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The Effects of Time Constant and Absorption on Stress Measured by X-ray Diffraction Method

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Synopsis

The diffracted intensity of X-ray depends upon several physical and geometrical factors such as structure, multiplicity, absorption and Lorentz-polarization and measuring conditions such as time constant and scanning speed of detector on counter method[1]. For analyzing on the X-ray stress measurement, especially, profile shape of X-ray diffraction which is affected by geometrical factors such as absorption and Lorentz-polarization is very important. In order to eliminate these factors affecting the stress measured by using X-ray, the correcting factors were introduced and those theoretical values were calculated.

After this theoretical calculation, it is found that as the half value breadth increases the difference between the stress measured by using X-ray and the corrected one becomes larger and larger under same measuring condition. When the ideal diffracted intensity of X-ray is assumed Cauchy distribution the measured stress depends upon measuring condition for same specimen, but it is independent of measuring condition in Gauss distribution. Consequently, it is found that the stress measured by using X-ray must be

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corrected under each measuring condition and the method of correction is made clear and proved experimentally in this paper.

1. Introduction

It is well known that X-ray stress measurement is one of the most effective methods for measuring stress in polycrystalline materials. It is practically the only nondestructive method of stress measurement that can be applicable in determining the local stress distribution. So it is widely used in material engineering studies. For the characteristic of this X-ray diffraction technique, however, there are a few basic problems to be further investigated.

X-ray stress measurement is a technique to obtain the stress from the lattice strain determined by diffraction angles. In practice, the diffraction angle 2θ is determined by the peak position of X-ray diffraction profile for several incident angles ψ_o. The plot of 2θ against $\sin^2\psi$ is theoretically linear(ψ is the angle between the normals of specimen and diffraction plane). The X-ray stress can be calculated by the product of the gradient M of this regression line and the constant K(σ=K•M).

Many factors which will affect the stress obtained by the method of X-ray stress measurement have been discussed and investigated. Concerning to the physical factors, the structure factor which depends upon the scattering power and the spatial arrangement of the atoms in the compound and the multiplicity factor which represents the number of symmetrically identical planes, they all have no effect on the peak shift of diffraction profiles. So they are ordinarily constant for measurement, on the other hand, the geometrical factors which are given as the function of θ and ψ affect the peak shift of X-ray diffraction profiles. However, the effect of these geometrical factors, especially about absorption factor, are not investigated and not discussed quantitatively.

On the X-ray stress measurement it is desirable that the profile shape of X-ray diffraction may be smooth and the time required for measurement may be short. So that, the time constant must be larger and scanning speed of detector must be faster in technologically. It is reported that the peak shift of X-ray diffraction profiles is dependent on the product of time constant and scanning speed and the shift is proportional to the magnitude of this product[2]. It was confirmed by an investigation that the time constant and scanning speed had no effect on the measured stress using the product of these measuring conditions up to about 32 deg/min•sec. In case of large time constant and high scanning speed, however, the stress measured by using X-ray is not evaluated sufficiently.

After consideration on this respect, in the present paper, the authors introduce the correcting function for absorption and Lorentz-polarization factros that affect the measured stress and theoretically discussed the relation between measuring condition and the stress measured by using X-ray as technological problems on the X-ray stress measurement.

2. Experimental Procedures

The specimens used in this experiment are the powder of iron and induction hardened steel. After being machined, the 0.45 % carbon steel specimen was fully annealed in a vacuum at 850°C for 1hr. and perfectly quenched by induction hardening and tempered at 170°C for 2.5hr.. The powder of iron was also annealed at 300°C for 1hr. in a vacuum furnace.

The specimens were furnished with X-ray stress measurement and the diffraction angles at various angles of incident X-ray beam were measured from the profile of X-ray diffraction by counter method. The conditions of X-ray diffraction are that the characteristic line of X-ray is CrKα, the time constant is 16 sec. and the scanning speed of detector is 2 deg/min.

3. Theoretical Calculations

Generally, the diffracted intensity I(x) of X-ray can be written as following equation

$$I(x)=F(x)G(x)+B(x) \qquad (1)$$

where variable x is the diffraction angle. F(x) is the correcting function including absorption and Lorentz-polarization factors, G(x) is the ideal diffracted intensity of X-ray and B(x) indicates the intensity of background. B(x) can be expressed by polynominal approximation as

$$B(x)=a+bx+cx^2+\cdots\cdots \quad (2)$$

On these functions, diffraction angle is shown as

$$x=2\theta-2\theta_0 \quad (3)$$

where θ is the anguler position of detector and θ_0 is the peak position of X-ray diffraction profile by characteristic line of X-ray.

The diffracted intensity of X-ray on recorder dy is proportional to $I(x')$ in an infinitesimally small time from $t=t'$ to $t=t'+dt'$.

$$dy=KI(x')dt' \quad (4)$$

where K is an arbitrary constant and x' is the diffraction angle at $t=t'$. It decreases exponentially till measuring time t, so that, Equation(4) becomes

$$dy=KI(x')\exp\left(\frac{t'-t}{T}\right)dt' \quad (5)$$

where T is the time constant. The diffracted intensity on recorder is obtained by integration in the form

$$y=\int_0^t KI(x')\exp\left(\frac{t'-t}{T}\right)dt' \quad (6)$$

The profile of X-ray diffraction can be obtained as the function of variable x on recorder. So that, in the Equation(6), variable t is exchanged for variable x by using the relation between t and x as follows

$$x=St+x_0 \quad (7)$$

where S is the scanning speed of detector and x_0 is the beginning angle for measurement, i.e. the measuring angle at t=0. Therefore, Equation(6) is transformed as follows

$$y=\int_{x_0}^x KI(x')\exp\left(\frac{x'-x}{ST}\right)\frac{dx'}{S} \quad (8)$$

Assuming that the diffracted intensity of X-ray increases a unit on the recorder for detecting one count per second(1cps), the arbitrary constant K is given as

$$K=\frac{1}{T}$$

Since, the diffracted intensity on recorder can be rearranged by substituting Equation(1) and (9) into Equation(8) as follows

$$y=\int_{x_0}^{x}\frac{1}{ST}I(x')\exp\left(\frac{x'-x}{ST}\right)dx'$$

$$=\frac{1}{ST}\int_{x_0}^{x}F(x')G(x')\exp\left(\frac{x'-x}{ST}\right)dx'+\frac{1}{ST}\int_{x_0}^{x}B(x')\exp\left(\frac{x'-x}{ST}\right)dx' \qquad (10)$$

It is clear from this equation that intensities of diffraction and background can be separated perfectly, the component of background is of the form

$$y_b=\frac{1}{ST}\int_{x_0}^{x}(a+bx'+cx'^2+\cdots\cdots)\exp\left(\frac{x'-x}{ST}\right)dx'$$

$$=\{(a-bST+2cS^2T^2-\cdots\cdots)+x(b-2cST+\cdots\cdots)+x^2(c-\cdots\cdots)\}$$

$$\times\{1+\exp\left(\frac{x_0-x}{ST}\right)\} \qquad (11)$$

The term of $\exp\{(x_0-x)/ST\}$ in Equation(11) that expresses the corrective one on nonequilibrium state is nearly equal to zero in case of x_0 of sufficiently small compared with x.

Thus, standardizing the range in which the intensity of background can be regarded as an equilibrium state, i.e. the beginning angle for measurement is very smaller than the diffraction angle, it is approximated in the form of a power series in x multiplied by a power of x which can hold to a fairly good approximation. If the intensity of background is assumed to be linear that is generally used, it is clear from Equation(11) that the component of background also appears as the linear function on recorder.

The real profile of X-ray diffraction can be calculated by subtracting the approximate value of y_b from y.

$$y_d=y-y_b$$

$$=\frac{1}{ST}\int_{x_0}^{x}F(x')G(x')\exp\left(\frac{x'-x}{ST}\right)dx' \qquad (12)$$

If the intensity of background is corrected by the method described above, it is sufficient to examine only for y_d. It is so difficult to integrate Equation(12) that it is numerically integrated by dividing the diffraction angle x into the infinitesimal angle Δx in order to simplify the equation.

Thus, Equation(12) can be put in a convenient form by using the summation notation as follows

$$y_{d_i}=\frac{1}{ST}\sum_{j=0}^{i}F(x_j)G(x_j)\exp(\frac{x_j-x_i}{ST})\Delta x$$

$$=\frac{1}{ST}\sum_{j=0}^{i-1}F(x_j)G(x_j)\exp(\frac{x_j-x_{i-1}-\Delta x}{ST})\Delta x+\frac{1}{ST}F(x_i)G(x_i)\Delta x$$

$$=y_{d_{i-1}}\exp(\frac{-\Delta x}{ST})+\frac{1}{ST}F(x_i)G(x_i)\Delta x \qquad (13)$$

Therefore, if y_{d_0} is given, $y_{d_1}, y_{d_2}, y_{d_3}, y_{d_4} \cdots\cdots y_{d_i}$ is calculated by one after another and the profile of X-ray diffraction is obtained by means of computer.

4. Methods of Calculations

It is assumed that the detector counts $I(x)\Delta t$ in the interval Δt and the indicator decreases exponentially in the same interval on the recorder. The profile of X-ray diffraction on computer can be obtained by repeating this process.

This theoretical calculation is applied to the stress measurement of ferritic materials by using characteristic line of Kα doublet that is constituted of $K\alpha_1$ and $K\alpha_2$. The maximum intensity and its position of the profile where $K\alpha_1$ and $K\alpha_2$ diffraction profile usually overlap each other are the value of the greatest intensity and that position and the half value breadth which shows the breadth of half value of the maximum intensity excluding the background is obtained as shown in Fig.1. When the true integral breadth is larger than about 1 deg. there are two intersection points of the half value height of the maximum intensity excluding the background. On the other hand, when it is less than about 1 deg. there are four intersection points of its height under certain measuring conditions. In that case, the middle position of the half value breadth is determined by the first two points that almost depend upon characteristic line of X-ray $K\alpha_1$.

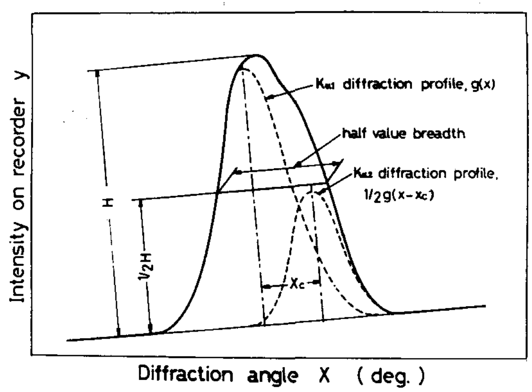


Fig.1 Illustrating the profile of X-ray diffraction on recorder

The ideal diffracted intensity G(x) of X-ray can be chosen from among many functions, however, G(x) used in this theoretical calculation is assumed by two distribution curves that are often discussed among investigators dealing with X-ray diffraction profile analysis. The characteristic line of $K\alpha_1$ has two times as intensity as $K\alpha_2$ one, so they can be shown as follows[3]:

Gauss distribution;

$$G_G(x)=G_0\exp(-\frac{\pi}{\beta^2}x^2)+\frac{1}{2}G_0\exp\{-\frac{\pi}{\beta^2}(x-x_c)^2\}$$

Cauchy distribution;

$$G_C(x)=\frac{G_0}{1+(\frac{\pi}{\beta}x)^2}+\frac{1}{2}\frac{G_0}{1+\{\frac{\pi}{\beta}(x-x_c)\}^2}$$

where β is the true integral breadth which is given by separating the diffraction lines into $K\alpha_1$ and $K\alpha_2$ one and x_c is the distance between diffraction angles by characteristic lines of $K\alpha_1$ and $K\alpha_2$ (cf. Fig.1). This theoretical calculation is applied to the stress measurement of (211)plane of α-iron by using CrKα line. So the numerical values are x_c=0.95 deg., $2\theta_0$ = 156.1 deg. where θ_0 is the peak position of X-ray diffraction profile by $CrK\alpha_1$.

It is well known that the absorption of X-ray depends upon the pass of X-ray beam in the specimen[4], so that in this paper, the absorption factor is expressed as the function of the pass length and is given as the value of integration, $\int_0^\infty \exp\{-(x_1+x_2)\}dy$ presented in Fig.2. After integration the absorption factor is given as follows

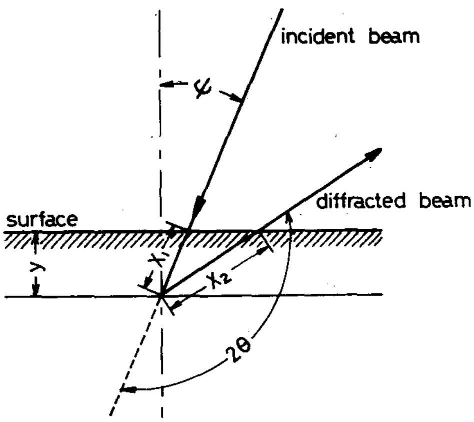


Fig.2 Pass of X-ray beam in the specimen

$$F_1(\theta)=\frac{1}{\sec\psi-\sec(\psi-2\theta)}$$

where θ is the diffraction angle and ψ is the angle of incident X-ray beam. Similarly, Lorentz-polarization factor which depends solely on θ and can be corrected easily is given in the form theoretically,

$$F_2(\theta)=\frac{1+\cos^2 2\theta}{\sin 2\theta \sin\theta}$$

Thus correcting function is given as the product of $F_1(\theta)$ and $F_2(\theta)$ as follows

$$F(\theta)=\frac{1}{\sec\psi-\sec(\psi-2\theta)}\cdot\frac{1+\cos^2 2\theta}{\sin 2\theta \sin\theta}$$

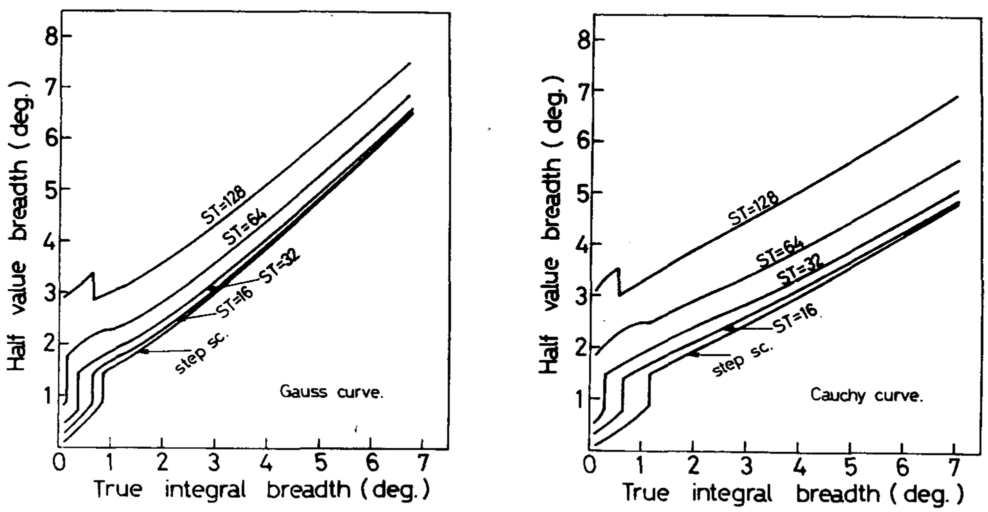
In practical calculation, the correcting function is introduced as the function F(x) by using Equation(3).

The angles of incident X-ray used in this calculation are 0, 15, 30 and 45 deg. to the normal of the measuring surface. The diffraction angle for each incident angle can be obtained from the middle position of the half value breadth of diffraction profile on computer. The stress is calculated from the product of the gradient M of the regression line on 2θ-$\sin^2\psi$ diagram that is determined by the least squares method and the constant K. The value of K is -32.44 kg/mm²·deg and the specimen is assumed to be stress-free condition(σ=0 kg/mm²).

5. Results and Discussions

5.1 Results of Theoretical Calculations

The peak position of X-ray diffraction profile is determined by the middle position of half value breadth on the X-ray stress measurement. So that, the results using half value breadth are described and discussed in this paper.

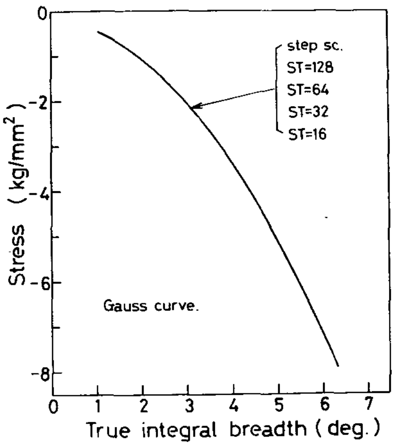


(a) Gauss distribution (b) Cauchy distribution

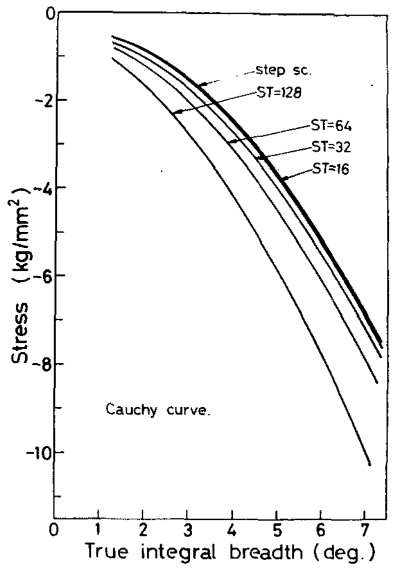
Fig.3 Effects of measuring condition on half value breadth

Fig.3 shows the relation between true integral breadth and half value breadth in case of Gauss and Cauchy distribution. On the measurement of same specimen which has same integral breadth, the half value breadth depends upon the value of ST(product of scanning speed and time constant), that is, it increases as the value of ST increases and its tendency is stronger in Cauchy distribution than Gauss one. When the integral breadth decreases less than about 1 deg., the relation between true integral breadth and half value one is very complicated. In that case, the profile of X-ray diffraction almost becomes to be separated into $K\alpha_1$ diffraction profile and $K\alpha_2$ one, there are four intersection points of half value height of the maximum intensity excluding the background and the half value breadth is determined by the distance of first two points. For the reason that the half value breadth almost depends upon $K\alpha_1$ diffraction profile, it increases suddenly in case of step scanning, ST=16, 32, 64 deg/min•sec. Although the diffraction profile separated into $K\alpha_1$ and $K\alpha_2$ one, there are two intersection points of its height in case of ST=128 deg/min•sec. So the half value breadth decreases suddenly as shown in Fig.3.

Fig.4 is the results of calculation for the specimen which is assumed to be stress-free condition(σ=0 kg/mm^2). They show the relation between measured stress of stress-free specimen and true integral breadth in case of Gauss and Cauchy distribution, respectively. On the measurement of same specimen which has same integral breadth, the half value breadth changes with the value of ST, however, the measured stress is not affected by the value of ST in case of Gauss distribution. That is, the measured stress is solely affected by the state of specimen, and so, it is sufficient to use only the true integral breadth as the



(a) Gauss distribution



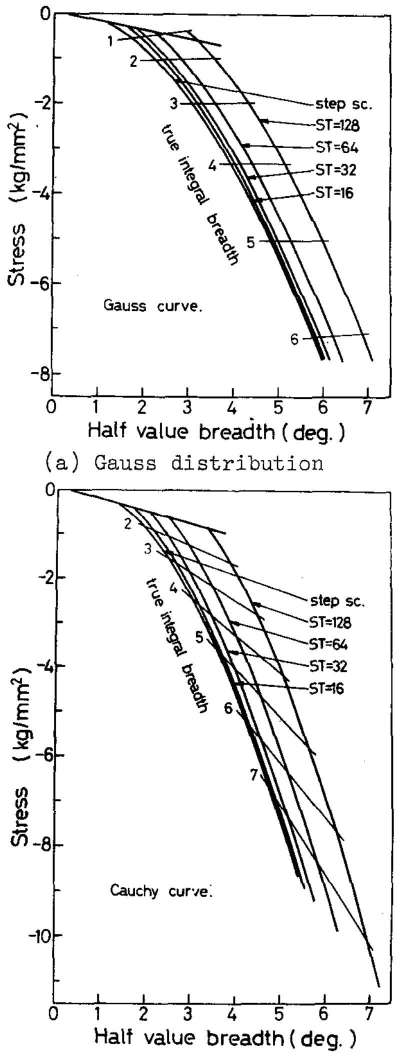
(b) Cauchy distribution

Fig.4 Effects of measuring condition on measured stress parameter estimating the measured stress in case of Gauss distribution. On the assumption of Cauchy distribution, however, the measured stress becomes smaller and smaller as the value of ST increases for same specimen, namely the measured stress depends upon both the measuring condition(ST) and the state of specimen. Therefore, both the measuring condition and true integral breadth must be used as the parameter estimating the measured stress in case of Cauchy distribution.

In order to obtain true integral breadth on experiment, it is necessary to get the profile of X-ray diffraction by step scanning and to separate the profile into $K\alpha_1$ and $K\alpha_2$ diffraction line. It is convenient to use the value of ST and half value breadth that can be obtained easily by experimental procedure instead of the true integral breadth as the parameter estimating the measured stress.

Fig.5 shows the relation among measured stress of stress-free specimen, half value breadth, true integral breadth and the value of ST in case of Gauss and Cauchy distribution. It is obvious that the measured stress becomes smaller and smaller as the half value breadth increases under same measuring condition in both Gauss and Cauchy distribution. Comparing Fig.5(a) and (b), this tendency mentioned above is stronger in Cauchy distribution than Gauss one. Hereon, the measured stress indicates the difference between the stress measured by using X-ray and the true stress, because it is assumed that specimen is stress-free condition. Therefore, when the corrected stress is required the stress measured by using X-ray must be corrected about this value of measured stress(hereinafter referred to as the value of correction, $\Delta\sigma$) shown in Fig.5.

For example, in the case where the X-ray diffraction profile has 5 deg. of its half value breadth and measuring condition(ST) is 64 deg/min·sec, the value



(a) Gauss distribution

(b) Cauchy distribution

Fig.5 Effects of half value breadth on measured stress of correction is about -4.4 kg/mm^2 in Gauss distribution and -5.8 kg/mm^2 in Cauchy one. The stress measured by using X-ray must be corrected about this value in order to obtain the corrected stress, however, the difference between the value of correction on the assumption of Gauss and Cauchy distribution is very small(about 1.4 kg/mm^2 in the case where the half value breadth is 5 deg.), so that, which distribution curve will do as long as the measured stress is corrected. When the accurate value of corrected stress is required, the profile of X-ray diffraction must be determined which distribution curve is closer to it. Fig.6 shows the relation between half value breadth of incident angle of 0 deg. and the ratio of quarter value breadth to half value breadth. The distinction of the ratio between Gauss and Cauchy distribution can be recognized on the same half value breadth, so that, the type of diffraction profile can be determined which distribution curve is closer to it by using Fig.6. The value of correction is obtained by the type of diffraction profile, the half value breadth and value of ST, however, it can be calculated easily by following equation after the type of diffraction profile is determined.

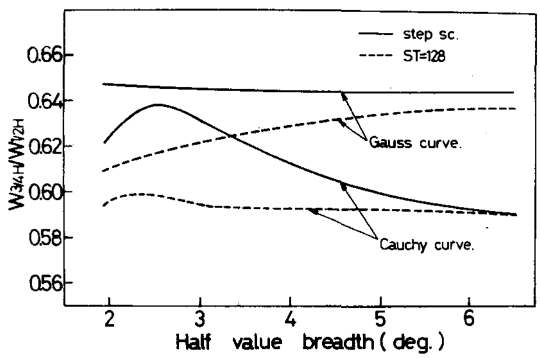


Fig.6 Distinction between the assumptions of Gauss and Cauchy distribution

Gauss distribution;

$$\Delta\sigma_G = -(0.0000894ST+0.207)W_{\frac{1}{2}H}^2 + (0.00412ST-0.130)W_{\frac{1}{2}H} + (0.000947ST+0.202) \qquad (14)$$

Cauchy distribution;

$$\Delta\sigma_C = -(0.000112ST+0.318)W_{\frac{1}{2}H}^2 + (0.00360ST+0.0724)W_{\frac{1}{2}H} + (0.0136ST-0.0577) \qquad (15)$$

where

$\Delta\sigma$: value of correction for measured stress (kg/mm^2)

$W_{\frac{1}{2}H}$: half value breadth of incident angle of 0 deg. (deg.)

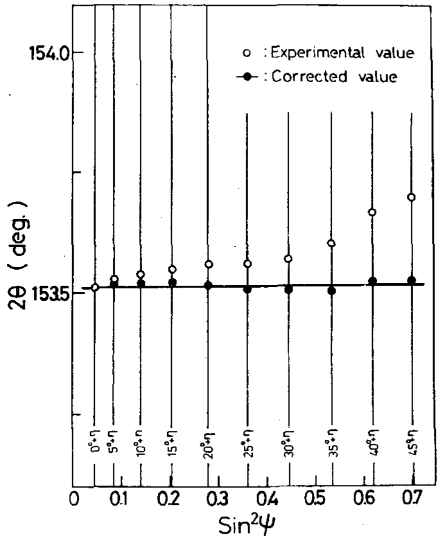
ST : product of scanning speed and time constant (deg/min·sec)

Considering the accuracy of correction, correction of the diffraction profile itself may be thought, however, it is practically better to correct the stress measured by using X-ray for the diffraction angle on each angle of incident X-ray beam(cf. Fig.7). After this method of correction, it is enable to calculate the corrected stress by the least squares method with a high accuracy.

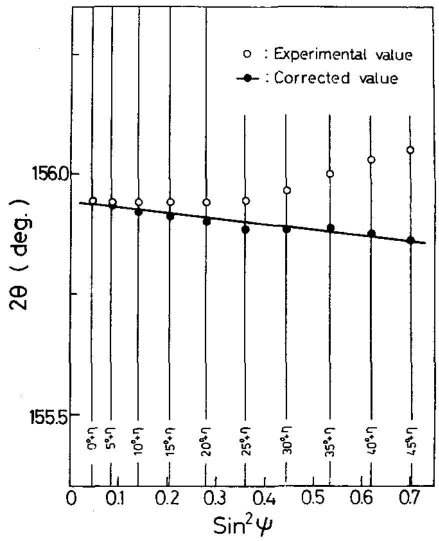
5.2 Experimental Results

Fig.7(a) shows 2θ-$\sin^2\psi$ diagram on the X-ray stress measurement of the powder of iron which is theoretically stress-free condition(σ=0 kg/mm^2). The measuring condition is that the scanning speed is 2 deg/min and the time constant is 16 sec.(ST=32 deg/min·sec). The specimen has about 5.38 deg. of half value breadth and 3.28 deg. of quarter value breadth, so that, it is found that the diffraction profile of powder is nearly equal to Cauchy distribution from Fig.6. The plot of experimental value on 2θ-$\sin^2\psi$ diagram is not linear and the measured stress using them is about -8.1 kg/mm^2 by the least squares method. On the other hand, the plot of corrected value is almost linear and the corrected stress is about -0.16 kg/mm^2 that is equal to the true stress within the range of measuring error. If Equation(15) is used, $\Delta\sigma_C$=-7.9 kg/mm^2, so that the corrected stress is about -0.2 kg/mm^2.

Fig.7(b) is similarly for the induction hardening material. The specimen has about 5.5 deg. of half value breadth and the value of ST is 32 deg/min·sec. The diffraction profile is also nearly equal to Cauchy distribution and the measured stress is about -5.3 kg/mm^2. The plot of corrected value is linear and the corrected



(a) Powder of iron



(b) Induction hardening material

Fig. 7 Theoretical and experimental results of 2θ versus $\sin^2\psi$

stress is about 3.5 kg/mm^2. If Equation(15) is used, the corrected stress is calculated about 3.0 kg/mm^2.

6. Conclusions

The correcting function for absorption and Lorentz-polarization factors that affect the measured stress is introduced and the relation between measuring condition and the stress measured by using X-ray is discussed as technological problems on the X-ray stress measurement.

The results obtained through this theoretical and experimental investigations are summarized as follows;

(1) On the measurement of same specimen which has same integral breadth, the half value breadth becomes larger and larger as the value of ST increases, i.e. it depends upon the measuring condition.

(2) When the ideal diffracted intensity of X-ray is assumed as Cauchy distribution the measured stress depends upon the measuring condition for the measurement of same specimen, but it is independent of measuring condition in Gauss distribution.

(3) The difference between the stress measured by using X-ray and the corrected one becomes larger and larger as the half value breadth increases under same measuring condition. This tendency is stronger in Cauchy distribution than Gauss one.

(4) The value of correction for measured stress can be expressed as the function of half value breadth and product of scanning speed and time constant. It is convenient and enough to use the equation mentioned above for the method of correction.

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