Proc. Int. Symposium on Advances in Neutron Scattering Research, Tokai, 2000
J. Phys. Soc. Jpn. **70** (2001) Suppl. A pp. 514–516

Measurement of Strain Free Lattice Spacing in Neutron Residual Stress Analysis by Sample Rotation Method

Toru SAITO[1,2], Nobuaki MINAKAWA[2], Yukio MORII[2], Toshihiko SASAKI[3] and Yukio HIROSE[3]

[1] *Graduate Student, Dept, of Materials Science and Engineering, Kanazawa University Kakuma-machi kanazawa, 920-1192, Japan*
[2] *Advance Science Research Center, Japan Atomic Energy Research Institute Tokai, Ibaraki 319-1195, Japan*
[3] *Department of Materials Science and Engineering, Kanazawa University Kakuma-machi, kanazawa, 920-1192, Japan*

In a high accuracy residual stress measurement by the neutron diffraction method, it is important to obtain the $d_{0(hkl)}$, the lattice spacing of strain free in the measurement material, exactly. We attempted new method to measure the $d_{0(hkl)}$ by neutron diffraction while sample is rotating at random as an easy method. In this study, the $d_{0(hkl)}$ of the extra super duralumin with texture was measured by our method. This technique is a method of calculating $d_{0(hkl)}$ from a_0 obtained by the extrapolation method. To examine the reliability of this new method, the a_0 obtained by new technique was compared with the a_0 obtained by the conventional powder neutron diffraction. These difference were 8×10^{-5} nm or less. This result confirms that a_0 and $d_{0(hkl)}$ with enough accuracy are obtained by this new technique for the neutron residual stress analysis.

KEYWORDS: neutron diffraction, strain free, lattice spacing, extra super duralmin, texture

§1. Introduction

Even the material is produced from the raw material, the stress is introduced in most cases. However, only the neutron diffraction technique is capable of establishing residual stress in the interior of metallic components nondestructively. Diffraction-based stress analysis utilizes the distance between atomic planes of a crystalline specimen as an internal strain gauge. For any reflection hkl, the lattice spacing $d_{(hkl)}$ can be determined from the observed Bragg angle θ of the appropriate diffraction peak through Bragg's law; $\lambda = 2d_{(hkl)} \sin\theta$, where 2θ is diffraction angle and λ is the neutron wavelength. Differentiation of the Bragg equation gives expressions for the lattice strain:

$$\varepsilon = \Delta d/d = (d_{(hkl)} - d_{0(hkl)})/d_{0(hkl)} = -\cot\theta \cdot \Delta\theta \quad (1.1)$$

where the $d_{0(hkl)}$ is a strain free lattice spacing, $\Delta\theta$ is the shift in Bragg angle corresponding to the lattice strain ε. If $d_{0(hkl)}$ is not accurately obtained, the resolution of strain is remarkably influenced. Therefore, to measure $d_{0(hkl)}$ accurately, it is necessary to use the strain free material. Generally, the following method is used for making the strain free material. 1) An appropriate heat-treating to the test specimen which was cut out from a part of the material. 2) The stress is released by making fine powder from material. 3) After making fine powder from material, heat-treating is given to it. However, preparation of high quality powder sample is actually difficult in that labor, time, knowledge, and technique are required.

We developed a method to measure the $d_{0(hkl)}$ by rotating the sample at random. This technique is a method of calculating $d_{0(hkl)}$ from obtained lattice parameters by the extrapolation method. An aluminium alloy A7075P with strong texture was used in this experiment. Since A7075P has the fcc structure, $d_{0(hkl)}$ can be evaluated from the measurement of a_0. Therefore, only a_0 was measured. To examine reliability of the new method, the a_0 obtained in the experiments was compared with the a_0 obtained by the conventional powder neutron diffraction.

§2. Material and Sample

A7075P is an extra super duralumin. This material is Al-Zn-Mg-Cu alloy of the precipitation hardening type. Two samples of the A7075P were used in this experiment. The first sample was made as a tension test specimen for new technique. Figure 1 shows the form of the test specimen. A texture measurement by neutron diffraction revealed that this material had a remarkable rolling texture which was introduced during the manufacturing process. The second sample was a powdered A7075P by a file. The powder method is a general method for $d_{0(hkl)}$ measurement. The average particle size of the powder was 80 μm.

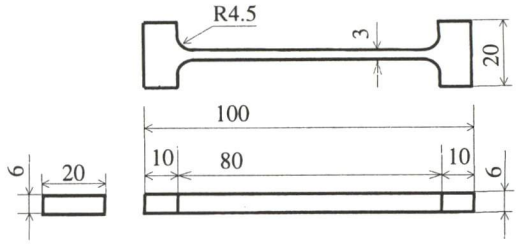
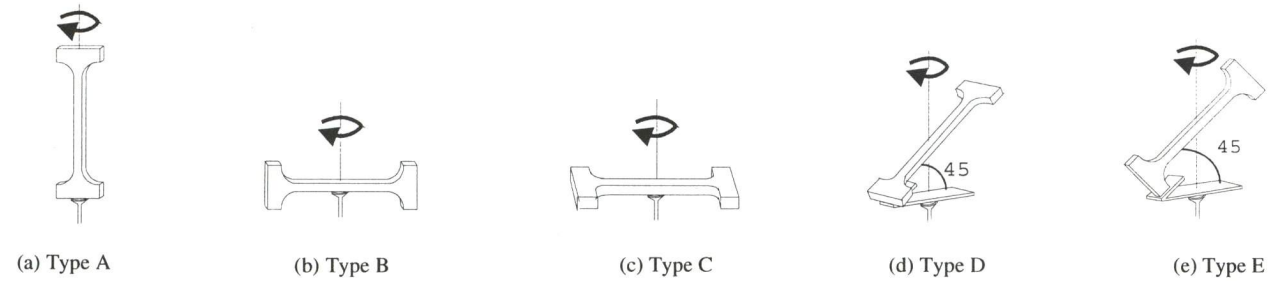


Fig. 1. Form of test specimen.



(a) Type A (b) Type B (c) Type C (d) Type D (e) Type E

Fig. 2. Five kinds of mounting arrangements.

§3. Measurement Equipment

Neutron diffractometer for the residual stress analysis, RESA,[1] installed at thermal neutron guide tube T2-1 of the JRR-3M reactor of Japan Atomic Energy Research Institute was used. Table I shows the measurement conditions.

Table I. Neutron diffraction conditions.

Equipment	RESA
Monochromator Crystal	Si311
Wavelength of Neutron (nm)	0.20696
Beam Collimation	0.2°—0.33°—0.33°
Detector	Zero dimentional ^{3}He
Diffraction Index	Al 111, 200, 220, 311, 222
measuring time (sec.)	60∼120
Scanning Speed (deg/step)	0.1
Slit Size (mm×mm)	15(H)×10(W)
Sample for wavelength calibration	NSB Si Powder
Room Temperature(°C)	23±0.5

§4. The a_0 Measurement by Neutron Diffraction

4.1 Measurement for tension test specimen

In the new technique, it is preferable to measure $d_{(hkl)}$ while sample is turning at random. However, a rotating table used for this experiment has only one axial rotation mechanism. Therefore, the $d_{(hkl)}$ were measured by five kinds of mounting arrangements. Figure 2 shows five kinds of mounting arrangements. The rotating speed was 16 rpm. Figure 3 shows the optical system for $d_{0(hkl)}$ measurement. Diffractions Al111, 200, 220, 311 and 222 were measured from A7075P in this method. The 2θ was obtained by the gaussian fitting.

The a_0 was calculated by the extrapolation method concerning $\cos^2\theta/\sin\theta$.[2] The lattice constant a is calculated by substituting $d_{(hkl)}$ for eq.(4.1).

$$a = d_{(hkl)}\sqrt{h^2+k^2+l^2} \tag{4.1}$$

The calculated a should be linear to $\cos^2\theta/\sin\theta$. This straight line is written by the next equation;

$$a = a_0 + K\cos^2\theta/\sin\theta \tag{4.2}$$

where K is a constant. It is clear that the a becomes the true a_0 at 2θ of high diffraction angle. Namely, a_0 can be obtained at $\cos^2\theta/\sin\theta$=0.

Subsequently, the $d_{0(hkl)}$ can be calculated by using the next equation;

$$d_{0(hkl)} = a_0/\sqrt{h^2+k^2+l^2} \tag{4.3}$$

4.2 Measurement for powder sample

The powder sample is enclosed with the vanadium container of 5 mm in diameter, 50 mm in height, and 0.04 mm in thickness. Diffractions Al111, 200, 220, 311 and 222 were measured from powdered A7075P. Figure

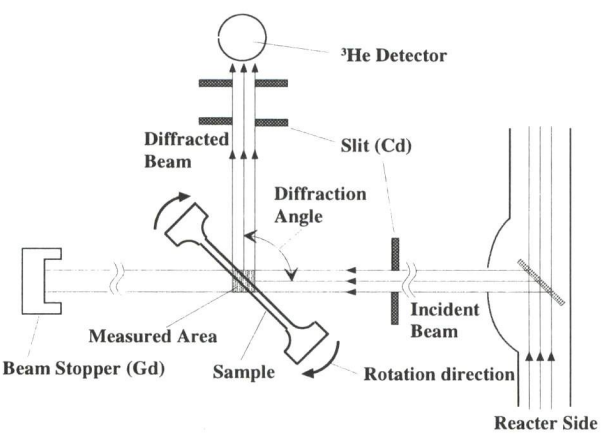


Fig. 3. Optical system for $d_{0(hkl)}$ measurement.

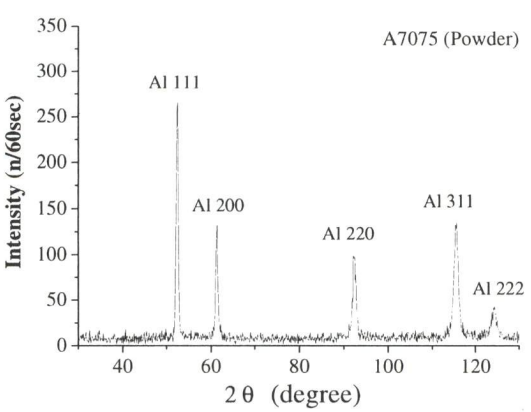


Fig. 4. Diffraction pattern of powder sample.

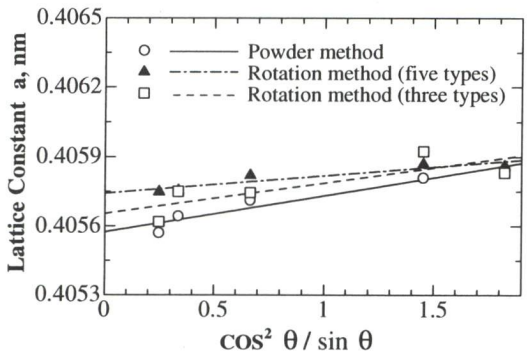


Fig.5. Extrapolation method to calculate lattice constant a_0.

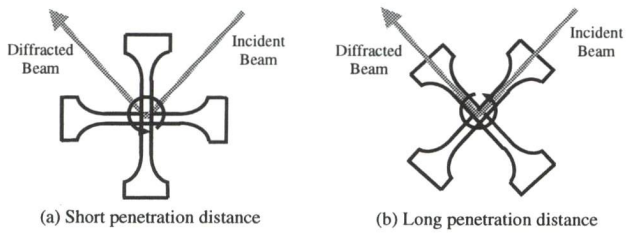


Fig.6. Relation between sample and neutron penetration distance.

TableII. Difference between the a_0 of the powder method and the a_0 of the rotation method. (unit: nm)

Powder method	Rotation method (Five types)	Difference
0.40558	0.40574	0.00016

TableIII. Difference between the a_0 of the powder method and the a_0 of the rotation method by three types. (unit: nm)

Powder method	Rotation method (Three types)	Difference
0.40558	0.40566	0.00008

4 shows the neutron diffraction pattern of the powder sample. The a_0 measurement by the powder method was calculated by the extrapolation method concerning $\cos^2\theta/\sin\theta$, in the same way as the rotation method.

§5. Result of Experiment and Calculation

5.1 The a_0 measurement by the rotation method

The accurate lattice constant a_0 obtained by the powder method and by the rotation method with five kinds of mounting arrangements were calculated by the extrapolation method. Figure 5 shows the graph of the extrapolation method to decide a_0. A solid and an alternate long-short dash line showe the approximation straight line obtained by the powder method and the rotation method, respectively. The intercepts of these lines correspond to a_0 in the graph. Table II shows the calculation results of the powder method and the rotation method. Difference between the a_0 of the powder method and one of the rotation method was 0.00016 nm. In a_0 measurement generally, the measurements accuracy of a_0 should be below 1×10^{-4} nm. The a_0 calculated from the rotation method can't be used for residual stress analysis. It seems that this result was influenced with the form of the test specimen. The penetration distance of the incident beam or the diffraction beam become remarkably long at type B and C in Fig. 2 when 2θ is measured. The absorption coefficient μ is 0.0117 at neutron wavelength 0.21 nm. There is great difference between Fig. 6(a) short penetration distance and (b) penetration distance. The peak position determined by the gaussian fitting is slightly different because of the absorption of the neutron.

Subsequently, the determination of a_0 was attempted by using 2θ measured from type A, D and E without an influence of the penetration distance of the neutron. The approximation straight line calculated from the average diffraction angle obtained with three types (Type A,D and E in Fig. 2) is shown as a short dashes line in Fig. 5. The intercept of this short dashes line approached closer to that of the powder method. Table III shows the calculation results of the powder method and the rotation method with three types. This difference is a half of the value obtained by the rotation method with five types.

§6. Conclusion

We attempted method to measure a_0 easily by neutron diffraction. This technique is a method of calculating a_0 by the extrapolation method. In our method, the a_0 of the extra super duralumin which have the structure of the texture was measured. To examine the reliability of this new method, the a_0 estimated by our method was compared with the a_0 estimated by the powder method. These difference were 8×10^{-5} nm or less. There is a possibility to approach truer a_0 and $d_{0(hkl)}$ if the test specimen not influenced by the neutron penetration distance is measured by the rotation method with five kinds of mounting arrangements. This result confirms that a_0 and $d_{0(hkl)}$ with enough accuracy are obtained by this new technique for the neutron residual stress analysis.

1) M. Hayashi, M. Ishiwata, N. Minakawa, S. Funahashi and J. H. Root: J. Soc. Mat. Sci. Japan. **44** (1995) 504.
2) B. D. Cullity: *Elements of X-Ray Diffraction, 2nd ed.* (Addison-Wesley, Reading, Massa. 1978)