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AN ANALYSIS OF INSTRUMENTAL ERRORS AFFECTING THE PERFORMANCE OF A SCHULTZ-TYPE TEXTURE GONIOMETER

by

J.W. KELLY

ABSTRACT

The performance of a Schultz-type texture goniometer is shown to be adversely affected by a number of experimental errors all of which result in defocusing of the diffracted beam. These errors result from the tilting of the specimen, from lack of precision in positioning it and from its absorption coefficient. An experimental procedure is outlined which minimizes these errors and results in optimum performance of the instrument.

CONTENTS

1. INTRODUCTION

In the quantitative determination of the degree and type of texturing in, for example, extruded bar, the X-ray intensity diffracted by a selected atomic plane in any one direction is compared with that diffracted from a randomly oriented specimen in the same direction. By measuring this ratio in a large number of directions, lines of equal intensity-ratio can be drawn on a pole figure and a complete picture of the type and degree of texturing can be obtained for any atomic plane.

The major uncertainty in this type of work is the magnitude of the absorption correction which varies with the angle between the normal to the specimen surface and the plane of the incident and diffracted beam. Several attempts have been made to calculate these corrections accurately (Decker, et al., 1948; Schultz, 1949; Schwartz, 1955), and to minimize them (Norton, 1948; Field and Merchant, 1949) and one of the most successful of the latter is the technique of Schultz (1949, p. 1030). Instruments based on his design are now available commercially from several manufacturers such as Siemens and Halske, and Philips.

Schultz claimed that absorption corrections were unnecessary when his instrument was used in the reflection region. This claim was based on the result that the X-ray intensity reflected from a random specimen remained constant to within 10 per cent. of its initial value when the normal to the specimen surface was tilted by up to 75° out of the plane of the incident and diffracted beam. (The remaining 15° had to be determined by the transmission method or by the reflection method using other specimens cut at right angles to the original specimen from the textured material.) However, when using the Siemens instrument to determine the texture of hot-pressed beryllium oxide, it was found that the intensity reflected from the random specimen decreased to less than 90 per cent. of its initial value for angles of tilt as low as 30° to 40°. Since this result drastically reduced the usefulness of the texture goniometer, an analysis was made of the factors causing this discrepancy. The results of this analysis are presented below.

2. BASIS OF THE SIEMENS TEXTURE GONIOMETER

In Schultz's technique, the specimen is placed at the centre, O, of a vertical ring, A, (see Figure 1) such that at all times the specimen surface contains the axis of the ring and the normal to the surface lies in the plane of the ring. The ring is rotated about a vertical axis, which passes through O, until the specimen surface makes the appropriate Bragg angle θ with the incident X-ray beam I_o, and the detector is rotated about the same vertical axis to the 2θ position. Both ring and detector are then locked in position, thereby fixing the geometry of the detection system. The X-ray beam is horizontally collimated at both the aperture and detector slits, and vertically collimated by the main slit placed immediately in front of the specimen. The specimen is then rotated around the normal to its surface and at the same time the normal to the specimen surface is tilted at a slower rate out of the plane of the incident and diffracted beam. In this way the intensity of the diffracted beam is recorded along a helical path on a pole figure, the centre of the pole figure corresponding to the pole of the specimen surface.

Following Schultz's analysis, let W and S be the horizontal and vertical widths, respectively, of the beam on the specimen (see Figures 2a and 2b). Then the scattering volume dV at a distance t below the surface of the specimen is

$$dV = \frac{W\,S\,dt}{\sin\theta\cos\phi},$$

where ϕ is the angle of tilt. The corresponding X-ray path length in the specimen is

$$\frac{2t}{\sin\theta\cos\phi}.$$

Then if I is the intensity scattered per unit volume and μ the linear absorption coefficient,

$$dI = I_o \exp(-2\mu t/\sin\theta\cos\phi)\,dV$$

$$= I_o \exp(-2\mu t/\sin\theta\cos\phi)\,(W\,S/\sin\theta\cos\phi)\,dt,$$

and the total diffracted intensity is

$$I = I_o \frac{W\,S}{\sin\theta\cos\phi} \cdot \int_0^\infty \exp(-2\mu t/\sin\theta\cos\phi)\,dt$$

$$= I_o \cdot \frac{W\,S}{2\,\mu}$$

Thus, the intensity diffracted by a randomly oriented specimen is expected to be constant and independent of the angle of tilt. In practice, Schultz found that the intensity slowly decreased to 90 per cent. of its original value at $\phi = 75^\circ$ and thereafter decreased rapidly as a result of the specimen surface intersecting the incident beam.

There are, however, two assumptions in the analysis of Schultz. They are:

(a) that the width of the diffracted beam is constant for all angles of tilt, and

(b) that the surface of the irradiated specimen contains the axis of the vertical ring and lies on the focusing circle.

Factors which affect these assumptions and hence the performance of a Schultz-type texture goniometer in the reflection region are discussed in the following sections.

3. FLATNESS OF SPECIMEN

In any true focusing arrangement, the specimen is bent to the radius of the focusing circle and ground to its diameter. In the Schultz method, neither of these conditions is met, since a flat specimen is used. However, since only small areas of the specimen surface are irradiated (approx. 1 mm^2), it can easily be shown that the error in the 2θ position due to defocusing by the flat specimen is only of the order of 0.05° for an aperture slit of about 1 mm. This is negligible since the 2θ position cannot be read to better than 0.1°.

However, in the Siemens instrument, the specimen is shuttled backwards and forwards over a length of about 1 cm in order to increase the area scanned by the beam and so improve the statistical accuracy of the results. Hence, for this instrument, it is important that the specimen be ground or polished flat to within a few thousandths of an inch over an area of almost 1 cm^2 in order that the surface of the specimen should lie on the focusing circle at all times. In practice this is readily obtained by good mechanical metallographic polishing procedures.

4. DEPTH OF PENETRATION

Since the instrument uses focusing X-ray optics, it is important that the source, the specimen, and the detector lie on the focusing circle. This is largely built into the instrument. However since it is only the surface of the specimen that lies on the focusing circle, it is essential that the depth of penetration below the surface of the specimen be small. If a large fraction of the diffracted intensity originates from below the surface, the effect is to increase the radius of the focusing circle as illustrated in Figure 3. This results in an asymmetrical broadening in the direction of smaller values of 2θ.

If it is assumed that a specimen of infinite thickness, t_{∞}, (as used in Schultz's analysis) is equivalent to a specimen whose thickness is such that the intensity diffracted from its lower surface is one thousandth of that diffracted from its upper surface, it is easily shown that

$$t_{\infty} = \frac{3.45 \sin \theta}{\mu}$$

Then with $\theta = 20^{\circ}$, t_{∞} for Al and BeO are 0.004 and 0.020 inch respectively, using copper Kα radiation. Thus for aluminium all the diffraction intensity effectively originates at the surface, but for beryllium oxide a large fraction is due to the sub-surface layers.

The magnitude of this asymmetrical line broadening effect for materials of low absorption coefficient was determined by measuring $\delta(2\theta)$, the change in the position of the detector when a material of high absorption coefficient was moved radially from its initial position on the focusing circle of radius R to a new position corresponding to a radius (R + 0.020) inches. Filtered CuKα radiation was used and the change in the maximum intensity position of the (111) reflection of aluminium was determined by step scanning through the 2θ positions in each of the above cases with the detector acceptance angle reduced to 0.1°. It was found that $\delta(2\theta)$ equalled 0.3° approximately. Since in texture goniometry it is the integrated intensity from a given area rather than the spatial resolution of the reflected beam that is important, it is clear that, for materials of low absorption coefficients, detector apertures must be used which are coarse by normal X-ray diffractometry standards.

5. POSITIONING OF SPECIMEN

It is a necessary condition of Schultz's analysis that the centre of the outside ring (and hence the axis of tilt) should lie both in the specimen surface and in the plane of the incident and diffracted beam. It was found however, that unless considerable care was taken in setting up the specimen on the goniometer, the surface of the specimen could be up to 0.010 inch away from this position owing to stickiness in the specimen mount and to play in the shuttling carriage which supports the mount in the Siemens instrument. When the angle of tilt (ϕ) is zero, it is possible to compensate for this error by moving the detector to a new position ($2\theta'$). But when $\phi > 0$, this correction no longer suffices since the centre of the specimen, as seen through the main slit, moves either further away from or closer to the correct axis of tilt depending on the direction of tilt and whether the specimen surface is initially in front of or behind the correct axis of tilt. In both cases there is an asymmetrical broadening of the 2θ position. This effect is illustrated in Figure 4 for the above case when the axis of tilt lies in the plane of the incident and diffracted beam, represented in the figure by the centre line of the main slit. The asymmetrical broadening is accentuated if, in addition to the incorrect positioning of the specimen, the axis of tilt lies at some point outside this plane.

The importance of this error was evaluated by deliberately setting a specimen 0.010 inch behind and above the correct axis of tilt and measuring the variation in 2θ for the (111) reflection of an aluminium specimen as ϕ was increased from 0 to 65° of tilt. Using narrow entrance and detector apertures, the 2θ value was found to decrease by 0.2 to 0.3° at $\phi = 65°$. Thus, small errors in the location of the specimen can lead to large errors in 2θ for large values of ϕ and so great care must be exercised in specimen alignment. Unlike the preceding error, this error can exist irrespective of the type of material being examined.

6. EFFECT OF TILTING

When the errors discussed previously have been minimised, a further error can still arise as a result of the tilting of the specimen. In Figure 5, the specimen is viewed through the main slit along a direction lying in the specimen surface. It can be seen that as ϕ is increased from ϕ_o to ϕ_1, a portion of the X-ray beam is diffracted from an area lying in front of the axis of tilt and another portion from behind it. Thus part of the specimen gives higher apparent angles of 2θ and the other part gives lower angles of 2θ than the correct value which is determined when $\phi = 0$. The overall result is that the 2θ position is symmetrically defocused and the angular resolution decreased. For a given angle ϕ the defocusing increases, the larger the width of the main slit.

The importance of this source of error was tested by varying the width of the main slit. Three slit widths were used, 0.050 inch, 0.020 inch (Siemens slit) and 0.010 inch. The effect of these slit widths on the count rate versus angle of tilt curve was determined for the (0002) reflection of a randomly oriented specimen of BeO and is illustrated in Figure 6. From these curves it is clear that the Siemens slit represents an optimum value: little advantage is to be gained by using the finer slit since this reduces the area irradiated and hence the statistical accuracy of the count rate, and makes instrument alignment more difficult.

7. DISCUSSION

A detailed examination of a Schultz type texture goniometer has shown that accurate specimen adjustment and the correct use of the detector and aperture slits is essential if defocusing of the diffracted beam is to be minimised and full advantage is to be gained from the use of the Schultz technique in the reflection region. Defocusing may result from the low absorption coefficient of the material being examined, from imperfect positioning and flatness of the specimen, and from the tilting of the normal to the surface of the specimen out of the plane of the incident and diffracted X-ray beam.

Once the initial mechanical adjustments have been made (as detailed by the manufacturer) it is recommended that a randomly oriented specimen be mounted with the normal to its surface lying in the plane of the incident and diffracted beam and the aperture and detector slits set to a width of about 1 mm (the actual value depends on the reflectivity of the material and the dead time of the counter). The detector slit should then be adjusted until the count rate just reaches its maximum value corresponding to the detector intercepting the full diffracted beam. The specimen should then be tilted to about 65 to 70° and the detector slit readjusted until a count rate of about 90 per cent. of the previous value is just attained. It can then be assumed that the instrument is functioning correctly. If this value cannot be attained then the mechanical adjustments must be repeated.

Particular care must be taken when using non-cubic materials. With these materials, the main reflections are relatively closely spaced; for example with BeO, the main reflections are from the $(10\bar{1}0)$, $(10\bar{1}1)$, and (0002) planes and occur at Bragg angles of 19.3, 20.6 and 22.0° respectively and care must be taken that the detector slit is not so wide as to include neighbouring reflections. This difficulty is accentuated if the material has a low absorption coefficient and hence requires the use of wide detector slits owing to the broadening of the diffracted beam. Moreover, since it is necessary to measure the background intensity before the true reflected intensity is known and since this is done by averaging the intensities measured a short angular distance away from, and on either side of, the main diffraction line, it may be necessary to reduce the width of the detector slit during this measurement at different angles of tilt, or even to measure it at zero tilt only, in order to avoid incorrect background intensity measurements due to overlap.

8. REFERENCES

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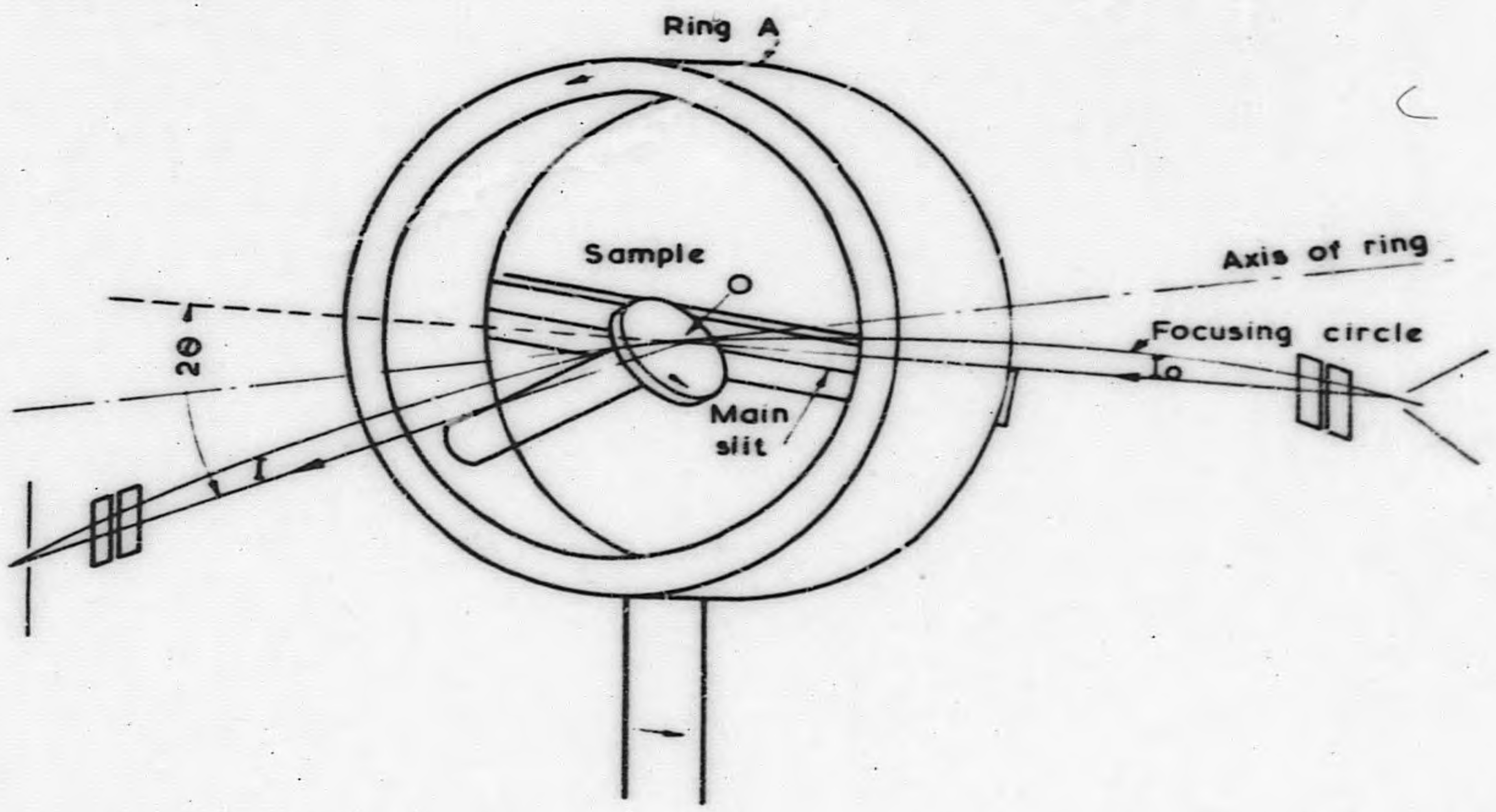
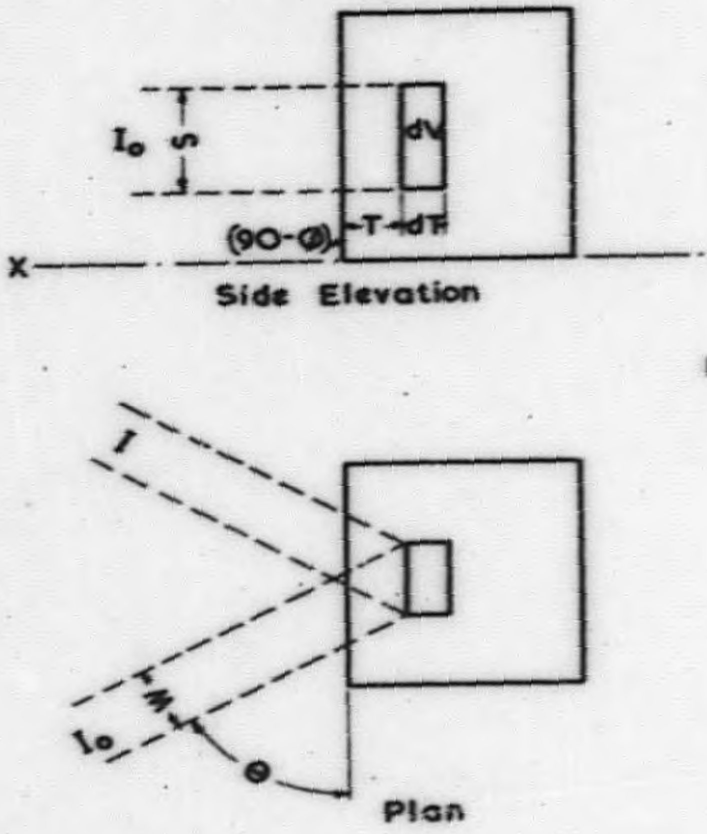


FIGURE 1.
DIAGRAMMATIC LAYOUT OF SCHULTZ'S TEXTURE GONIOMETER



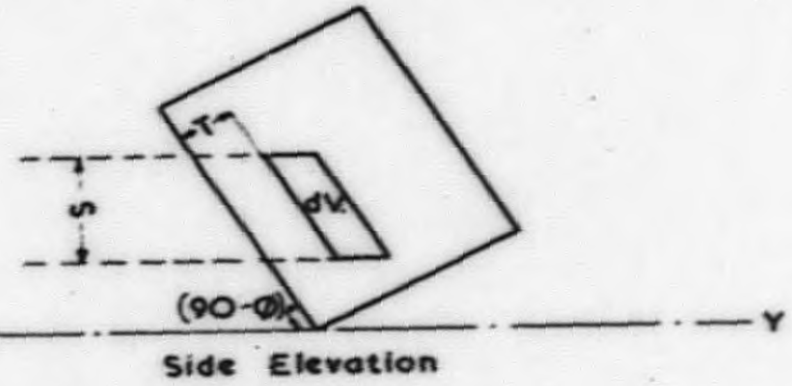


FIGURE 2B.
LOCATION OF ELEMENTAL VOLUME (dV) WHEN $\phi = \phi$ AND X-RAY PATH LENGTH EQUALS $\frac{2t}{\sin\Theta} \times \frac{1}{\cos\phi}$

FIGURE 2A.
LOCATION OF ELEMENTAL VOLUME (dV) WHEN $\phi = 0$ AND X-RAY PATH LENGTH EQUALS $\frac{2t}{\sin\Theta}$

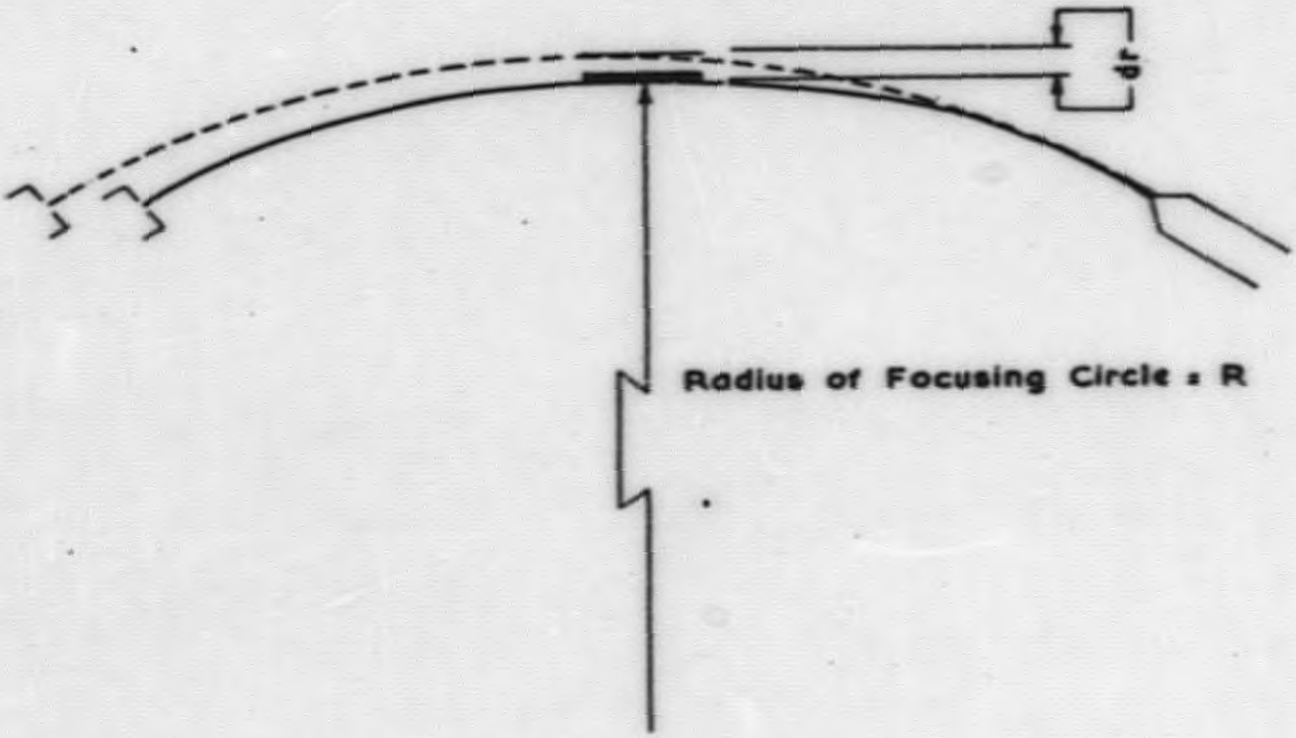


FIGURE 3.
INCREASE IN FOCUSING CIRCLE OWING TO FINITE PENETRATION (dr) OF BEAM INTO SPECIMEN.
(Radius increase from R to R + dr)

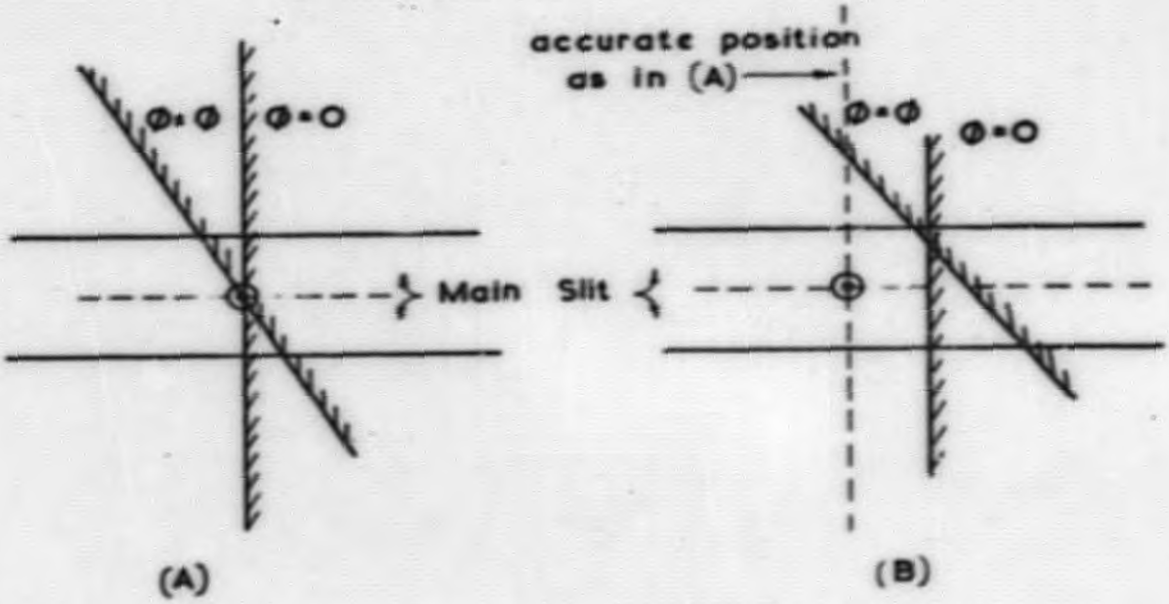


FIGURE 4.

EFFECT OF MISALIGNMENT OF SPECIMEN

(A) Accurate alignment: Centre of specimen does not change as ϕ is varied.

(B) Poor alignment: Centre of specimen moves away from accurate position owing to tilting.

Note: ⊙ is the actual axis of tilt.

R = Radius of focusing circle when $\phi = 0$

R - dR = Radius of focusing circle when $\phi = \phi_1$ from upper half of specimen.

R + dR = Radius of focusing circle when $\phi = \phi_1$ from lower half of specimen.

FIGURE 5.
DEFOCUSING AS A RESULT OF TILTING.

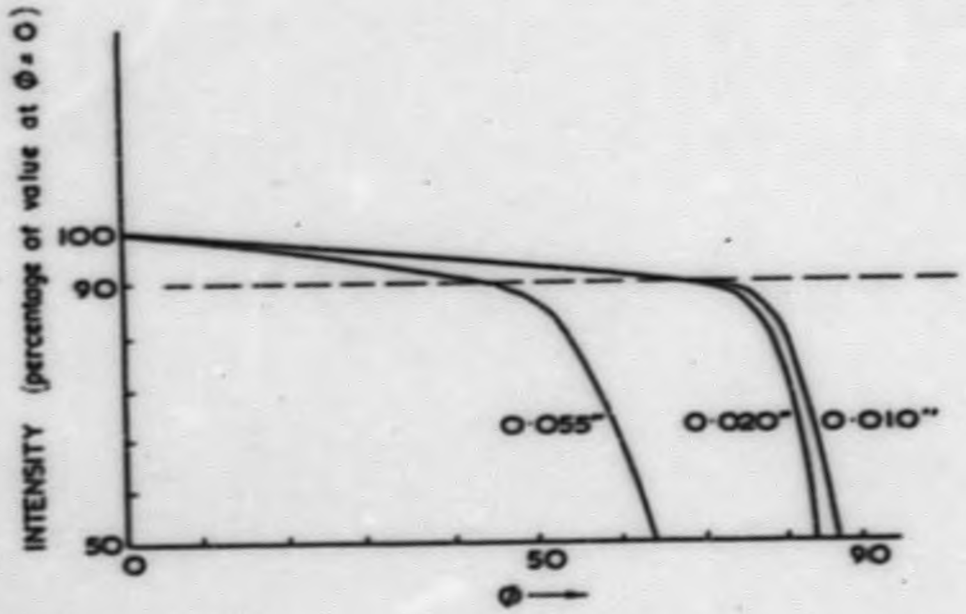


FIGURE 6.
THE EFFECT OF THE WIDTH OF THE MAIN SLIT
ON THE INTENSITY - ϕ CURVE

END