PREFERRED ORIENTATIONS IN ROLLED METALS. CONSTRUCTION OF POLE FIGURE BY BACK-REFLECTION METHOD

by

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I. INTRODUCTION

When a poly-crystalline metal has been subjected to a considerable amount of cold work which is directional in nature, such as in rolling, drawing or extruding, a reorientation of the grains with a tendency to approach some certain direction occurs. The greater the amount of cold work the higher is the degree of reorientation. This effect is known as preferred orientation which has great influence on the physical properties of the material. For some industrial purpose, preferred orientation is a very desirable characteristic of the material, and also some special orientation is more favorable than the others. However, in other cases, the directional properties, which are associated with the presence of preferred orientation of grains may be a distinct disadvantage. Consequently, it is evident that the quality of the finished products may be improved through the knowledge of the specific effects produced by various fabrication methods which directly influence the preferred orientation.

The existence of preferred orientation is indicated by the concentration of the diffraction images into certain regions, since there are more grains in position to reflect to certain segments of the diffraction ring than the others. In the case of drawn wire, the preferment in orientation is only in one direction, along the wire, since the nature of working is identical in all directions perpendicular to the wire. However, in rolled sheet metal, preferment will occur with respect to two directions - the rolling direction and the cross rolling direction - owing to the fact that the treatment is not identical for all directions perpendicular to the rolling direction. The only method of describing two-dimensional preferment is to construct the so-called 'pole figure' which is a summarization by stereographic projection of data obtained from a series of X-ray photographs taken at different angles to the sheet.

Previous Work

The usual method in constructing a pole figure is done by means of the data from transmission lane patterns. Usually, one is taken with the rolling direction and the other with the traverse direction perpendicular to the axis of rotation of the sample. The thickness of the sample has to be reduced to between 5 and 6 mils so as to cut down the absorption of X-rays to a minimum. The film is placed from the sample at such a distance as to obtain the Debye ring of low θ value with a radius of 3 - 4 cm. Nevertheless, the exposure time is still considerably long, i.e., about 15 hours. The pole figure is usually plotted from the Debye rings of the (110) plans which are considered to give the best result on account of the greatest atomic density. This is accomplished by taking a series of diffraction patterns with the specimen tilted increasing angles of 5 or 10°. The number of exposures in such a series may vary from 5 to 20, depending upon the detail required in the pole figure.

The great handicap of this method arises from the effect of absorption of X-rays by the sample. As the angle of rotation of specimens is increased the intensity will be decreased according to the law of absorption ($I = I_0 e^{-\mu x}$). Besides, with the sheet samples, except for the case when the X-ray beam is perpendicular to the rolling plane, the absorption is not the same for all points on the Debye ring, since, in passing the sample, the diffracted rays traverse paths of unequal lengths. Maximum absorption occurs for the beam diffracted to the point on the cross-rolling direction of the Debye ring opposite the portion of the sample inclined toward the film, minimum absorption for the beam diffracted to the opposite point of the Debye ring. As a result of the variation of absorption, the time of exposure for each photograph has to be varied for each position of the sample. This is not practical.

<u>Summary</u>.

It is the purpose of this experimental work to construct a pole figure of the cold rolled steel sheet by means of the back-reflection method so as to avoid the trouble about unequal absorption. A cylindrical camera was so designed that a collimated beam of X-rays reflects from the sample and the diffraction pattern is recorded on photographic film held on the cylinder. Cobalt target was chosen bacause of suitable value of θ (61.9) of the required (220) planes. Owing to the fact that the reflection circle ($90 - \theta$) for high angle Debye ring is small compared to the circle of the pole figure, three sets of diffract patterns must be taken from cylindrical samples mounted with the rolling direction perpendicular, at 45° and parallel to the axis of rotation respectively. The exposure time is about 10 - 40 minutes depending on the size of the sample. The number of photograms taken at 10° ~~minute~~ intervals is twenty-eight. The pole figure so obtained is quite satisfactory according to the purpose.

II. EXPERIMENTAL

1. Equipment

a.) X-ray Source. The X-ray tube used in the experimental purpose is of the cold cathode or gas type. Cobalt was found to be a suitable source of X-radiation on account of 2θ of the (220) planes being 123.8°. It is evident that in choosing the target the value of 2θ ought neither to be much larger than 120°, nor less than 100° when considering: 1) the intensity which varies inversely with the square of the distance, 2) the absorption within the sample, which will increase as 2θ approaches 90°, and 3) the reflection circle $(90 - \theta)$, which, in this case, will become smaller as θ increases. The target is water cooled, whereas the focussing cup which is at high potential is cooled by a stream of air. The tube is operated between 8 - 10 milli amperes and a voltage of from 35-45 kilovolts after the proper pressure of about 0.01 mm. of mercury has been maintained.

b.) Camera. The camera used in this experiment is a simple cylindrical camera as shown in Figure I. The diameter of the cylinder upon which the film can be held firmly was made at 57.3 mm. so that one millimeter on the film corresponds 2° of arc. The shield has a 2 cm. slit through which the desired diffraction ring may pass to the film through the black screen pasted on the shield to make the camera light-tight. The sample can be mounted on the holder which is so equipped that it can be rotated to any

Figure I. Camera with a sample on the holder desired angle about the vertical axis and also can be shifted horizontally parallel to the X-ray beam, thus permitting the sample to be set at any required distance from the pinhole. The pinhole and camera are mounted on the same plate, and in case of loading a film, the plate is removed from its platform by loosening its holding screw. The loading of film is done by pulling out the shield, and then inserting the film of 3 × 17 cm. in its place. The film is afterwards spread tightly on the camera by means of a screw.

c.) Pole Figure Chart. The pole figure may be defined as a circle which is the stereographic projection of an imaginary sphere at the center of which a crystal is to be placed. It is known that when a beam of light falls on a plane, the incident beam, the reflected beam and the pole of the reflecting plane all lie in the same plane. Since the angle of incidence θ, of the beam on the crystal plane, is determined by the Bragg Law $n\lambda = 2d\sin\theta$, whenever reflection occurs, it follows that the poles of all planes capable of reflecting must lie at a constant angle of $90 - \theta$ from this incident beam and must, therefore, intersect the reference sphere only along the circle known as the reflection circle, a circle on the projection $90 - \theta$ from the centrally located beam. If the plane of the sample is normal to X-ray beam, the projection of the σ reflection circle will therefore, by the law of stereographic projection, be concentric with the boundary circle of the pole figure. From this principal, the pole figure chart may be constructed in the following manner.

Having placed a piece of tracing paper over the Wulff not of the size desired for the pole figure, trace onto it the equator, vertical meridian and the boundary of the net. On the pole figure chart, draw the 0° circle with a radius of (90 - θ or 28.1°) as measured on the equator, or the vertical meridian of the Wulff net.

To determine the parallels of latitude for the pole figure chart, divide each quadrant of the 0° circle into 10° arcs by drawing radial lines to the boundary circle. The intersection of the radii with 0° circle are the points where the 10° parallels of latitude for the pole figure chart. Draw in parallel of latitudes by tracing them from those of the Wulff net.

Now, when the sample is rotated through an angle of n°, the Debye ring is still circular but is no longer parallel to the sample. According to the theory of stereographic projection, its projection will be a circle whose center must lie on the equator or the equator produced. The radius of this circle remains unchanged, i.e., at (90° - θ). However, it will intersect the equator at points located n° to the right or to the left of the 0° circle, depending on the direction of rotation of the sample. Thus, the n° circle must have its center at the middle point of the line joining the two points. This point may be located by means of a pair of dividers. Furthermore, this circle will also be tangent to each of the tangent latitudes (or areas of the tangent circles) of the 0° reflection circle and the point of tangency is located at a point n° to the right (or to the left) of the vertical meridian, measured on the tangent latitudes.

The prepared chart was made with the reflection circles at intervals of 10° as shown in Figure II. The radius of each circle is equal to 28.1°.

2. Preparation of sample

Owing to the fact that the reflection circle for Debye ring of high angle is small when compared with the circle of the pole figure, such a series of photograms made from a specimen which has been rotated at intervals of 5 or 10 degrees about two principal axes, e.g., the rolling and traverse directions will not give sufficient data to complete the pole figure. Therefore, in this case, it is necessary to acquire further information about one or more axes which make definite angles with the previous axis. The other series of photograms taken from the sample rotating about the axis at an angle of 45° with the rolling direction was found to supply sufficient data for the missing spaces in the pole figure.

Nevertheless, if much more details are required, the series of diffraction patterns about the other two axes at 30° and 60° with the rolling direction are recommended.

From the above requirement, three samples were made with the rolling direction perpendicular, parallel and at 45° with the vertical axis respectively. For the purpose of eliminating any effect due to increased absorption as the inclination of the sample to the beam is increased, the sample was prepared in the shape of a rod rather than a sheet. It was also found that if the sample was in the shape of a sheet, the recorded pattern became shorter as the angle of rotation was increased because of the interference of the plane of the sheet sample with the reflected rays, causing a sine-curve shade. (See Figures III and IV). Besides, the rays reflected from the sheet sample become more unsymmetrical about the axis of rotation as the inclination of the sheet increases.

The sample was reduced to the desired shape according to the following steps:

1.) Cutting to a square rod.

2.) Filing to an approximately cylindrical shape.

3.) Polishing with an alundum polishing stone, and

4.) Etching with 25% HNO_3.

The final etch will remove all traces of material worked during the earlier operations. Since the presence of oxides will introduce some confusing effect, it is desirable to keep the surface of the sample clean at all times.

3. **Nature of Pattern**

According to Bragg's Law, when a beam of X-radiation of known wave length is permitted to fall upon a sample, the beam is bent or diffracted through an angle of θ , which is given by the following formula:

$$n\lambda = 2d\sin\theta = \frac{2a_0 \sin\theta}{h^2+k^2+l^2}$$

where λ = the wave length of X-radiation

d = interplanar spacing for set of planes concerned

hkl = Millerian indices of the planes

a_0 = lattice parameter

n = order of diffraction ring.

(calculation of θ is given in appendix)

The Bragg angle θ for the (220) plane of iron using Co radiation is equal to 61.9°, and, consequently, the reflected beam will make an angle of 2θ or 123.8°. This Debye ring, recorded on the film placed on the cylindrical camera, appears as a straight line of varying intensities when the film is spread out. Now since the diameter of the camera is made at 57.3 mm., one millimeter on the film will represent 2° angle of the Debye ring. The intensity at any one point is proportional to the number of planes which are in position to reflect rays. Consequently, if a photogram contains intensity varying on the diffraction line, it signifies that many more grams are oriented to reflect X-rays to other portions of the diffraction circle, and thus is said to have preferred orientation. Therefore, in the case of random orientation, the diffraction line will be of uniform intensity throughout; whereas, the pattern of ideal orientation will consist of spots of maximum intensity separated by regions of zero density. The position of any plane which causes a reflection to a point on the Debye ring is given by the cosine law.

$$\cos \delta = \frac{\cos \alpha}{\cos \theta}$$

where α = angle between normal to reflecting planes and rotation axis of sample.

δ = angular departure of spot from the fiber axis of film (azimuth angle)

θ = Bragg angle of incidence

For the planes of low values of θ as commonly used in pole figure, cos θ is nearly equal to one, and α and δ may be regarded as identical. As a result, the measurement of an angle on the film is a direct determination of the angle corresponding directions in the sample. However, this assumption cannot be applied to the back-reflection method where the value of cos θ becomes an important factor.

In setting up the apparatus the sample is brought in as near to the pinhole as possible on account of the intensity which varies inversely as the distance. The time of proper exposure was determined by trial. In this case, since the sample was very small compared to the collimated beam, the proper exposure time was found to be about 40 minutes, while it requires only 12 minutes for the sheet sample.

4. Plotting of Pole Figure.

The intensity distribution along the diffraction line of each photogram was determined by dividing it into zones of equal intensity. This was done by visual estimation rather than with the aid of a standard density scale or by means of a microphotometer, for it was found to be the more satisfactory because it was less tedious and of sufficient accuracy for the purpose. In this case, the intensities were classified into 4 zones - rating as high, medium, low and no intensity. Since the joining of the film is at 90° to the axis of rotation of the sample, the reference point on the diffraction line is chosen at the center of the film. The measurement of the length of the line to the zone boundaries from this reference point in millimeters will correspond to twice the number of degrees on the Debye ring. To decrease the labor of measurement of each film, a chart calibrated in degrees was made as shown in Figure V. The data of each photogram was read directly from the chart, on which the film was placed with its reference point on the 0° division.

In plotting the pole figure, a trace paper was placed on the pole figure chart, and the boundary circle, the rolling direction and the cross rolling direction were then traced onto it. The data taken from each film were recorded on the corresponding reflection circles of the pole figure chart in the following way.

Considering the 0° film taken with the rolling direction perpendicular to the axis of rotation, the equator of the pole figure chart will then correspond to the rolling direction of the sample, and the data of this film must be recorded on the 0° reflection circle. The reference point on the diffraction line, which coincides with the 0° division on the chart, must represent the point where the 0° reflection circle intersects the equator of the pole figure chart. From this point the data obtained by the measurement of the length to the boundaries of the intensity zones are plotted directly as read from the chart. The 90° division on the chart will represent the two points where the reflection circle intersects the vertical meridian. Hence, suppose the maximum intensity zone read from the chart as from 70° to 110°, the maximum zone on the reflection circle will then be plotted on the pole figure as 20° measured from either side of the points of intersection of the vertical meridian and the reflection circle. It will be seen that on the 0° film the pattern is symmetrical with respect to both the rolling and cross rolling directions, but as the sample is rotated through an angle, the pattern will be symmetrical only about the rolling direction. However, for film taken with respect to 45° axis, the pattern produced would not be symmetrical about the reference point. Thus, in plotting the data, the position of the film with respect to the position of the sample must be marked so that the data will be plotted on the right part of the reflection circle. In this respect, the shade produced by the sample holder was taken as a mark on the film, and consequently, the data as read from 0° to 180° from the film with the shade on was thus plotted on the lower half of the reflection circle with respect to the 45° axis. Moreover, the direction of rotation must be noted so as to know exactly where the reference point on the film corresponds to the reference point on the reflection circle.

After the plotting of the data have been accomplsihed, the pole figure of the sample may be obtained by drawing the boundary lines of those intensity zones. In the case of two lines of different intensities crossing each other, the higher intensity line is considered to predominate, and consequently is taken as the area of the higher intensity zone. The pole figure which is obtained by this method is as shown in Figures VI and VII.

III. DISCUSSION OF RESULTS

The pole figure of (220) planes so produced proves to be quite satisfactory. Regarding to the time of exposure, it is practical for an ordinary purpose, since the proper exposure can be done in a relatively short time, i.e., 12 - 40 minutes depending on the thickness of the sheet, and if the sample is a true cylinder, the exposure time remains unchanged, regardless of the position of the sample, as a result of equal absorption; whereas, the usual method requires change of exposure time and some corrections on the intensity of the film. However, the pattern obtained from the (220) planes does not show distinct sharpness of the intensity zone, and therefore, the estimation of intensity zone will depend greatly on careful visual judgment and proper control of exposure so that each classified zone will have equal intensity on each film. The information received from an individual film is much less than that obtained with the usual method, and consequently, the usual method will give more accurate details for equal number of photograms. Moreover, the film cannot record the whole Debye ring on one film because part of it falls on the protecting cylinder (shield) opposite to the joining ends of the film, but, in plotting, reasonable interpolation may be done since the missing data is only 30°. Nevertheless, this can be avoided by changing the position of the camera so that the joining ends of the film are on the vertical axis, thus giving the whole upper half of the reflection circle full information which is symmetrical to that of the lower half, or else the other photogram may be again taken with the sample rotated through the same angle in opposite direction.

The main handicap to this method arises from the fact that a true cylindrical sample is nearly impossible to obtain especially for the very thin sheet, and so proper control of exposure is greatly involved. Besides, care must also be taken in working down the sample so that the actual texture of the sample will not be destroyed. Above all, the specimen should have a fine-grained texture, otherwise it has to be oscillated through a slight angle to bring more grains into reflecting position. At any rate, the sample is assumed to have no modification in texture between the surface and the center of the metal. This is not always true since it has been found that preferred orientation in rolled sheets thicker than 1 mm. are likely to have a texture on the surface which differs from that in the interior.

IV. CONCLUSIONS

Aside from possible errors as indicated, the back-reflection method of construction of a pole figure of the (220) planes may be well considered to be applicable for some practical purpose which only requires the knowledge of approximate texture of the material. Considering the labor of construction, it is much more practical for industrial purposes than the usual method since it can be produced in a relatively short time and requires no correction in absorption whatsoever.

V. SUGGESTION FOR FURTHER WORK

The sample should be made as a true cylinder as possible. Series of photograms are to be taken with respect to four axes, i.e., rolling direction, traverse direction, and at 30° and 60° to the rolling direction. Compare the accuracy in details with that by the transmission method. The camera should be so equipped that the sample can be rotated in synchromizing with the film, thus allowing the whole data to be recorded on one film for each axis.

VI. APPENDIX

Calculation of θ for the (220) ring of rolled steel sheet, using Co K_α radiation.

Known data $n = 1$

$kkl = 220$

$a_o = 2.8608\ \mathring{A} = 2.8608 \times 10^{-8}$ cms.

$\lambda = 1.7853\ \mathring{A} = 1.7853 \times 10^{-8}$ cms.

$$\sin\theta = n\ \lambda \frac{h^2+k^2+l^2}{2a}$$

$$= \frac{2\sqrt{2} \times 1.7853}{2 \times 2.8608}$$

$$= .8824$$

$$\theta = 61.9°$$

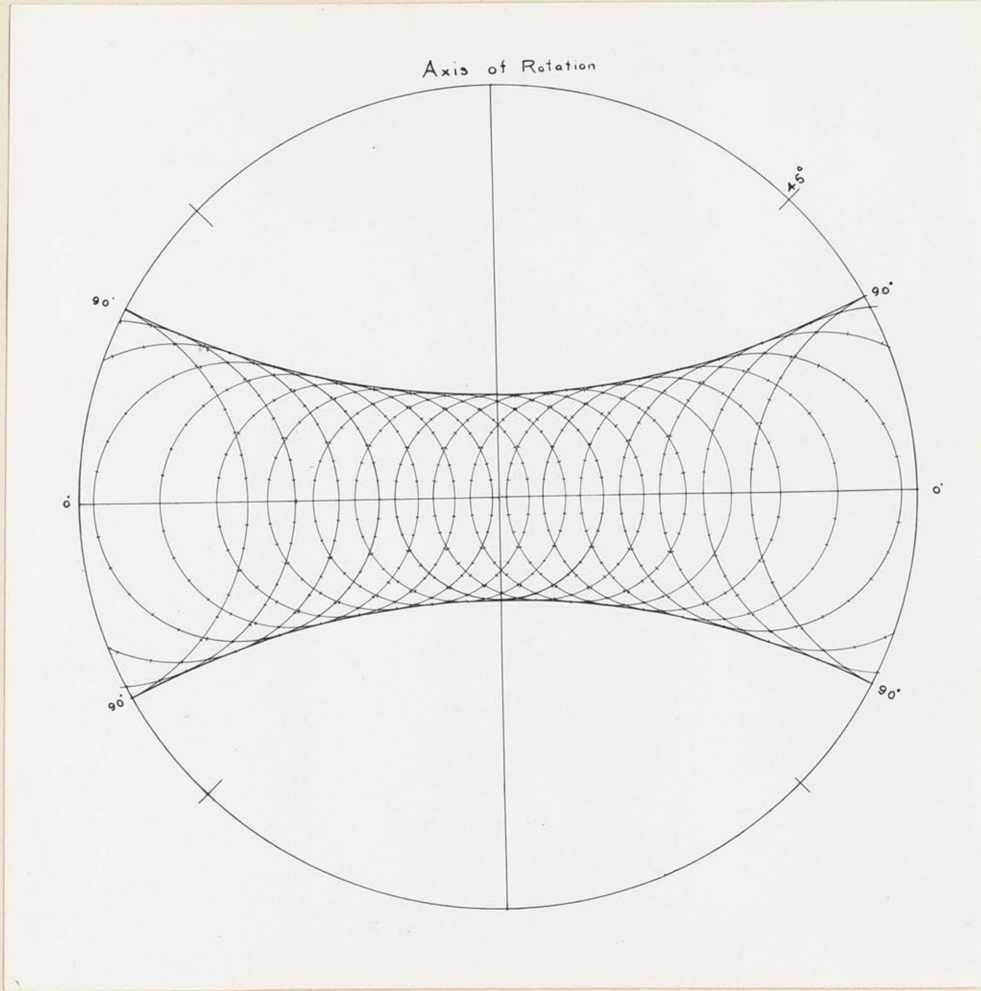

Figure II. Pole-Figure Chart for Co Radiation Reflecting from (220) planes of iron Θ = 61.9°

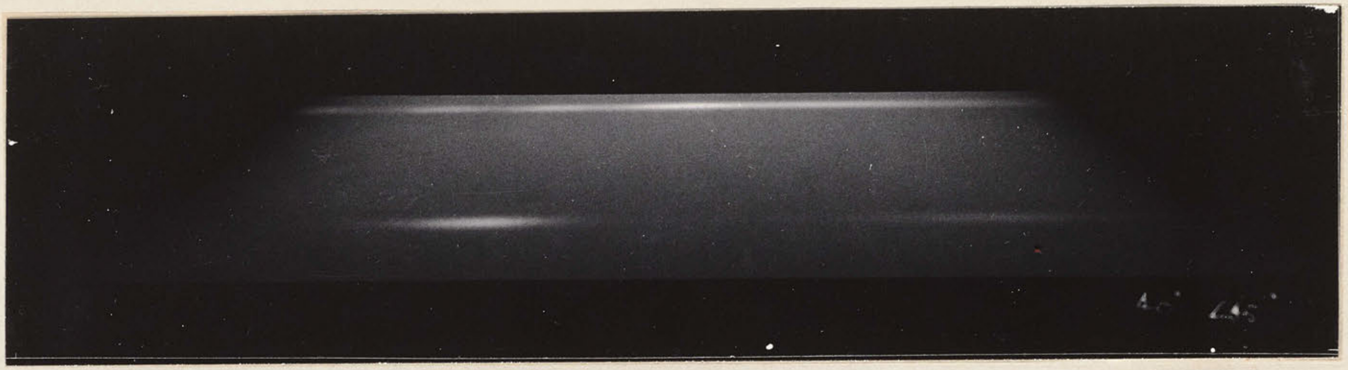

Figure III. Pattern produced from sheet sample rotated through 40° with rolling direction perpendicular to the vertical axis.

Figure IV. Pattern produced by a cylindrical sample rotated through 20° with rolling direction perpendicular to the vertical axis.

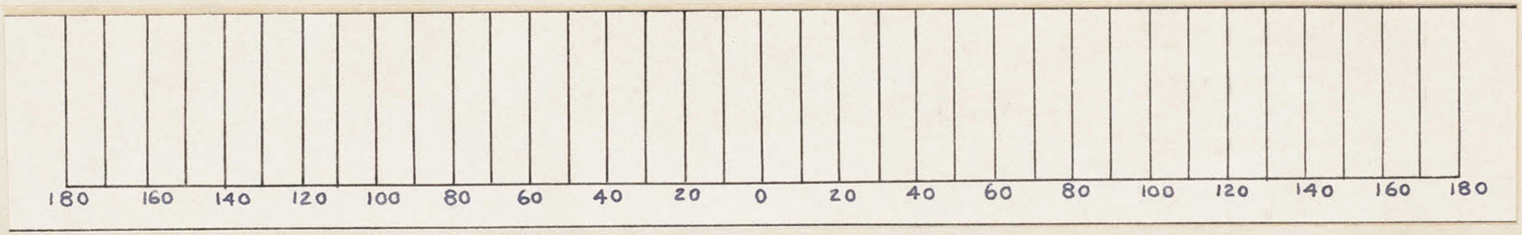

Figure V. Chart for Measuring Patterns

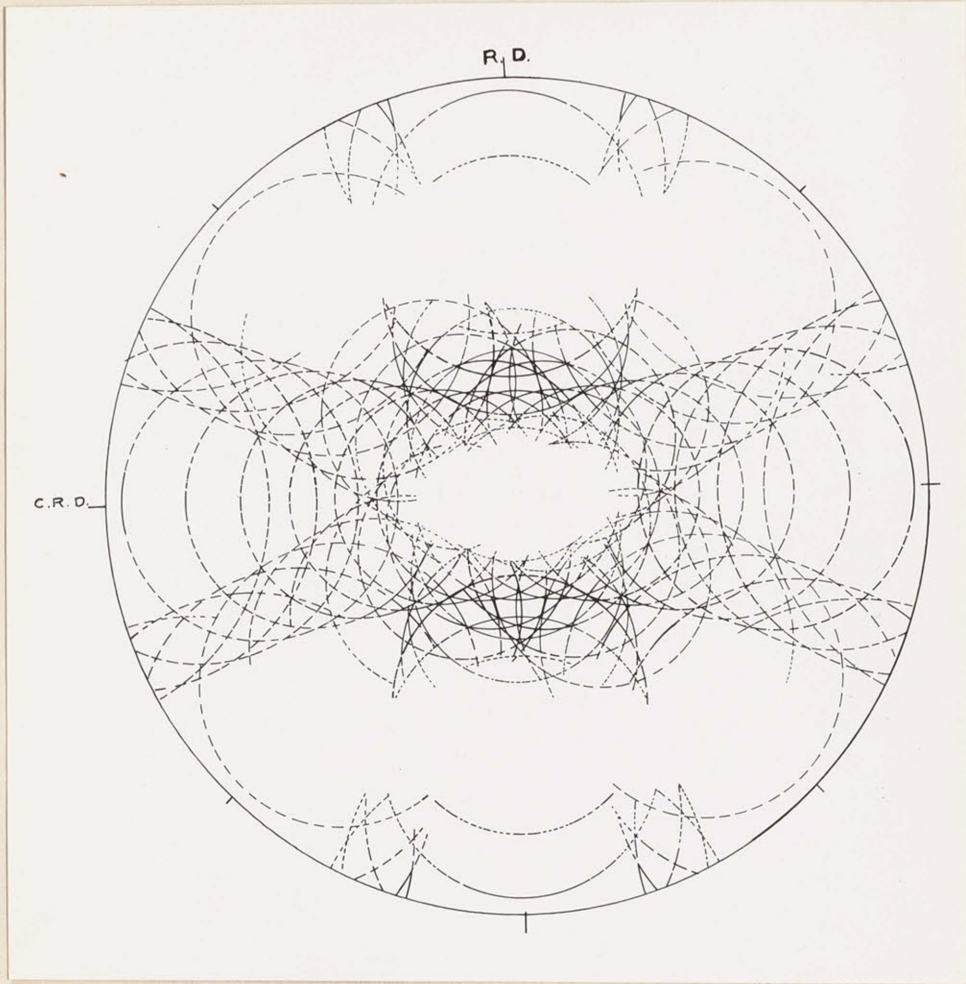

Figure VI. (220) Pole Figure of steel sheet cold-rolled per
86 cent reduction (as plotted).

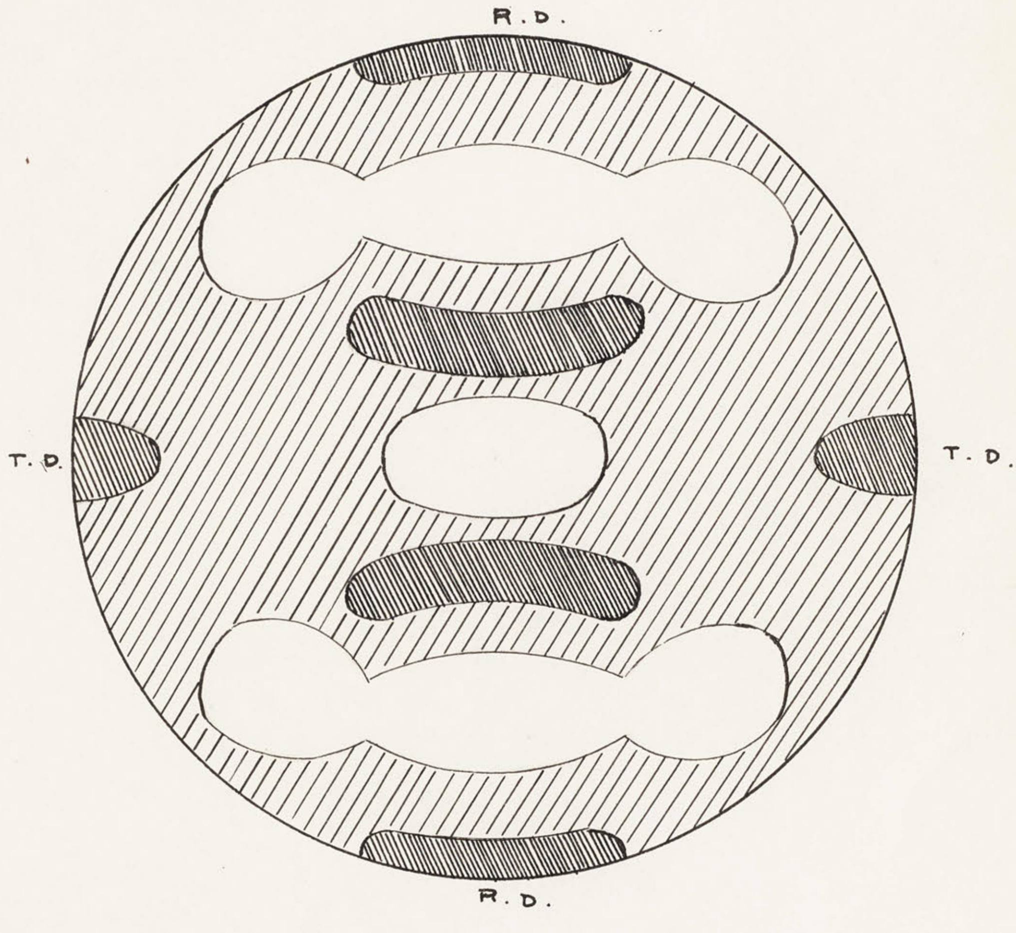

Figure VII. (220) Pole-Figure of steel sheet cold-rolled

86 per cent reduction.

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