

On the Arrangement of the Micro-crystals in Rolled Platinum Plate. Part I.

By

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ABSTRACT.

By means of X-rays the arrangement of the micro-crystals in rolled platinum foil was tested by the powder method on the one hand, and by taking photographs of the X-ray spectra caused by the reflection of the rays from the surface of the foil on the other hand. The X-ray patterns thus obtained were explained by the following consideration:—The greater part of the micro-crystals in the foil are so arranged that their dodecahedral face is parallel to the surface of rolling and the normal to a trapezohedral face, which is contained in the dodecahedral face just considered, is parallel to the direction of rolling; and consequently the trigonal axis in the same dodecahedral face, which is perpendicular to the normal to the trapezohedral face just considered, is perpendicular to the direction of rolling in the rolled surface. The orientation of some of the micro-crystals deviates slightly from such ideal orientation.

Using the X-rays, many authors have investigated the arrangement of the micro-crystals in metals and they have arrived at the conclusion that the micro-crystals which were arranged rather irregularly in the metal at the beginning could be put in some regular arrangement by being subjected to some regular working such as rolling or drawing. However, it seems to the writer that the relation between the manner of the regular arrangement and the direction of the rolling or drawing is not yet known very clearly.

In 1916 Debye and Scherrer¹ and also Hull² discovered the powder method of X-ray crystal analysis and determined completely the lattice forms of the crystals of various metals. By using the results obtained by these authors the present writer attempted to determine the arrangement of the micro-crystals in thin rolled platinum plates from the X-ray

¹ Physik. Zs. **17** 277 (1916).

² Physic. Rev. **10** 661 (1917).

patterns. The X-ray examination of platinum foils was first made by Hupka,¹ and he stated that the impression on a photographic plate consisted of a central patch and many strips radiating outwards from it. Moreover he observed that, after annealing the foil, these radiating strips disappeared and many irregular spots appeared on the plate. Next M. de Broglie² observed that each of these radiating strips consisted of the spectrum of the characteristic X-rays of the anticathode employed. And Knipping³ said that the surface of the platinum foil was probably formed of the octahedral faces of the micro-crystals of this metal.

Recently H. Mark and K. Weissenberg⁴ and N. Uspenski and S. Konobejewski⁵ investigated mainly about Ag and Al foils, and determined the orientation of the micro-crystals in these foils. Moreover they concluded that the micro-crystals of other metals belonging to the face-centered cubic lattice would be arranged in a similar orientation under similar working. Former authors had declared that there were two non-related groups of lattice positions, of which the first was characterised by the $[112]$ axis parallel to the direction of rolling and the (110) plane parallel to the rolled plane, and the second, by the $[100]$ axis parallel to the direction of rolling and the (100) plane parallel to the rolled plane. But this second group was not found by the latter authors.

The specimen of the platinum foil used in the present experiment was prepared as follows:—The pure platinum was melted in the furnace and hammered in a hot state to a parallelepiped of suitable size, the thickness of which was about 0.5 mm. After being heated once more to a temperature just below the melting point, it was quenched in cold dilute hydrochloric acid. Then this was very carefully rolled down to a thin foil of 0.03 mm. thickness by being passed through the rollers 15 times. The roller was composed of two steel bers 15 cm. in diameter, which were rotated 10 times in a minute. In the present experiment the Coolidge tube having a molybdenum anticathode was excited by an induction coil. The maximum voltage applied to the tube was about 50–60 K. V., and the current through it was 2–3 milliamperes.

First the writer adopted the ordinary transmission method of photographing the Laue spot, the specimen being exposed to a small cylindrical beam of X-rays of 2 mm. in diameter and the interference

1 *Physik. Zs.*, **14**, 263 (1913).

2 *C. R.*, **158**, 333 (1914).

3 *Physik. Zs.*, **14**, 996 (1913).

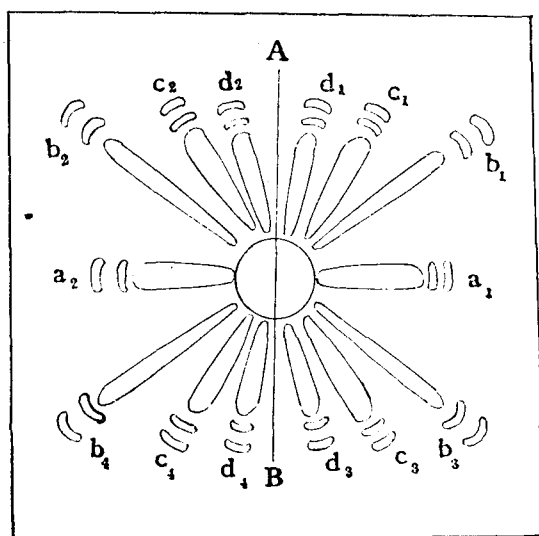
4 *Zs. f. Physik.*, **14**, 328 (1923); **16** 314 (1923).

5 *Zs. f. Physik.*, **16**, 215 (1923).

figures being taken on a photographic plate placed at some distance behind the specimen. The specimen with which the experiment was performed had been rolled up and down along one straight line. When the photographic plate and the specimen were set perpendicular to the incident beam, the photograph reproduced in Fig. 1 was obtained after an exposure of about 20 hours, and it was represented diagrammatically in Fig. 1'.

It will be easily noticed that 14 prominent bands radiate outwards from the central spot and that each band has two clear spectral lines near its end.

Fig. 1'.



near its end. In order to explain this interference figure it will naturally be considered that:—1. the micro-crystals in the metal are mostly so arranged that they have a definite common axis parallel to the direction of rolling and any reflecting plane is inclined with a definite angle to this direction but that in other respects the orientation of the reflecting plane is irregular, 2. each radiating band is produced by the reflection of X-rays from a

certain crystal face and the two spectral lines are K_{α} and K_{β} doublets of molybdenum.

The analysis of the interference figure thus caused was given by S. Nishikawa and S. Ono¹ and lately by A. Ono² from the angular distribution of the radiating bands. But in addition to the angular distribution of the radiating bands the writer was now able to determine the reflecting plane of the crystals belonging to each radiating band from the position of the spectral lines. And the common axis of the crystals supposed to be arranged in the direction of rolling, was found with these data.

In the present experiment the distance between the specimen and the

1 Su. Butu. Kw. K., Tokyo., 7, 131 (1913-1914).

2 Mem. Coll. Engin. Kyushu., 2, 241 (1922).

photographic plate was 7.24 cm. Then by knowing the distance l between the central spot and any spectral line, the glancing angle θ of the X-rays on the reflecting plane of the crystals corresponding to this line will be calculated by the following equation,

$$\tan 2\theta = \frac{l}{7.24} \dots\dots\dots (1)$$

Since the wave lengths λ of K_α and K_β doublets of molybdenum are 0.710 and 0.630 Å. U. respectively, the distance d between the consecutive atomic planes will be obtained at once by Bragg's equation,

$$\lambda = 2d \sin \theta. \dots\dots\dots (2)$$

The numerical values of l observed and the values of θ and d thus calculated are tabulated in Table I.

Table I.

Radiating Band	$l_{\text{cm.}}$		θ	$d_{\text{Å.}}$ (obs.)	Indices of Planes	Result obtained by Hull	
						$d_{\text{Å.}}$	Indices of Planes
a_1, a_2	K_α	2.38	$9^\circ 6'$	2.24	(III)	2.266	(III)
	K_β	2.06	$7^\circ 59'$	2.26			
b_1, b_2, b_3, b_4	K_α	2.75	$10^\circ 25'$	1.96	(100)	1.964	(100)
	K_β	2.43	$9^\circ 18'$	1.95			
c_1, c_2, c_3, c_4	K_α	4.10	$14^\circ 47'$	1.38	(110)	1.390	(110)
	K_β	3.55	$13^\circ 6'$	1.39			
d_1, d_2, d_3, d_4	K_α	2.36	$9^\circ 4'$	2.25	(III)	2.266	(III)
	K_β	2.04	$7^\circ 57'$	2.27			

By means of the powder method, Hull¹ determined the lattice form of the crystal of platinum and showed that it belongs to the face centered cubic lattice. The distances d , obtained by him, between the consecutive atomic planes of various indices are given in the last column of the same table. Comparing these data with the present ones, it will readily be seen that the values of d corresponding to each band coincided very well with those obtained by Hull for the prominent reflecting planes. Thus the same indices as those given by Hull were assigned to each reflecting plane which has the same value of d . The indices thus obtained are given in the fifth column of Table I.

1 Physic. Rev., 17, 571 (1912).

The angles between the bands and the line of symmetry AB in Fig. 1', which is parallel to the direction of rolling of the specimen, are given in Table 2.

Table 2.

Radiating Band	Angle φ
a_1, a_2	90°
b_1, b_2, b_3, b_4	54°
c_1, c_2, c_3, c_4	36°
d_1, d_2, d_3, d_4	$17^\circ 30'$

Since the atomic arrangement of the crystal of platinum follows that of the face centered cubic lattice, it was supposed at first that the common axis of the crystals, which was supposed to be parallel to the direction of rolling, would be the tetragonal, the trigonal or the diagonal axis. But it was found immediately that, with any of them, it was impossible to explain satisfactorily the angular distribution of the bands above mentioned, because when the tetragonal or the trigonal axis is taken as the common axis, no radiating band formed by the reflection from the plane (111)

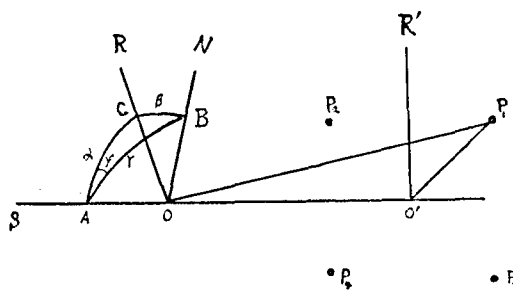
should appear at the angle of 90° from the line AB, which is contrary to the actual observation. Next when the diagonal axis is taken as the common axis, a radiating band formed by the reflection from the plane (100) should appear at the angle of 45° ; whereas no trace of such band was detected in the figure.

Next a line passing through a corner atom and the atom lying at the center of one of the opposite faces of the elementary cube was taken as the common axis of the crystals. The common axis thus considered coincides with the normal to one of the

planes (112). And, on the supposition that the micro-crystals in the foil are arranged at random in other respects, a mathematical calculation was made to see whether the angular distribution of the radiating bands could be accounted for by these considerations or not.

Now the analysis made by the authors before mentioned is repeated briefly. In Fig. 2, SO represents the X-ray beam which makes an angle α with the common axis of the crystals OR. ON is the normal of a reflecting plane and makes an angle β and γ with OR and SO respectively. Then the ray reflected from this plane will proceed along

Fig. 2.

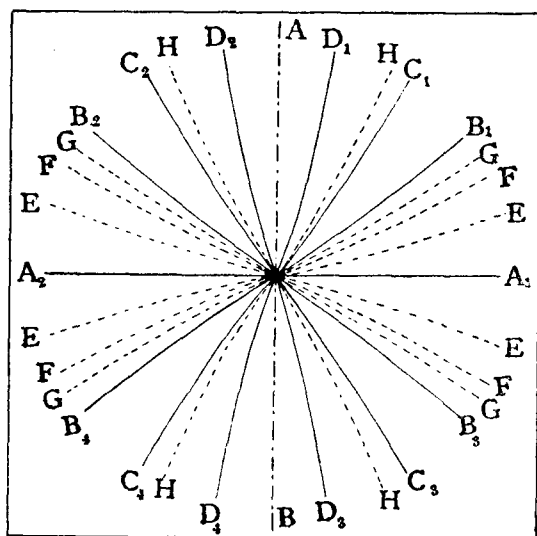


$\bullet P_2$ $\bullet P_3$

OP_1 which is in the plane of incidence and makes the angle γ with ON . Thus the angle γ is the complement of the glancing angle θ . The intersections of the planes SOR and SON with the photographic plate standing normally to the incident beam at O' are represented by $O'R'$ and $O'P_1$ respectively. In the spherical triangle ABC , we have the following relation

$$\cos \beta = \cos \gamma \cos \alpha + \sin \gamma \sin \alpha \cos \varphi, \dots\dots\dots (3)$$

Fig. 3.



where φ is the angle between the planes SOR and SON or the angle $R'O'P_1$.

If the values of α , β and γ are given, the value of φ will be obtained by the equation (3), and the position of P_1 on the photographic plate can be denoted by this value of φ and the distance l of P_1 from the position of the central spot O' . This value of l will be calculated by the equation (1) or

$$-\tan 2\gamma = \frac{l}{OO'}. \dots\dots\dots (4)$$

In the present case the normals to the planes denoted by the definite indices of the micro-crystals are supposed to be distributed on the surface of a cone formed by the revolution of the line ON around the axis OR . Thus if the reflection of the X-rays of a definite wave length λ from a certain reflecting plane occur at a certain position in the plane, four different orientations of such a plane will be possible generally, and four different spots as P_1, P_2, P_3 and P_4 in Fig. 2 will be impressed on the photographic plate. The positions of these four points will at once be determined by the equations (2), (3) and (4). For the X-rays of various wave lengths we obtain four different groups of interference spots which are nothing but the four curves radiating outwards from the central spot on the photographic plate.

Table 3.

Planes	Cos β	β	Curves in Fig. 3
100, 010	0.408	65° 55'	F
001	0.816	35° 20'	C ₁ C ₂ C ₃ C ₄
110	0.577	54° 45'	B ₁ B ₂ B ₃ B ₄
$\bar{1}10$	0	90°	A ₁ A ₂
101, 011.	0.866	30°	H
$\bar{1}01, 0\bar{1}1.$	0.289	73° 15'	E
111	0.943	19° 20'	D ₁ D ₂ D ₃ D ₄
$\bar{1}\bar{1}1, 1\bar{1}\bar{1}.$	0.473	61° 50'	G
$\bar{1}\bar{1}\bar{1}$	0	90°	A ₁ A ₂

Now taking the [112] axis as the common axis OR of the crystals, and putting $\alpha=90^\circ$, the curves represented in Fig. 3 corresponding to various possible values of β as given in Table 3 were obtained. The values of β thus used were calculated in the following manner. Taking a corner atom as the origin, and the edges of the cubic lattice as the rectangular coordinate axes, the direction cosines of the normal ON to a plane with the indices $h_1 h_2 h_3$ are

$$l = \frac{h_1}{\sqrt{h_1^2 + h_2^2 + h_3^2}}, \quad m = \frac{h_2}{\sqrt{h_1^2 + h_2^2 + h_3^2}}, \quad \text{and} \quad n = \frac{h_3}{\sqrt{h_1^2 + h_2^2 + h_3^2}}.$$

Now let $l', m',$ and n' be the direction cosines of the common axis OR of the micro-crystals, which are supposed to be arranged in the direction of rolling. Then the angle β between OR and ON will be given by

$$\text{Cos } \beta = \frac{h_1 l' + h_2 m' + h_3 n'}{\sqrt{h_1^2 + h_2^2 + h_3^2}} \dots\dots\dots (5)$$

Now every corner atom of the elementary cube of the crystal may be taken equally well as the origin of the coordinates and any three edges of the elementary cube of the crystal may be taken equally well as the three axes of the rectangular coordinate axes, as they are in the same

geometrical position with respect to the atomic orientation in the elementary cube. Consequently there are a number of different ways of taking the (112) plane in the elementary cube. This number will be 24 in all, and any one of the normals to these 24 planes may be considered as the common axis of the micro-crystals supposed to be arranged in the direction of rolling. Now if we take a corner atom of the elementary cube as the origin of the coordinates and the three edges of the cube meeting at this corner atom as the coordinate axes, the direction cosines of these normals will be obtained by the different combinations of the following numbers $\pm\frac{1}{\sqrt{6}}$, $\pm\frac{1}{\sqrt{6}}$ and $\pm\frac{2}{\sqrt{6}}$. Thus, for examples,

$l' = \frac{1}{\sqrt{6}}$, $m' = \frac{1}{\sqrt{6}}$ and $n' = \frac{2}{\sqrt{6}}$ will be one combination. Assigning

proper values to the direction cosines of the normals to the planes (100), (110) and (111), and taking every possible combination of the values of l' , m' and n' , all the possible values of β corresponding to the above three reflecting planes will be calculated by the equation (5). So far as we are concerned with the calculation of all possible values of β , it will be the same if we give the fixed values $l' = \frac{1}{\sqrt{6}}$, $m' = \frac{1}{\sqrt{6}}$ and $n' = \frac{2}{\sqrt{6}}$

for the direction cosines of any one of the normals to the planes (112), and take all possible sets of indices for each group of similar reflecting planes. The values thus

Table 4.

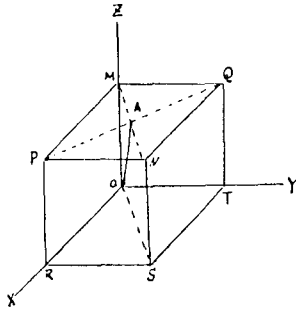
Indices of Planes	φ	
	calc.	obs.
111	17° 10'	17°
001	33° 50'	35°
110	53° 20'	53°
11 $\bar{1}$	90°	90°

calculated are tabulated in Table 3. Here it is to be noted that the two planes denoted by indices of opposite signs are parallel to each other, and consequently we have taken only the positive values of $\cos \beta$. By comparing the curves of Fig. 3 with Fig. 1 or Fig. 1', it was found that the curves corresponding to $\beta = 19^\circ 20'$; $\beta = 35^\circ 15'$; $\beta = 54^\circ 45'$ and $\beta = 90^\circ$ coincided exactly with the radiating bands denoted by $d_1 d_4 d_3 d_2$, $c_1 c_4 c_3 c_2$, $b_1 b_4 b_3 b_2$, and $a_1 a_2$ in Fig. 1' respectively. The values of φ calculated for these values of β and those observed on the photograph for the K_α doublet of molybdenum are tabulated in Table 4. We can see from this table that the agreement is within the limit of experimental errors.

There are many curves in Fig. 3 which do not appear on the photograph as radiating bands. The reason for this may be explained as follows:—

In Fig. 4 OX, OY and OZ are the rectangular coordinate axes parallel to the edges of the elementary cube. Now suppose that the common axis OA, whose direction cosines are $\frac{1}{\sqrt{6}}, \frac{1}{\sqrt{6}}, \frac{2}{\sqrt{6}}$, becomes parallel to the direction of rolling and the plane $\bar{1}10$ (MNSO) becomes parallel to the surface of rolling. The beam of X-rays projected normally to this surface will impinge along the diagonal axis PQ whose direction cosines are $\frac{-1}{\sqrt{2}}, \frac{1}{\sqrt{2}}, 0$. The glancing angles θ of this ray to the predominant reflecting atomic planes of the crystals are calculated and given in Table 5 together with the values of β .

Fig. 4.



These values show that the incident beam is parallel to the planes $001, 110, 111$ and $11\bar{1}$, the values of β corresponding to these planes being $35^{\circ}20', 54^{\circ}45', 19^{\circ}20'$ and 90° respectively. As these values coincide exactly with those given in Table 4, we can conclude that the reflection of the X-rays is detectable on the photograph only when the reflecting planes are nearly parallel to the incident beam. If there are some micro-crystals oriented a little differently from the above position, the reflection from the above four

planes will occur, but the other planes will not cause the reflection because of their great inclination to the incident beam. Now we shall consider this fact more precisely. If the arrangement of the micro-crystals is such as has just been considered, the dodecahedral faces (110) of the crystal may be perpendicular to the incident beam, i. e. they are arranged in the plane of rolling as is seen from Table 5 and the normal to the trapezohedral faces (112) which is supposed to be arranged in the direction of rolling is contained in the face (110) . There are two kinds of such orientation of the micro-crystals and they are represented diagrammatically in Fig. 5 and Fig. 6.

The one orientation is obtained from the other by the rotation of 180° around the common axis QO . In both cases a dodecahedral faces $LQNS$ is parallel to the surface of rolling and the X-ray beam is supposed to impinge along a diagonal axis \vec{MK} or \vec{KM} which is parallel to the faces $KLMN, KMS, KMRP$ and KMQ . Now suppose that the crystal in Fig. 5 is rotated clockwise around OQ with a small angle, the reflection of the X-ray beam from the faces above mentioned will occur and the

interference figure represented diagrammatically in Fig. 7 will be obtained. When the rotation is counter-clockwise the interference figure represented in Fig. 8 will be obtained. Similarly, Fig. 9 and Fig. 10 will be obtained from the crystal of Fig. 6 by rotating it clockwise and counter-clockwise respectively around the common axis. The curves in Fig. 3 denoted by the same marks as those in these figures correspond to the same interference figure.

Table 5.

Planes	θ	β
100	45°	65° 55'
010	45°	65° 55'
001	0	35 20'
110	0	54° 45'
$\bar{1}10$	90°	90°
101	30°	30°
$\bar{1}01$	30°	73° 15'
011	30°	30°
0 $\bar{1}1$	30°	73° 15'
111	0	19° 20'
$\bar{1}\bar{1}\bar{1}$	0	90°
$\bar{1}11$	54° 45'	61° 50'
1 $\bar{1}\bar{1}$	54° 45'	61° 50'

Among the curves in Fig. 3 all those marked by the letters $A_1 A_2, B_1 B_2 B_3 B_4, C_1 C_2 C_3 C_4$ and $D_1 D_2 D_3 D_4$ appeared on the photograph as was stated before. As all these curves are obtained by the superposition of those represented in the figures from Fig. 7 to Fig. 10, and it will be natural to consider that the arrangement of the micro-crystals in the metal is such as that composed of all the kinds of rotation above mentioned.

The reason for the absence of the interference bands represented by the dotted lines in Fig. 3 has already been considered to be due to the fact that the reflecting planes of the micro-crystals for these bands are too much inclined to the incident rays. If so, these must appear on the photograph when a sample is placed in a position rotated at a proper angle about the common axis, that is, about the direction of rolling. In this case the X-rays strike the surface of rolling or MNOS in Fig. 4 obliquely. Now

suppose that the X-ray whose direction cosines are l, m, n , makes the angle α and Φ with the axis OA and the trigonal axis MS in Fig. 4 respectively. As the values of the direction cosines are $\frac{1}{\sqrt{6}}, \frac{1}{\sqrt{6}}$ and $\frac{2}{\sqrt{6}}$ for the axis OA, and $\frac{1}{\sqrt{3}}, \frac{1}{\sqrt{3}}$ and $\frac{-1}{\sqrt{3}}$ for the trigonal axis MS, we obtain the following simultaneous equations,

$$\frac{l}{\sqrt{3}} + \frac{m}{\sqrt{3}} - \frac{n}{\sqrt{3}} = \cos \Phi,$$

Fig 5.

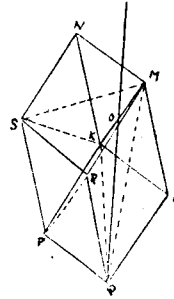


Fig. 7.

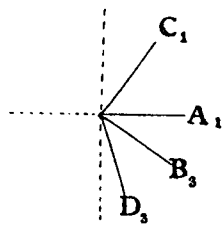


Fig. 8.

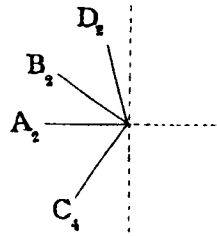


Fig. 6.

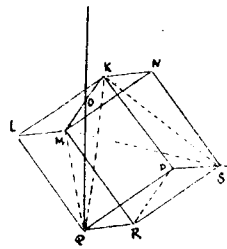


Fig. 9.

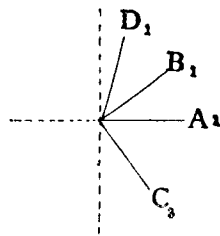
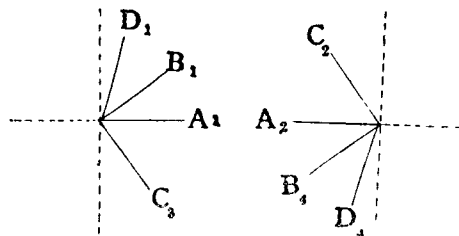


Fig. 10.



$$\frac{l}{\sqrt{6}} + \frac{m}{\sqrt{6}} + \frac{2n}{\sqrt{6}} = \cos \alpha,$$

$$l^2 + m^2 + n^2 = 1.$$

Hence we have

$$\left. \begin{aligned} l &= \frac{2\sqrt{3} \cos \Phi + \sqrt{6} \cos \alpha \pm \sqrt{18(1 - \cos^2 \Phi - \cos^2 \alpha)}}{6}, \\ m &= \frac{2\sqrt{3} \cos \Phi + \sqrt{6} \cos \alpha \mp \sqrt{18(1 - \cos^2 \Phi - \cos^2 \alpha)}}{6}, \\ n &= \frac{\sqrt{6} \cos \alpha - \sqrt{3} \cos \Phi}{3}. \end{aligned} \right\} \dots\dots\dots (6)$$

When the X-ray is projected normally to the direction of rolling, the equation (6) will be simplified as follows by putting $\alpha = 90^\circ$,

$$l = \frac{2\sqrt{3} \cos \Phi \pm 3\sqrt{2} \sin \Phi}{6},$$

$$m = \frac{2\sqrt{3} \cos \Phi \mp 3\sqrt{2} \sin \Phi}{6},$$

$$n = \frac{-\sqrt{3} \cos \Phi}{3}.$$

By calculating the values of l , m , n , corresponding to 60° , 70° , 80° , 90° , 100° , 110° and 120° for the value of Φ , we are able to calculate the glancing angle θ of the X-ray to the planes (hkl). The values of θ thus calculated are tabulated in Table 6. In the case of normal incidence of the X-rays to the rolled surface of the specimen, we have considered that the axis (112) of the micro-crystals are arranged in the direction of rolling and the faces (110) of the crystals are situated roughly parallel to the rolled surface of the foil. Though the majority of the faces (110) will be nearly parallel to the rolled surface, some of them will be situated in the position rotated through some angles around the direction of rolling *i. e.* the common axis of the crystals. If the specimen is placed in the position rotated through 30° for example, from the position of normal incidence, around the direction of rolling, the values of Φ for all the micro-crystals will not always be $60^\circ = 90^\circ - 30^\circ$ or $120^\circ = 90^\circ + 30^\circ$. Some will be smaller or greater than these values, and they will be distributed around these two values. Thus the reflection of the X-rays will occur from the faces of the crystals which have a small glancing angle θ in table 6 corresponding to the values 60° and 120° of Φ . Thus, taking up the values of θ smaller than 20° , for example in the third and the last column of table 6, we can expect that the radiating bands produced

by the planes 100, 010, $\bar{1}01$, $0\bar{1}1$ will be obtained first in addition to those obtained in the case of the normal incidence. This experiment was actually carried out and the expectation was verified. When the specimen was rotated at a certain angle around the direction of rolling, the interference bands became longer and some new bands appeared on one side of the photograph and those on the opposite side became shorter and gradually disappeared in accordance with the consideration before mentioned.

Table 6.
The values of θ

Planes.	β	Φ						
		60°	70°	80°	90°	100°	110°	120°
001	35° 20'	17°	11°	6°	0°	6°	11°	17°
010	65° 55'	19°	28°	35°	45°	51°	60°	66°
100	65° 55'	66°	60°	51°	45°	35°	28°	19°
110	54° 45'	24°	16°	8°	0°	8°	16°	24°
$\bar{1}10$	90°	61°	70°	73°	90°	73°	70°	61°
101	30°	26°	28°	29°	30°	29°	28°	26°
$\bar{1}01$	73° 15'	58°	49°	39°	30°	20°	11°	2°
011	30°	26°	28°	29°	30°	29°	28°	26°
$0\bar{1}1$	73° 15'	2°	11°	20°	30°	39°	49°	58°
111	19° 20'	10°	7°	3°	0°	3°	7°	10°
$\bar{1}11$	61° 50'	63°	62°	57°	55°	46°	41°	33°
$1\bar{1}1$	61° 50'	33°	41°	46°	55°	57°	62°	63°
$11\bar{1}$	90°	30°	20°	10°	0°	10°	20°	30°

One of the photographs is reproduced in Fig. 11. The specimen was rotated at 30°, from the position of normal incidence, around the direction of rolling in this case. The calculated and the observed values of φ corresponding to the K_{α} doublet of molybdenum in one quadrant of the photograph are tabulated in Table 7. As the bands are very diffuse and moreover the intensities of some of them are very weak on the photograph, it is very difficult to measure the values of φ accurately. Thus taking these facts into consideration, the agreement seems to be within the limit of experimental errors.

Table 7.

Planes	φ	
	Calc.	Obs.
111	17° 10'	17°
001	33° 50'	35°
110	53° 20'	52° 30'
$\bar{1}\bar{1}\bar{1}$, $1\bar{1}\bar{1}$	61° 25'	} 63°
100, 010	65° 25'	
$\bar{1}0\bar{1}$, $0\bar{1}\bar{1}$	72° 40'	74°
11 $\bar{1}$	90°	90°
$\bar{1}\bar{1}0$	90°	90°

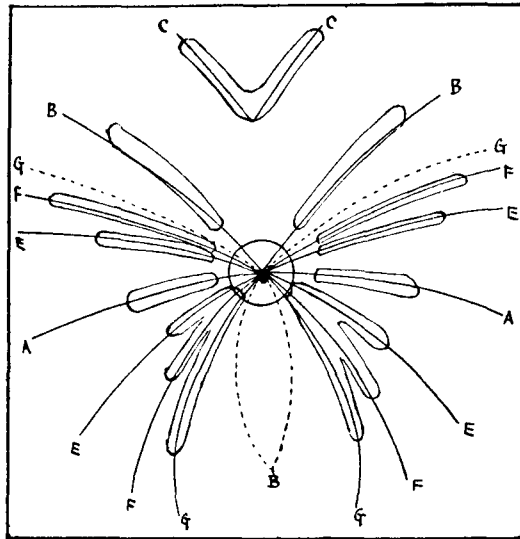
accurately the position of the spectral lines in Fig. 13, the writer placed the curve upon the photograph and sketched the radiating bands on the curves as shown in Fig. 12. The radiating bands corresponding to the dotted lines were not detected on the photograph. This may be explained in a similar way as in the former case. The glancing angles θ between the X-ray and the reflecting planes (hkl) were calculated by putting $\alpha = 45^\circ$, and $\Phi = 60^\circ, 70^\circ, 80^\circ, 90^\circ, 100^\circ, 110^\circ, 120^\circ$, respectively in the equation (6) and tabulated in Table 8.

As was considered before, the faces (110) of all the micro-crystals are not arranged exactly parallel to the rolled surface of the foil. Some of them are rotated at some angle about the common axis. Thus the values of Φ are not exactly 90° for all the crystals, they are greater or smaller for some crystals.

Next if the sample is rotated about the line perpendicular both to the common axis and the incident ray i.e. about the line parallel to the trigonal axis MS in Fig. 4. the value of α is different from 90° , and curves obtained from the above equations (3) and (4) are rather complicated. Assigning 45° to the value of α we obtain the curves represented by the continuous and the dotted lines in Fig. 12. The photograph taken under such conditions is reproduced in Fig. 13. The distance between the specimen and the photographic plate was 6.8 cm. in this case.

As it was impossible to observe

Fig. 12.



Keeping this fact in mind the writer calculated the values of θ corresponding to the various values of Φ ranging from 60° to 120° . From table 8 it will easily be recognised that the reflection from the planes $\bar{1}10, 101, 111, 1\bar{1}1$ can not occur because of the great glancing angle θ . Moreover we can not detect the presence of the radiating bands caused by 011 on the photograph. The reason may be partly due to the fact that this face has not a small glancing angle to the X-rays, and partly

Table 8.
The values of θ

Planes	Φ							
	β	60°	70°	80°	90°	100°	110°	120°
001	$35^\circ 20'$	16°	22°	28°	35°	42°	51°	60°
010	$65^\circ 55'$	12°	2°	5°	12°	17°	20°	21°
100	$65^\circ 55'$	68°	66°	61°	52°	42°	30°	21°
110	$54^\circ 45'$	54°	43°	33°	24°	15°	6°	$0'$
$\bar{1}10$	90°	30°	37°	43°	45°	43°	37°	$30'$
101	30°	60°	66°	74°	76°	74°	66°	$60'$
$\bar{1}01$	$73^\circ 15'$	27°	22°	16°	8°	0°	11°	21°
011	30°	21°	18°	16°	15°	16°	18°	21°
$0\bar{1}1$	$73^\circ 15'$	3°	14°	24°	34°	44°	53°	60°
111	$19^\circ 20'$	56°	51°	47°	42°	38°	32°	30°
$1\bar{1}1$	$61^\circ 50'$	35°	46°	58°	66°	73°	70°	$65'$
$\bar{1}11$	$61^\circ 50'$	14°	16°	16°	14°	10°	2°	$5'$
$1\bar{1}\bar{1}$	90°	30°	20°	10°	0°	10°	20°	30°

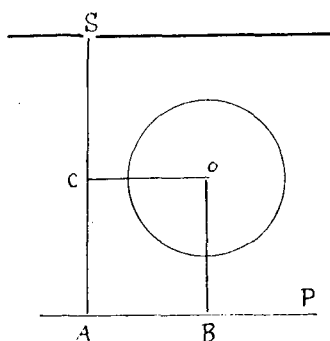
due to the weak intensity. Next as regards the bands by B and G in Fig. 12 we can detect only one part of them respectively and the remaining parts beyond the central spot can not be observed on the photograph. This may be caused partly by the weak intensity due to the lack of the reflecting planes of the crystals responsible for the reflection of X-rays, as the zero-value of θ corresponding to the value of Φ deviated too much from 90° in both cases. Disregarding these minor ambiguous cases we may say, from Fig. 12 that the prominent radiating bands occupied nearly the positions expected by the theory.

Thus considering, it may be concluded that the micro-crystals in the

rolled platinum plate are mostly so arranged that the normal to the plane (112) is parallel to the direction of rolling and their dodecahedral faces are arranged mostly parallel to the surface of rolling. This latter statement is not true for all the micro-crystals: the dodecahedral faces of some of them take the positions rotated through some angles, about the direction of rolling, from being parallel to the surface of rolling.

These considerations were also tested by the method of reflection of X-rays from the surface of the rolled foils. The writer employed the same apparatus as the bent mica X-ray spectrometer which was used by Prof. Yoshida¹ and others.² A slightly divergent X-ray beam from the slit *S* in Fig. 14 was projected on the surface of the platinum foil which was carefully wound on the surface of a wooden cylindrical rod *O*.

Fig. 14.



With this apparatus the reflection occurs at different parts of the foil with various incident angles. These reflected X-rays were made to strike the photographic plate *P* placed behind the cylinder. The radius of the cylindrical platinum foil was 36.16 mm., the perpendicular distance *SA* from the slit to the film side of the photographic plate was 13.00 cm., the perpendicular distance *OB* from the center of the cylindrical foil to the film side of the photographic plate was 5.21 cm. and the perpendicular distance *OC* from *O* to the line *SA* was 4.17 cm. With an exposure of about twelve hours four prominent spectral lines which were supposed to be those of K_{α} and K_{β} of molybdenum appeared on the photographic plate after development. Now supposing that the reflecting planes are parallel to the surface of the rolled foil, and if we measure the distance f from the zero position to the spectral lines, the spacings d of the atomic planes corresponding to the each spectral line can be calculated from the data given above. The results of calculation are tabulated in Table 9.

These results show that the first two spectral lines are those reflected from the plane (110) as was expected from our consideration and the last two are those reflected from the planes (210). Here it must be noted that the lines reflected from the planes (210) are very weak com-

¹ These Memoirs, 4, 343 (1921).

² Yoshida and Tanaka: These Memoirs, 5, 173 (1922). Tanaka and Tsutsumi: These Memoirs, 7, 1 (1923).

pared with those reflected from the plane (110). The fact that the planes (210) are arranged parallel to the surface of the foil seems to be explainable by our previous consideration. The angles between the planes (110) and (210) are $18^{\circ}30'$ and $72^{\circ}30'$, and the planes (210) which make an angle of $18^{\circ}30'$ with the plane (110) are not very much far from being parallel to the latter. If the planes (110) of some of the micro-crystals are situated in the positions rotated at some small angles about the direction of rolling as was supposed before, the planes (210) of some such crystals will become parallel to the surface, but other planes of higher atomic density will not become parallel to the surface, for the

Table 9.

No. of lines	Distance f_{cm}	Pt. foil.		Calculated spacing \AA U.	Planes
		Spacing $d_{\alpha}^{\text{\AA U.}}$ from K_{α}	Spacing $d_{\beta}^{\text{\AA U.}}$ from K_{β}		
Rolled Pt. foil.					
1	2.67		1.37	1.39	(110)
2	3.08	1.37			
3	4.70		0.880	0.88	(210)
4	5.55	0.905			
Beaten Au foil.					
	1.70		2.00	2.04	(100)
	1.95	1.98			

angles between these planes and the planes (110) are too great. This seems to be the reason why the spectral lines reflected from the planes (110) and the planes (210) are obtained.

The photograph, were taken under two different conditions. At first the slit was made parallel to the direction of rolling of the foil and next it was made perpendicular. Though all the spectral lines before mentioned appeared at the same positions on the photographic plate, there was a little difference in their sharpness in the two cases. In the former case the spectral lines were more diffuse in their breadth but the upper and

lower ends of the lines were more sharply terminated than in the latter case. This seems to be easily understandable from the fact that the planes (110) are not all arranged exactly parallel to the rolled surface, but some are rotated at some angles about the direction of rolling, as was considered before. The photographic plate taken when the slit was made perpendicular to the direction of rolling of the foil, is reproduced in Fig. 15.

Lastly the above cylindrical platinum plate was wrapped in a thin gold leaf of 0.0001 mm, in thickness, and a photograph was taken. The intensities of the spectral lines due to the reflection from the platinum foil were not much weakened and moreover the lines reflected from the crystals in the gold leaf appeared on the photograph as is seen from Fig. 16.

This shows that the cause of the reflection of the X-rays from the platinum plate is not necessarily limited to the micro-crystals situated at the surface of the foil.

The distances between zero position and the spectral lines reflected from the crystals in the gold leaf were 1.95 and 1.70 cm. The spacings calculated from the preceding data are given in the lower half of Table 9.

From this Table it will be seen that the reflecting surfaces are the planes (100) of gold crystals. This result is coincident with that obtained by W. H. Bragg¹ with the method of rotating crystals. As was stated by Bragg, it is possible to obtain sharp spectral lines even with the micro-crystals distributed at random in the foil with the method of rotating crystals. But with the cylindrical spectrometer now used, it seems to be impossible to obtain a sharp spectral line from the micro-crystals distributed at random in the foil for all the position of the photographic plate. Thus this ambiguity is eliminated in our case by placing the photographic plate in a proper position. This was actually tested with annealed platinum foil. Actually in the present experiment the divergency of the beam was about 4° and with this foil no sharp spectral line was detected, and only some very faint diffused bands corresponding to the reflection from the irregularly arranged crystals were found on the photographic plate.

In conclusion the author wishes to express his sincere thanks to Prof. Mizuno for the interest he has taken in the present research, and also to Prof. Yoshida for his kind guidance.

¹ Nature, **113**, 639 (1924).

Fig. 1.

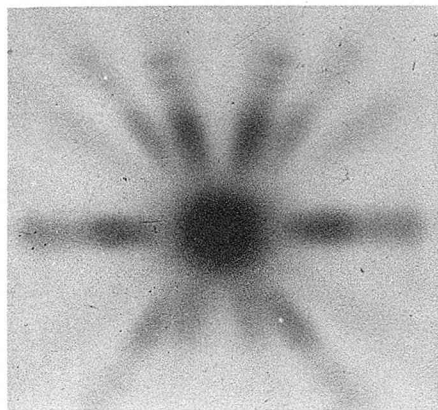


Fig. 13.

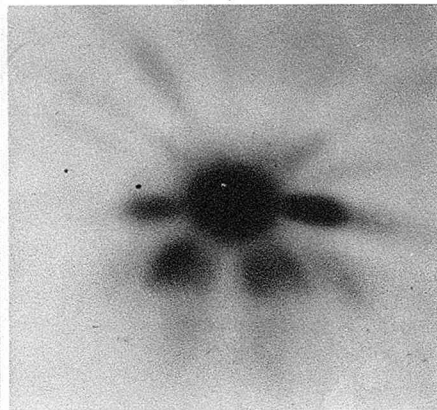


Fig. 11.

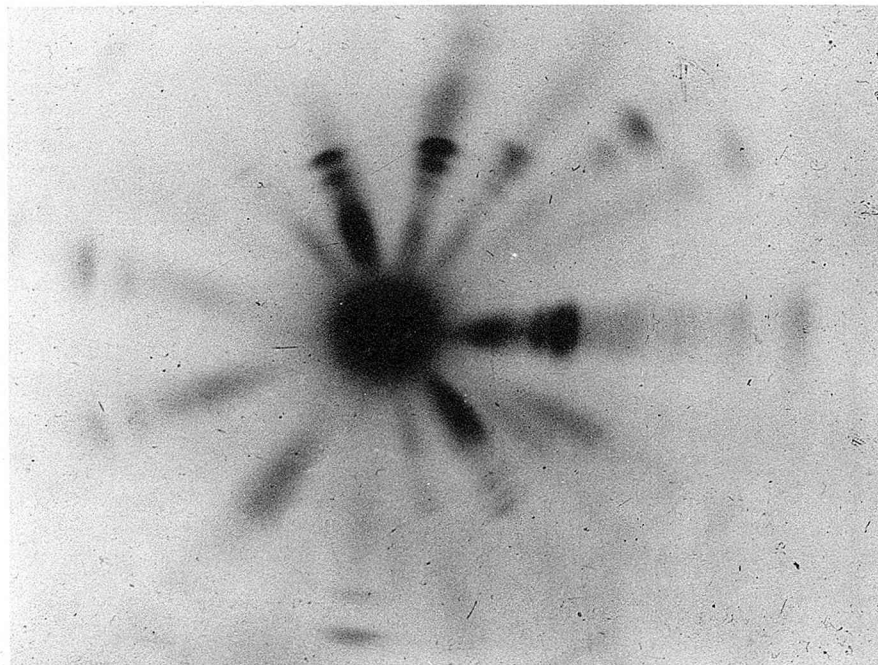
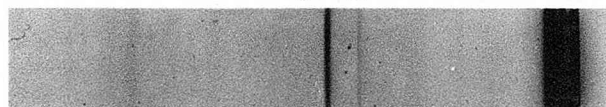


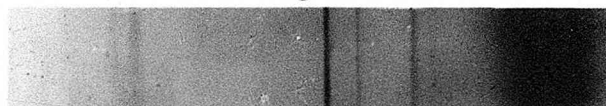
Fig. 15.



(210)

(110)

Fig. 16.



(210)

(110)

(100) Au