Short communication

PENETRATION DEPTH OF X-RAYS FOR STRESS MEASUREMENT OF SILICON NITRIDE

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1 INTRODUCTION

The residual stress of ground ceramics has a steep stress gradient [1]. Since the X-ray method measures the weighted average of lattice strain within the penetration depth, the detected strain changes with the X-ray tilt angle ψ . The 2θ - $\sin^2 \psi$ diagram taken from materials with a steep stress gradient becomes nonlinear. Yoshioka *et al.* proposed the method to estimate the stress gradient from the analysis of the nonlinearity of 2θ - $\sin^2 \psi$ diagram [2]. Their analysis requires the exact value of the penetration depth of X-rays.

In this paper, the known stress distribution was generated by bending a thin plate of silicon nitride, and the exact value of the penetration depth was determined from X-ray analysis of the applied stress gradient.

2 X-RAY STRESS ANALYSIS

The effective X-ray penetration depth T is defined by the following equation in the iso-inclination method [2].

$$T = \frac{\sin^2 \theta - \sin^2 \psi}{2\mu \sin \theta \cos \psi} \quad (1)$$

Here, we approximate T by

$$T = T_0 + T_1 \cdot \sin^2 \psi \quad (2)$$

where $T_0 = 36.4\mu\text{m}$ and $T_1 = -32.7\mu\text{m}$ for 323° diffraction by Cu-K α radiation.

The applied stress distribution in the bent specimen is linear. The principal stresses are given by

$$\sigma_1 = \sigma_{10} + A_1 \cdot z \quad (3)$$

$$\sigma_2 = \sigma_{20} + A_2 \cdot z \quad (4)$$

where σ_{10} , σ_{20} are surface stresses, A_1 , A_2 are stress gradients, z is the depth from the surface. Due to the absorption of X-rays, the measured stress $\langle \sigma_{ij} \rangle$ is the weighted average over the penetration depth and is given by [3]

$$\langle \sigma_{ij} \rangle = \frac{\int_0^{z_1} \sigma_{ij}(z) \cdot \exp(-z_1/T) dz}{\int_0^{z_1} \exp(-z_1/T) dz}$$

Fig. 1. Bending jig.

$$\begin{aligned} &= \sigma_{ij0} + A_{ij} T \frac{(1 + z_1/T) \exp(-z_1/T) - 1}{\exp(-z_1/T) - 1} \\ &= \sigma_{ij0} + A_{ij} W \cdot T \end{aligned} \quad (5)$$

where W is the coefficient determined by the actual penetration depth z_1 .

The relation between 2θ and $\sin^2 \psi$ will be approximated by parabola of $\sin^2 \psi$. The stress distribution is calculated from the coefficients of the parabola equation. Comparing the applied stress with the stress distribution obtained from the above procedure, the appropriate W value can be determined.

3 EXPERIMENT

3.1 Specimen and Bending

The material was the pressureless sintered Si_3N_4 [4]. Its bulk density was 3.25g/cm^3 . Young's modulus and Poisson's ratio were 321GPa and 0.28 , respectively. The test specimen had a thickness of $100\mu\text{m}$, a width of 10mm and a length of 58mm .

Thin plate specimens were cylindrically bent on a plastic cylinder with 60mm diameter as shown in Fig. 1. The applied surface stress σ_{10} and the stress gradient A_1 are 580MPa and $-11.6\text{MPa}/\mu\text{m}$.

3.2 X-Ray Stress Measurement

The X-ray stress was measured by the fixed plane normal method with the iso-inclination arrangement.

Table 1. X-ray conditions for stress measurement.

Characteristic X-ray	Cu- $\text{K}\alpha_1$
Diffraction	3 2 3
Diffraction angle	141.260 deg
Tube voltage	40 kV
Tube current	30 mA
Filter	Ni
Divergent angle	0.64 deg
Preset time	2 sec
Scanning speed	1deg/min
Stress constant	-811 MPa/deg
Irradiated area	$4 \times 10\text{mm}^2$

Fig. 2. 2θ - $\sin^2\psi$ diagrams before and after bending.

The X-ray conditions are summarized in Table 1. X-ray elastic constants used in this experiment were $E = 339\text{MPa}$, $\nu = 0.285$ and the linear absorption coefficient μ is $136/\text{cm}$ for 3 2 3 diffraction by $\text{Cu-K}\alpha_1$. The value of the stress constant was determined by experiment.

4 RESULTS AND DISCUSSION

The $\sin^2\psi$ diagram measured for the bent specimen is shown in Fig. 2. The relation between 2θ and $\sin^2\psi$ shows nonlinearity because of the stress gradient. The data in this figure include the initial residual stresses.

To cancel the initial state, shifts in the diffraction angle before bending from $2\theta_0 = 141.26\text{deg}$ were subtracted from values of diffraction angles after bending. The results are plotted in Fig. 3.

Approximating the relation between 2θ and $\sin^2\psi$ by parabola, surface stresses and stress gradients were calculated by the coefficients of the parabola equation. The results of calculation are presented in Table 2. Stresses σ_{10} and σ_{20} are independent of the W value. The value of the corrected stress is close to the applied stress 580MPa . The stress gradient A_1 depends on W , because A_1W is constant. Since the applied

Fig. 3. 2θ - $\sin^2\psi$ diagram of bent specimen after canceling the initial state.

stress gradient is $-11.6\text{MPa}/\mu\text{m}$, the best value of W is 0.779 as shown in Table 2.

The depth z_1 equals ∞ for $W = 1.00$ and z_1 equals T for $W = 0.42$. In the case of $W = 0.779$, the depth z_1 is $87\mu\text{m}$ where the intensity of X-ray diffraction become 8 percent of the intensity of X-ray diffraction from the surface. Therefore the appropriate penetration depth z_1 in Eq.(5) is equivalent to the depth of 8 percent intensity.

Table 2. Measured result of stress distribution.

	Weight	Surface stress (MPa)		Stress gradient (MPa/ μm)	
	W	σ_{10}	σ_{20}	A_1	A_2
Measured stress	1.000	562	144	-9.04	-0.68
	0.779	562	144	-11.6	-0.87
	0.420	562	144	-21.5	-1.61

5 CONCLUSION

The plate of silicon nitride with a thickness of $100\mu\text{m}$ was cylindrically bent. Comparing the applied stress distribution and the distribution measured by X-rays, the appropriate penetration depth was determined to be $87\mu\text{m}$ where the diffracted X-ray intensity was reduced by absorption down to 8 percent of that from the surface.

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