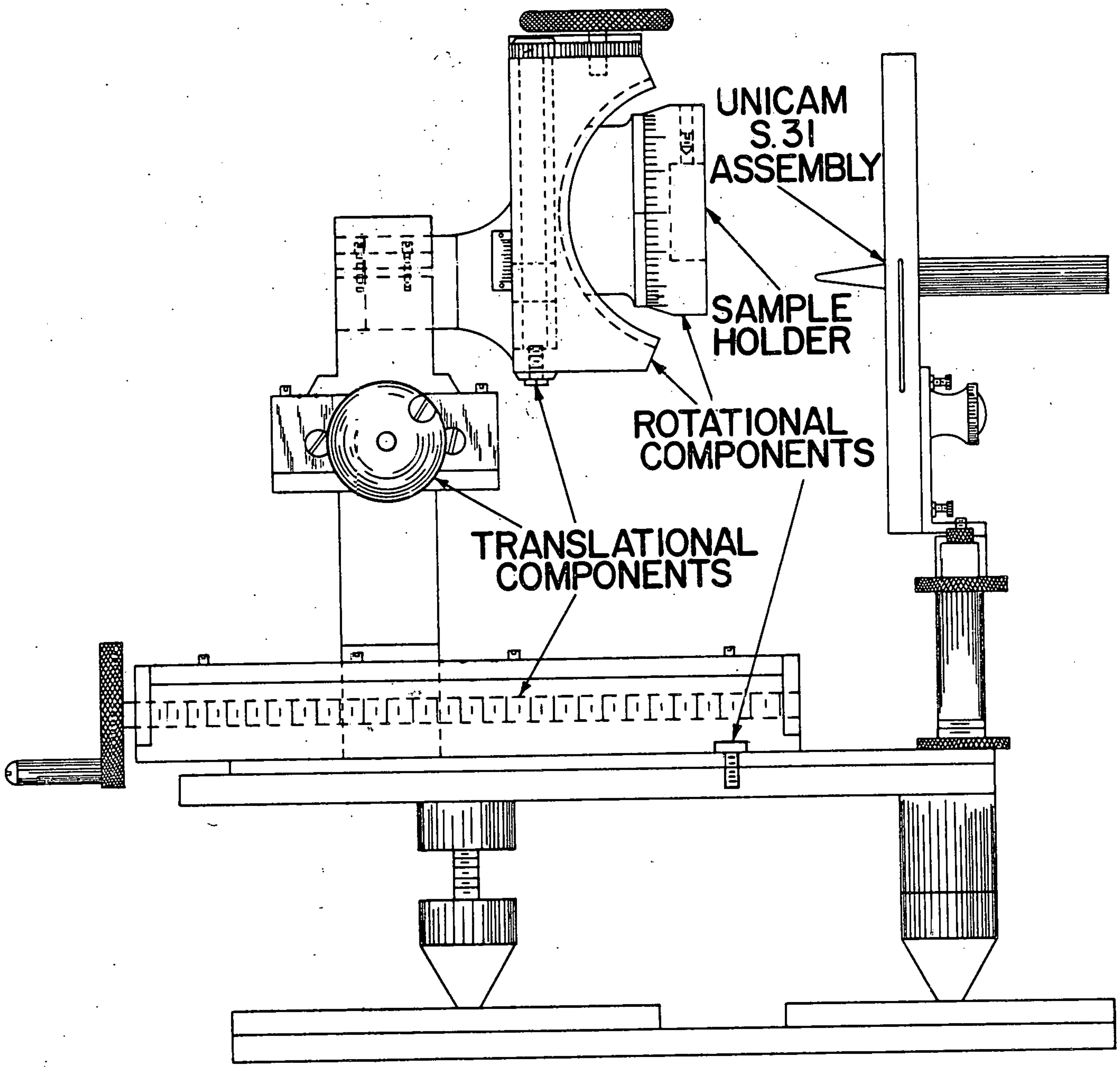
UNICAM S.31 ASSEMBLY
SAMPLE HOLDER
ROTATIONAL COMPONENTS
TRANSLATIONAL COMPONENTS

Figure 1

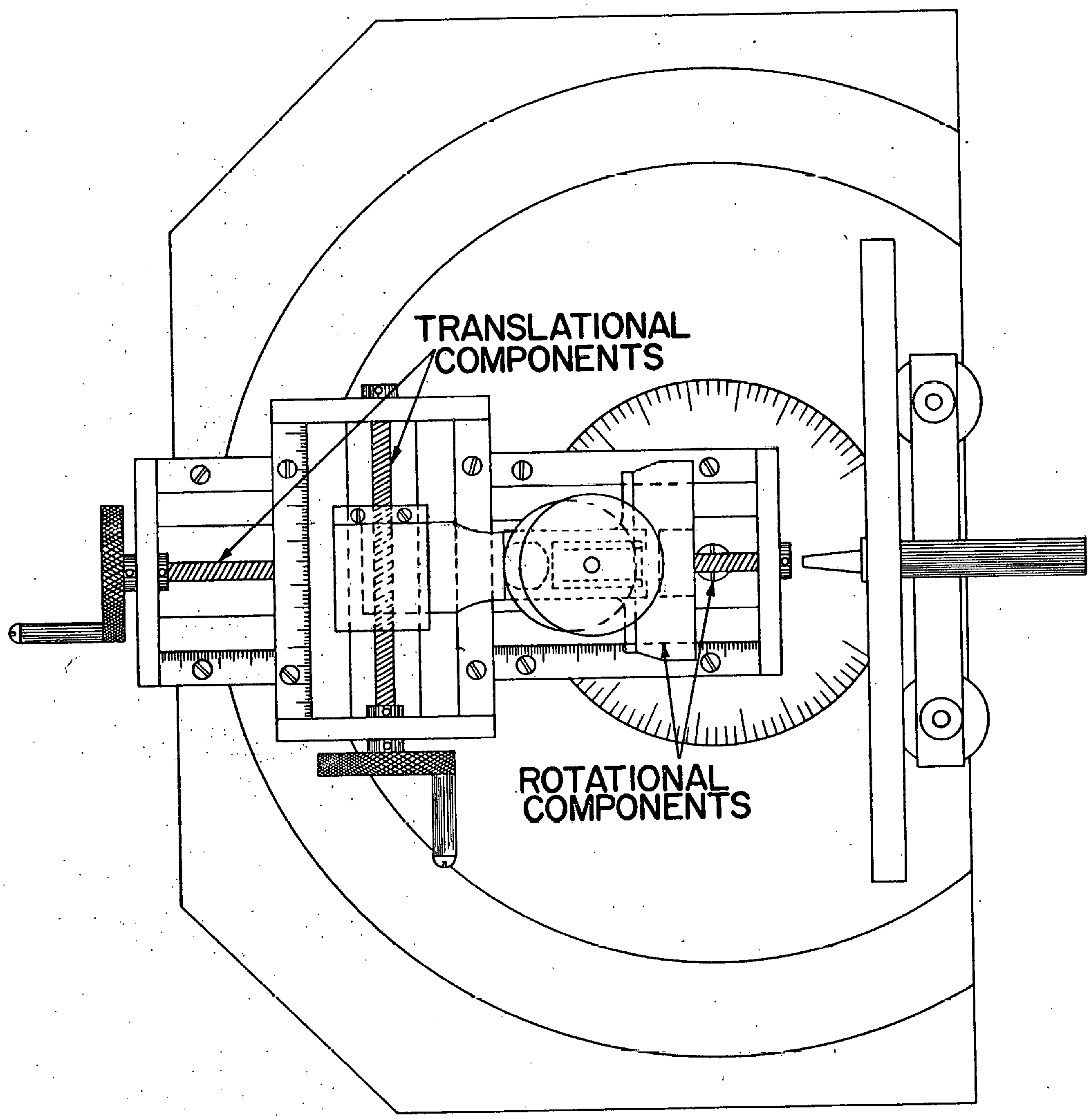

Figure 2

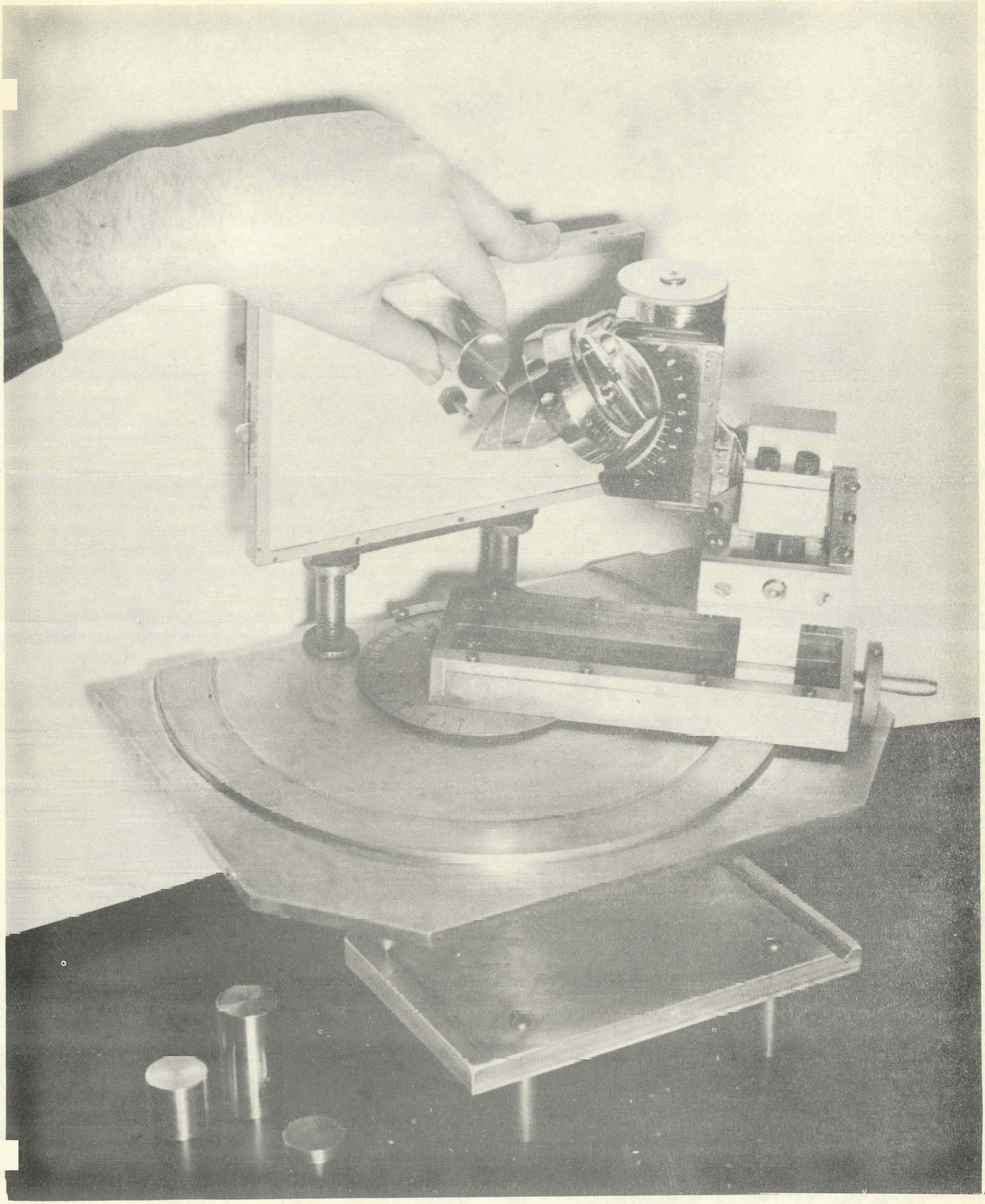

Figure 3