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A VERTICALLY ROTATING DOUBLE CRYSTAL
X-RAY SPECTROMETER

by

M. C. Wittels, F. A. Sherrill and A. C. Kimbrough

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A Vertically Rotating Double Crystal
X-Ray Spectrometer

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Abstract

A versatile double crystal x-ray spectrometer has been developed for the precise measurement of x-ray diffraction line-widths to tenths of seconds. The device can be employed in either the parallel or anti-parallel arrangement for rocking curve studies and can also be used in anomalous x-ray transmission experiments with nearly perfect crystals.

A detailed description of the instrument is given as well as some results concerning the Darwin theory of x-ray diffraction line-widths and Borrmann effects.

Introduction

In his measurements of absolute integrated reflections Compton¹ was the first to employ the principle of the double crystal spectrometer. Some of the optical and geometrical aspects of the instrument have been treated by Schwarzschild², Spencer³, Laue⁴, and Smith⁵, while a full theoretical account is given by Compton and Allison⁶.

* Oak Ridge National Laboratory is operated by Union Carbide Corporation for the United States Atomic Energy Commission.

The instrument described in this paper makes use of the previously established principles of the double crystal spectrometer, and, in addition, contains some features which give it further versatility.

Instrument Description

A Norelco wide range diffractometer is employed to hold the first crystal and to provide a fixed axis of rotation for setting successive orders of reflection. A worm gear on the rear axis of the diffractometer is operated either manually or synchronously for setting the first crystal in the proper position for reflection, but a detailed description of this mechanism is not important to this discussion and is omitted.

The second crystal arrangement is the crux of the instrument and allows the vertical rotation of the second crystal from -6° to $+16\frac{1}{2}^{\circ}$ about the fixed rotation axis of the first crystal. This range covers nearly all possible reflections. A schematic view of this portion of the instrument is shown in Fig. 1. It is important to note that both crystals are held on conventional small goniometers which permit quick, preliminary orientation of crystals to be achieved by Laue and Precession methods. For lightness of weight the large sections of machined metal are 24S aluminum with thicknesses reduced only to the extent that rigidity is not endangered. The base, A, contains two portable magnets which are useful during the horizontal alignment of the instrument, after which it is bolted through the table top. The face plate, B, provides several important features. It contains the bearing surface

and rotation axis of the rotating plate, C. In addition, it is graduated, as shown, in increments of 1° , to furnish the 2θ settings for the second crystal.

The rotating plate, C, contains the rocking device for the second crystal which includes the following: (1) An ω drive, D, adapted from a Weissenberg attachment, which has a gear reduction of 180:1, and which holds the second crystal on its small goniometer shaft, (2) a Boston Gear Reducer LW9, E, with a 100:1 gear reduction, and (3) a Hurst 1/6 rpm synchronous motor, F. For easy replacement the latter three parts are interconnected with the sliding couplings shown. A rocking speed of 12 sec/min for the second crystal results when these gear ratios are applied, and for most angular measurements in the second range, it is an ideal arrangement. In addition, the sliding couplings permit one to make coarse adjustments through by pass of part of the gear reduction. Between the face plate and rotating plate a wool felt pad is cemented to reduce unwanted friction.

A shutter arm, G, for opening and closing the incident x-ray port, extends through the face plate, B, and rotating plate, C, and since it lies on the pivot axis of the rotating plate, it does not interfere with its motion. This pivot axis, of course, is co-aligned with the diffractometer axis, or rotation axis of the first crystal, and can be vertically adjusted by means of the slotted holes at the bottom of the face plate.

X-rays from the tube target are vertically confined by the incident radiation slit (Fig. 2), which with the dimensions shown, gives a total vertical divergence of 110 seconds at a take-off of 3° . The slit surface was ground from a chromium-plated cold-rolled steel, and it is

therefore comparatively free of corrosion problems.

The scattering cover, H, for personnel protection from scattered radiation, also contains two horizontally adjustable rings, which limit the horizontal width of the incident beam and also permit the selective radiation of certain portions of the crystals. In a similar manner, at the detector arm there is placed a horizontally adjustable Pb slit holder, I, which not only limits the horizontal dimension of the doubly reflected beam, but also assists in proper horizontal alignment of the instrument. A clamp at the rear of this slit holder is used for locking film or nuclear emulsion cassettes into place when an optical image of the beam is desired.

Experimental Results

Halfwidth Measurements

In a recent publication⁷ measurements were reported for the halfwidths of successive orders of (111) in nearly perfect crystals of copper together with the theoretical predictions of Darwin⁸ for perfect crystals. The results of that investigation were obtained with the instrument described here, and some of these results are reproduced below. Two kinds of double reflection curves were made. One kind utilized copper crystal pairs in the parallel arrangement (1, -1), and the other involved the use of the (333) reflection from a perfect silicon crystal in the first crystal position together with (222) reflections from copper crystals in the second crystal position. Fortunately, this latter condition results in a parallel (1, -1) arrangement even closer

than cutting errors allow, namely $< 0.5^\circ$. By eliminating the broadening inherent in a slightly imperfect copper first crystal, a more accurate determination of the true half-width of the (222) for copper was thus obtained.

Table I contains the paired copper crystal results together with the values calculated from the Darwin⁸ for perfect crystals. His theoretical predictions give a range of total reflection with width

$$Y = \frac{2N \lambda^2 (e^2/mc^2) |F| K}{\sin 2\theta} \quad (1)$$

where N is the number of unit cells/cm³, λ is the x-ray wavelength, F is the structure factor, θ is the angle of reflection, K is the polarization factor (1 or $\cos 2\theta$, depending upon the polarization of the incident beam), and e , m , and c are the well-known physical constants. Y , in Darwin's expression, is conventionally termed halfwidth. We used the Fermi-Thomas-Dirac⁹ statistical model for the atomic scattering factors used in these calculations. In the paired copper measurements an x-ray beam of cross-section approximately 6mm x 0.5mm impinged on the second crystal, the radiation was $\text{MoK}\alpha_1$, and a Krypton filled Geiger tube was employed for counting purposes. The dislocation densities indicated were determined by etch-pit counts. Table I also gives the results for the measurements with the perfect silicon (333) in the first crystal position, but in this example the x-ray beam cross-section was reduced to 2mm x 0.5mm so that the number of dislocations seen by the x-rays was reduced or eliminated depending upon the dislocation density of the specific crystal examined. The absolute perfection of this sili-

con crystal was confirmed with the instrument and its halfwidth found to agree with the value reported¹⁰ earlier, namely 0.9 sec for (333) using MoK α_1 . In the calculations using the Darwin⁸ theory the polarization vector was assumed to lie in the plane of incidence of the primary beam. Temperature and absorption corrections were neglected. The calculated values for the copper pairs in the table also include a multiplying factor $\sqrt{2}$ which results from the assumption that the reflection curve for each crystal is Gaussian with full-width at half-maximum, Y, giving a measured convoluted¹¹ width $\sqrt{2}Y$ for the single crystal reflection curve. The true halfwidth (Y) was determined by use of the expression¹²

$$Y = (\beta^2 - \Phi^2)^{1/2} \quad (2)$$

where Y = true halfwidth of the second crystal,

β = measured halfwidth of the second crystal, and

Φ = true halfwidth of the first crystal.

Since the halfwidth for perfect silicon (333) is considerably smaller than those halfwidths for copper (222), the measured values are very near the true values.

Typical double crystal reflection curves are shown in Figs. 3, 4, and 5. Several independent sets of measurements revealed that a region of uniform perfection extended at least 1 cm across the (111) surfaces of Cu(A) and Cu(B). From the data (Table I) for copper pairs it was shown that reasonably good agreement was obtained with the values predicted by Darwin for perfect copper crystals. The measured values were somewhat larger than the Darwin values, roughly to the same extent as

those reported for silicon¹⁰ crystals with similar imperfection concentrations. It should be noted that Cu(B), used as a first crystal in these measurements, had $\sim 4 \times 10^3$ dislocations/cm², and probably prevented even closer agreement with the Darwin predictions. A second set of measurements, utilizing the Si(333) reflection from a perfect silicon crystal in the first crystal position and Cu(222) for a parallel (1, -1) condition, resulted in a marked narrowing in the (222) rocking curves for Cu as shown in Table I and Fig. 4. The halfwidths for Cu(A) and Cu(B) are slightly smaller than those predicted by Darwin for perfect copper crystals. It should be noted that the copper pair and silicon-copper pair measurements are not strictly comparable, because x-ray beams of different cross section were employed, and more imperfections were seen in the former case. In addition the measurements would be somewhat altered with polarization conditions different from those we assumed.

A third set of measurements with Cu(A) and Cu(B) was made with $\text{CuK}\alpha_1$ in order that a further check on the applicability of the Darwin theory would be attained. Again, the parallel (1, -1) condition was employed with Cu(A) in the first crystal position and the results for the (111) and (222) rocking curves for Cu(B) are shown in Fig. 5. Using Darwin's expression (1) for perfect crystals, we find that the predicted halfwidth for (111) and (222) in perfect copper crystals should be 31.7 sec and 15.0 sec, respectively. Our measurements, using an x-ray beam of approximately 2mm x 0.5mm cross section, gave values in agreement with the theoretical predictions within the experimental limits of observation. These findings are in concordance with the results¹⁰ on

silicon crystals which revealed that the sensitivity with which dislocations can be detected is greater for $\text{MoK}\alpha_1$ radiation than for $\text{CuK}\alpha_1$ radiation, primarily because of the depth of penetration of the shorter wavelength.

Anomalous X-Ray Transmission Measurements

The anomalous transmission of x-rays is the phenomenon in perfect or nearly perfect crystals for which the apparent absorption coefficient of x-rays becomes abnormally small when an incident beam undergoes a Bragg reflection during passage through the crystal. Borrmann¹³ first observed this phenomenon in quartz and the effect has been given his name. Following Borrmann's discovery, experiments of a similar nature were performed on Calcite¹⁴⁻¹⁶, germanium¹⁷, and silicon¹⁸.

Borrmann effects reported here were observed by two techniques. In one case crystals in the second position of the double crystal spectrometer were rocked at an angular speed of 12 sec/min through either (111) or (220) reflecting positions and the intensities of the transmitted and reflected beams were simultaneously recorded as a function of rocking angle. Two separate detectors and recorders were employed in these measurements. In the second case the two beams were recorded simultaneously on emulsions when crystals were fixed at the peak ($\bar{1}11$) or ($\bar{2}20$) reflecting positions. The incident radiation was $\text{CuK}\alpha_1$ or $\text{MoK}\alpha_1$ which was monochromated by (111) reflection from either a perfect silicon crystal or a nearly perfect copper crystal.

The results of a Borrmann experiment, with a dislocation-free silicon crystal of 1.5mm thickness, are shown in Fig. 6. $\text{CuK}\alpha_1$ radiation, monochromated by (111) reflection from a perfect silicon crystal, was directed towards a (111) face of the 1.5mm crystal, which was oriented for ($\bar{2}20$) reflection in transmission. The figure shows the simultaneously transmitted and reflected beams recorded as the crystal was rocked through the total range of reflection. It is seen that the anomalous transmitted and reflected beams are approximately equal in intensity, in agreement with previous¹⁴ findings. The somewhat enhanced background for the anomalous transmitted beam was due to the weak normal transmitted beam in the region near 0° .

The anomalous transmission of x-rays in copper¹⁹ crystals continues to be of interest and some of that earlier work is reproduced here. The possibility of observing these Borrmann effects became apparent when Lang²¹ topograph studies²⁰ were conducted. Figures 7A, 7B, and 7C are reproduced¹⁹ from the later work on a 1mm thick copper crystal. The Borrmann effect for this crystal (Fig. 6A) shows the images of the transmitted and reflected beams together with direct beam image at the bottom when the crystal and collimation was removed from the path. Gradual variation in intensity along the length of the transmitted beam image is due to a substantial change of imperfection concentration along the length of the crystal through which the transmitted beam traversed. In Fig. 7B apparent individual imperfections are revealed as light images in the magnified view of Fig. 7A. Utilizing the reflected ($\bar{1}11$) beam associated with the anomalous transmission of $\text{AgK}\alpha_1$ radiation on this 1mm thick crystal, a Lang type topograph of the entire crystal was made

and is shown in Fig. 7C. The large white area in the lower portion of the photograph is the image of a region so dense in imperfections that they were not resolved. The section of the crystal from which Fig. 7A was obtained is indicated by the short arrows in Fig. 7C, and corresponds to an area in which dislocations are resolved. From this topograph it is evident that a considerable number of imperfections were present in this 1mm thick crystal that exhibited anomalous x-ray transmission.

In Fig. 8 the anomalous transmitted and reflected beams are shown for a nearly perfect copper crystal 0.006 in. thick with a (110) face, and a ($\bar{1}11$) plane normal to this surface was utilized as the reflecting plane. $\text{CuK}\alpha_1$ radiation, monochromated by reflection from a perfect silicon (111), was employed as the incident beam, and the crystal was rocked through the ($\bar{1}11$) peak position at an angular speed of 12 sec/min. As frequently found when using longer wavelength radiation, the peak intensity of the anomalous transmitted beam is somewhat greater than that for the reflected beam. In Fig. 9 is shown a Lang topograph of another copper crystal $\sim 0.3\text{mm}$ thick with a small angle boundary running diagonally. It was apparent that the dislocation density at a short distance from the boundary was $\sim 10^3/\text{cm}^2$, and it therefore was of interest to observe the Borrmann effect at either side and across the small angle boundary. This is shown in Figs. 10-12. The crystal surface in this crystal was (111) and from a set of ($\bar{1}11$) planes at an angle of $70^\circ-32'$ from this surface, the Bragg conditions were set for simultaneous observation of the anomalous transmitted and reflected beams on one side of the small angle boundary and also across it. Fig. 10 shows the

results of such a measurement taken through the central portion of the large sub-grain, which was away from the boundary. The Borrmann effect was also seen when a small segment of the incident beam was directed across the boundary. This is indicated in Fig. 11 where the anomalous peaks are separated by ~ 17 sec. By successively translating the crystal across the monochromatic x-ray beam, without changing the crystal orientation, a series of these anomalously transmitted beams was obtained on both sides of the small angle boundary (Fig. 12).

Discussion

Information concerning lattice perfection in nearly perfect crystals can be acquired by several methods of investigation. In this study it was apparent that the double crystal spectrometer could be used advantageously as a complementary tool with Lang techniques in investigations of this nature. Since the double crystal device described here makes use of conventional small goniometers for crystal supports, by its use one can conveniently carry out the necessary precise crystal alignments with a minimum of difficulty.

The experimental results on linewidths in nearly perfect copper crystals demonstrated the significance of selecting a perfect crystal for the first crystal position while still maintaining the parallel (1, -1) arrangement to eliminate dispersion. There was some evidence indicating a finite half-width increase corresponding to a dislocation density increase in copper, but that evidence was only tentative. Correlations of the halfwidth measurements with the Darwin predictions

for perfect copper crystals were remarkably good and certainly indicate that the Darwin theory is applicable to metals as well as semiconductors and insulators. Since θ_D , the Debye characteristic temperature for Cu, is only 315°K, some thermal narrowing might have occurred even during the room temperature measurement and $|F|$ in Eq. (1) should probably be replaced by $F_0 e^{-M}$. Simple estimates show that this effect would not be large enough to produce the narrowing noted, however, and thermal studies on crystals such as these could prove interesting.

The Borrmann effect studies surprisingly revealed that this phenomenon could be observed in reasonably thick metal crystals containing considerable dislocation concentrations. It also demonstrated that the anomalous reflected beam associated with the Borrmann effect could be utilized to obtain good Lang topographs under conditions where $\mu t > 1$.

Summary

1. A versatile double crystal x-ray spectrometer has been described which is capable of measuring x-ray diffraction line widths to tenths of seconds, as well as studying Borrmann effects with counters or emulsion.
2. As an adjunct to the Lang camera, it is a valuable tool in the investigation of crystal perfection.
3. Line widths in nearly perfect crystals of copper have been measured as low as 3.0 sec with this instrument and Borrmann effects have been seen in crystals up to 1.3 mm thick.

4. It was demonstrated that both Borrmann effects and narrow line widths in the angular range of seconds could be observed in copper crystals containing considerable numbers of defects.

5. Borrmann effects were observed even across small angle boundaries in nearly perfect copper crystals.

Acknowledgements

It is a great pleasure to thank F. W. Young, Jr. for the remarkable copper single crystals which were used in this study.

For the perfect silicon crystals we are indebted to the generosity of the late W. C. Dash.

TABLE I

Halfwidths for Copper Crystals Using $\text{MoK}\alpha_1$

Crystal Position 1	Crystal Position 2	Reflection	Measured Halfwidths (sec)	Calculated Halfwidths $\times\sqrt{2}$ (Darwin) (sec)	Calculated Halfwidths, β $\beta = \sqrt{Y^2 + \phi^2}$ where Y = 3.6 sec for Cu (222) after Darwin
Cu(B) Dislocation density $\sim 4 \times 10^3/\text{cm}^2$	Cu(A) Dislocation density $\sim 2 \times 10^2/\text{cm}^2$	Cu(A) (111) (222) (333) (444) (555)	Cu(A) 14.4 7.9 5.4 4.5 3.9	 13.8 5.0 2.6 1.8 1.6	

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Si(333) Dislocation-free measured half- width = 0.9 sec	Cu(A) Dislocation density $\sim 2 \times 10^2/\text{cm}^2$	Cu(A) (222)	3.0	3.6
	Cu(B) Dislocation density $\sim 4 \times 10^3/\text{cm}^2$	Cu(B) (222)	3.2	3.6
	Cu(C) Dislocation density $\sim 10^4/\text{cm}^2$	Cu(C) (222)	5.0	3.6
	Cu(D) Dislocation density $\sim 3 \times 10^4/\text{cm}^2$	Cu(D) (222)	5.2	3.6

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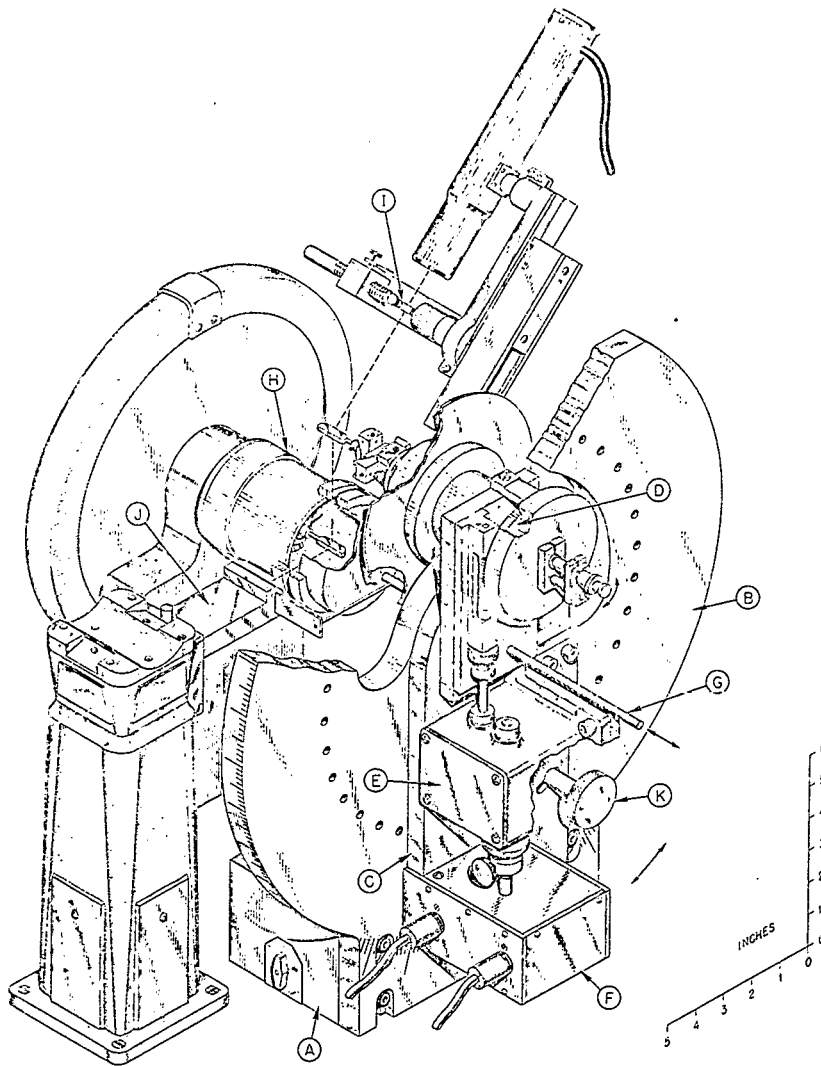
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FIGURE CAPTIONS

- Figure 1 A Vertically Rotating Double Crystal X-ray Spectrometer.
- Figure 2 Incident Radiation Slit.
- Figure 3 Reflection Curves for Cu(A) Using $\text{MoK}\alpha_1$, Which Show the Effects in Increasing Orders of Reflection. Cu(B) was used as first crystal.
- Figure 4 (222) Reflection Curves for Cu(A) and Cu(C) Using $\text{MoK}\alpha_1$ Radiation. Silicon (333) from a perfect crystal was used as first crystal.
- Figure 5 Reflection Curves for Cu(A) Using $\text{CuK}\alpha_1$, Which Show the Effects in Increasing Orders of Reflection. Cu(B) was used as first crystal.
- Figure 6 Rocking Curves. Anomalous transmitted (T) and reflected beams (R) for 1.5 mm thick silicon crystal. Crystal face (111), reflection plane ($\bar{2}20$). $\lambda = 1.54 \text{ \AA}$.
- Figure 7A 16-h Exposure for Borrmann Effect in 1 mm Thick Copper Crystal. Crystal face (111), reflecting plane ($\bar{1}11$). Upper line - anomalous reflected beam (R), middle line - anomalous transmitted beam (T), lower line - direct beam with crystal removed. Ilford L-4 nuclear plate, 100- μ emulsion. $\lambda = 1.54 \text{ \AA}$. Magnification 3X.

- Figure 7B Portion of Anomalous Transmitted Beam (T) and Direct Beam Shown in Fig. 7A. Magnification 30X.
- Figure 7C Lang Topograph of 1 mm Thick Copper Crystal. Crystal face (111), reflection plane ($\bar{1}\bar{1}1$). Positive photographs, 168-h exposure. Ilford L-4 nuclear plate, 100- μ emulsion. $\text{AgK}\alpha_1$, magnification 9X.
- Figure 8 Rocking Curves. Anomalous transmitted (T) and reflected beams (R) for 0.006 in. thick copper crystal. Crystal face (110), reflecting plane ($\bar{1}\bar{1}1$). $\lambda = 1.54 \text{ \AA}$.
- Figure 9 Lang Topograph of 0.3 mm Thick Copper Crystal. Crystal face (111), reflecting plane ($\bar{1}\bar{1}1$). Negative photograph, 24-h exposure, Ilford L-4 nuclear plate, 100- μ emulsion, $\text{AgK}\alpha_1$, magnification 20X.
- Figure 10 Rocking Curves. Anomalous transmitted (T) and reflected (R) beams for 0.3 mm thick copper crystal. Crystal face (111), reflecting plane ($\bar{1}\bar{1}1$). $\lambda = 0.71 \text{ \AA}$. Curves taken from center portion of large grain. (Fig. 9).
- Figure 11 Rocking Curves. Anomalous transmitted (T) and reflected (R) beams for 0.3 mm thick copper crystal (Fig. 9) taken across a small angle boundary. Crystal face (111), reflecting plane ($\bar{1}\bar{1}1$). $\lambda = 0.71 \text{ \AA}$.

Figure 12 Rocking Curves. Anomalous transmitted (T) beams of three different portions of 0.3 mm copper crystal (Fig. 9), showing result of a small angle boundary on Borrmann effect. Crystal face (111), reflecting plane ($\bar{1}11$). $\lambda = 0.71 \text{ \AA}$.



- A _ BASE PLATE
- B _ FACE PLATE
- C _ ROTATING PLATE
- D _ ω , SECOND CRYSTAL DRIVE
- E _ GEAR REDUCER
- F _ SYNCHRONOUS MOTOR
- G _ SHUTTER ARM
- H _ SCATTER COVER
- I _ HORIZONTAL SLIT HOLDER
- J _ VERTICAL DIVERGENCE SLIT
- K _ LOCKING SCREW

Figure 1

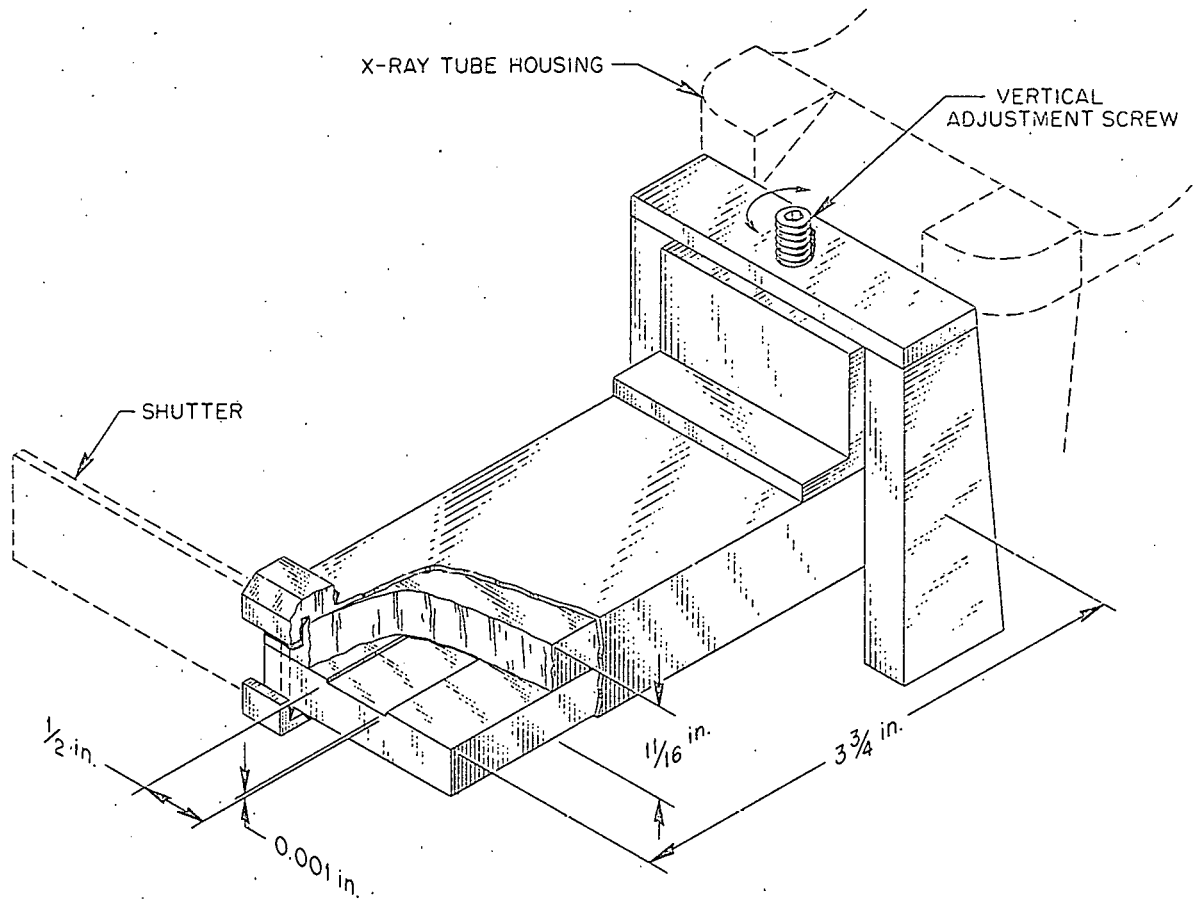


Figure 2

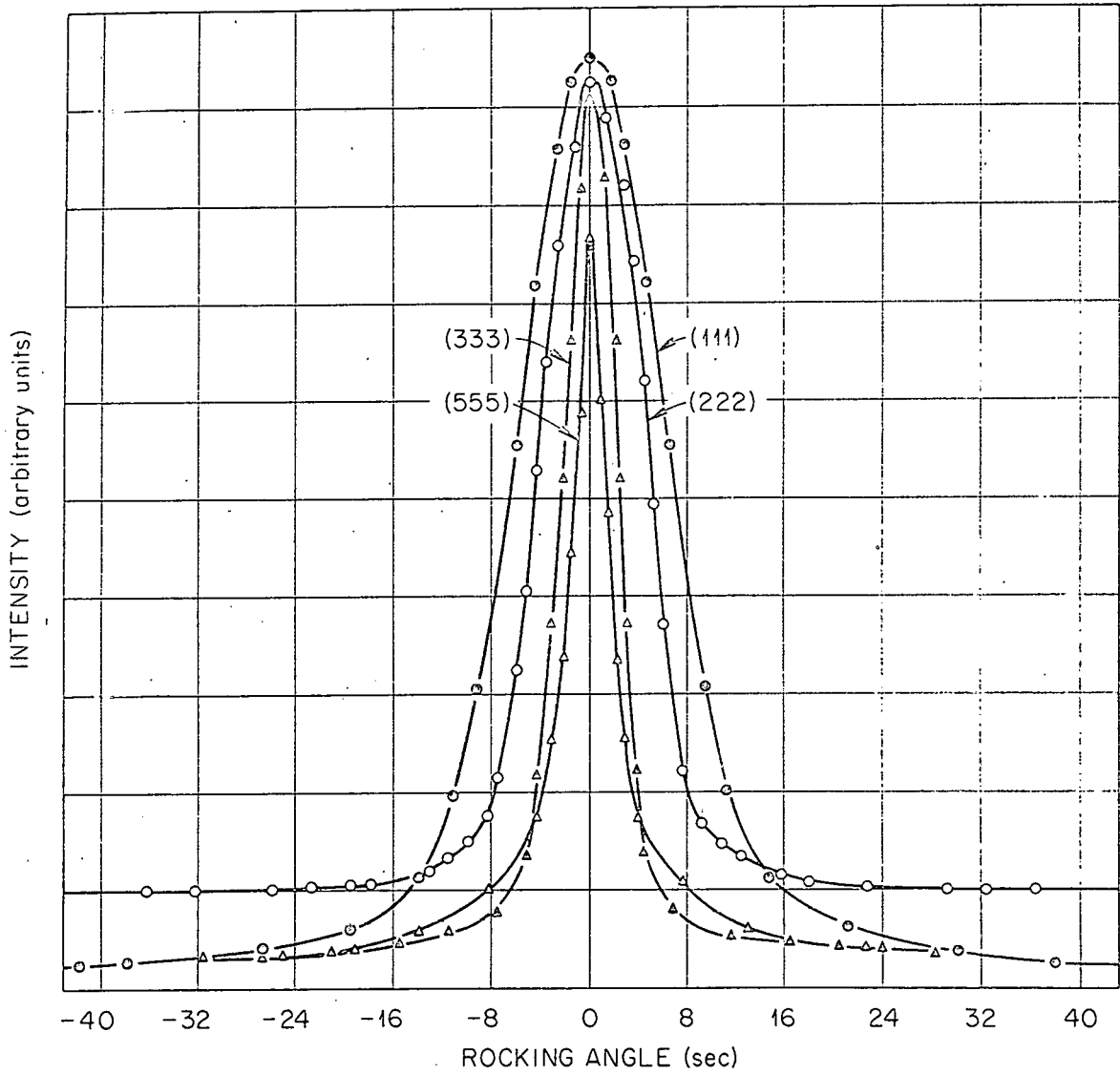


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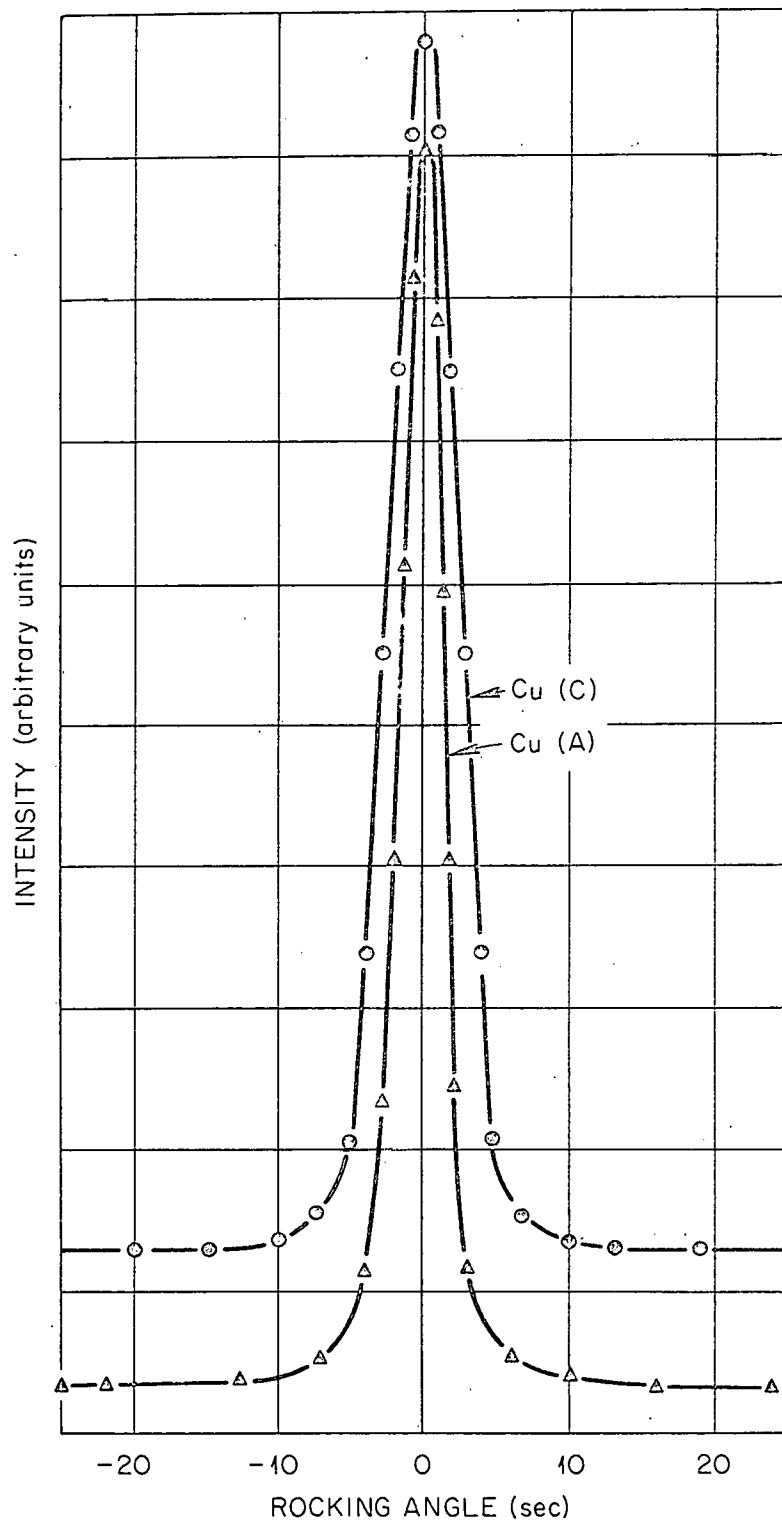


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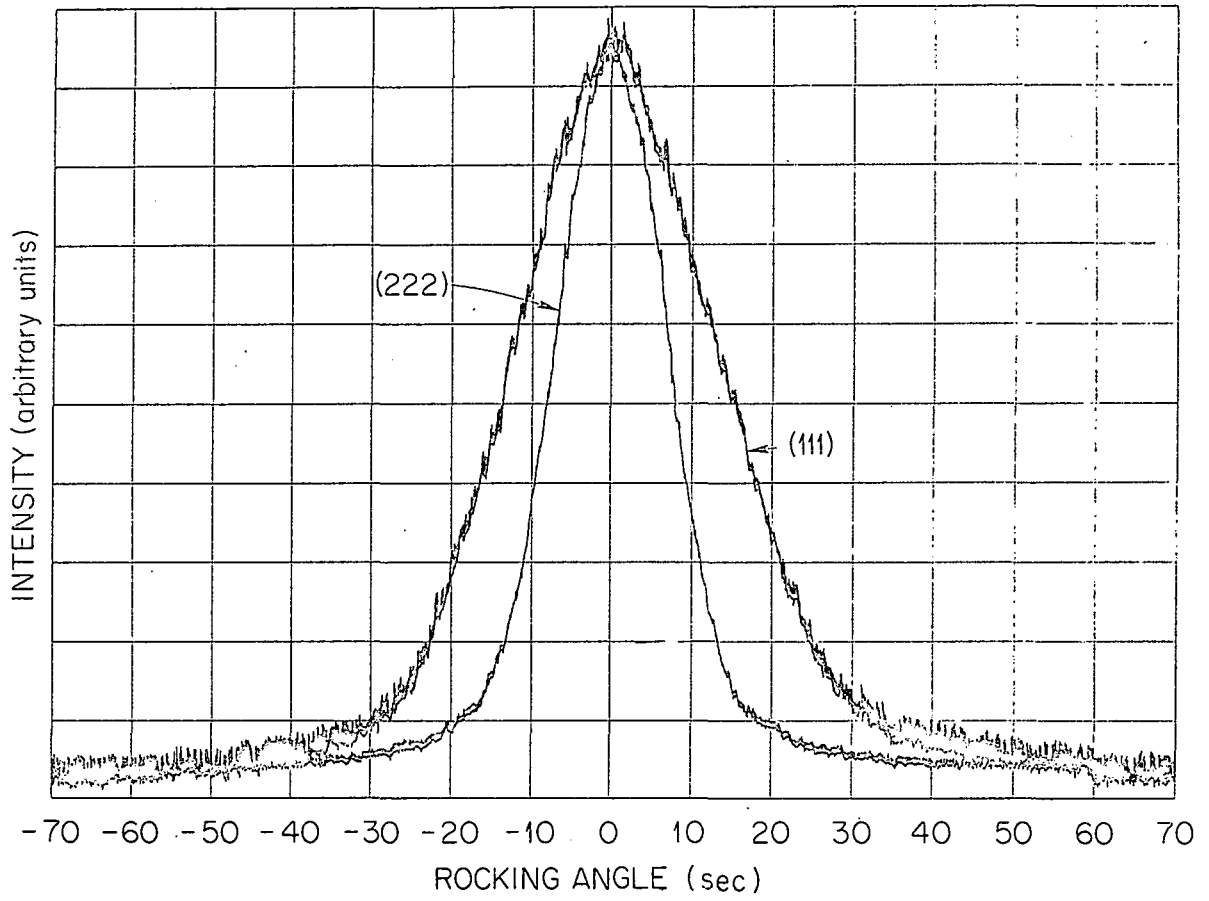


Figure 5

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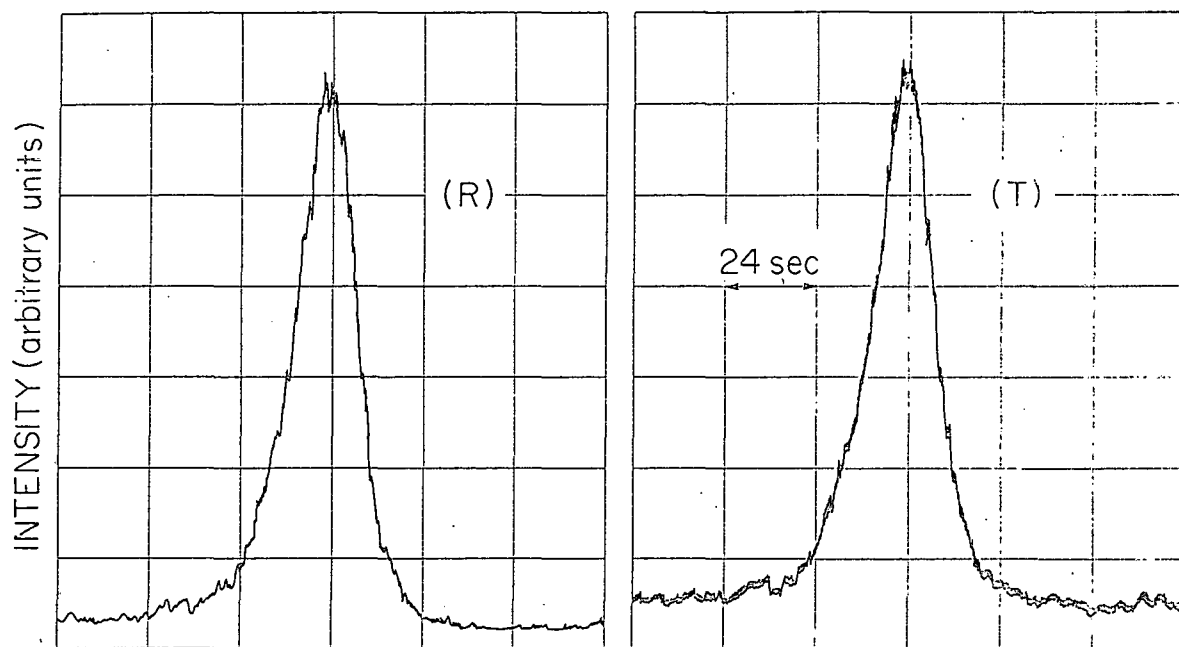


Figure 6

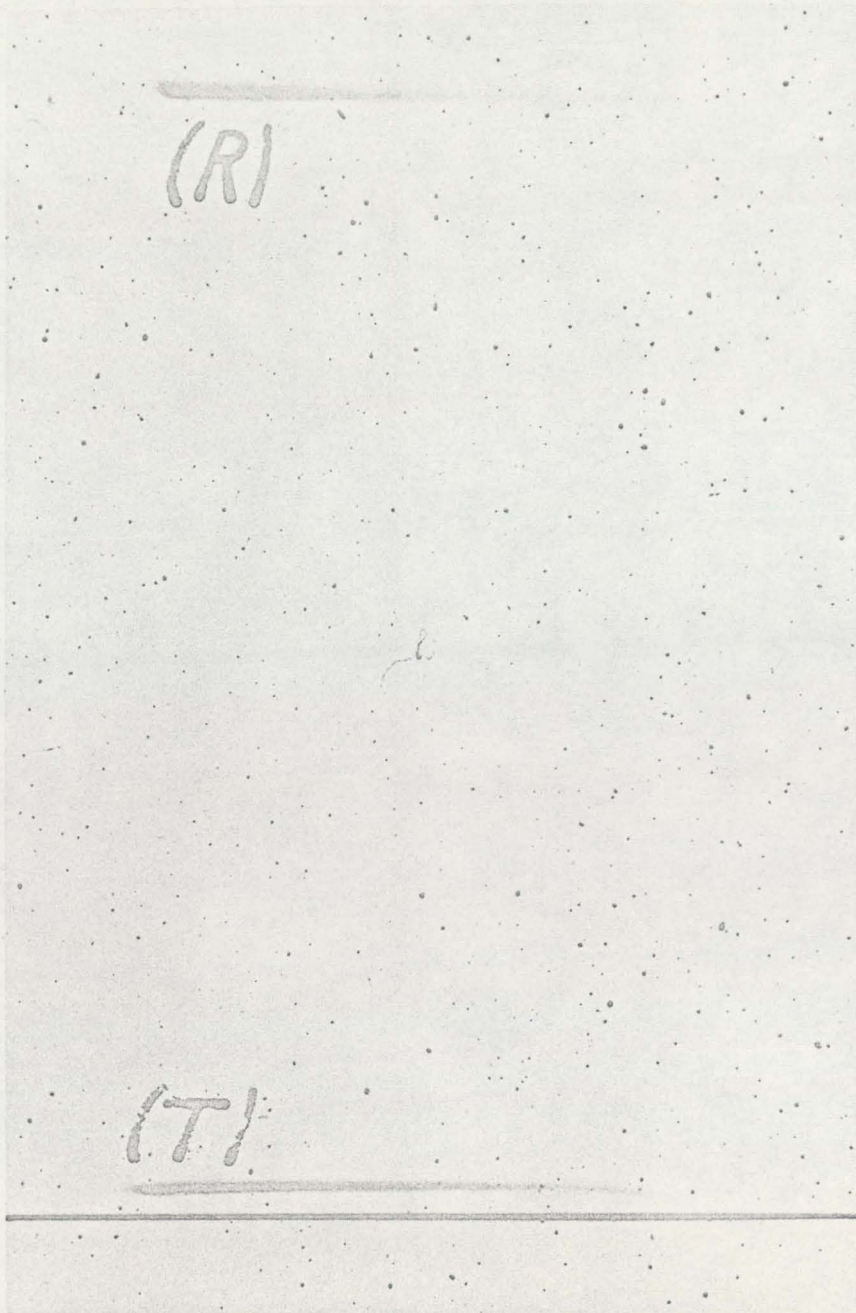


Figure 7a

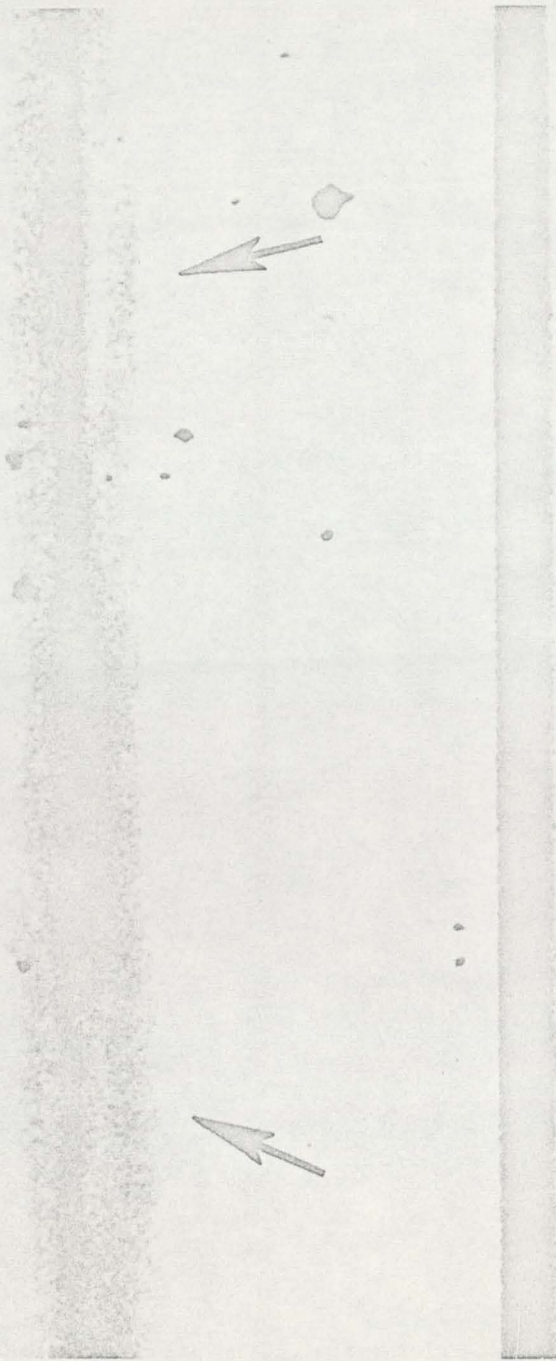


Figure 7b



Figure 7c

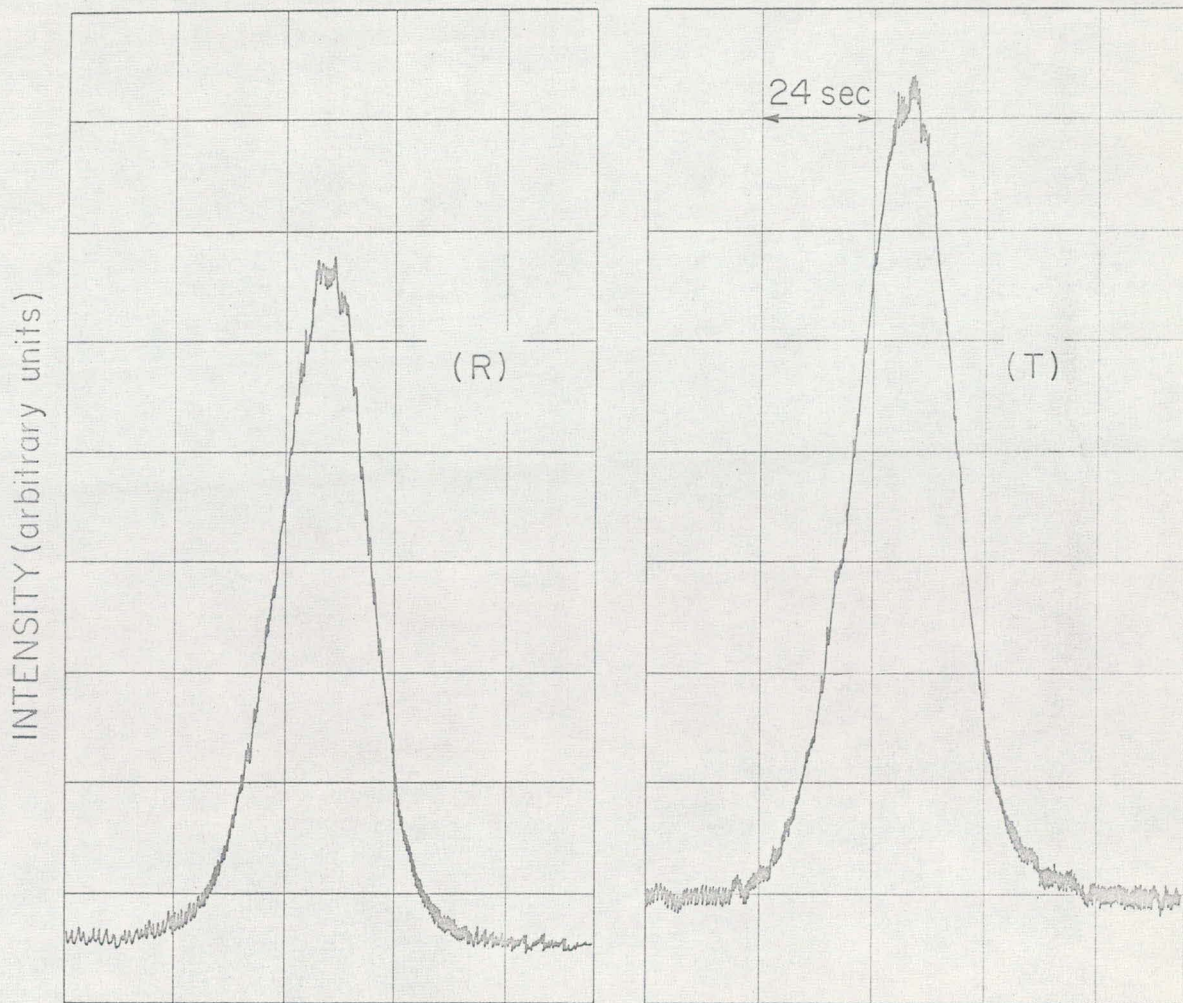


Figure 8

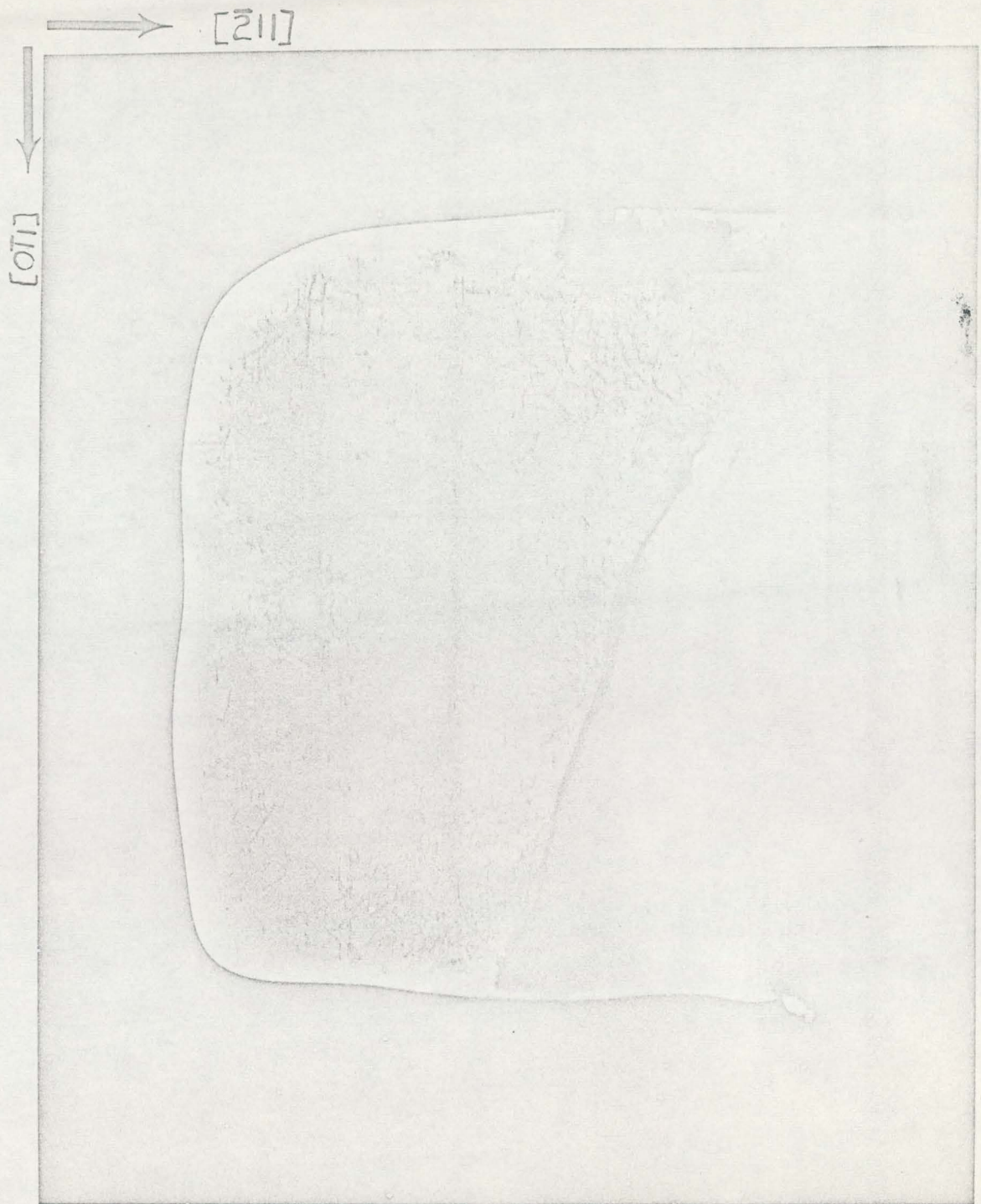


Figure 9

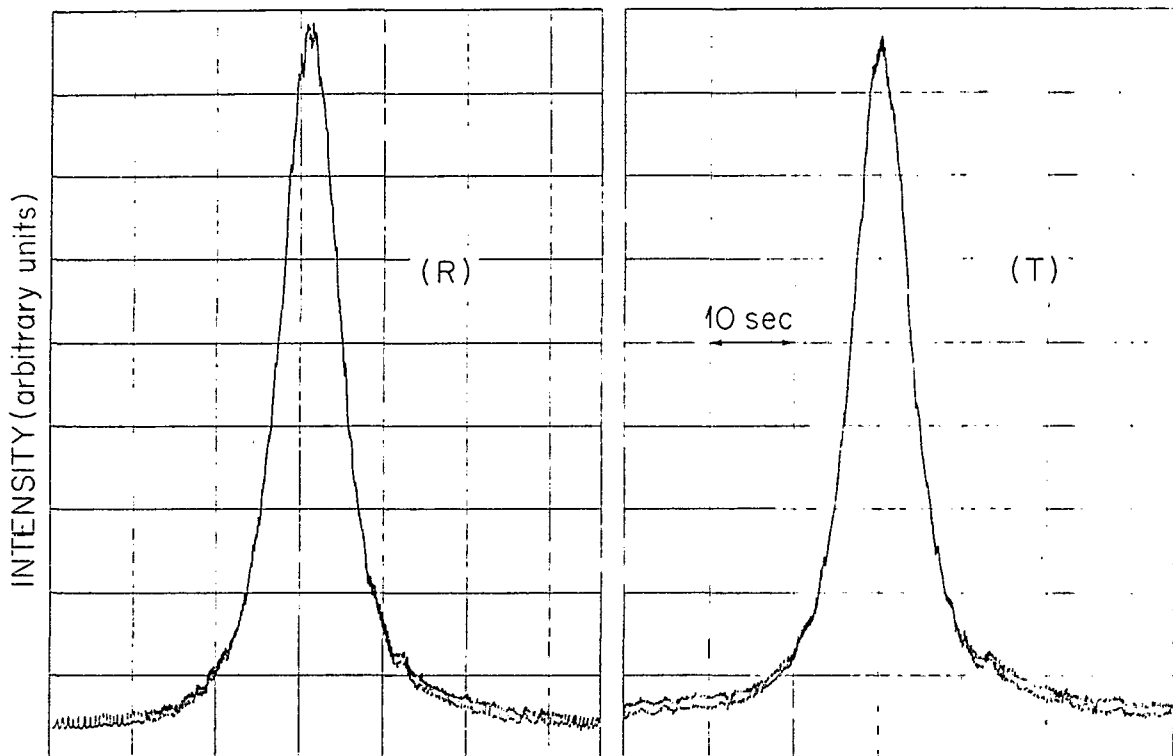


Figure 10

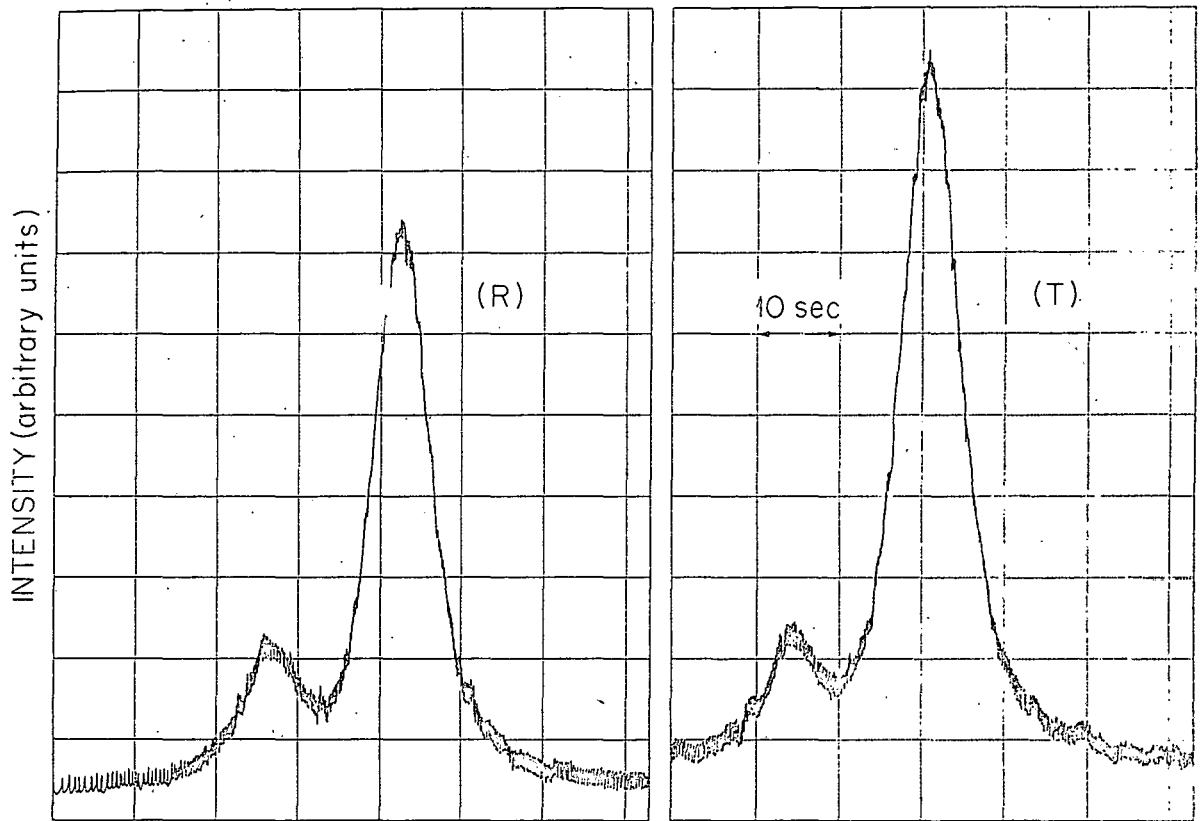


Figure 11

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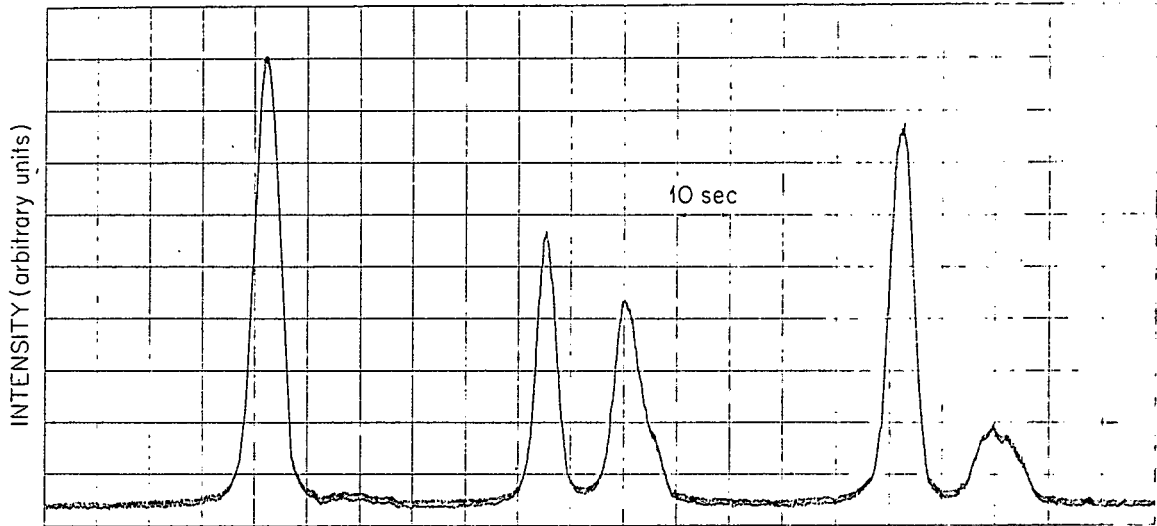


Figure 12