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FEASIBILITY OF ISOTOPIC X-RAY SOURCES FOR PORTABLE STRESS ANALYSIS EQUIPMENT

By
R. A. Semmler

April 1967

IIT Research Institute
Chicago, Illinois

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Prepared by

R. A. Semmler

for

Division of Isotopes Development
United States Atomic Energy Commission

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April, 1967

FOREWORD

This report was prepared by IIT Research Institute and is the final report under Contract AT(11-1)-578, Project Agreement No. 28 with the Division of Isotopes Development, U. S. Atomic Energy Commission. IITRI project number for this work is A6150.

Respectfully submitted,

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ABSTRACT

The feasibility of using a 10 millicurie Fe-55 x-ray source for x-ray diffraction was examined. Diffraction lines from iron were easily measured in a few minutes' time to within 0.2° using a proportional counter and rudimentary apparatus, but experiments with film were unsuccessful because of the low Mn K_α photon output (10^8 /sec). Further improvement in speed and resolution is possible for general diffraction work but the diffraction angles are poorly situated for stress work. Other source and film characteristics measured in the study include the angular dependence of the radiation, x-ray transmission of the Polaroid XR-7 cassette, Polaroid film sensitivity, and a comparison of intensifier screens. Suggested collimator arrangements to permit using a larger area source with less self-absorption are given. In conjunction with a smaller diffraction camera than used here, it is concluded that the present source would be adequate for a portable diffraction instrument of modest resolution for field work with either pulse counters or film. A film system would be particularly simple and highly portable. Film exposure times producing a weak but definite line would be of the order 10 hours.

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1. INTRODUCTION AND RESUME OF CURRENT DEVELOPMENTS

The primary purpose of this work was to evaluate the use of an Fe-55 x-ray source in conjunction with Polaroid film for portable stress analysis equipment. Estimates of response originally assumed the availability of a source emitting 100 millicuries of x-rays and indicated such a system might now be practical.

The available source, however, was not as strong as anticipated. For this, and other reasons discussed later, the available source was not adequate for this type of work. On the other hand, the source does have sufficient intensity for routine x-ray diffraction if pulse counting techniques are used. With only a modest relaxation in the resolution requirements, and with a more compact diffraction geometry, it appears that a highly portable diffraction unit can be designed using an isotopic x-ray source and a film detector. Relatively long times, say, 2-20 hours, will be required to form the film image but the unit would be extremely compact and independent of power, cooling water, or darkroom facilities if Polaroid film is used.

Because isotopic x-ray sources for diffraction seem to be on the threshold of development into practical devices, the first few sections of this report give an expanded summary of x-ray diffraction techniques and terminology. Particularly for those with a background in isotopes applications rather than x-ray diffraction, these sections will provide a quick review of x-ray diffraction analysis, terminology, and the various geometries that are used for diffraction.

These sections are followed by a discussion of the experimental work performed to establish current isotopic capabilities and suggestions and recommendations for a portable diffraction unit.

The following paragraphs give a brief review of developments in the general area of diffraction using isotopic sources to help clarify the current status of this work.

X-ray diffraction is an analytical tool which is as old as the Bragg law which dates back to 1912. The substitution of an isotopic source for an x-ray machine is an obvious extension but has been delayed by the relatively low x-ray output of radioisotope sources. The problem is apparent if one calculates the source activity required to produce the same x-ray output as an x-ray tube. A recent example of such an approach is a paper by J. Dunne¹ who considered isotopic sources for lunar analysis equipment. The calculated source is 200 curies in a volume of 10^{-3} cm^3 . This is certainly a formidable requirement. However, this approach exaggerates the difficulty since it makes no allowance for the fact that 1) isotopic sources have no glass envelope and can be placed adjacent to or in the collimation system and 2) a system intended for qualitative analysis can use less stringent collimation with a higher transmission efficiency. These considerations do not eliminate all problems but at least leave the door open to consider something less than a research quality diffraction instrument.

The next step was to actually use an isotopic source to see just what could be achieved in an actual system. After much difficulty, this was finally achieved by Preuss² using pulse detection and equipment especially designed for use with isotopes. The counting times were long, however, and amounted to nearly an hour at each angular setting. A source of 10^8 photons/sec was used and peak counting rates were of the order 10 counts/min from LiF powder.

The same source was used in the present contract with the initial intention of employing film detection. This plan was based on an erroneous value for the photon output some 30 times greater than the 10^8 value actually measured during the feasibility study. Although simple film detection could not

be used in the geometries planned, there appeared to be ample intensity for pulse detection and a very crude diffraction geometry was assembled. This immediately yielded diffraction peaks on the first experiment with an intensity more than an order of magnitude greater than the counting rates reported by Preuss. One minute counts were adequate and a complete peak could be scanned in 5-10 minutes. (Further details are contained in a later section.) This demonstration of diffraction in a few minutes time using unsophisticated equipment gives a more realistic picture of the true potential of isotopic sources.

Very recently, it was found that similar developments are in progress in Russia. A brief feasibility study was reported by Lobov and Tsukerman³ and experimental data was reported later by Lobov, Funin, and Tsukerman.⁴ Special miniature cameras with 1-2 cm radii have been constructed with only limited collimation to compensate for the low intensity. Although of poor quality, diffraction from Fe, Pt, and Ta was detected on film with exposures of 5-20 hours. The photon output is not clearly specified but the effective strength of the source is given as 3-5 mc. Portable equipment for geological survey work is also envisioned by the Russian experimenters.

The need for analytical instruments on field expeditions is apparent when one realizes that even truck mounted spectrometers are utilized. If a survey team is operating in an area without roads, however, size and weight limitations severely limit the equipment which can be used. A portable diffraction instrument with no power requirements would be most welcome.

Since the angular width of diffraction lines is also influenced by crystal characteristics such as defect density, there are other possible applications in the metals industry. X-ray line widths have been used in much the same way as hardness tests to follow the annealing process. Cold-work also produces lattice distortions which broaden the diffraction

line. These effects are generally easier to see than a shift in line position. Although cold-work such as produced by cyclic stresses produce a broadening of the diffraction line, it must be pointed out that "diffraction patterns taken in the latter stages of cyclic stressing give no clear-cut indication of the impending onset of fatigue failure."⁵ There are thus limitations on the information available but diffraction line broadening has provided much useful data on the effects of cold-work.

2. REVIEW OF DIFFRACTION SYSTEMS

The following paragraphs briefly review the most common geometries encountered in x-ray diffraction. Further details can be obtained in any textbook on x-ray diffraction.

Transmission and Back Reflection Laue Method

Laue photographs require a beam with a continuous distribution of x-ray energies and a single crystal as the target. Only those energies which satisfy the Bragg condition will be diffracted from the crystal resulting in an array of spots on the film. Pinhole collimators are normally used to produce a very narrow beam of x-rays. The geometry of transmission and back reflection Laue cameras is shown schematically in Fig. 1a and 1b.

Debye-Sherrer Method

The Debye-Sherrer technique uses monochromatic x-rays and a polycrystalline target usually in the form of a thin rod about 0.5 mm in diameter and 10 mm long. Because of the many crystals present, the diffracted beams form a cone of radiation about the initial beam axis. A segment of both forward and back reflected beams are recorded at the same time on a narrow strip of film. Slit collimators are sometimes used rather than pinhole collimators in order to reduce exposure time although the resolution is somewhat decreased because the image is formed by overlapping circles (umbrella effect). A schematic diagram of a Debye-Sherrer camera is shown in Fig. 1c. The effect of a slit collimator is shown exaggerated in Fig. 2.

Seemann-Bohlin Camera

The Seemann-Bohlin technique uses monochromatic x-rays and a polycrystalline sample. In contrast to the previous

FIGURE 1 SCHEMATIC DIAGRAMS OF GEOMETRIES USED FOR DIFFRACTION.

FIGURE 2 SCHEMATIC DIAGRAM ILLUSTRATING THE OVERLAPPING IMAGES FORMED BY ELONGATED SAMPLES. A DEBYE-SHERRER GEOMETRY IS SHOWN ALTHOUGH THE EFFECT OCCURS IN OTHER GEOMETRIES WITH A SLIT COLLIMATOR AS WELL.

methods, this camera uses a focusing technique to enhance the intensity of the diffracted beam. Only a limited range of diffracted angles can be accurately measured with one camera, however. The resolving power of this camera is twice that of a Debye-Sherrer camera of the same radius and in addition the exposure times are much less because of the large sample irradiated. Although a curved sample is necessary to exactly satisfy the focusing requirements, it is not essential. A flat specimen tangent to the focusing circle can be used with only slight loss of resolution. A schematic diagram of the proper curved sample and an acceptable flat sample is shown in Figs. 1d, 1e, and 1f.

Pinhole Camera

A pinhole camera is identical to a Laue camera except that monochromatic x-rays and polycrystalline samples are used. The camera therefore appears the same as Figs. 1a and 1b. Continuous rings rather than spots are formed on the film as in any technique using polycrystalline samples. With polycrystalline samples a divergent beam is sometimes used with a pinhole camera as shown in Fig. 1g. Only one diffraction ring on the flat film will be in focus for a given film position but the exposure time is reduced.

Diffractometer Method

Diffractometer techniques use monochromatic radiation and polycrystalline samples. The geometry is essentially the same as a Debye-Sherrer camera except that a movable GM counter is used instead of film and focusing is employed. Normally, the sample to detector distance is held constant. Since the detector does not move on the focusing circle (circle through source and tangent to sample), slits must be located at the focal points. A line source is usually used with modern diffractometers although the original diffractometer models viewed

the x-ray tube focus end-on to produce a point source. In order to use an extended line source with a focusing geometry, the divergence out of the plane must be limited. This is achieved with a Soller slit. A Soller slit contains many horizontal parallel plates which effectively limit the divergence in the vertical direction but allow the beam to spread in the horizontal plane.

Summary

Table I summarizes the characteristics of these techniques. Briefly, pinhole techniques irradiate only a small sample but have very low intensity. A slit with limited vertical dimensions can significantly increase (by, say, a factor of 20) the intensity with only slight loss of resolution due to the overlapping of images shown in Fig. 2. The intensity is also enhanced by irradiating a larger sample with a divergent beam and using focusing techniques. The length of the entrance slits can be increased if Soller slits are used to limit the vertical divergence of the beam.

Table I - SUMMARY OF DIFFRACTION GEOMETRIES

Method	Radiation	Collimation	Sample form	Sample position	Usual detector	Detector position	Comment	Focusing action
Laue	white	pinhole, well collimated	single crystal	_____	film	perpendicular to beam	_____	No
Debye-Sherrer	monoenergetic	pinhole	slender cylinder of polycrystalline or powdered material	at center of cylindrical camera	"	on circumference of cylindrical camera	_____	No
"	"	slit, limited height	"	"	"	"	intensity greater than pinhole by about a factor of 10.	No
Seemann-Bohlin	"	"	curved surface on circumference of camera	on circumference of cylindrical camera	"	on circumference of cylindrical camera	_____	Yes
"	"	"	flat surface tangent to circumference of camera	_____	"	"	focusing action slightly, but not objectionably, decreased	Yes
Pinhole	"	pinhole, well collimated	flat surface	_____	"	perpendicular to beam	_____	No
"	"	pinhole, divergent cone of radiation	"	_____	"	"	focusing action only at one angle	Yes
Diffractionmeter	"	extended slit (Soller slits used to limit vertical divergence)	flat surface tangent to focusing circle	_____	GM or other pulse counter	at constant distance from sample and on focusing circle	_____	Yes

3. COLLIMATORS

3.1 General Comments

The x-ray beam intensity is strongly influenced by the collimators and the required collimation is, in turn, influenced by the diffraction geometry as seen in the last section. If only a limited area of the sample is to be irradiated, there is not much choice but to use a well collimated pinhole system. However, this is not necessary to obtain a useable diffraction pattern and other geometries give considerably more intense x-ray beams with only a slight loss in resolution.

In particular, divergent beams in a two dimensional plane can be accommodated using a focusing geometry as in the Seemann-Bohlin camera. This geometry normally uses a relatively short slit to limit the vertical divergence. However, an extended source slit is possible if a Soller slit is used to effectively chop the source into a series of shorter slits as in a modern diffractometer. Vertical divergence in diffractometers is of the order of 2° . Horizontal divergence of the order $3^\circ - 4^\circ$ is used for diffraction in the backward direction.

The use of a converging slit collimator to permit using sources with a large area for diffraction work is also possible as indicated by the early work of Frevel.⁶ Frevel obtained diffraction lines using a focusing geometry and an x-ray beam shaped by a set of converging slits. (See Fig. 1h.) The entrance window (toward the x-ray source) was 2mm by 12mm and the exit window was 2 mm x 6 mm. The potential of this arrangement was sufficient for him to speculate that, with refinement, large sources of monoenergetic x-rays obtained by fluorescence might even be utilized.

Although Frevel could only use this technique with a transmission type geometry, which is unsuitable for stress measurements, the compact size of isotopic sources permits using such a collimator in a back diffraction geometry as shown in Fig. 1i. This reduces the source to sample distance by about a factor of 4 compared to the geometry of Fig. 1d.

3.2 Collimator Divergence Angles

The characteristics and typical examples of collimators used with x-rays are reviewed here. The simplest collimator is the pinhole system shown in Fig. 3a.

For a simple collimator with equal entrance and exit widths, the maximum divergence when the source is at the entrance slit is given by

$$\tan \frac{\alpha}{2} = \frac{d_2}{L}, \quad (1)$$

or

$$\alpha \doteq \frac{2d_2}{L}.$$

The maximum convergence is

$$\tan \frac{\beta}{2} = \frac{d_1/2}{L+x_2}, \quad (2)$$

or

$$\beta \doteq \frac{d_1}{L+x_2}.$$

For a collimator with unequal slits, the maximum divergence is

$$\tan \frac{\alpha}{2} = \frac{d_1 + d_2}{2L}, \quad (3)$$

or

$$\alpha \doteq \frac{d_1 + d_2}{L}.$$

The maximum convergence is given by either

$$\tan \frac{\beta}{2} = \frac{d_1/2}{L+x_2} \quad \text{or} \quad \tan \frac{\beta}{2} = \frac{d_2/2}{x_2}, \quad (4)$$

whichever is smaller. The second form will be smaller when

$$d_2 < \frac{d_1}{1 + (L/x_2)}.$$

FIGURE 3 a) PINHOLE COLLIMATOR WITH SOURCE AT THE ENTRANCE,
b) COLLIMATOR WITH DIVERGENCE ANGLE RATHER THAN SLIT
OPENINGS SPECIFIED.

The approximate values are

$$\beta \doteq \frac{d_1}{L + x_2} \quad \text{or} \quad \beta \doteq \frac{d_2}{x_2}$$

Some feeling for the amount of divergence which can be tolerated in a useable system can be obtained from Table II which gives values of α , β , slit widths, and distances for pinhole and slit collimators.

The horizontal divergence of typical pinhole collimators and the slit systems in Table II is of the order of 1° . These collimators were intended for Debye-Sherrer cameras, however, which do not focus the scattered x-rays. The allowable horizontal divergence of focusing cameras is greater and even when the target is flat, a horizontal divergence of 4° is used.

Vertical divergence and the vertical extension of the slit lead to a loss of resolution because of the overlapping of the diffraction cones as shown in Fig. 2. This occurs in focusing cameras as well as Debye-Scherrer cameras. The amount of acceptable divergence is $4^\circ - 7^\circ$ from the examples in Table II. Although these effects produce a spread in the measured line, it is worth noting that Garrod⁸ found the same variation (0.02%) in the measurement of interplanar spacing for both the pinhole and slit collimators. The exposure time, of course, was reduced by a factor of 20 for the slit systems.

Even with focusing, the diffraction line has a finite width which is largely dependent on the maximum separation of parallel rays through the collimator. One must keep in mind that a broad source and a divergent beam will still give sharp lines in a focusing geometry if a narrow slit is used as in Fig. 1d. In the examples from Table II the maximum separation of parallel rays in the horizontal plane varies from 0.12 to 0.75 mm. These are mostly determined by the narrowest slit but the smallest value of 0.12 mm is due to viewing the 1 mm width of the focal spot at a low angle. This is the technique used in modern diffractometers

Table II

Typical Collimator Characteristics

System	Source	S	d ₁	d ₂	x ₁	L	x ₂	α	β	Maximum separation of parallel rays	Plane of divergence	Relative intensity	Cross section at target
2 pinhole, end-on view	Alexander ⁷	1 1	0.75 0.75	0.75 0.75	60 60	40 40	50 50	1° 1°	0.4° 0.4°	0.75 0.75	Hor. Vert.	10.5	1.6 x 1.6
2 slit, end-on view	"	1 1	0.75 5.5	0.75 5.5	60 60	40 40	50 50	1° 3.9°	0.4° 0.4°	0.75 1.0	Hor. Vert.	100	1.6 x 9.2
2 slit, side view	"	0.12 8.0	0.75 5.5	0.75 5.5	60 60	40 40	50 50	0.5° 7.7°	0.05° 3.1°	0.12 5.5	Hor. Vert.	97.5	1.2 x 12.2
2 pinhole, end-on view	Garrod ⁸	1 1	0.75 0.75	0.75 0.75	(50) (50)	50 50	(50) (50)	(1°) (1°)	(0.4°) (0.4°)	0.75 0.75	Hor. Vert.	5.6	1.6 x 1.6
2 slit, end-on view	"	1 1	0.5 5.5	1.5 5.5	(50) (50)	56 56	(50) (50)	(1.4°) (3.5°)	(0.3°) (0.4°)	0.5 1.0	Hor. Vert.	100	2.6 x 8.5
2 slit, side view	"	0.12 8.0	0.5 5.5	1.5 5.5	(50) (50)	56 56	(50) (50)	(0.7°) (7.3°)	(0.04°) (2.9°)	0.12 5.5	Hor. Vert.	100	1.8 x 12

Note: All dimensions in mm.

Values in parentheses are either assumed or based on assumed values.

S = source dimension

L = collimator length

d₁ = first slit dimension

x₂ = second slit to target

d₂ = second slit dimension

α = maximum divergence

x₁ = source to first slit

β = maximum convergence

to give sharp line sources. Rather than permit vertical divergences of 7° , modern diffractometers also introduce Soller slits which chop the beam like a venetian blind and give, effectively, a stack of short slits arranged end to end. Vertical slit lengths of 10 mm can then be used.

In terms of collimator limitations, the primary factors to consider are 1) the maximum separation of parallel rays, 2) the maximum horizontal divergence which can be approximately focused even by a flat plate, and 3) the maximum vertical divergence and vertical extension which can be tolerated before umbrella effects become objectionable. From reported experience, it appears that one would want to keep these parameters within the ranges given in Table III.

Table III

APPROXIMATE RANGE OF COLLIMATOR PARAMETERS

1) Maximum separation of parallel rays in horizontal plane	0.5 - 0.75 mm
2) Minimum separation of parallel rays in vertical plane	5 mm without Soller slits, 10 mm with Soller slits.
3) Horizontal divergence	Any in focusing geometry; up to 4° in back direction with approximate focusing from flat plate; usually about 1°.
4) Vertical divergence	Up to 7°.

3.3 Transmission of Collimators for Isotopic Systems

Several geometries will be examined in this section to further illustrate the capabilities and limitations of isotopic sources. The performance in all of the examples could be improved by tilting the source to reduce the apparent size, but this technique has not been used in order to simplify the calculations. (Tilting the source will increase the self-absorption but not objectionably.) These examples are cited to illustrate the variety of instruments which can be considered and to indicate the approximate transmission of the appropriate collimation system.

Case 1, Parallel Soller Slit

In this and the following case, it is assumed that the collimation requirements can be relaxed slightly for a field instrument compared to laboratory analysis. Horizontal divergence and vertical divergence are allowed to be 2° and 10° respectively. The maximum separation of parallel rays is limited to 0.75 mm. In the present case, the limited horizontal divergence is achieved by a narrow source and the vertical divergence is controlled by a Soller Slit. In the following example horizontal divergence is controlled by a converging Soller slit and vertical divergence is controlled by limiting the vertical source dimension. Also, the present case is basically for use in a non-focusing geometry with a small sample as in a Debye-Sherrer camera.

Assuming a 1 mm x 10 mm source and a 0.75 mm slit to control the maximum separation of parallel rays, the required collimator length is

$$L = \frac{0.75 + 0.5}{0.0349} = \frac{1.25}{0.0349} = 35.8 \text{ mm}$$

If Soller slits are now added to limit the vertical divergence to 10° in each segment, the separation, s , of

the individual slots must be at most

$$s = \frac{\alpha L}{2} = \frac{(0.1745)(35.8)}{2} = 3.12 \text{ mm}.$$

This separation is readily accomplished. If 3 slats (4 channels) are used, the approximate transmission is

$$T = \frac{(0.75)(10/4)}{4\pi(35.8)^2} = 0.116 \times 10^{-3} = 1.16 \times 10^{-4}$$

Case 2, Converging Soller Slit (Frevel).

A few arbitrary assumptions are made in this case to permit examination of a specific example. In particular, the overall width of the exit side of the collimator is set at 5mm (i.e. the source will illuminate a sample about 5mm wide, the focal length is set at 6 cm, and the source area is identical to the last example (10 mm²)).

The collimator width on the source side is $5 + (5/60)L$ and the allowed height (for divergence α) is $\alpha L/2$. Therefore for a 10mm² source, one has

$$\left(5 + \frac{5L}{60}\right) \left(\frac{0.1745 L}{2}\right) = 10.$$

Solving for L gives L = 17.6 mm. The horizontal and vertical source dimensions are thus 6.47 mm and 1.54 mm. The horizontal divergence in each channel is

$$\alpha = \frac{O_s + O_B}{L},$$

where O_s and O_B are the horizontal dimensions of the slit openings on the small and large ends of the collimator respectively. Using

$$O_B = O_s + \frac{2.5}{30} \frac{L}{n},$$

where n is the number of slats and assuming slats of 1 mil thickness gives

$$n = \frac{603 + 5L}{60\alpha L + 3}.$$

Evaluating this equation for $\alpha = 0.0349$ (2°) and $L = 17.65$ mm gives

$$n = 17.3.$$

The slit opening, O_s , at the small end of the collimator is 0.27 mm which can be easily achieved. This is also the maximum horizontal separation of parallel rays.

If 18 slats are used, the approximate transmission of this collimator is

$$T = \frac{(1.54)(0.27)}{4\pi(17.65)^2} = 1.06 \times 10^{-4}.$$

This system therefore has essentially the same transmission as the last case but has several advantages: The maximum separation of parallel rays is less (0.27 mm compared to 0.75), 2) the vertical dimension is considerably less (1.64 mm compared to 10 mm) and 3) larger source areas can be accommodated by using a longer collimator; the limitation to 10mm^2 was merely to make a comparison with case 1 easier. Even spreading the same activity over, say, twice the area to reduce self absorption is practical.

Case 3, Soller Slit, Diverging Beam

If small diffraction angles can be avoided then the usual Seemann-Bohlin geometry with a thick target can be used. A diverging beam can be used but the maximum separation of parallel rays should be kept small, say, 0.5 mm. A 10° horizontal divergence is assumed here; this will illuminate a target 1 cm

wide at about 5 cm. Soller slits to limit the vertical divergence to 7° are used. For a 1mm x 10mm source, the required collimator length is

$$L = \frac{d_1 + d_2}{\alpha} = \frac{1.5}{0.1745} = 8.6 \text{ mm}.$$

In order to limit the vertical divergence to 7° , the separation s , of the individual slats in the Soller slit must be at most

$$s = \frac{\alpha L}{2} = \frac{(0.122)(8.6)}{2} = 0.52 \text{ mm}.$$

If 19 slats (20 channels) are used, the approximate transmission of the collimator is

$$T = \frac{(0.5)(10/20)}{4\pi(8.6)^2} = 2.7 \times 10^{-4}.$$

Examination of a case similar to Case 3 was the basis for the original optimism for isotopic sources. At that time it was erroneously believed that the x-ray output of the available 1 x 10mm Fe-55 source was 3.7×10^9 photon/sec (= 100 mc).. Such a source used in a system with a transmission of 3×10^{-4} as in Case 3 would contain $(1.8 \times 10^9)(3 \times 10^{-4}) = 5.4 \times 10^5$ photon/sec in the collimated beam. Allowing for diffraction losses of the order $10^{-4} - 10^{-5}$ would still leave 5 - 50 photons/sec for image formation. The required 10^5 photons for a minimal film image could then be obtained in 0.2 - 2 day. This is short enough to encourage further development. As noted earlier the actual x-ray output was only about 3mc rather than 100mc.

Case 4, Diverging Soller Slit

A broad source can also be used with a diverging Soller slit as shown in Figure 1i. To permit easier comparison with the last case which uses the same basic focusing geometry,

many of the same parameters are used here. A beam spread of 10° is again assumed; this permits a 5mm wide source at 28.6 mm from the focal point. If the focal point width is limited to 0.5 mm to achieve about the same resolution as the last case, then the maximum horizontal divergence in each individual collimator channel is approximately determined by

$$r\alpha_H = 0.5,$$

where r is the distance to the focal point. Using $r = 28.6$ mm, this gives $\alpha_H = 1^\circ$. In order to limit the vertical divergence to 7° , the allowed height of the source (with the exit slit height equal to the source height) is

$$d = \frac{\alpha_v L}{2} = 0.061L.$$

A length, L , of 32.8 mm is required to accommodate a 10 mm^2 source as before. The source dimensions are 2mm x 5mm. The number of channels to achieve the desired horizontal divergence can be determined from

$$\alpha_H = \frac{d_1 + d_2}{L},$$

where d_1 and d_2 are the slit widths at the entrance and exit of the collimator. Writing d_1 and d_2 in terms of the number of channels, n , and allowing for a 1 mill slit thickness gives $n = 25$. The slit width at the exit is therefore 0.4mm and the transmission, T , is approximately

$$T = \frac{(2)(0.4)}{4\pi(32.8)^2} = 0.0591 \times 10^{-3} = 0.59 \times 10^{-4}.$$

This is less than the last case but the sample area illuminated is also less. The maximum separation of parallel rays in this case is also less, about 0.2mm compared to 0.5mm.

One can argue for either design; the choice will depend on the diffraction angles to be observed, size limitations on the diffraction sample and other factors which can only be evaluated for a specific application. A combination collimator could also be made with diverging (or converging) plates to generate a line focus and parallel plates to control divergence in the vertical direction.

In any case, collimation systems for large sources with only slightly less than typical resolution (and certainly better than in the elementary system used in Section 11) can be achieved with transmission of about 10^{-4} . This is about one third the transmission of the system used in Section 11. If the resolution of Section 11 is adequate or nearly adequate (merely putting a slit at the focal point rather than at the detector would improve both the resolution and counting rate), then transmission factors of at least 5×10^{-4} should be possible. With no improvement in source strength over the Fe-55 used in this work (about 3 mc of photons/ 10mm^2), the beam intensity for the collimator with 10^{-4} transmission would be

$$3\text{mc} \left(\frac{3.7 \times 10^7 \text{ photon/sec}}{\text{mc}} \right) 10^{-4} = 1.11 \times 10^4 \frac{\text{photon}}{\text{sec}} .$$

The fraction diffracted into the detector is of the order $10^{-5} - 10^{-4}$ giving response rates 6 - 60 per minute.

4. INTENSITY OF DIFFRACTED X-RAYS

The intensity of a diffracted x-ray beam is considerably less than the intensity of the initial beam. Also, the diffracted intensity usually decreases as the scattering angle increases although the sample geometry must be considered and in some cases the intensity at large scattering angles begins to increase. Absolute measurements of the scattered intensity are not common, but a few measurements have been made and compared with theoretical values. Calculated and experimental values for the diffracted intensity are viewed here to indicate the order of magnitude of a diffracted x-ray beam.

The starting point for most calculations is an expression for the number of photons per second diffracted by a single small crystal with volume V situated in a beam of unpolarized x-rays. For a crystal oriented to give diffraction at a scattering angle of 2θ , the amount diffracted is given by

$$\frac{I}{\phi_0} = \left(\frac{e^2}{mc^2} \right)^2 \frac{1 + \cos^2 2\theta}{2} F_{hkl}^2 \frac{M^2 \lambda^3}{\sin 2\theta} nV = QV, \quad (5)$$

where

I = photons/sec in the diffracted cone.

ϕ_0 = photons/cm²-sec in the incident beam.

e = electron charge.

m = mass of the electron.

c = velocity of light.

$e^4/m^2c^4 = 7.9 \times 10^{-26} \text{ cm}^2$.

2θ = scattering angle ($n\lambda = 2d_{hkl} \sin \theta$).

F_{hkl} = structure factor = amplitude of wave scattered by the plane (hkl) relative to the amplitude of wave scattered by a single electron. The calculation of F includes the atomic scattering factor, f , which relates the amplitude of scattering by an atom to the scattering by a single electron.

M = number of unit cells per cubic centimeter.

λ = wavelength (cm).

n = multiplicity = number of sets of planes with different indices but the same spacing and therefore producing diffraction at the same angle.

V = volume (cm³).

Terms to allow for temperature effects and absorption are sometimes included.

For a polycrystalline sample, consideration of the fraction of crystals properly oriented to give diffraction at 2θ introduces an additional $(\cos \theta)/2$ factor. The number of photons scattered by a polycrystalline sample of volume V into the cone at 2θ is then

$$\frac{I}{\phi_0} = QV \frac{\cos \theta}{2}. \quad (6)$$

In a Debye-Sherrer camera, the fraction of this diffracted radiation striking an arc of length ℓ at a distance r from the sample is given by $\ell/2\pi r \sin 2\theta$. Altogether, the radiation reaching a line of length ℓ in a Debye-Sherrer camera with radius r is

$$\begin{aligned} \frac{I}{\phi_0} &= QV \frac{\cos \theta}{2} \frac{\ell}{2\pi r \sin 2\theta} = \frac{Q\ell V}{8\pi r \sin \theta} \\ &= \left(\frac{e^2}{mc^2} \right)^2 \frac{1 + \cos^2 2\theta}{\sin^2 \theta \cos \theta} \frac{F^2 M^2 \lambda^3 n \ell V}{32\pi r}. \end{aligned} \quad (7)$$

The function $(1 + \cos^2 2\theta) / \sin^2 \theta \cos \theta$ initially decreases as θ increases but passes through a minimum at about 50° and then increases as θ approaches 90° . The fraction of the radiation incident on the target which is diffracted in a Debye-Sherrer camera is

$$\frac{I}{\phi_0 A_T} = \frac{Q\ell}{8\pi r \sin \theta} \frac{V}{A_T}, \quad (8)$$

where A_T is the target area.

For a polycrystalline sample in the form of a thick slab, attenuation of the diffracted beam must be considered. From Fig. 4, the total path length in the sample is $x (\csc \alpha + \csc \beta)$. The attenuation is then

$$e^{-\mu x (\csc \alpha + \csc \beta)}, \quad (9)$$

where μ is the linear attenuation coefficient. For a beam with a cross section of $A \text{ cm}^2$, the volume element is $A \csc \alpha dx$. The contribution to an arc of length ℓ at distance r from the element is therefore

$$\frac{I}{\phi_0} = Q(A \csc \alpha dx) \frac{\cos \theta}{2} \frac{\ell}{2\pi r \sin 2\theta} e^{-\mu x (\csc \alpha + \csc \beta)}. \quad (10)$$

Integrating from $x = 0$ to $x = \infty$ gives

$$\frac{I}{\phi_0} = \frac{Q\ell}{8\pi r \sin \theta} \frac{A}{\mu (1 + \frac{\sin \alpha}{\sin \beta})}. \quad (11)$$

The fraction of incident photons diffracted into the arc ℓ is given by

$$\frac{I}{\phi_0 A} = \frac{Q\ell}{8\pi r \sin \theta} \frac{1}{\mu (1 + \frac{\sin \alpha}{\sin \beta})}. \quad (12)$$

Sample calculations of relative and absolute intensities as well as further details can be found in Refs/ 9-12 as well as in most standard texts. Guinier (Ref. 9, p. 126) evaluates Eq. 7 in detail for the 111 line in aluminum using Mo $K\alpha$ radiation and quotes relative intensities for other lines. In Guinier's example, the sample is assumed to be a cylinder

FIGURE 4 X-RAY BEAM PATH IN A THICK TARGET.

1 mm high and 0.6 mm in diameter in a Debye camera with a 3 cm radius. The value of Eq. 7 (with attenuation and a temperature factor included) is

$$\frac{I}{\phi_0} = 2.6 \times 10^{-6}. \quad (13)$$

Assuming the diffracted line has a full width of 0.5 mm and approximately a triangular distribution, the ratio of peak intensity, ϕ_m photon/cm²-sec, at the center of the line to the intensity of the incident beam is

$$\frac{\phi_m}{\phi_0} = 1.0 \times 10^{-4}. \quad (14)$$

If the beam cross section is the same as the sample cross section, the fraction of incident radiation diffracted into a line (of 1 cm height) is

$$\frac{I}{\phi_0 A_T} = \frac{2.6 \times 10^{-6}}{(0.06)(0.1)} = 4.3 \times 10^{-4}. \quad (15)$$

Other lines of higher order are less intense. The fraction diffracted into the (511) line, for example, is about 2.1×10^{-5} .

The fraction of radiation diffracted by a thick slab is greater. Taking the ratio of Eq. 12 to Eq. 8 gives

$$\frac{\text{Fraction diffracted by thick slab}}{\text{Fraction diffracted by Debye cylindrical sample}} = \frac{A_T}{\mu \left(1 + \frac{\sin \alpha}{\sin \beta}\right) V} \quad (16)$$

Using values from the above example $\mu = 5.2$, $A_T = 6 \times 10^{-3}$, $V = 28 \times 10^{-5}$ and assuming $\sin \alpha / \sin \beta = 1$, the ratio is 2.1. The fraction of radiation in, say, the (511) line would then be about 4.4×10^{-5} . Assuming 10^5 photons are required to form a barely detectable line on the film, the exposure time for the weaker (511) line with a beam of 10^6 photons/sec would be

$$t = \frac{10^5}{10^6 (4.4 \times 10^{-5})} = 2270 \text{ sec} = 38 \text{ min.}$$

For a thick slab, therefore, the intensity is significantly increased compared to the thin samples used in a Debye-Sherrer camera but of more importance is the fact that the beam in a focusing camera does not have to be confined to an area as small as 1 mm x 0.6 mm.

5. X-RAY ATTENUATION COEFFICIENTS

5.1 Literature

Besides information on diffraction geometries, information on x-ray attenuation is also needed. Mass absorption coefficients for x-rays are available from several sources. The most commonly used source is probably the compilation by S.J.M. Allen.¹³ Using Allen's data in the energy range 0.2 to 8 keV, Henke, et al.,¹⁴ have computed interpolation functions which permit the calculation of mass absorption coefficients for elements with atomic numbers up to 36. Recent measurements in this low energy region indicate, however, that some of the data in Allen's compilation is in error by as much as 25%. Revised interpolation functions for Al, C, O, and F are given in Ref. 15.

Some data for low energy x-rays are also given in Refs. 16 and 17, although the emphasis is on energies above 10 keV.

A limited amount of experimental data for the attenuation of x-rays from radioisotope sources has been published by Weissmantel and Wuenschmann.¹⁸ Iron-55 as well as 4 other sources with x-ray energies up to 84 keV were used.

For energies above the K absorption edge, the tables prepared by J.A. Victoreen¹⁹ are also useful. These tables have been extended below the K absorption edge and to higher Z elements in Ref. 20. This last reference is one of the most complete and gives attenuation coefficients not only for x-rays from specific elements but also for the wavelength range 0.30 Å to 2.75 Å in steps of 0.05 Å for $Z = 1$ to $Z = 83$. Other references include the tabulation of x-ray emission and absorption energies in Refs. 21, 22, and 23, and the graphical display of attenuation coefficients in Ref. 24.

The many gaps in knowledge of low energy x-ray coefficients and the need for additional work in this area is emphasized by Heinrich in Ref. 25. The recent tabulation of coefficients prepared by Heinrich²⁶ is quite extensive and is claimed to be the most reliable set of data currently available. This

tabulation gives the attenuation coefficients of all elements except hydrogen and helium for K, L, and M radiation in the range of 0.7 to 12 angstroms.

Table IV gives the absorption coefficient for Mn K_{α} x-rays in a few common compounds. The absorption coefficient in air, in particular, is large and significantly influences the use of Fe-55 x-ray sources. Only 22 cm of air is required to reduce the intensity by one-half. The half-thickness for other materials is also given in Table IV.

TABLE IV
Mn K_α ABSORPTION COEFFICIENTS AND HALF-THICKNESS VALUES
FOR AIR AND SEVERAL COMPOUNDS

Material	ρ (gm/cm ³)	μ/ρ (cm ² /gm)	$t_{1/2}$ (cm)
Air	1.29×10^{-3}	24.2	22.2
Water	1.00	25.3	0.0274
Nylon	1.11	13.6	0.0459
Polystyrene	1.07	10.4	0.0623
Hydrogen	8.99×10^{-5}	0.510	15,100.
Helium	1.77×10^{-4}	0.673	5,820.
Beryllium	1.84	3.52	0.107
Nitrogen	1.25×10^{-3}	18.59	29.8

5.2 Moisture

The effect of moisture in the air is negligible. For example, at 50% relative humidity, 70°F, and 1 atmosphere pressure, the moisture density is

$$\begin{aligned}\rho_{\text{water}} &= (\text{Relative humidity})(\text{density of saturated vapor at } 70^\circ\text{F}) \\ &= (0.5)(0.00115 \text{ lb/ft}^3) = 5.8 \times 10^{-4} \text{ lb/ft}^3.\end{aligned}\quad (17)$$

The partial pressure of this water is given by

$$\begin{aligned}P_{\text{water}} &= (\text{Relative humidity})(\text{vapor pressure of water at } 70^\circ\text{F}) \\ &= (0.5)(0.361 \text{ lb/in}^2) = 0.182 \text{ lb/in}^2.\end{aligned}\quad (18)$$

The air density is therefore

$$\begin{aligned}\rho_{\text{air}} &= \frac{P_{\text{air}} A}{RT} = \frac{(P_{\text{total}} - P_{\text{water}}) A}{RT} \\ &= \frac{(14.7 \text{ lb/in}^2 - 0.18 \text{ lb/in}^2)(144 \text{ in}^2/\text{ft}^2)(29)}{1545(530)} \\ &= 0.074 \text{ lb/ft}^3.\end{aligned}\quad (19)$$

where A is the molecular weight of air and R is the gas constant. The air density is $0.074/0.00058 = 130$ times as great as the water density. This is more than sufficient for air to dominate the total cross section even though water has a slightly greater mass absorption coefficient than air.

For hot, humid conditions, say, 90°F and 90% relative humidity, the densities are

$$\begin{aligned}\rho_{\text{water}} &= 0.00192 \text{ lb/ft}^3 \\ \rho_{\text{air}} &= 0.069 \text{ lb/ft}^3\end{aligned}$$

The moisture content is still too low to affect the cross section.

6. DIFFRACTION ANGLES

The diffraction angles, 2θ , commonly used for stress analysis in various materials are tabulated in Table V. For the Mn K_{α} x-rays from Fe-55, a lattice spacing of at least $d > (\lambda/2) = 1.05 \text{ \AA}$ is necessary for diffraction. Many of the lines commonly used are thus inaccessible.

The two largest diffraction angles which can be produced with Mn K_{α} radiation are given in Table VI using lattice formation in Ref. 27. Two additional lines are given for Ti since the last 3 lines are particularly weak. Very few lines, it may be noted, are available at the large diffraction angles which are required for accurate stress analysis. With both iron and aluminum the maximum angle is $2\theta = 128^{\circ}$ or a deviation of 52° from the backward direction.

Table V

TYPICAL DIFFRACTION ANGLES USED FOR STRESS ANALYSIS

Element	Radiation	Lattice Plane	d	2 θ	Supplement of 2 θ
Fe	Cr K $_{\alpha}$	(211)	1.1702 A	156.1°	23.9°
Al	Cu K $_{\alpha}$	(511)	0.779	162.8	17.2
Ti	Co K $_{\alpha}$	(114)	0.9175	154.3	25.7
Ag	Cr K $_{\alpha}$	(222)	1.1796	152.1	27.9
Ag	Cu K $_{\alpha}$	(511)	0.787	156.3	23.7
Pt	Co K $_{\alpha}$	(331)	0.900	167.3	12.7

Table VI

LARGEST DIFFRACTION ANGLES POSSIBLE WITH Mn K_{α} RADIATION

Element	Radiation	Lattice Plane	d	2θ	Supplement of 2θ
Fe	Mn K_{α}	(200)	1.4332 A	94.4°	85.6°
	"	(211)	1.1702	127.9	52.1
Al	"	(311)	1.221	118.9	61.1
	"	(222)	1.1690	128.2	51.8
Ag	"	(311)	1.231	117.3	62.7
	"	(222)	1.1796	126.1	53.9
Ti	"	(201)	1.233	117.0	63.0
	"	(004)	1.1708	127.8	52.2
	"	(202)	1.1220	139.2	40.8
	"	(014)	1.0653	161.5	18.5
Pt	"	(311)	1.1826	125.5	54.5
	"	(222)	1.1325	136.4	43.6

7. TRANSMISSION OF POLAROID CAMERA

A Polaroid XR-7 cassette was used for most of the experimental work. Figure 5a shows a cross section view of a Polaroid camera and bracket designed for general x-ray work. A source holder is also shown attached to the bracket in this figure. Along the path of the x-rays from the source through the camera to the target and back to the film, the beam must pass through several camera components as shown in Fig. 6. The transmission of these components for Mn K_{α} radiation at normal incidence was measured (using a GM detector) as follows:

First light shield	85%
Polaroid negative	68%
Air Pillow	85%
Second light shield	84%

The total transmission of these components is 40%. In a conventional diffraction geometry, the x-ray beam passes through the last three components twice - once on the way to the target and again returning from the target to the intensifier screen. The transmission for this complete path is 20%. This can be raised to about 35% by punching a hole in the negative and eliminating the first light shield which is not required in a sealed system. A further increase to 50% transmission can be achieved by placing the source in front of, rather than behind, the camera.

FIGURE 5a CROSS SECTIONAL VIEW OF XR-7 CASSETTE AND STANDARD BRACKET.

FIGURE 5b SAME CROSS SECTIONAL VIEW AS IN FIG. 5a WITH VERTICAL SCALE MAGNIFIED BY FACTOR OF FOUR AND SOURCE TILTED 30° .

FIGURE 5c SAME CROSS SECTIONAL VIEW AS IN FIG. 5a WITH VERTICAL SCALE MAGNIFIED BY FACTOR OF FOUR AND WITH SOURCE HOLDER NEXT TO CAMERA.

FIGURE 6 SCHEMATIC VIEW OF XR-7 CASSETTE COMPONENTS.

8. TRANSMISSION OF PINHOLE SYSTEM

With the source mounted in the rotating source holder and attached to the camera bracket as in Fig. 5, the 1 mm pinhole subtends a solid angle of $0.26 \times 10^{-2}\%$ of 4π steradians. The pinhole transmission as well as the transmission of other components in this particular geometry are tabulated in Table VII for the complete path from source to intensifier with the target at 5/8 in. from the film. The return path assumes $2\theta = 128^\circ$ (iron). Transmission through camera components is 13%. Transmission of the total air path is 77%. If one had a 100 mc x-ray source (3.7×10^9 γ /sec), the number of x-rays incident on the sample would be

$$(3.7 \times 10^9)(0.26 \times 10^{-4})(0.34) = 0.33 \times 10^5 \text{ } \gamma/\text{sec.}$$

Assuming a fraction $10^{-4} - 10^{-5}$ would be diffracted into a 1 cm length on the diffraction ring, the incidence rate at the film would be 0.9 - 0.09 / sec. The time to accumulate 10^5 photons to form a line in this conventional geometry would be 1.2 - 12 days. (1 day = 0.86×10^5 sec).

If the bracket in Fig. 5 is removed and the source holder is mounted directly onto the back of the camera as in Fig. 5c, the 1mm pinhole subtends a solid angle of $0.90 \times 10^{-2}\%$ of 4π steradians - about a factor of 3 higher than when using the bracket. Exposure times would be 0.3 - 3 day.

At the time the source was believed to have a 100mc x-ray output, several attempts were made to detect diffraction using the above pinhole geometry both with and without the bracket. These proved unsuccessful. Exposure times up to 24 hours were used with iron and aluminum samples. In light of subsequent source intensity measurements which indicated a 3 mc x-ray output, the negative response is quite understandable.

Table VII
TRANSMISSION OF ELEMENTS IN FIG. 5a

Element	Transmission
pinhole	0.26×10^{-4}
1st light shield	0.85
film	0.68
pillow	0.85
2nd light shield	0.84
air (source-to-target = 2.67 in.)	0.82
	} 0.34
2nd light shield	0.75
pillow	0.77
film	0.53
air (target-to-intensifier = 1.02 in.)	0.93
	} 0.28
Total transmission	$= 2.5 \times 10^{-6}$

9. SEEMANN-BOHLIN SYSTEM

The source holder shown in Fig. 5 was also mounted on a Seemann-Bohlin camera with a slit transmission of 0.5×10^{-4} . A top view of this camera is shown in Fig. 7. The estimated exposure time for a 100mc source was 1.5 - 15 hours. A helium atmosphere was used in the camera and an iron sample was exposed for 21 hours. Polaroid film and a piece of Patterson Hi-Speed, Series - 2 intensifying screen were cut to fit the normal film carrier in this camera. No diffraction line was evident for the same reason noted in Section 8.

10. SOURCE AND FILM CHARACTERISTICS

Following the completely negative results using both the pinhole and slit systems, it was decided to measure the source and film parameters which were assumed in the calculated estimates of response. Transmission of the x-ray beam through camera components was also remeasured using a proportional counter and detection of the Mn K_{α} peak rather than a GM counter as used originally. For this and other pulse counting measurements, the following components were used: LND #455BX xenon filled proportional counter with 2 mil Be window, Hammer #N354 preamplifier, Hammer #N300 amplifier, and Nuclear Data #130A 512 channel analyzer. High voltage (2100v) was taken from a Nuclear Chicago #192A Ultrascaler. The escape peaks present in spectra measured with a xenon filled tube are a nuisance but can be tolerated to get the high efficiency possible with xenon. Also, one must watch for the possible presence of Ni x-ray lines from the Monel body of the proportional counter. Although several sources were used to check the calibration, the most convenient was Zn-65 which gives a Cu K_{α} line at 8.05 kev and an L escape peak at 3.94 kev which nicely brackets the Mn K_{α} line at 5.90 kev.

FIGURE 7a CROSS SECTIONAL VIEW OF SEEMANN-BOHLIN CAMERA.

FIGURE 7b SAME CROSS SECTIONAL VIEW AS IN FIG. 7a WITH VERTICAL SCALE MAGNIFIED BY FACTOR OF FOUR.

10.1 Camera Attenuation

Attenuation measurements using the Mn K_α peak were repeated for most of the camera components. These values are given in Table VIII and are essentially the same as originally measured using a GM counter. These values are all for normal incidence.

Table VIII.

Mn K_α TRANSMISSION IN CAMERA COMPONENTS

Component	Transmission
2nd light shield	83.7%
emulsion	68.1%
pillow	84.4%

10.2 Source Strength and Angular Distribution

The arrangement shown in Fig. 5a with the camera removed and the proportional counter placed directly in front of the collimator was used to measure the source strength. The shutter was also removed so that the entire 1 mm x 10 mm active area of the source was viewed. Neglecting self-absorption, the detector response, R, and source strength, S, are related by

$$R = S\omega \frac{A}{4\pi r^2} \frac{e^{-\mu a} \epsilon}{2.22 \times 10^9} , \quad (20)$$

where

R = detector response

S = source disintegration rate

ω = fluorescence yield = 0.32

A = cross sectional area of collimator

r = distance from source to collimator = 1.96 in.

μ = linear absorption coefficient for air = 0.0724 in^{-1}

a = path length in air = 2.15 in.

ϵ = detection efficiency = (fraction transmitted through window) (fraction absorbed of those transmitted through window) (fraction of counts in main peak) = (0.968)

$$(1.0)(0.967) = 0.935$$

$$2.22 \times 10^9 = \text{c/m per mc.}$$

Pinhole collimators of 0.015 in. and 0.030 in. were used giving $S = 10.0 \text{ mc}$ and 11.0 mc respectively. In terms of the x-ray output, the source strength is $S\omega = (10.5 \text{ mc})(.32) = 3.4 \text{ mc}$ of x-rays. This is approximately a factor of 30 less than assumed in the estimates and readily explains the lack of response.

The angular distribution was also measured in this same geometry. Figure 8 shows the effect of tilting the source to reduce the apparent width using several different pinhole collimators. The intensity does not begin to drop significantly until the viewing angle is about 70° from the normal. Part of the reduction is caused by increased self absorption, part is caused by the fact that the active material is deposited in a depressed groove on the disc.

10.3 Film Response

Using the source holder and bracket of Fig. 5 but with the Polaroid camera turned around and shifted laterally so that the direct beam would strike the intensifier screen, a series of exposures was made to determine the minimum x-ray flux for a detectable film response. The beam intensity was first measured directly as $1.14 \times 10^5 \text{ } \gamma/\text{min}$ with the proportion counter. Again using 0.935 as the detection efficiency, the x-ray flux is $1.22 \times 10^5 \text{ /min}$. Allowing for the additional attenuation factors given in Table IX, the x-ray flux striking the intensifier screen is $0.57 \times 10^5 \text{ /min}$.

FIGURE 8 VARIATION OF BEAM INTENSITY AS THE SOURCE IS ROTATED IN THE GEOMETRY OF FIGURE 5 WITH SHUTTER REMOVED.

Table IX

ADDITIONAL ATTENUATION FACTORS IN FILM GEOMETRY
COMPARED TO COUNTER GEOMETRY

Component	Transmission
Additional 17/32" air path	96%
Front camera shield	84%
Pillow	85%
Polaroid film	68%
Total	= 47%

Exposure from 30 sec to 8 minutes indicated that 2 minutes was sufficient for a weak but definite exposure. A barely visible spot was visible in half this time. The total x-ray flux hitting the intensifier screen during a two minute exposure is 1.1×10^5 photons. The number of photons striking the film for this same exposure is 1.7×10^5 photons. The assumed figure of 10^5 photons thus appears to be quite reasonable.

10.4 Image Intensifier Screens

Since there was some doubt as to the efficacy of the image intensifier screen normally used with the Polaroid Camera at the very low x-ray energy involved here ($\text{Mn K}_{\alpha} = 5.90 \text{ kev}$), a few common screens were compared. The screens examined are listed in Table X.

The first of these is the screen normally mounted in the camera. The other screens were also placed in the camera and used for a similar series of test exposures. The Mn K_{α} flux out of the collimator was measured as $1.22 \times 10^5 \text{ } \gamma/\text{min}$. Allowing for attenuation in the air, light shield, pillow, and emulsion, the flux on the intensifier screen was about $0.57 \times 10^5 \text{ } \gamma/\text{min}$.

From this choice of screens, the preferred ones are the Fluorazure and CB-2. The other screens did not produce any obvious intensification.

Table X

COMPARISON OF INTENSIFIER SCREENS

Screen	Minimum* Exposure	Photons Hitting film
Dupont Fluorazure	2 min.	1.7×10^5
Dupont Industrial	8	6.7×10^5
Dupont CB-2	2	1.7×10^5
Patterson Hi-speed, series-2, thick type	8	6.7×10^5
No screen (camera screen covered with 1.5 mil tin foil)	8	6.7×10^5

*Gives weak but definite spot; half these values would give barely detectable spot.

A small effort was made to find better phosphors. A card covered with small buttons (powder under Scotch tape) of a variety of phosphors was placed in the camera covering the usual screen. These phosphors included the following:

1. 100% ZnS (Cu)
2. 80% ZnS, 20% CdS (Cu)
3. 40% ZnS, 60% CdS (Cu)
4. 100% CdS (Cu)
5. P-11
6. P-13
7. P-14
8. P-22
9. Violite No. 747
10. ZnS (Ag)

A hole was also punched in the card to expose the Fluorazure screen. Polaroid film was exposed in the camera both with and without x-ray exposure to insure that any response was not due to residual luminescence. In this geometry, buttons 1, 2, and 10 produced at least 4 times the intensity of the fluorazure screen, button 7 was also more intense than the fluorazure. Button 5 produced a barely detectable response, weaker than the fluorazure. The other buttons gave no detectable enhancement. A repeat run was made with a button from a fluorazure screen added to the top of the card so that it was also in direct contact with the film (except for Scotch tape). The results were the same. Although the phosphor buttons may have been thicker than the fluorazure screen, it appears that for optimum sensitivity to Fe-55, a ZnS (Ag) or ZnS (Cu) screen should be fabricated rather than the Fluorazure screen supplied with the camera. With ZnS, the overall gain in sensitivity is approximately 16 compared to the response with no screen whatever.

10.5 Interferences

In all of the time exposures with Polaroid film lasting 1 day or longer, the photographs contained what appeared to be sharp pinhole images. The origin of these spots is not certain although they are not from true pinholes. A one hour exposure with a strong light source directed at the camera produced no spots. Time exposures both with and without an x-ray source present showed similar spots although a long time exposure with the pillow raised (film is then close but not in intimate contact with the intensifier screen) showed no spots. Exposure to a uniform beta flux (10 mc Sr-90 at 12 in. for 30 sec) and a uniform gamma flux (0.2 mc Cs-137 at 1.8 in. for 2.2 hours) produced a gradual fogging with no enhancement of the pinhole-like structure.

11. PULSE DETECTION OF DIFFRACTED RADIATION

Although the strength of the source examined is inadequate for use with film in the geometries attempted, it is ample for pulse counting techniques. It was found that diffracted x-rays can be detected with the most rudimentary arrangement in a few minutes time. The simple setup shown in Figure 9, consisting simply of a source and protractor on a rotating turntable, was assembled in about an hour from miscellaneous hardware and immediately produced diffraction peaks from iron both in transmission and reflection. The exit slit was 0.5 x 7.0 mm and was located 2.89 cm from the source. This gives a maximum horizontal divergence of 3° . The distance from exit slit to target was about 3.6 cm and from target to counter was about 5.0 cm. No effort was made to put slits at optimum positions (focal points) or to reduce (or subtract) the background in any way. Soller slits were not used. The resolution could, of course, be improved by proper placement of slits and with better collimation. Figure 10 shows diffraction peaks obtained with one minute counts at each angular setting using a 0.005 in. iron foil as a target. The angular separation of the (110) line and (211) line is 65.5° , as expected.

Figure 11 is the (211) line from a sample of stress free and a sample of machined iron. Despite the rather poor resolution of this rudimentary equipment, it appears that the expected line broadening effect of the surface work has perceptably reduced the peak height.

It should be noted that the source used in this work is the identically same source used in the diffraction work reported in Ref. 2. The resolution found here is essentially the same but the count rates here are roughly a factor of 15-20 higher. It is felt that almost any change would further improve both resolution and intensity.

FIGURE 9 ISOMETRIC VIEW OF SOURCE, SAMPLE, AND DETECTOR FOR PULSE DETECTION OF DIFFRACTED RADIATION.

FIGURE 10 DIFFRACTION PEAKS FROM IRON FOIL USING 1 MINUTE COUNTING PERIODS AT EACH ANGULAR SETTING.

FIGURE 11 DIFFRACTION PEAKS FROM ANNEALED AND MACHINED IRON.

12. MACHINE X-RAY INTENSITY

Although the weak source strength precluded the originally planned comparison of machine-produced and isotope produced radiation, the same basic information has been gathered in another way. The same proportional counter detector used to measure the isotopic source strength was also used to measure the K_{α} flux in an x-ray beam used to give a detectable diffraction pattern.

In order to make the machine radiation as close as practicable to the Fe-55 source, an x-ray tube with a chromium target and vanadium filter was used. There was considerable difficulty reaching a compromise between a beam intensity which was measurable and yet would give a diffraction pattern in a reasonable time. There was also difficulty with fogging to the Polaroid film when using the machine radiation. Low voltage and heavy filtering were generally used to minimize fogging which is apparently caused by the general background of white radiation from the machine.

With the x-ray spectrum shown in Figure 12, a barely detectable diffraction ring was obtained from an iron sample at 3 cm from the film using the conditions given in Table XI.

FIGURE 12 X-RAY SPECTRUM USING CONDITIONS OF TABLE XI BUT WITH 0.005 IN. COLLIMATOR.

TABLE XI

MACHINE CONDITIONS FOR
MINIMUM DETECTABLE DIFFRACTION PATTERN FROM IRON

Voltage	20,000 V
Current	10ma
Collimator	1mm(0.040 in.)
Tube-to-collimator	13.6 cm
Film-to-target	3 cm
Filter	Fe, V
Exposure time	7.0 hr

The Fe filter was used to reduce the continuous radiation above the Cr line. This was necessary for spectrum measurements. The beam intensity was measured in this same geometry with the camera removed and the proportional counter next to the final collimator except the final collimator was reduced from 0.040 in. to 0.005 in. for the counter measurements. The measured spectrum for the conditions of Table XI (except with the smaller collimator) is given in Figure 12. The total counts in the Cr K_{α} peak of this 1 minute spectrum are 8.4×10^4 . Allowing for a detection efficiency of 93%, the photon intensity is 9.1×10^4 photon/min. Allowing for the change from a 5 mil collimator to a 40 mil collimator for the film exposure gives an intensity of 5.81×10^6 photon/min. To achieve a typical 1 hour exposure would require 7 times this intensity or 0.41×10^6 photon/second. Considering that this exposure produced a barely detectable diffraction ring, the beam intensity estimate given by Boeing of 10^6 /second appears reasonable.

Based on the above measured x-ray intensity, with corrections for the geometrical efficiency and for absorption in vanadium,

iron, and air, the approximate value for the tube photon emission is $0.3 \times 10^{12}/\text{sec}$.

The Polaroid camera of course, strongly attenuates the chromium radiation as will any film holder. The measured transmission of the camera components was 20% for manganese radiation. The transmission for chromium radiation is therefore approximately $(0.20)^{1.3} = 0.12$ where 1.3 is the ratio of the chromium x-ray absorption coefficient to the manganese coefficient. Elimination of some of the camera components as suggested earlier would raise the transmission to about 0.40.

Allowing for the difference in camera attenuation, the beam intensity which would be required to obtain the same pattern in 7 hours using Mn K_{α} radiation is approximately $(0.12/0.20) (5.8 \times 10^6/\text{min}) = 3.5 \times 10^6/\text{min}$. It might be noted that the present Fe-55 source can produce a collimated beam of about $1.2 \times 10^5/\text{min}$ and that if the source had been 30 times stronger as expected then the diffraction pattern using an isotopic source would have been observable on film as estimated.

13. SOURCE IMPROVEMENTS

The source could be improved in several ways: 1) deposit the activity on a greater area to reduce self-absorption 2) reduce the amount of inert iron in the source to reduce self-absorption 3) increase the total activity of the source. Since there is roughly 7 times as much inactive as active iron in the present source, the specific activity can be improved considerably. Also, if 100 mc of Fe-55 was actually deposited initially (about 1 year ago) and it appears now as only 10 mc from the external radiation, much of the radiation is being lost in self-absorption. Since self-absorption in this source is large, merely depositing the same activity over a larger area will significantly increase the source strength. A gain of at least 2 should be possible. Between these two factors and a nominal increase in the total amount of material deposited, the actual x-ray output can be increased by about an order of magnitude. This source would be something like 200 mc of Fe-55 deposited on a 5mm x 20mm area producing an x-ray output of 30 mc or 10^9 photons/sec.

With collimator transmissions of 10^{-4} , the beam intensity would be 10^5 photon/sec. and the diffracted intensity would be about 1-10/sec. This is sufficient to consider film in some cases since 2×10^5 photons can be accumulated in about 6 hours with a flux of 10/sec. Overnight exposures should thus give some detectable lines on Polaroid film. A 90° scan with pulse counting equipment could probably be made in half an hour.

14. POTENTIAL DEVELOPMENTS

The important question is whether isotopic sources are now suitable for practical diffraction work. Obviously, a qualified but affirmative answer must be given in light of the work reported in Section 11 even though the available diffraction angles using Fe-55 are not suited for stress analysis. Presently existing sources even in a very poor geometry have sufficient intensity to scan a diffraction peak and determine its position within $0.1^\circ - 0.2^\circ$ in a few minutes time with pulse counting equipment.

The essential parameters in a discussion of an isotopic diffraction system are summarized below:

SOURCE: Present source produces 10^8 photon/sec from 10mm^2 ; possible new source produces 10^9 photon/sec from 100mm^2 .

COLLIMATION: Several systems possible with a transmission of 10^{-4} and a divergence only slightly worse than laboratory systems.

FRACTION DIFFRACTED: Varies with compound but nominal value is $10^{-4} - 10^{-5}$; for a "miniature" system, nominal value is $10^{-3} - 10^{-4}$.

FILM SENSITIVITY: Weak but distinct line requires 2×10^5 photons using film with Fluorazure intensifying screen. ZnS screen would require 0.5×10^5 photon.

If it is assumed that air paths are short and a film camera is designed to eliminate absorption in light shields, etc., the "worst case" would use the present source in a nominal geometry with a Fluorazure intensifying screen. The film dose rate is approximately

$$(10^8)(10^{-4})(10^{-4}) = 1 \text{ photon/second.}$$

The time to accumulate 2×10^5 photons for a weak line is 2×10^5 sec or about 2 days. By using a miniature system this would be reduced to 0.2 days or 5 hours. With an improved intensifying screen the time is reduced to 1-2 hours. This exposure is with the present source. Any improvement in source strength will reduce the time further and if a full order of magnitude improvement is achieved, the time is down to 0.1 - 0.2 hours or about 10 minutes. Even allowing for additional exposure time to intensify the lines still gives quite reasonable exposure times.

The basic conclusion is that a truly portable diffraction system can now be developed for use with film using the presently available sources.

An example of how one might arrange the source, sample, and film to achieve a specified range of diffraction angles is shown in Fig. 13. This is only one of the several geometries which are suitable for geological samples.

FIGURE 13 SCHEMATIC DIAGRAM OF A DIFFRACTION GEOMETRY FOR GEOLOGICAL SAMPLES.

15. SUMMARY

The initial purpose of this investigation was to examine the feasibility of using an available Fe-55 x-ray source with Polaroid film for stress analysis under field conditions. This application did not prove practical because of the low intensity of the present source (1.2×10^8 photon/sec) and the poorly situated diffraction angles (more than 50° from the back direction). With pulse counting equipment, however, diffraction lines from iron were determined to within 0.1 - 0.2° in a few minutes time with elementary equipment susceptible to considerable improvement.

Although not critical to future development work, significant improvement in the x-ray output of the source is possible by using a larger source area and a higher specific activity. Both of these factors reduce the self absorption and coupled with approximately a twofold increase in the total deposited activity should yield about a tenfold increase in x-ray output or 10^9 photons/sec.

A comparison of intensifier screens for Mn K_α radiation showed the DuPont CB-2 to be essentially equivalent to the DuPont Fluorazure screen used in the Polaroid XR-7 cassette. Neither produced the enhancement of ZnS (Cu or Ag) powder, however. These powders produced about four times the intensification of either the Fluorazure or CB-2 screen.

The overall transmission of Polaroid XR-7 camera components Mn K_α radiation has been measured as 20% when the camera is used in the normal back reflection modes. Transmission of the individual components is given.

Collimation systems have been examined and several which are capable of accepting a broad source seem better suited for use with isotopic sources than trying to imitate the narrow line source present in an x-ray tube. Collimator transmissions are generally of the order 1×10^{-4} . Coupled with a source of 10^8 photon/sec, this will yield diffracted intensities of 1-10 photon/sec or about $1 \cdot 10 \times 10^5$ photon/day in a miniature camera.

The minimum dose to form a weak but definite dot (approximately 1.2mm diameter) on Polaroid film with a Fluorazure intensifying screen was measured as 1.7×10^5 Mn K_{α} photons. A barely visible spot would require half this value. With a further gain of 4 using a screen of ZnS powder, a weak but definite spot would require about 0.4×10^5 Mn K_{α} photons. This dose can be achieved in 12 hours or less with a miniature camera or with the stronger source mentioned above.

At present the available Fe-55 source is practical in a conventional geometry only if used with pulse counting equipment. By using a miniature camera (or a stronger source), relaxing the resolution requirements slightly compared to laboratory equipment (but still determine peak positions to 0.1°), and improving the intensifier screen, film detection as well as pulse detection appears practical.

The development of a portable x-ray diffraction camera with no power requirements whatever is recommended.

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