

### 3-6. FILM LOADING

Two holes are punched along the center line of the film to receive the beam collimating and beam receiving tubes. The film is 1-3/8 inches (35 mm) in width and by using the film punch and cutter, is cut to length and punched to fit either the large or small camera. The Powder Camera, with previously aligned specimen inside, is taken into the darkroom and the beam collimating and beam tubes are removed.

#### CAUTION

Use extreme care in handling. Improper film patterns will result if the beam collimating and beam receiving tubes are damaged or exposed to dirt.

The film is gently lowered into the camera along the inner circumference, Fig. 3-1, so that the holes in the film coincide with the mounts into which the beam collimating and beam receiving tubes are inserted. One end of the film is placed so that it presses against the fixed pin of the film expanding device; and the other end of the film is placed beneath the movable pin of the film expanding device. The external adjusting screw, of the film expanding device is then gently pulled tight and screwed down. This insures that the film will be held in close contact with the interior of the camera body. Next, the beam collimating and beam receiving tubes are carefully inserted into the camera body, and the camera cover is replaced.

The camera can now be taken from the darkroom and placed onto the camera track assembly. Tighten the camera base knurled screw, and proceed with the X-ray exposure. The specimen holder, can be rotated by means of a belt, which is placed over the specimen rotating pulley, and the drive motor pulley, of the camera track. For operation of the drive motor which is inside the camera tract assembly, plug the supply cord, directly into one of the convenience receptacles located on the back or sides of the Diffraction Unit.

### 3-7. EXPOSURE

The average exposure time with the 114.6 mm Powder Camera will be from one to three hours, and the 57.3 mm Powder Camera will give well defined lines with exposure time from five to sixty minutes.

The exposure time desired will depend on the type of target, tube loading specimen, and X-Ray film used.

## 3-8. MEASUREMENT AND CALCULATION (Figures 3-3 &amp; 3-4)

Any film, after development and drying will shrink to a degree that is dependent upon a number of factors. By making use of the Straumanis technique, it is possible to correct for uniform film shrinkage and the film becomes self-calibrating. In this technique, the 114.59 mm diameter Powder Camera is designed so that 2 mm measured on the film is equivalent to 1° Bragg angle.

The Type 170 114 00 Film Reader and Measuring Device is an optional camera accessory to aid in reading diffraction lines of varying intensity.

The film is laid flat on the film measuring device and a 0° reference point in the forward reflection region is established by bisecting the distance between the corresponding diffraction line on each side of the beam receiving tube hole in the film. To determine the 180° reference point, the same procedure is followed for the back reflection region, without changing the position of the film. Bisect the distance between the corresponding diffraction line on each side of the beam collimating tube hole in the film. The distance between the 0° reference point and the 180° reference point should be 180 mm. If this does not measure 180 mm, the correction factor will be the ratio of 180 mm to the actual measurement. For example, if the film length is found on measurement to be 178 mm, then all measured distances must be multiplied by  $(180) / (178)$  to get their true value. The distance of the various diffraction lines from the zero reference point is determined by reading from the scale on the film measuring device. The correction factor is then applied.

The resulting series of numbers divided by 2 are equal to  $\theta$ , the Bragg angles. (The resulting series of numbers are equal to  $\theta$ , the Bragg angles when the 57.3 mm camera is used since 1 mm measured on the film equals 1° Bragg angle).

From Bragg's Law:  $\lambda = 2d \sin \theta$

Now 
$$d = \frac{\lambda}{2 \sin \theta}$$

Where  $d$  = interplaner spacing in Angstrom Units

$\lambda$  = wavelength of characteristic radiation in Angstrom Units

$\theta$  = angle between incident X-ray beam and crystal planes in the crystallite of the specimen.

Reciprocal Lattice Notation:  $d = \frac{\lambda}{2 \sin \theta}$

so that:  $d^* = 2 \sin \theta$

### 3-9. FILM INTERPRETATION METHODS

#### a. VISUAL

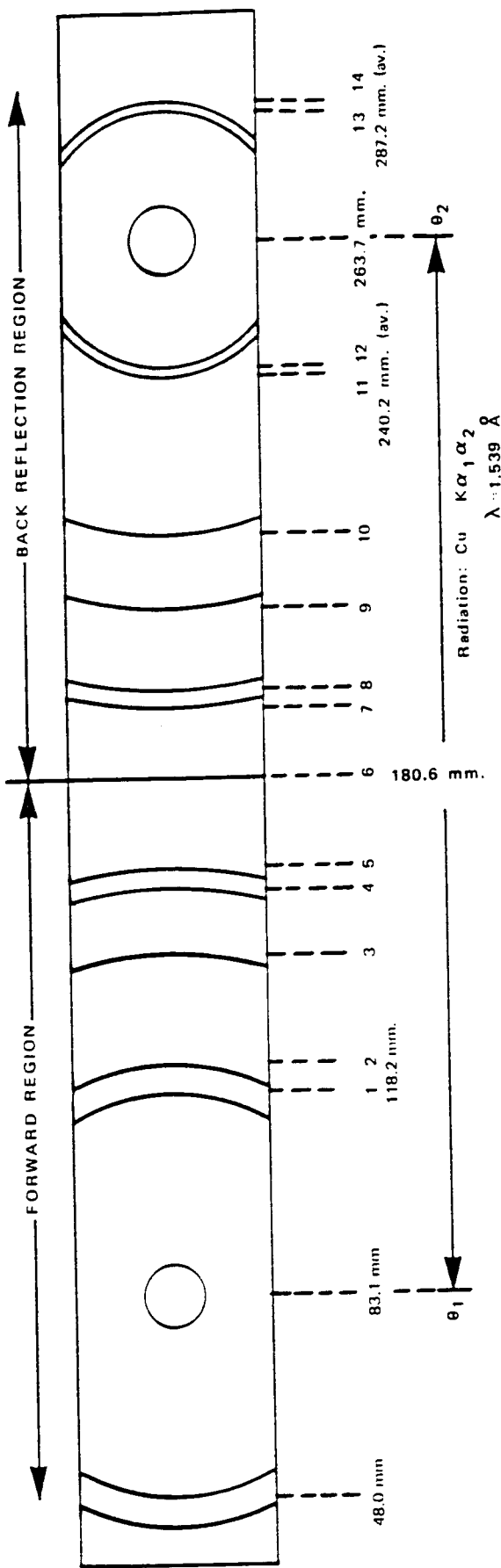
This method is very simple, since the type of lines produced on the film will suggest the type of material in the specimen. In general, under average conditions, the better the ultimate crystalline structure, the sharper the lines will be.

1. Diffuse lines indicate that the specimen is either amorphous, that the particles are very small, or have become imperfectly crystallized and are under strain.
2. Preferred orientation such as might be present in a rolled or drawn solid specimen, can produce lines that will have alternating dark and light blackening on any one line. If the grains that compose the specimen have exceeded a certain size, the visible lines will be rather spotty instead of being uniform.
3. Heavy background fog, obtained under conditions that are known to normally give a good film, is probably due to fluorescence of the specimen. For example, the presence of iron in a sample studied with copper radiation causes X-ray fluorescence, while many materials such as calcium tungstate give off visible light.

#### MEASUREMENT AND CALCULATION

From the procedure for measurement and calculation of the Bragg angles, as described above, a series of "d" values are obtained that will be characteristic of the specimen under examination. The "d" values for a large number of chemical identities have been indexed and published, and are very useful for this type of identification. With some specimens, by means of Hull-Davey Charts<sup>7</sup>, the lines present can be indexed by their Miller indices and the crystal structure of the substance determined.

7. See reference work page 3-15.



**SAMPLE CALCULATION**

1. Locate positions of lines employing millimeter scale, reading along center line of film. Zero mark on scale need not coincide with any line. Enter values so obtained as S<sub>1</sub> values in column 2.

2. Determine pattern centers θ<sub>1</sub> and θ<sub>2</sub>

$$\theta_1 = \frac{48.0 + 118.2}{2} = 83.1 \text{ mm}; \theta_2 = \frac{240.2 + 287.2}{2} = 263.7 \text{ mm.}$$

θ<sub>2</sub> - θ<sub>1</sub> = 263.7 - 83.1 = 180.6 mm., therefore, since correct length on exposure was 180 mm., each measurement must be multiplied by  $\frac{180}{180.6} = 0.996$ .

3. Subtract 83.1 mm. (θ<sub>1</sub>) from each S<sub>1</sub> value. Enter in column 3 as S (uncorrected).

4. Multiply each value in column 3 by 0.996 to get S (corrected). Enter in column 4 as S (corrected).

5. Divide values of S by 2 and enter in column 5. This value gives the Bragg angle in degrees since 2 mm. equals 1° Bragg. (In the 67.3 mm. camera the figure in column 4 would be employed since 1 mm. on the film equals 1° Bragg.)

6. In column 6 enter corresponding values of Sin θ.

7. In column 7 enter 2 × Sin θ.

8. In column 8 enter values of "d" from formula  $d = \frac{\lambda}{2 \times \text{Sin } \theta}$

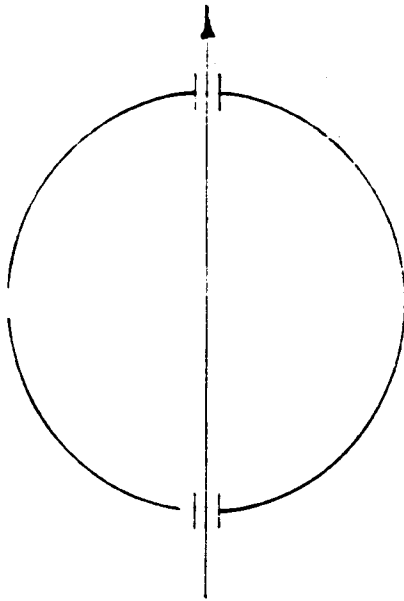
9. In column 9 enter relative intensity values of lines

10. Using data in columns 8 and 9, ASTM (Hanawalt) tables may be consulted.

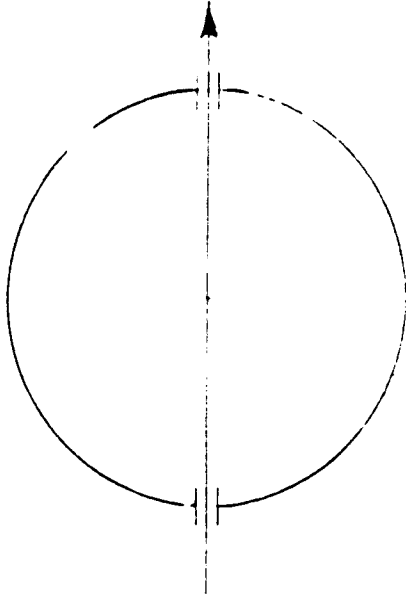
Line #	S <sub>1</sub> mm.	Uncorr. (S <sub>1</sub> - θ <sub>1</sub> )	Scorr.	S (θ°)	Sin θ	2Sin θ	d	I
1	118.2	35.1	34.95	17.47	.3000	0.600	2.57	1.0
2	123.7	40.6	40.43	20.21	.3455	0.691	2.22	0.8
3	142.0	58.9	58.71	29.35	.4901	0.980	1.57	0.8
4	153.5	70.4	70.12	35.06	.5743	1.148	1.34	1.0
5	157.0	73.9	73.6	36.8	.5990	1.198	1.29	0.4
6	171.0	87.9	87.55	43.77	.6913	1.383	1.11	0.2
7	181.5	98.4	98.00	49.00	.7547	1.509	1.02	0.6
8	185.0	101.9	101.49	50.74	.7740	1.548	0.997	0.6
9	199.7	116.6	116.13	58.06	.8485	1.697	0.909	0.7
10	211.8	128.7	128.18	64.09	.8993	1.798	0.858	0.8
11	240.2	157.1	156.47	78.23	.9789	1.958	0.785	0.5

Figure 3 - 3

a)



b)



FILM SHRINKAGE CORRECTION METHODS

- a) STRAUMANIS TECHNIQUE
- b) WILSON TECHNIQUE (MODIFIED STRAUMANIS TECHNIQUE FOR USE WITH SPECIMENS WHICH YIELD NO DIFFRACTION LINES IN THE BACK REFLECTION REGION)

Figure 3-4

Table 3-1

CHARACTERISTIC WAVELENGTHS

TARGET	CHARACTERISTIC LINE $\alpha_1$	$\alpha_2$	$\beta$	WEIGHTED VALUE $\alpha_1$	$\alpha_2$	FILTER	FILTER THICKNESS INCHES x 10 <sup>-3</sup>	DO NOT USE FOR
Cr	2.2850	2.2889	2.0806	2.287		Va	0.75	Ti
Fe	1.9321	1.9360	1.7530	1.934		Mn	0.65	Cr
Co	1.7852	1.7891	1.6174	1.787		Fe	0.70	Mn
Cu	1.5373	1.5412	1.3893	1.539		Ni	0.75	Co, Fe
Mo	0.7078	0.7128	0.6310	0.709		Zr	7.00	Y