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# On the Development of X-Ray Stress Measurement Technique Using X-Ray Diffraction by Crystal Oscillation Method. I,

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## Synopsis

In the present paper, to provide information on the stress measurement in coarse grained materials by X-ray micro-beam diffraction technique using a crystal oscillation method, the authors first examined experimentaly and theoretically the relation between the sizes of X-ray beam and crystal to obtain the particular diffraction ring in the case of use of crystal oscillation method.

The specimen attachment of X-ray camera used in this experiment can be oscillated automatically around a horizontal and vertical axes with high accuracy centering around an illuminated position on the specimen surface. Accordingly it is possible to increase the number of the diffraction spots without changing the area and position of the specimen illuminated.

Experiments were carried out for three kinds of annealed low carbon steel with grain sizes of about 15, 30 and 50$\mu$ in diameter, and with X-ray beam collimated by pinhole slits of 0.12, 0.30 and 1.00 mm in diameter, using CrK$\alpha$ characteristic X-rays.

On the other hand, a theoretical analysis was carried out according to the X-ray diffraction theory which have been proposed by P. B. Hirsch *et al.*

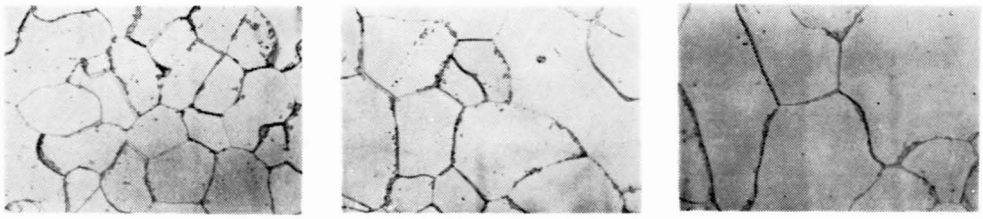
As the conclusion, it is found that the crystal oscillation method is extremely useful for X-ray stress measurement of coarse grained materials. Moreover, the conditions of the crystal oscillating operation were clarified theoretically for any pair of the sizes of X-ray beam and crystal.

## . Introduction

X-ray stress measurement is sole method of ɜ local stress measurement. The value of the ess obtained by this method is related to the sition of X-ray diffraction line which consis- of the spots diffracted from the crystals sati- ing the Bragg's condition. Accordingly, it ɜn indispensable condition for the stress me- ɹrement by this method to determine the sitions of the X-ray diffraction lines. The- ɾre, it is to be desired that the lines are clea- continuous Debye-Sherrer rings, and so ɜ size of the X-ray beam ought to be larger ɜn the crystal size of the material considera- /.

After considerations on these respects, it is ɜar that the stress measurement of the extre- ɛly localized region as a tip of crack using ray micro-beam diffraction techinque is ɾy difficult.

Now, in previous papers[1),2)], the authors have investigated qualitatively the mechanism of the fatigue crack propagation from the features of the plastic deformation in the crystals surounding the tip of the crack. As the authors have pointed out in the papers, however, the stress states between external and internal stresses existing at the tip of the fatigue crack are unavoidable factor to make the mechanism of the crack propagation clear during fatigue process, too. Therefore, although the authors have always given their mind to approach the mechanism from the point of the stress states, it has been impossible to measure the extremely localized stress such as the tip of the crack under the techniques in actual operation. However, X-ray stress measurement using crystal oscillation method which was cultivated and developed by the authors[3)] and the others[4)] made stress measurement of the material with coarse grains possible.



(a) (b) (c)

Fig. 1. Optical micrograghs of used specimens.
(a) Average grain size of 15$\mu$ in diameter.
(b) Average grain size of 30$\mu$ in diameter.
(c) Average grain size of 50$\mu$ in diameter.

In the present paper, to provide information on the stress measurement by X-ray micro-beam diffraction technique using the crystal oscillation method, the authors first examined experimentaly and theoretically the relation between the sizes of X-ray beam and crystal to obtain the clearly continuous Debye-Sherrer ring in the case of use of the method.

## § 2. Experimental Procedures

### §§ 2.1. Specimen Preparation

The specimen used in the present examinations were fully annealed low carbon steels (0.07%C) with grain sizes of about 15, 30 and 50$\mu$ in diameter. The optical micrographs of the specimens are shown in Fig. 1. The specimens which were removed 50$\mu$ in thichness from the surface layer were used for X-ray observation.

### §§ 2.2. X-Ray Diffraction Technique

X-ray camera is composed of the camera casette, specimen holder, optical microscope and GM counter. The specimen holder can be moved up and down and right and right and left, and specimen attachment also can be rotated around horizontal and vertical axes with high accuracy centering around an illuminated position on the specimen surface. Accordingly it is possible to direct the beam to the desired position on the specimen surface with microscope. In this experiment, however, the microscope is not necessary. The outside view of X-ray camera and the schematic representation of the the oscillation axes of specimen are shown in Figs. 2 and 3, respectively. The observation of X-ray diffraction pattern was carried out on (211) diffraction plane using CrK$\alpha$ radiations.

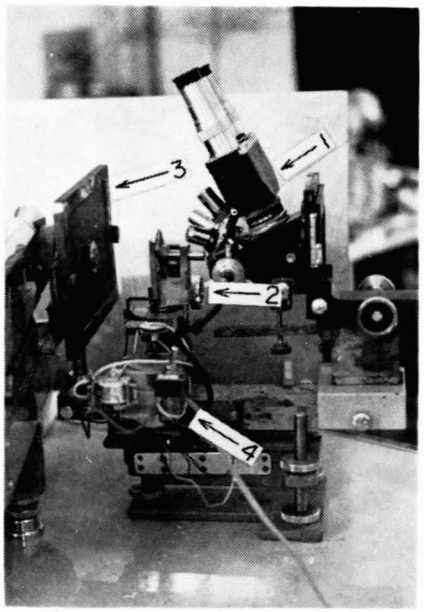



Fig. 2. X-Ray micro-beam camera with optical microscope.
(1) Optical microscope. (2) Specimen holder.
(3) Cassette. (4) Motor.

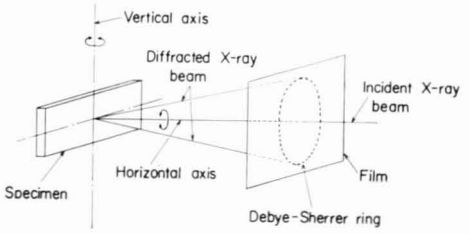
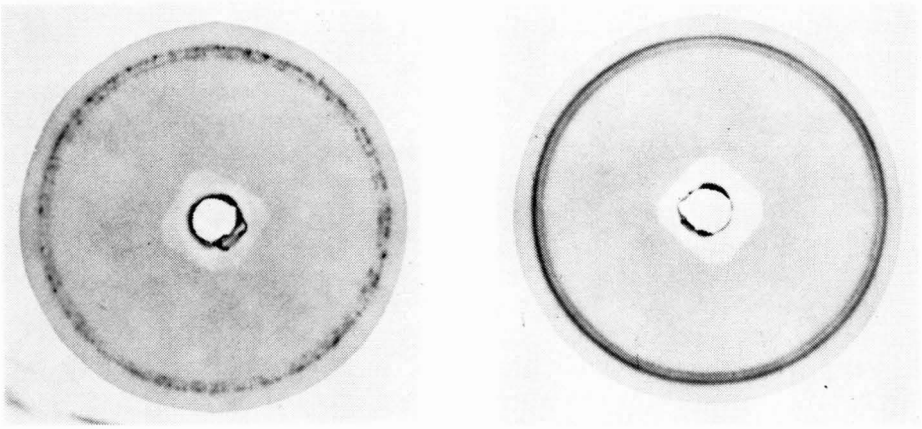



Fig. 3. Schematic representation of oscillation axis.

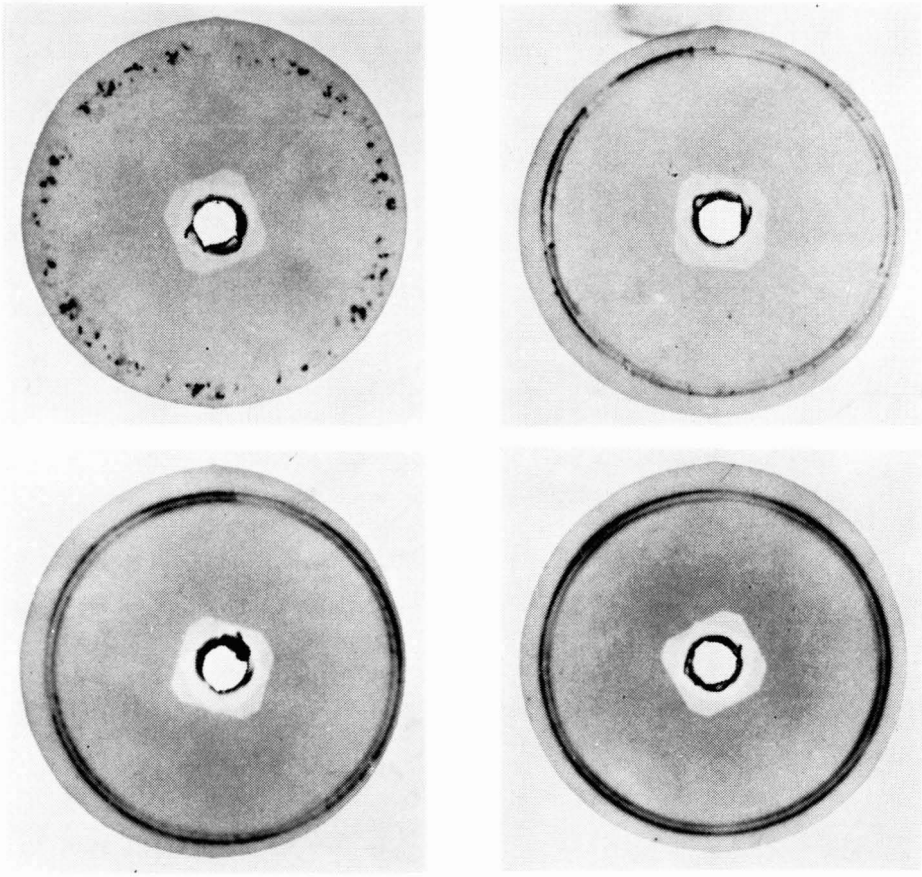
## § 3. Experimental Results and Discussions

Fig. 4 (a) and (b) are X-ray diffraction patterns which were obtained from the fixed



(a) (b)

Fig. 4. X-Ray diffraction patterns obtained from specimen having grain size of 15$\mu$ in diameter by using film oscillation and no crystal oscillation methods with 0.30 mm $\phi$ slit.
(a) Film oscillation around incident X-ray beam (F. O.) ; 0°
(b) F. O. ; ±15°



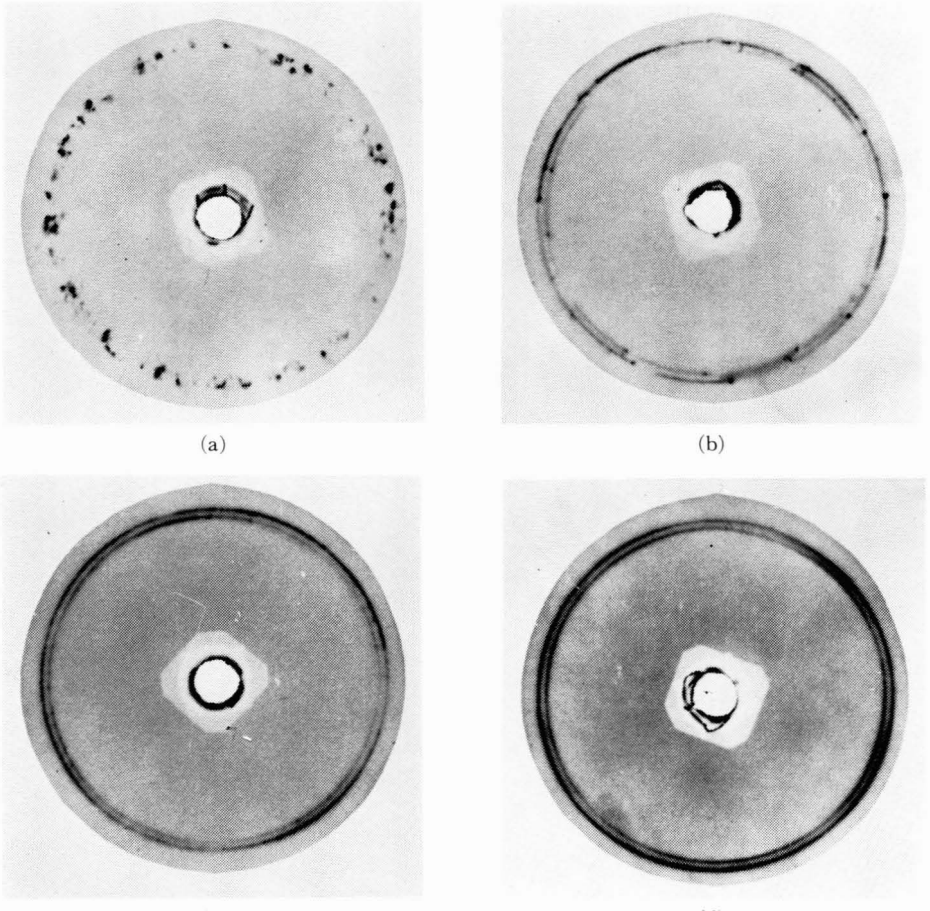
(a) (b) (c) (d)

Fig. 5. X-Ray diffraction patterns obtained from specimen having grain size of 30$\mu$ in diameter by using crystal oscillation method with 0.30 mm $\phi$ slit.
(a) Crystal oscillation around vertical axis (C. O. V. A.) ; 0° Crystal oscillation around horizontal axis (C. O. H. A.) ; no step, F. O. ; 0°
(b) C. O. V. A. ; 0°, C. O. H. A. ; no step, F. O. ; ±15°
(c) C. O. V. A. ; ±1.5, C. O. H. A. ; no step, F. O. ; ±15°
(d) C. O. V. A. ; ±1.5, C. O. H. A. ; 3 steps, F. O. ; ±15°

specimen with the grain size of 15 $\mu$ in diameter using no and film oscillation techniques in 0.30 mm $\phi$ single pinhole slit. The oscillation was carried out in the range of $\pm 15°$ around X-ray incident beam. The film oscillation has been operated to obtain the smoothly continuous Debye-Sherrer ring in the previous method. The photograph (b) shows that X-ray stress measurement is possible in the specimen with the grain size of below 15 $\mu$ without intr-oducing the crystal oscillation method.

Figs. 5 (b) and 6 (b) obtained from the specimen with the grain sizes of 30 and 50 $\mu$ in diameter respectively, were taken using the film oscillation only. The patterns in both figures could not be regarded as the continuous diffraction ring. Accordingly, it is understood that it is impossible to measure the stress with high accuracy for the materials with the grain sizes of 30 $\mu$ in diameter by the previous method on stress measurement. But it is clear that the diffraction rings become gradually contin uous with increasing of the oscillating angle around two axes (for example, Figs. 5 and 6). Paying attention to this fact, it may be easily imagined that the X-ray stress measurement is possible in the materials with the grain sizes of above 30 $\mu$ in diameter as well as the materials with those of below 15 $\mu$ in diameter.

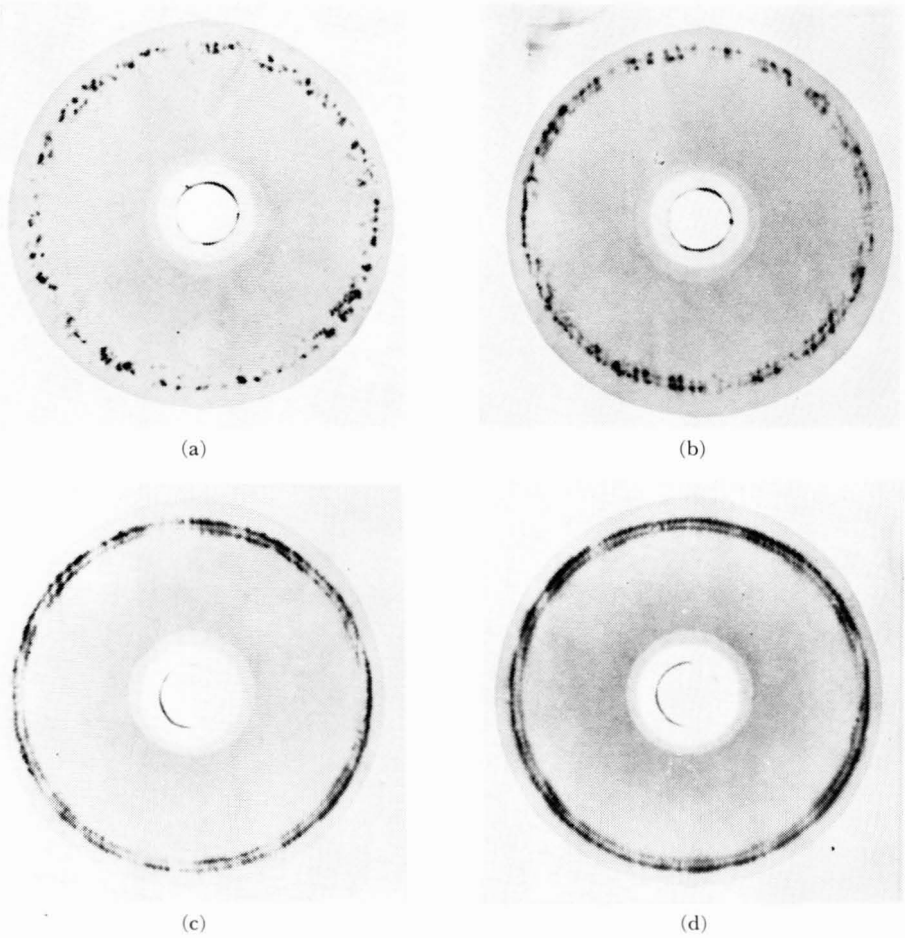
It is difficult to say that the diffraction ring shown in Fig. 7 (d) are clearly continuous. However, if the operation of the film oscillati-



(a) (b)

(c) (d)

Fig. 6. X-ray diffraction patterns obtained from specimen having grain size of 50 $\mu$ in diameter by using crystal oscillation method with 0.30 mm $\phi$ slit.
(a) C. O. V. A. ; 0°, C. O. H. A. ; no step, F. O. ; 0°
(b) C. O. V. A. ; 0°, C. O. H. A. ; no step, F. O. ; $\pm 15°$
(c) C. O. V. A. ; $\pm 1.5°$, C. O. H. A. ; no step, F. O. ; $\pm 15°$
(d) C. O. V. A. ; $\pm 1.5°$, C. O. H. A. : 4 steps, F. O. ; $\pm 15°$



(a) (b)

(c) (d)

Fig. 7. X-Ray diffraction patterns obtained from specimen having grain size of 30$\mu$ in diameter by using crystal oscillation and no film oscillation methods with 1.00 mm $\phi$ slit.
(a) C. O. V. A. ; 0°, C. O. H. A. ; no step
(b) C. O. V. A. ; ±1°, C. O. H. A. ; no step
(c) C. O. V. A. ; ±1°, C. O. H. A. ; 4 steps
(d) C. O. V. A. ; ±2°, C. O. H. A. ; 4 steps

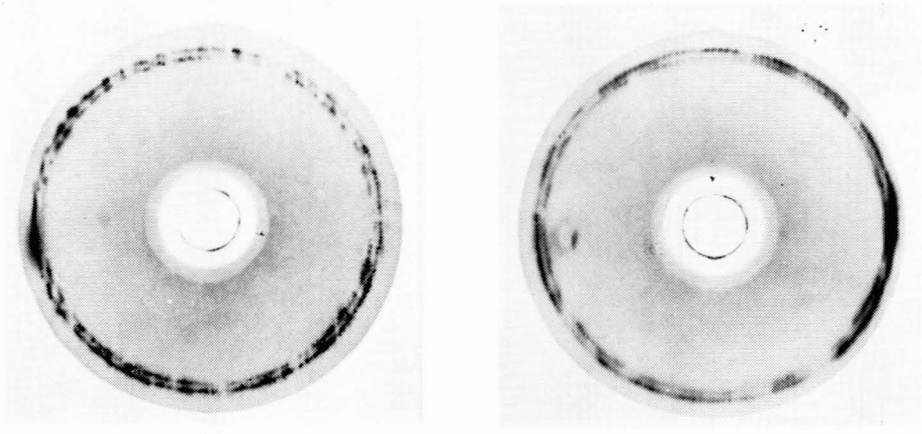
on is superpose on that of the crystal oscillation, or if the crystal oscillation around the horizontal axis is operated automatically, it may be imagined that the spotty rings as shown in Fig. 7 become continuous lines.

A real aim of the present experiments is the stress measurement of the extremely localized region such as tip of crack. Therefore, it is requried to introduce the micro-beam technique to this method. Figs. 8 (a) and (b) are the diffraction patterns which were obtained from the specimen with grain sizes of 30 and 50$\mu$ in diameter using 0.12 mm $\phi$ pinhole slit. It is undersood that the diffraction rings in both figures become also smoothly continuous lines considering Fig. 4 (a). After all, it was made clear that when the micro-beam technique is introduced to the crystal oscillation method it is possible to measure the stress at the extremely localized region such as a tip of crack.

Moreover, although this method makes the stress measurement in one grain possible, the details are given elsewhere[4].

Where, the authors are intended to described the meanings of the oscillations of the crystal from the stand point that the number of the diffraction spot is increased by the crystal oscillation.

To oscillate the specimen around a vertical axis is the same significance to change the direction of incident X-ray beam continuously in the range of the oscillating angle. Accor-



(a) (b)

Fig. 8. X-ray diffraction patterns obtained from specimens having grain sizes of 30 and 50$\mu$ in diameter by using crystal oscillation and no film oscillation methods with 0.12 mm $\phi$ slit.
(a) C. O. V. A. ; $\pm 1°$, C. O. H. A. ; 4 steps
(b) C. O. V. A. ; $\pm 1.5°$, C. O. H. A. ; 5 steps

dingly, this operation makes the diffraction from other crystal and the crystal plane of the same form in the crystal which does not diffract in the fixed specimen possible. In this experiment, however, the later diffraction is neglected because that the oscillating angle is very small.

After consideration on mentioned above respects, it is clear that the number of the crystal concerning to diffraction increases with the increment of the oscillating angle. But as the data which are obtained by means of this method are averaged values of the crystals existing in the range of the oscillating angle, it is undesirable to enlarge the angle in practical use of the X-ray stress measurement. The allowable range of the angle would be about 4° at most.

On the other hand, to oscillate the specimen around horizontal axis is equivalent to the oscillation of the film around the axis of incident X-ray beam. That is, this operation is intended to change the position of the diffraction spot on the film along the direction of the circumference on Debye-Sherrer ring. The allowable range of that oscillating angle would be similar to the case of the oscillation around the vertical axis.

As mensioned above, it is concluded experimentaly that the crystal oscillation method is useful for the stress measurement.

In the next place the authors are intended to describe theoretically the relation between the sizes of X-ray beam and crystal in order to obtain the continuous X-ray diffraction ring.

If a beam of X-ray of divergence $d\theta$ falls on a perfect particle (crystal), a reflection (at Bragg's angle $\theta$) can take place only if the normal to the diffraction planes lies within the volume between two cones of semi-angle $(\pi/2-\theta)$ and $(\pi/2-\theta+d\theta)$, with axes along the direction of the incident X-ray beam as shown in Fig. 9. If the incident radiation has a wavelength spread $d\lambda$, the crystal can reflect

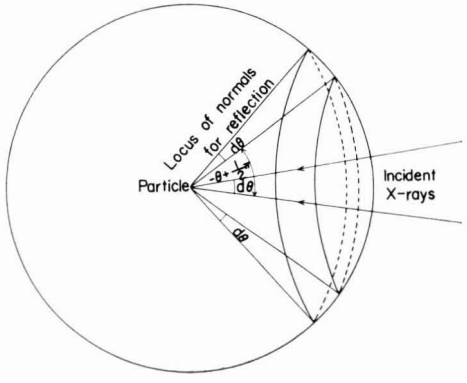



Fig. 9. Diagram illustrating the probability of reflection of a particle.

radiation over a range of angles $d\lambda = \tan\theta \cdot d\lambda/\lambda$, and for reflection to be the normal must lie within the volume between two cones of semi-angle $(\pi/2-\theta)$ and $(\pi/2-\theta+d\theta+d\lambda)$. The difference between the semi-angle of the two

cones can be written as $d\theta \times \Delta$, where $\Delta$ is equal to the range of the angles over which the particle can reflect owing to its imperfections or small size, and owing to the wavelength spread of the radiation.

If the crystals are randomly orientated, the probability of radiation for a particular particle, for a given set of diffraction planes, is equal to

$$\frac{2\pi\cos\theta(d\theta+\Delta)}{4\pi}=\frac{1}{2}\cos\theta(d\theta+\Delta). \quad (1)$$

If $p$ is the multiplicity factor in the lattice planes, the probability for a particle to reflect at an angle $\theta$ is

$$\frac{1}{2}p\cos\theta(d\theta+\Delta). \quad (2)$$

If $V$ is the volume of specimen illuminated, and $v$ is the mean volume of particle, the number of the reflections, $N$, occuring diffraction ring considered is

$$N=\frac{1}{2}\left(\frac{V}{v}\right)p\cos\vartheta\,(d\theta+\Delta), \quad (3)$$

Now $V$=area of cross-section (A)$\times$ penetration of X-ray beam (l) ; since l cannot be determined, it is adopted as 15$\mu$.

While, the tangential widths of the spots give a direct measure of the average divergence of the beam during the exposure ; it has been shown in other paper[3] that the tangential spot width $S_T$ is given by

$$S_T=\frac{R_0}{|\cos 2\theta|}(d\vartheta+B_T) \quad (4)$$

where $B_T$ is the physical broadening due to distortion and shape of particle, and $R_0$ is the specimen-film distance.

If the following condition is satisfied the spotty diffracton ring is to be obtained, because the circumference of the ring is given by $2\pi R_0$ tan $2\theta$ :

$$NS_T<2\pi R_0 \tan 2\vartheta \quad (5)$$

Here, the authors are intended to introduced above mentioned theory to this experiment.

Calculated results of the divergences, the areas and volumes of the specimens illuminated for three pinhole slits used in the present experiment are shown in Table I. In this calculation the penetration length of X-ray

Table I. X-ray beam conditions for three pinhole slits.

| Diameter of slit (mm) | Angle of divergence ($\times 10^{-2}$rad) | Area of irradiation (mm$^2$) | Volume of irradiation ($\times 10^{-2}$mm$^3$) |
|---|---|---|---|
| 0.12 | 1.3 | 2.4 | 3.6 |
| 0.30 | 1.6 | 3.5 | 5.3 |
| 1.00 | 1.7 | 5.5 | 8.3 |

beam and the specimen-film distance are taken as 15$\mu$ and 100mm, respectively. The number of $p$ and value of $\theta$ are obtained from (211) diffraction plane of iron specimen by CrK$\alpha$, radiation are 12 and 78° 0′ 30″, respectively. Number of the diffraction spots which are obtained theoretically to put those values into equation (4) are tabularized in Table II. It is

Table II. Calculated number of diffraction spots for each slit and crystal size.

| Crystal size ($\mu$) | Diameter of slit (mm) | | |
|---|---|---|---|
| | 0.12 | 0.30 | 1.00 |
| 15 | 200 | 370 | 610 |
| 30 | 25 | 46 | 77 |
| 50 | 6 | 10 | 17 |

worthy to note that the number of the calculated spots agree approximately with the number which were obtained from the count of the spot on Debye-Sherrer ring, although the counting was carried out with the condition of $S_T=R_0 d\theta/\cos 2\theta>109 d\theta$.

On the other hand, a condition on the number of diffraction spot to form the particular diffraction ring is $N\geqq 2\pi R_0|\tan 2\theta|/S_T=2.55/d\theta$. Therefore $N$ which are theoretically calculated for each pinhole is as follows :

For 0.12mm pinhole ; $N_{th}\geqq 196$
For 0.30mm pinhole ; $N_{th}\geqq 160$
For 1.00mm pinhole ; $N_{th}\geqq 150$

Comparing these results to the values listed up in Table II, it is considered the diffraction spots obtained from the material with the grain size 15$\mu$ in diameter are able to form a continuous ring even if any pinhole is used, but it is not always to say so for the materials with the grain sizes of 30 and 50$\mu$. As mentioned above, it is also possible to obtain the continuous rings for each as coarse grained materials using the crystal oscillation method.

If a specimen is oscillated in the range of angle of $\pm\beta/2$ around the horizontal and vertical axes, it is considered that the diffraction spot increases in number approximately by $\beta/d\theta$ times in comparison with that of the fixed specimen. Thus, the minimum oscillating angles around either axis to form the particular diffraction rings are calculated, and tabularized in Table III for each pinhole slit and grain size. As shown in this table, Debye-Sherrer rings in material with the grain size of $15\mu$ are continuous using 0.30 and 1.00 mm pinhole slits but any other case are not, because that it is desired that the oscillation angle around axis is smaller than 4°. However this problem is solved by the oscillation around another axis. That is, as the increment of the diffraction spot due to the oscillation around another axis is the same as that of one axis, it is easy to estimate the oscillation angle around that axis. If the oscillating operation around the horizontal axis is not automatic as the present experiment, however, the qutients obtained by dividing the values of angle tabularized in Table III by 4 indicate the number of times for inclinating of specimen within the angle of $\pm 2°$ to the horizontal axis. Moreover, when the film oscillation technique is superposed on the crystal oscillation method, there is no doubt to get much more clear ring as shown in Figs. 5 (b) and 6 (b).

Table III. Values of minimum angle of oscillation around vertical axis for each slit and crystal size.

| Crystal size ($\mu$) | Diameter of slit (mm) | | |
|---|---|---|---|
| | 0.12 | 0.30 | 1.00 |
| 30 | 5.9 | 3.2 | 1.9 |
| 50 | 24.6 | 14.9 | 8.8 |

## § 4. Conclusions

X-ray crystal oscillation method was applied to the examination in oder to obtain the smoothly continuous diffraction ring with specimen made of a annealed low carbon steel having different grain sizes, the followings were concluded.

(1) The smoothly continuous diffraction ring was obtained by specimens with grain size of $15\mu$ and not 30 and $50\mu$ in diameter, using the film oscillation around a axis of incident X-ray beam, only.

(2) If the crystal oscillation technique is applied to X-ray stress measurement with combined the film oscillation method, it is possible to measure stress in materials with coarse grained such as 30 and $50\mu$ in diameter.

(3) Considering a practical use of X-ray stress measurement although the allowable range of the angle of the oscillation around horizontal or vertical axes would be about 4° at most, respectively, the ranges are able to calculate theoretically from the grain size of material and the angle of divergence of used X-ray beam.

(4) Introducing X-ray micro-beam technique to this method, it is able to clear theoretically and experimentaly that X-ray stress measurement in localized region may be possible.

(5) By using crystal oscillation method, the stress in one grain are possible to measure also, although details of this method and result have been given elsewhere.

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