## UNCLASSIFIED



Melvin H. Mueller, Harold W. Knott, and Paul A. Beck
蠋
This report was prepared as an account of Government sponsored work. Neither the
United States, nor the Commission, nor any person acting on behalf of the Commission:
A. Makes any warranty or representation, express or implied, with respect to the ac-
curacy, completeness, or usefulness of the information contained in this report, or that the
use of any information, apparatus, method, or process disclosed in this report may not in e
fringe privately owned rights; or
B. Assumes any liabilities with respect to the use of, or for damages resulting from the
use of any information, apparatus, method, or process disclosed in this report.
As used in the above, person acting on behalf of the Commission" includes any em-
ployee or contractor of the Commission to the extent that such employee or contractor
prepares, handles or distributes, or provides access to, any information pursuant to his em-
ployment or contract with the Commission.


Final Report - Program 4.1.12

METALLURGY DIVISION

Operated by The University of Chicago under
Contract W-31-109-eng-38
1-2

## DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

## DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

# 为 <br> PREFERRED ORIENTATION IN $300^{\circ} \mathrm{C}$ ROLLED AND IN RECRYSTALLIZED URANIUM SHEET 

## ABSTRACT

The rolling and recrystallization textures in $300^{\circ} \mathrm{C}$ rolled uranium sheet were investigated using a Geiger counter diffractometer with the modified Schulz reflection technique. Seven sections of sheet material were used in order to obtain sufficient data for quantitative pole figures by the reflection technique. A special integrating specimen table was used for obtaining and recording the data automatically. The rolling texture was described in terms of several "ideal orientations" in which the stronger orientations have either the (110) or (010) poles in the vicinity of the rolling direction. The recrystallization texture of the sheet uranium was also described by several "ideal orientations" which are somewhat different, except in one case, than the as rolled "ideal orientations." Either approximate (010), (140), or (553) poles are near the rolling direction in the recrystallized texture.

## INTRODUCTION

An initial photographic X-ray texture study was made by A. D. Fischer, which is included as Appendix $A$ of this report. It was found that uranium sheet rolled at $300^{\circ} \mathrm{C}$ and subsequently recrystallized at $575^{\circ} \mathrm{C}$ showed considerable scatter of the (001) planes from the plane of rolling. This work had also indicated that the (010) planes were nearly perpendicular to the rolling direction.

Preliminary texture determinations by KAPL were made on uranium sheet reduced $60-75 \%$ at room temperature and at $250^{\circ} \mathrm{C}$, using the Schulz method with a X-ray diffractometer. (1) Some of the important peaks, however, were not sufficiently resolved to give satisfactory results. Therefore it was decided to use the photographic technique for the material reduced $65 \%$ at room temperature. (2) The ( 001 ) pole figure showed an intensity maximum of elliptical shape around the center and the (010) pole figure had high intensity near the rolling direction. Both the (110) and (021) pole figures appeared nearly random. It appeared that this room temperature rolled texture agreed with that reported previously by A. D. Fischer for $300^{\circ} \mathrm{C}$ rolled and recrystallized material.

More recent diffractometer orientation studies on sheet using a modified Schulz method have been reported by W. E. Seymour. (3) The texture developed in uranium foil rolled to a $91 \%$ reduction at room temperature was
described as (102) [010] and (012) [031]. The (001) pole figure showed four areas of maximum intensity; two were approximately $45^{\circ}$ on either side of center towards the transverse direction which may be described as a $45^{\circ}$ rotation about the [010] direction. The other two maxima were approximately $20^{\circ}$ on either side of center along the rolling direction which may be described as a $20^{\circ}$ rotation about the [100] direction.

The Norton technique was used in determining the preferred orientation of uranium sheet which had been reduced $85 \%$ at $275^{\circ} \mathrm{C}$ at 100 feet per minute. The orientation found ${ }^{(3)}$ was approximately the same as that described for the foil above except the four maxima on the (001) pole figure appeared approximately $10^{\circ}$ closer to the center in each case.

Preliminary data for uranium sheet rolled at $600^{\circ} \mathrm{C}$, reported by W. E. Seymour and J. F. Duffey, (4) indicated the (010) and (110) planes lie perpendicular to the rolling direction. The (001) planes were predominantly parallel to the rolling direction with considerable spread from the rolling plane. Data have been reported by the same authors $(5)$ for the center portions of pole figures of room temperature rolled uranium sheet recrystallized at $600^{\circ} \mathrm{C}$. Maximum intensity areas were found on the various pole figures in the following locations: in the center of the (100), displaced from center along the rolling direction in the (110) pole figure and in the four areas on the (001) previously described for the sheet as rolled, with the greatest intensity along the transverse direction. These data were interpreted in terms of a single (100) [010] "idealized orientation," leaving the maxima as shown in the (001) pole figure unexplained.

In view of the need for more complete data for rolled sheet, and particularly for the rolled and recrystallized material, it was decided to investigate these preferred orientations by means of the Schulz reflection method, using seven sections taken in different directions through the sheet. Because of the great drop-off of intensity with increasing tilting angle, (7) it is best to limit the range of angles covered by means of each section to a cone of $30^{\circ}$ aperture. By using seven well chosen sections, the quadrant of the pole figure can be covered adequately and accurately, with sufficient overlapping of data from the various sections to afford a good check on the accuracy. Although this method, used in the present work, is somewhat elaborate, it is justified by the accuracy and completeness of the results.

## EXPERIMENTAL

Uranium Sheet for Study - Uranium biscuit metal was melted in a graphite crucible previously coated with a MgO wash. The molten metal was then poured into a MgO washed graphite mold. The average chemical analysis of this 3 in . square casting was as follows:

|  | 130 |
| :--- | ---: |
| Carbon | 140 |
| Nitrogen | 50 |
| Iron | 30 |
| Silicon | 30 |
| Nickel | 70 |

The flat stock to be used for the final reduction at $300^{\circ} \mathrm{C}$ was fabricated from this casting according to the following procedure:


Material ${ }^{68} \mathrm{~A}^{\%}$ was then given an $87 \%$ reduction at $300^{\circ} \mathrm{C}$ which produced a strip 0.120 in . in thickness. The ends were reversed for each pass.

A portion of the $87 \%$ reduced material was annealed at $525^{\circ} \mathrm{C}$ for 1 hour in vacuum for use in studying the recrystallization texture. Microscopic evidence indicated that the material was completely recrystallized after annealing.

Specimen Preparation - In order to obtain sufficient data for a complete pole figure for sheet matexial, with the Schulz reflection method, it was necessary to use a number of sections. These sections were cut in different orientations with respect to the strip as shown in Figure 1. The shaded portion on each section shown in this figure indicates the surface used for obtaining the diffraction intensity data defined by the respective circle. Since three rod sections were used to cover $90^{\circ}$ of $\phi$ from the rolling direction to the cross direction in rod orientation determinations, it was convenient to use seven sections to cover a quadrant of a pole figure for sheet material. Six of these sections consisted of a number of pieces similarly cut and held together by means of a jig. This jig arrangement made it possible to form a plane surface $45^{\circ}$ or $90^{\circ}$ to the surface of the sheet sufficiently large so that the $X$-ray beam always remained on the plane surface while the specimen was moved back and forth for integration. The seventh section used was parallel to the rolled surface; it was prepared from a single piece of the sheet.

Two different types of jigs were necessary for the six assembled sections required. A sketch of the jig construction is shown in Figure 2. "A. ${ }^{20}$ type jig was used for sections 1,3 , and 5 and " $B$ " type jig for sections 2, 4, and 6. The small individual pieces were securely held in the jig by means of a clamping screw. In order to obtain a flat plane surface for diffraction, the assembled jigs were surface ground with light cuts. Hand grinding was then

used to take off approximately 0.025 in . of material followed by an electroetch and polish which removed an additional 0.002 in. During all the grind ing operations the top surface to be used for diffraction was maintained parallel, within $0.001 \mathrm{in} .$, to the bottom surface of the jig.

Preferred Orientation Instrument - The data for the pole figures were obtained with a X -ray diffractometer using the Geiger counter and the modified Schulz device previously described.(6) Although it is possible to use the specimen table shown in Figure 4 of the latter reference for sheetorientation study, it was necessary to design and construct a special table to accomodate the thick jigs which were used for this study. At the same time it was desirable to extend the automatic operation previously described (6) so that not only the $\phi$ angle may be changed automatically in $5^{\circ}$ steps, but also so that the $\alpha$ angle may be changed in $10^{\circ}$ intervals after completing each $60^{\circ}$ change in $\phi$. (The $\phi$ rotation axis is in the reflecting specimen surface and the $\alpha$ rotation axis is perpendicular to this surface.) With this automatic arrangement it was possible to obtain complete data for one of the seven areas for one reflection without attending the instrument.

A sketch of the specimen table used for accomplishing this automatic operation is shown in Figure 3. With this device, it was possible to move the specimen table, and hence the specimen, back and forth for integration over a sufficiently large number of grains in the specimen. This motion, shown by the arrows at the top of Figure $3 A$ and $3 B$, is accomplished by attaching a flexible shaft to the cam shaft (A) of Figure 3A. Since this table was designed to be interchangeable with the other specimen tables, it also was held in the inner ring of the instrument by an elevating screw attached to the under side of the table. As previously described, (6) the automatic rotation around an axis in the specimen surface ( $\phi$ rotation) is accomplished by the motor, pinion gear and cam assembly on top of the Schulz ring together with the ring gear attached to the inner ring. The automatic rotation of the specimen around an axis perpendicular to its surface ( $\alpha$ rotation) is accomplished as follows: after the extreme top or bottom $\phi$ step is reached some overtravel of the inner ring permits pivot arm $B$ or $C$ (Figure $3 C$ ) to strike a stationary stop on the outer ring, causing pivot arm $B$ or $C$ to move as indicated by the arrows (Figure 3C). The lever motion produces a motion on drive cam D, as indicated by the arrow in Figure 3A. This causes $E_{1}$ end of the locking pawl $E$ to move clockwise and the $E_{2}$ end to move away from table $F$. Since the rotating table $F$ is now unlocked, the pivoting motion produced by $E$ on ratchet cam $G$ allows the ratchet pawl $H$ to rotate table $F 10^{\circ}$. At this point the inner ring motion on the Schulz ring is reversed by a microswitch, described previously in (6), which permits drive cam $D$ to move away from the $E_{l}$ end of locking pawl $E$. This allows the ratchet cam $G$ and pawl $H$ to rotate counterclockwise slightly. The described movements are aided by springs 1, 2, and 3 (Figure 3B). The $E_{2}$ end of pawl $E$ then locks the table $F$ in position. With this fixed $\alpha$ movement the table then goes through the thirteen $\phi$ positions before again changing $\alpha$.

The assembled jigs were held on the table by a thin coat of tacky wax which seemed to be very satisfactory in spite of the rather heavy jigs and the integrating motion. A micrometer was used to set the jig surface parallel to the table. The table could then be inserted in the Schulz ring and alignment made with the alignment jig previously described.(6)

Plotting of the Data - For convenience, after aligning the specimen surface the table was moved to the lower or $-30^{\circ} \phi$ position. The table was rotated manually so that the jig clamping screw was at the top. The starting position for each section under these conditions is shown by the $S$ with the subscript number on the outer circle of each section (Figure 4).

The time necessary for a given number of counts for each $\alpha$ and $\phi$ position was recorded on a tape by the time interval printer as described in Reference (6). The counting time for each position was then plotted on standard polar co-ordinate paper according to the sequence shown for section 7 in Figure 4. This was done for each section and each reflection. A replot of this data was made on a second sheet of polar co-ordinate paper in terms of counts per second (intensity). A third plotting was then made on a master net which contains dots for each point from each of the seven sections. There are 585 points in one quadrant of a pole figure at which an intensity measurement was made (see Figure 5). The corrected intensity used for the last plotting on the pole figure was obtained by first subtracting background from the observed intensity and then multiplying each background corrected intensity by the appropriate correction factor as shown in Table I. This may be summarized as follows:

$$
\left(I_{\text {Observed }}-I_{\text {Background }}\right)(\text { Correction Factor } \phi)=I_{(\text {Corrected })}
$$

Contour lines of the pole figure were then drawn on a transparent sheetplaced over the net with the plotted intensities. It has been assumed that each of the four quadrants of a pole figure of this material would be practically identical due to the symmetry produced in reverse rolling.

The correction factors given in Table I were determined by measuring the drop-off in intensity with increasing $\phi$ angle of a strong reflection from a random sample of PbS powder. One of the factors which has an effect on the magnitude of this correction is the size of the slit system near the $\mathrm{G}-\mathrm{M}$ tube, which was described by Chernock and Beck. (7) Since it was necessary to use a rather narrow receiving slit to maintain the required resolution for certain uranium reflections the amount of correction at the higher $\phi$ angles was considerable.

This entire method of obtaining quantitative pole figures, by use of the reflection technique only, will be described more fully in a separate paper.

Table I
FACTORS USED TO CORRECT FOR THE DECREASE IN INTENSITY AS A FUNCTION OF $\phi$

| $\phi$ Angle | Correction Factor |
| :---: | :---: |
| 0 | 1.00 |
| 5 | 1.01 |
| 10 | 1.02 |
| 15 | 1.05 |
| 20 | 1.10 |
| 25 | 1.15 |
| 30 | 1.25 |

## DISCUSSION - $300^{\circ} \mathrm{C}$ ROLLED URANIUM SHEET

The quantitative pole figures, with intensity contours, for the $300^{\circ} \mathrm{C}$ rolled sheet described above, are reproduced in Figures 6 to 9. It is evident from the (010) and (110) pole figures that there is a considerable concentration of these two poles parallel to the rolling direction. However, in addition, there is a considerable spread of these poles away from the rolling direction, which is especially evident in the (110) pole figure. There is a sharp concentration of (001) poles tilted $30^{\circ}$ to $35^{\circ}$ away from the rolling plane towards the transverse direction and another (001) pole concentration tilted $15^{\circ}$ to $20^{\circ}$ away from the rolling plane towards the rolling direction.

A separate symbol has been used in each pole figure to mark "ideal orientations" corresponding to maxima or high pole concentrations. These symbols, without the contour lines, have been transferred to the pole figures shown in Figure 10 in order to show the compatibility of several "ideal orientations" with the high intensity areas which appear in the (010), (110), (001), and (100) pole figures.

The lines which form the quadrangles shown in Figure 10 connect one high intensity area from each of the pole figures mentioned above. The four symbols which form the corners of these quadrangles have the correct angular relationships to each other: namely, $90^{\circ}$ between the (001) and (010), (110), and (100); $90^{\circ}$ between the (010) and (100); and $64^{\circ}$ between the (010) and (110). Quadrangle $I$, formed from the dashed lines, corresponds to one type of "ideal orientation." In quadrangle II the set of high intensity areas joined together are different from those of quadrangle I except for the common location of the ( 001 ) pole, and they define a second type of "ideal orientation."

An entirely different set of high intensity areas are joined together to form quadrangle III, representing the third "ideal orientation." Quadrangle IV is different from the others with the exception that the (001) pole location is common with quadrangle III. "Ideal orientations" III and IV, represented by these quadrangles respectively, are largely made up of high intensity pole locations, but not necessarily maxima, especially in the case of the (010), (110), and (100) pole locations.

The pole intensity spreads around the highest intensity locations on the various pole figures may be accounted for by movement or rotation of the superimposed quadrangles shown in Figure 11. Quadrangle I may be rotated $64^{\circ}$ about one of the (001) maxima to coincide with II, which may produce a spreading of the intensity over the area indicated by the heavy lines with arrows. A rotation of the quadrangle II to III, as shown in Figure 11. would produce a smearing out of the pole intensity indicated by the heavy dashed lines. This rotation of quadrangle II to III may be described approximately as a $30^{\circ}$ rotation about the (122) pole which is nearly equivalent to the [322] direction. Quadrangle III may be rotated $26^{\circ}$ about the ( 001 ) to produce orientation IV if the four quadrants of pole figure are assumed to be symmetrical. This latter rotation would produce considerable smearing out of the intensity between III and IV.

The orientations which Seymour has reported (3) for uranium sheet reduced $85 \%$ at $275^{\circ} \mathrm{C}$ correspond approximately to the orientations repre= sented by quadrangles II and IV in Figure 10. The KAPL study did not show an orientation corresponding to quadrangle III, which may be considered as a large spread from orientation II. Seymour did not report the Type I ${ }^{\text {ch }}$ ideal orientation," and the differences, particularly in the (100) and (110) pole figures, between his results and those reported here remain unexplained.

It may be concluded from the present study that the greatest intensities shown on the four pole figures for as rolled material may be quite satisfactorily described in terms of four types of "ideal orientations" tabulated below and of the considerable scatter; a) between I and II, b) between II and III, and c) between III and IV. Orientation II is apparently stronger than I, IV, or III. The latter three seem to be approximately of equal intensity.

Table II
"IDEAL ORIENTATIONS" FOR URANIUM SHEET $300^{\circ} \mathrm{C}$ ROLLED

| "Ideal <br> Orientation" | Plane Parallel <br> to Rolling <br> Plane | Direction <br> Parallel <br> to Rolling <br> Direction | Plane <br> Perpendicular <br> to Rolling <br> Direction | Multiplicity <br> in Pole <br> Figures |
| :---: | :---: | :---: | :---: | :---: |
| I | $(4, \overline{17}, 26)$ <br> near (1"46) <br> $(9,0,25)$ | near [410] | $(110)$ | 4 |
| II | near (103) | [010] | $(010)$ | 2 |
| IV | $(\overline{4}, 14,45)$ <br> near (139) <br> near (038) | near [552] | $(4,17,5)$ <br> near (141) <br> near (041) | 4 |

## DISCUSSION - URANIUM SHEET $300^{\circ} \mathrm{C}$ ROLLED AND RECRYSTALLIZED

The pole figures for the $300^{\circ} \mathrm{C}$ rolled uranium sheet after recrystallization at $525^{\circ} \mathrm{C}$ are shown in Figures 12 to 15 . The ( 010 ) pole figure is very similar to that obtained for the as rolled sheet. The distribution of the (110) pole intensity has changed considerably from the rolled material. There is no longer a maximum of (110) poles parallel to the rolling direction; how ever, there are maxima in other locations. The ( 001 ) pole figure shows a maximum pole concentration approximately $35^{\circ}$ on each side of center towards the transverse direction which is similar to the pole figure obtained before recrystallization. Although there are high intensity areas $15^{\circ}$ to $20^{\circ}$ from the center of the (001) pole figure towards the rolling direction, there are no longer maxima in these locations, as there were in the as rolled sheet. The (100) recrystallized pole figure also appears considerably different from the (100) as rolled pole figure.

As explained above, symbols are shown to designate high intensity areas in each pole figure. Several different quadrangles are formed by connecting a symbol from each of the following pole figures (010), (110), (100), and (001). These quadrangles which represent "ideal orientations" are shown separately in Figure 16. The location of quadrangle II is identical with quadrangle II shown for the as rolled sheet. Apparently there is considerable amount of this orientation retained after recrystallization. The orientation represented by quadrangle VI has a common (001) location with II. The set of high intensity areas joined together to form quadrangle $V$ are different from II and VI, except the (010) location, which is common with II. This

orientation V is reminiscent of the "cube" texture in face centered cubic metals. The location of the high intensity areas joined to form quadrangle VII is different from those previously described. The symbols used for quadrangle VII are formed from dashed lines for easy recognition.

The spreads appearing between the several "ideal orientation" 10 cations may be accounted for by a gradual movement or rotation of the quadrangles as shown in Figure 17. Quadrangle II may be rotated $26^{\circ}$ about the (001) location to produce quadrangle VI. The intermediate orientations would account for the spreading out of the intensity over the area indicated by the heavy dashed arrows. Quadrangle VII may be rotated into quadrangle II, as shown in Figure 17, and the intermediate orientations account for the pole intensity along the heavy solid arrows. This rotation of quadrangle VII to II may be described as approximately a $47^{\circ}$ rotation about the (122) pole, which is nearly equivalent to the [322] direction. In addition to these two rotations, quadrangle II may be pivoted about the (010) pole in order to obtain quadrangle $V$. The smearing out of the pole intensity as a result of this latter rotation is not indicated in Figure 17.

The (131) pole figure for the recrystallized sheet is shown in Figure 18 with dots to indicate the locations of high pole concentrations required by the "ideal orientations' shown in Figure 16. There is a considerable spreading out of the intensity on the (131) pole figure which is to be expected as a result of the large number of (131) pole locations required by the various "ideal orientations" described, since (131) has a multiplicity of four.

The preferred orientation obtained in the recrystallized uranium sheet may be quite satisfactorily accounted for by the four "ideal orientations" tabulated below, with considerable spreading out of the intensity between a) II and VI, b) VII and II, and c) II and V.

## Table III

${ }^{6}$ IDEAL ORIENTATIONS" FOR URANIUM SHEET $300^{\circ} \mathrm{C}$ ROLLED AND RECRYSTALLIZED

| ${ }^{66}$ Ideal Orientation" | Plane Parallel to Rolling Plane | Direction Parallel to Rolling Direction | Plane <br> Perpendicular to Rolling Direction | Multiplicity in Pole Figures |
| :---: | :---: | :---: | :---: | :---: |
| II | $\begin{aligned} & (9,0,25) \\ & \text { near }(103) \end{aligned}$ | [010] | (010) | 2 |
| V | (100) | [010] | (010) | 2 |
| VI | $\begin{aligned} & (7,7,19) \\ & \text { near }(113) \end{aligned}$ | [110] | $\begin{aligned} & (3,13,0) \\ & \text { near }(140) \end{aligned}$ | 4 |
| VII | $\begin{aligned} & (2,2,15) \\ & \text { near }(116) \end{aligned}$ | near [411] | near (553) | 4 |

From a comparison of the intensities in various locations on the pole figures, it would seem that the relative amounts of the various "ideal orientations" in the recrystallized sheet may be expressed as follows:

$$
\mathrm{VI}>\mathrm{II} \cong \mathrm{VII}>\mathrm{V}
$$

It is interesting to note that the strongest texture, VI, has planes near the (140) perpendicular to the rolling direction. The (140) had been previously mentioned (8) as a texture component in recrystallized uranium rod. The (131) pole figure for the recrystallized sheet (Figure 18) is also in agreement with planes near the (140) perpendicular to the rolling direction. The angular distance between the (140) and (131) poles is $20^{\circ}$ and maximum intensity on the (131) pole figure is approximately $20^{\circ}$ to $25^{\circ}$ away from the rolling direction.

It is rather difficult to interpret the mechanism by which the recrys tallized textureforms from the as rolled texture. Only one of the "ideal orientations" used to describe the as rolled texture is found in the recrystallized texture. There are a number of rotations about the (001) and (010) poles, as shown in Figure 19, which may be used to describe the relationship between the various components of the recrystallized texture and the as rolled texture. However, it is possible that rotations about other poles are involved.

Orientation VI may arise from a $90^{\circ}$ rotation of I about the (001) or from a $26^{\circ}$ rotation of II about the same pole. Since orientation II appears in both the as rolled and recrystallized sheet, it is most likely that recrystallization occurs "in situ" although some of the recrystallized II might come from a $64^{\circ}$ rotation of I about ( 001 ) or a $70^{\circ}$ rotation of a symmetrical II orientation about the (010). In terms of the two suggested rotations, orientation V would arise from a $55^{\circ}$ rotation of II about the (010). Orientation VII, as indicated, could arise from a $44^{\circ}$ rotation of IV or a $70^{\circ}$ rotation of III around the (001).

## SUMMARY

1 - The texture in uranium sheet rolled at $300^{\circ} \mathrm{C}$ to an $85 \%$ reduction was described in terms of four "ideal orientations" with considerable scatter.

2 - The xecrystallized texture of the $300^{\circ} \mathrm{C}$ rolled material was also described by four "ideal orientations" and scatter. Only one of the "ideal orientations" used to describe the as rolled sheet was found after recrystallization.

## ACKNOWLEDGMENTS

The authors wish to express their appreciation to Mr. G. Erlebacher for working out the detailed design of the specimen table and to Mr. Robert Macherey and Mr. H. J. Luetzow under whose supervision the material for study was prepared. Thanks are also due Dr. F. Foote and Dr. S. S. Sidhu for their interest and encouragement.

## REFERENCES

1. KAPL-309, Progress Report for February 1950.
2. KAPL-415, Progress Report for September 1950.
3. W. E. Seymour, "Preferred Orientations in Uranium Sheet," KAPL-807, Report of Metallurgy Section, June-August 1952, p. 31.
4. W. E. Seymour and J. F. Duffey,"Preferred Orientations in Uranium Sheet, " KAPL-845, Report of Metallurgy Section, September-November 1952, p. 39.
5. W. E. Seymour and J. F. Duffey, "Preferred Orientation in Uranium Sheet," KAPL-930, Report of Metallurgy Section, March-May 1953. p. 49.
6. W. P. Chernock, M. H. Mueller, H. R. Fish, and P. A. Beck, ${ }^{6}$ An Automatic X-ray Reflection Specimen Holder for the Quantitative Determination of Preferred Orientation," Review of Scientific Instruments, 24, 925-928 (1953)
7. W. P. Chernock and P.A.Beck, "Analysis of Certain Errors in the $X-r a y$ Reflection Method for the Quantitative Determination of Preferred Orientations." Journal of Applied Physics, 23, 341-345 (1952).
8. W. P. Chernock and P.A. Beck, "Quantitative Determination of Rolling and Recrystallization Textures in $600^{\circ} \mathrm{C}$ and $300^{\circ} \mathrm{C}$ Rolled Uranium Rods," ANL-4839, August 1951.

14
Figure 1
pole figure showing area covered by the various sheet sections



Figure 2

## SCHEMATIC DRAWING OF THE TWO TYPES OF JIGS FOR HOLDING THE ASSEMBLED SHEET SECTIONS



Figure 3
INTEGRATING SPECIMEN TABLE FOR SHEET MATERIAL


C

Figure 4
POLE FIGURE SHOWING THE METHOD OF PLOTTING THE DATA



Figure 5
POLE FIGURE SHOWING THE POINTS AT WHICH INTENSITY DATA are obtained from the seven sheet sections


Figure 6
(010) POLE FIGURE FOR URANIUM SHEET ROLLED

AT $300^{\circ} \mathrm{C}$ TO AN $87 \%$ REDUCTION

(010)

## -

Figure 7
(110) POLE FIGURE FOR URANIUM SHEET ROLLED AT $300^{\circ} \mathrm{C}$ TO AN $87 \%$ REDUCTION

(IIO)

Figure 8
(001) POLE FIGURE FOR URANIUM SHEET ROLLED AT $300^{\circ} \mathrm{C}$ TO AN $87 \%$ REDUCTION



Figure 9
(100) POLE FIGURE FOR URANIUM SHEET ROLLED AT $300^{\circ} \mathrm{C}$ TO AN $87 \%$ REDUCTION

(100)

Figure 10
Pole Figures Showing the "Ideal Orientations" Used to Explain the Relationship Between the Intensities Found on the (010), (110), (001), and Pole Figures of the As rolled Sheet.

$O(010) \quad O$ (110) $\square(001) \quad \triangle(100)$

Figure 11
Pole Figure Showing Rotation or Movement of the "Ideal Orientations" Which May Account for the Intensity Seatier Between "Ideal Orientations" of As Rolled Sheet.


サ日णேण

$$
a
$$

Figure 12
(010) POLE FIGURE FOR URANIUM SHEET $300^{\circ} \mathrm{C}$ ROLLED AND RECRYSTALLIZED

(010)

$$
=16
$$

Figure 13
(110) POLE FIGURE FOR URANIUM SHEET $300^{\circ} \mathrm{C}$ ROLLED AND RECRYSTALLIZED

(IIO)
$+i$
Figure 14
(001) POLE FIGURE FOR URANIUM SHEET
$300^{\circ} \mathrm{C}$ ROLLED AND RECRYSTALLIZED

(001)


$$
2
$$

Figure 15
(100) POLE FIGURE FOR URANIUM SHEET $300^{\circ} \mathrm{C}$ ROLLED AND RECRYSTALLIZED

(100)

Figure 16
Pole Figures Showing the "Ideal Orientations" Used to Explain the Relationship Between the Infensities found on the (010), (110), (001), and (100) Pole Figures of the Recrystallized Sheet.

$O(010) \quad O(110) \quad \square(001) \quad \triangle(100)$
サேणिए

Figure 17
Pole Figure Showing Rotation or Movement of the "Ideal Orientations" Which May Account for the Intensity Scatter Between "Ideal Orientations" of the Recrystallized Sheet.

एकणिए

Figure 18
(131) POLE FIGURE FOR $300^{\circ} \mathrm{C}$ ROLLED URANIUM SHEET AND RECRYSTALLIZED

(131)

サGणே円?

Figure 19
SCHEMATIC DRAWING OF POSSIBLE ROTATIONS OF THE AS ROLLED "IDEAL ORIENTATIONS" WHICH MAY PRODUCE THE RECRYSTALLIZED "IDEAL ORIENTATIONS"
(OOI) ROTATION

(OIO) ROTATION

APPENDIX A

# PREFERRED ORIENTATION IN $300^{\circ} \mathrm{C}$ ROLLED URANIUM PLATE 

A. D. (Smigelskas) Fischer


#### Abstract

The preferred orientation texture of uranium $300^{\circ} \mathrm{C}$ soak rolled plate, $81 \%$ reduction, rolled in one direction was investigated by means of taking a series of $X$-ray photograms at varying angles to the plate surface and cross and rolling directions and plotting the pole figures for the (001), (010), and (021) poles. The texture was found to differ from that of $300^{\circ} \mathrm{C}$ soak rolled rod, $66 \%$ reduction, which has the single mean ( 001 ) pole radial to and a duplex mean (110) pole at an angle of $20^{\circ}$ with the rolling direction. This rolled uranium plate has duplex means of the (001) pole located $18^{\circ}$ either side of the normal to the plate and radial about the rolling direction as an axis. The mean (010) pole is located at a tilt of $18^{\circ}$ from the rolling direction and the mean ( 021 ) pole is in the corresponding position approximately $60^{\circ}$ from the ( 001 ) pole and approximately $30^{\circ}$ from the (010) pole.


## INTRODUCTION

Inasmuch as the preferred orientation textures of many uranium rods has been studied in connection with the "growth" problem of uranium upon thermal cycling, it seemed advisable to study the preferred orientation of a rolled plate under the same conditions. Rolled plate has fewer directions of freedom of plastic flow than a rolled rod; therefore, a less symmetrical texture would be expected which may lead to a better analysis as to what the structure mechanism of deformation by rolling may be. For this purpose the investigation of the texture of $300^{\circ} \mathrm{C}$ soak rolled uranium plate has been carried out. The thermal cycling growth results for the plate have been reported in TID 68, 4, $7-40,(1949)$. The texture of $300^{\circ} \mathrm{C}$ soak rolled rods and other textures will be compared with the plate texture discussed in this report.

## MATERIALS AND METHODS

The specimen history is as follows. An uranium block $1-1 / 16$ in. thick, $2-1 / 4 \mathrm{in}$. wide and $3-1 / 4 \mathrm{in}$. long was cut from a casting $2-1 / 4 \mathrm{in}$. x $3-1 / 4$ in. cross section. The piece was given approximately $10 \%$ reduction per pass at $300^{\circ} \mathrm{C}$ and was reheated 3 to 5 minutes between passes. The reduction in cross section was $81.7 \%$. The piece spread approximately

$11 \%$ during rolling. This specimen was surface ground and then sectioned into specimens 0.525 in . $x 0.425 \mathrm{in}$. with the rolling direction identified. Two of these specimens were submitted for preferred orientation determination.

A specimen annealed 2 hours at $575^{\circ} \mathrm{C}$ was shaped in the form of two rods projecting out of the edges of the plate with their axes in the rolling direction and the transverse direction, respectively. After the rods were machined from the specimen, they were electroetched in sulfuric acidglycerine bath to reduce the diameter 0.005 in . This amount was previously found adequate to remove the worked surface (ANL-5139), thus exposing the true orientation of the rod. Figure 1 A shows a sketch and the dimensions of the specimen. This method of shaping the sample is the same as that used by Bakarian. (1)

This shape was adopted because the absorption factor becomes a constant for exposures at all angles and may thus be eliminated. The absorption of X-rays by uranium is so great that only the reflected beam is recorded on the film. The intensity can be read only on $170^{\circ}$ of the diffraction ring; therefore, two sets of pictures are necessary for the pole figure of any one plane.

One hour exposures were made with a 0.025 in . diameter pinhole, using Cu radiation, $45 \mathrm{~K} . \mathrm{V} .=18 \mathrm{M} . \mathrm{A}$., type A film at 5 cm . Because of the recrystallized grain size, a large diameter rod with respect to the pinhole was necessary so that sufficient surface would be radiated. The pinhole had to be small in order to effect separation of diffraction rings which are about $3 / 4$ of a degree apart, and would otherwise overlap. Inasmuch as the beam could not cover the rod entirely, the rod was placed in the beam so that only half the beam impinged on the edge of the rod as may be seen in sketch of Figure 2A. The rod was carefully repositioned each time a photogram was taken at a different angle. Figure 3A shows a sketch of the different angular positions of the specimen with respect to the $X$-ray beam.

It was found necessary to oscillate the specimen $5^{\circ}$ about its own axis to reduce spottiness; however, even then, only two degrees of intensity could be read from the arcs on the films. Typical photograms are shown in Figure 4 A .

The pole figures were plotted in the usual way with the intensity of the various portions of the diffraction rings estimated by eye. Eleven films were made about each rod projection, the axis of rotation being the rolling direction and cross direction, respectively. All twenty-two films were read in plotting the pole figures. The series includes exposures made with the rod having the rolling direction vertical and the beam at $0,10,20,30 \ldots \ldots$ $110^{\circ}$ to the cross direction, respectively, and with the rod having the cross direction vertical and the same angulax intervals to the rolling direction.

The rolling, $81.7 \%$, is sufficient to develop a final texture. However, the texture plotted is a step integral of various depths of the sheet because the diameter of the rods cut out is the thickness of the sheet.

The crystallographic values used for orthorhombic alpha uranium $(a=2.852 \AA, b=5.865 \AA, c=4.945 \AA)$ were the following $\theta$ values:

| hkl | $\sin ^{2} \theta$ | Intensity | $\theta$ |
| :---: | :---: | :---: | :---: |
| (020) | 0.068712 | very weak | $15^{\circ} 20^{\prime}$ |
| (110) | 0.089844 | strong | $17^{\circ} 27^{\prime}$ |
| (021) | 0.092876 | strong | $17^{\circ} 45^{\prime}$ |
| (002) | 0.114008 | strong | $18^{\circ} 7^{\prime}$ |

The $\theta$ value is used to determine the reflection circle; thus, reflection circles for pole figures of planes plotted are

| Direction | Reflection | Degrees From the Center of Pole Figure |
| :---: | :---: | :---: |
| [001] | (002) | $90^{\circ}-18^{\circ} 71=71^{\circ} 531$ |
| [010] | (020) | $90^{\circ}-15^{\circ} 20^{\prime}=74^{\circ} 40^{\prime}$ |
| [021] | (021) | $90^{\circ}-17^{\circ} 45^{\prime}=72^{\circ} 15^{\prime}$ |

The angles between the poles of reflections studied and between planes used in determining the mean orientation are:

$$
\begin{aligned}
& \left.\begin{array}{l}
\mathrm{a} \wedge \mathrm{~b} \wedge \mathrm{c} \wedge=90^{\circ} \\
(100) \wedge(010) \wedge(001)=90^{\circ} \\
{[100] \wedge[010] \wedge[001]=90^{\circ}}
\end{array}\right\} \text { Orthorhombic Relations } \\
& {[001]_{\wedge}[021]=59.3^{\circ}} \\
& {[010]_{\wedge}[021]=30.7^{\circ}} \\
& {[001] \wedge[031]=68.1^{\circ}} \\
& \left.\begin{array}{l}
{[001] \wedge[013]=15.7^{\circ}} \\
{[001] \wedge[025]=18.6^{\circ}}
\end{array}\right\} \quad \begin{array}{l}
\text { Possible directions orienting } \\
\text { parallel to the rolling direction }
\end{array}
\end{aligned}
$$

## DISCUSSION OF RESULTS

The results of the $X$-ray determination are assembled in the pole figures of Figures 5A, 6A, and 7A.

The plane of the rolled plate is parallel to the plane of the stereographic projection in all the figures, with the rolling direction vertical and the transverse direction horizontal. The densities of the arcs on the films are indicated by the closeness of the cross hatching on the pole

figures. The orientation of three different sets of planes is shown, the three with most marked evidence of orientation in the films, (001), (010), and (021).

The texture found may be described in terms of a twin ideal, or mean, orientation plus a scatter, the ideal orientation being the pole of the (001) plane inclined toward the rolling direction from normal direction at a small angle, $18^{\circ}$, and the pole of the ( 010 ) plane inclined toward the normal direction to the rolling plane at $18^{\circ}$ from the rolling direction. This orientation may best be seen from the pole figures and a diagram of the mean orientation, Figure 8A.

The scatter from this mean orientation is such that the c -axis, (001) pole, tilts slightly, $8^{\circ}$, either side from the mean around cross direction as an axis and scatters $+90^{\circ}$ about the rolling direction as an axis as may be seen in Figure 5A. The b-axis shows a corresponding tilt and scatter.

Figure 9 A shows a diagram of a unit cell of alpha uranium with its axis tilted corresponding to the mean orientation of the pole figures for the rolled plate texture. From this diagram it may be seen that the (031) plane may be considered perpendicular to the cross direction; then the ( $01 \overline{3}$ ) plane is approximately at a $6^{\circ}$ tilt from the perpendicular to the rolling direction. The angle between the (031) and (013) is approximately $95^{\circ}$.

Two positions of plate direction axis, R.D. and C.D. and R.D.' and C.D.' are shown. Either is possible within the limits of scatter on the pole figures.

The plate texture compares with that of rods in the following manner:

1. While the mean (001) pole is single, radial and normal to the rolling direction in all rods investigated at ANL, it is duplex, radial, and at a tilt of $18^{\circ}$ to either side of the normal to the rolling direction in this plate.
2. The mean ( 010 ) pole of the plate is also duplex. However, it is not located similarly to that of the rod having a duplex orientation of this pole, i.e., the $300^{\circ} \mathrm{C}$ soak rolled rod which is discussed in ANL-5139. The tilt of the mean ( 010 ) pole of both that rod and of this plate are about the rolling direction. In the rod this pole has a greater angle of tilt as well as uniform radial scatter about the rolling direction, while in the plate this pole is located at a spot with the scatter about the spot.
3. The mean texture of the plate as a whole corresponds most nearly to that of the hot swaged uranium rod which has the (010) pole parallel to the rolling direction, and the (001) pole radial and normal to the rolling direction differs from it only by the twin $18^{\circ}$ tilts of the ( 001 ) pole from the normal to the plate and the ( 010 ) pole from the rolling direction.

## REFERENCE

## FIGURE IA



FORM OF SPECIMEN AFTER SHAPING AND ETCHING.

## FIGURE 2A

TOP VIEW



SKETCH OF X-RAY BEAM IMPINGING ON SPECIMEN. shows relative size and position of specimen AND BEAM.

FIGURE 3A


POSITION OF SPEGIMEN WITH RESPECT TO BEAM. SEVERAL ANGULAR POSITIONS, TOP VIEWS.

(a) $90^{\circ}$ to $\mathrm{R} . \mathrm{P}_{\text {. }}$
R. D. vertical, C. D. horizontal

(c) $90^{\circ}$ to R. P .
C. D. vertical, R. D. horizontal

(b) $20^{\circ}$ to R. $P_{0}$ R. D. vertical, C. D. horizontal

(d) $20^{\circ}$ to R. $\mathrm{P}_{\text {. }}$ C. D. vertical, R. D. horizontal

## 41

FIGURE 5A

(OOI) POLE FIGURE URANIUM ROLLED PLATE $81 \%$ REDUCTION - $300^{\circ} \mathrm{C}$ mean projection indicated by small circle

## 4

FIGURE 6A

(OIO) POLE FIGURE uranium rolled plate $81 \%$ REDUCTION - $300^{\circ} \mathrm{C}$

FIGURE 7A

(O21) POLE FIGURE uranium rolled plate $81 \%$ REDUCTION - $300^{\circ} \mathrm{C}$
שயாाए

FIGURE 8A


A STANDARD STEREOGRAPHIC PROJEGTION OF ALPHA URANIUM IS SHOWN BY OPEN CIRCLES.
THE STEREOGRAPHIC PROJECTION OF THE MEAN ORIENTATION OF the rolled plate is shown by the filled circles. THE ARROWS INDICATE HOW THE MEAN ORIENTATION IS OBTAINED FROM THE STANDARD STEREOGRAPHIC PROJECTION BY ROTATION OF $18^{\circ}$ ABOUT THE C.D. AS AN AXIS.

FIGURE 9A


