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State of the Art X-ray Diffraction Methods for Measuring Stress State in a Single Crystal

Muneyuki Imafuku, Koichi Akita* and Takahisa Shobu**

Materials Characterization Center, Nippon Steel Technoresearch Corporation,
20-1 Shintomi, Futtsu, Chiba 293-0011, Japan
Fax: 81-439-80-2767, e-mail: imafuku@nstr.co.jp
* Department of Mechanical Systems Engineering, Musashi Institute of Technology,
1-28-1 Tamazutsumi, Setagaya, Tokyo 158-8557, Japan
** Synchrotron Radiation Research Center, Quantum Beam Science Directrate,
Japan Atomic Energy Agency
1-1-1 Kohto, Sayo-cho, Sayo-Gun, Hyogo 679-5148, Japan

Two types of x-ray diffraction methods for measuring stress state in a single crystal are proposed. The first one is tuned to the laboratory x-ray diffraction system. The diffraction data for several equivalent diffraction indices are obtained by using our original apparatus and the multiple regression analysis method is applied for the solution algorism of the plane stress components. The second one is suitable for investigating the residual stress state in an inner part of a single crystal by using high energy synchrotron radiation facility. The stress components can be directly analyzed by solving the stress-strain equation for a single crystal with the diffraction data in three orthogonal directions. The experimental demonstrations are also shown for the residual stress analysis of laser-irradiated Fe single crystal.
Key words: x-ray diffraction, residual stress, single crystal, synchrotron radiation

1.INTRODUCTION

Residual stress state in engineering materials is a critical factor in their practical reliability and performance. The sophisticated x-ray diffraction method, so-called the 2θ-$\sin^2\psi$ method, has been developed and widely used for evaluating the residual stresses by measuring the elastic strains caused by the residual stresses in polycrystalline materials[1]. However, this method cannot be applied to a single crystal or coarse grains in principle. In order to break through this difficulty, we have developed new methods for measuring the stress states in a single crystal by using the laboratory x-ray equipment and the synchrotron radiation facility. In this paper, we intended to show the details of our measurement systems and the new theories for analyzing the stress components in a single crystal from the x-ray diffraction data and demonstrate some applications.

2. EXPERIMENTAL

2.1 METHOD-1

In principle, the stress state in a single crystal can be obtained when the absolute lattice displacements of the several directions can be measured precisely. Although several methods have been examined to investigate the stress state in a single crystal [2, 3], they don't become prevailing techniques because it is difficult to obtain the reliable lattice displacement values. Recently, Yoshioka *et al.* have been proposed a unique and practical x-ray stress measurement method for a single crystal [4]. Being based on the Yoshioka's method, we have improved the accuracy of the measurement system and sophisticated the data analysis algorism. The details of our method are as follows.

Figure 1 shows the crystal coordinate system X_i, the laboratory coordinate system L_i and the specimen coordinate system P_i. ϕ and ψ are the rotation angle between P_1 and L_1 and P_3 and L_3, respectively. A lattice strain of n-th plane, $\varepsilon^{L}_{33(n)}$ in the L_3 direction on the laboratory coordinate system in the plane stress condition is expressed with the stress components σ^{S}_{11}, σ^{S}_{12} and σ^{S}_{22} on the specimen coordinate system by the following equation for a cubic crystal [5]:

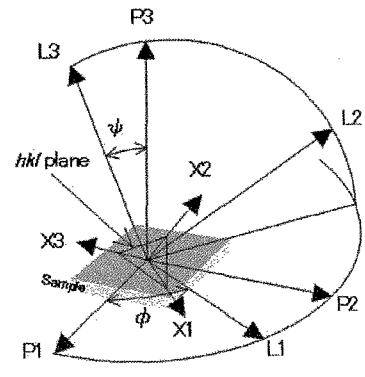

Fig.1 Three coordinate systems: the crystal coordinate system X_i, the laboratory coordinate system L_i and the specimen coordinate system P_i.

$$\varepsilon^{L}_{33(n)} = -(\theta_n - \theta_0)\cot\theta_0$$
$$= (S_{11} - S_{12} - \frac{1}{2}S_{44})[(\gamma^2_{31}\pi^2_{11} + \gamma^2_{32}\pi^2_{12} + \gamma^2_{33}\pi^2_{13})\sigma^S_{11} +$$
$$2(\gamma^2_{31}\pi_{11}\pi_{12} + \gamma^2_{32}\pi_{12}\pi_{22} + \gamma^2_{33}\pi_{13}\pi_{23})\sigma^S_{12} +$$
$$(\gamma^2_{31}\pi^2_{21} + \gamma^2_{32}\pi^2_{22} + \gamma^2_{33}\pi^2_{23})\sigma^S_{22}] + S_{12}(\sigma^S_{11} + \sigma^S_{22}) +$$
$$\frac{1}{2}S_{44}(\sigma^S_{11}\sin^2\phi - \sigma^S_{12}\sin 2\phi + \sigma^S_{22}\cos^2\phi)\sin^2\psi$$
$$= A_n\sigma^S_{11} + B_n\sigma^S_{12} + C_n\sigma^S_{22} \quad (1)$$

Where, S_{ij} is the elastic compliance of the cubic crystal. The transformation matrices, π and γ are between the specimen and crystal coordinate systems, and crystal and laboratory coordinate systems, respectively. In this equation, θ_0 is the half of the diffraction angle in the stress-free condition, and is usually unknown. A_n, B_n and C_n are the variables which can be determined by the Miller indices of the measured diffraction planes. θ_n is expressed as the following equation:

$$2\theta_n = -[(\frac{2\sigma^S_{11}}{\cot\theta_0}A_n + \frac{2\sigma^S_{12}}{\cot\theta_0}B_n + \frac{2\sigma^S_{22}}{\cot\theta_0}C_n)] + 2\theta_0 \quad (2)$$

By choosing the equivalent diffraction planes, σ^S_{11}, σ^S_{12}, σ^S_{22} and θ_0 can be calculated by the multiple regression analysis method. In this analysis, at least four equivalent diffraction planes should be measured. It should be noted that the θ_0 value don't need to be measured. Instead, it can be determined form the sample values.

Figure 2 shows our new x-ray stress measurement apparatus for a single crystal. Diffracted x-ray is detected by the one dimensional position sensitive proportional counter (PSPC). The $\phi\psi$-oscillation stage is installed in the tube-type x-ray generator system. In order to obtain a perfect x-ray diffraction profile of the target diffraction plane with PSPC, coupled two-axis oscillation of the sample is necessary. Some types of two-axis combination is possible. For example, our original prototype system adopted $\chi\psi$-oscillation stage [6]. Because the adjustment of the rotation center of the sample is simpler for $\phi\psi$-oscillation than for $\chi\psi$-oscillation, our new apparatus adopted the former oscillation mode. The accuracy of the rotation center of the stage is within ±5 μm error by using laser displacement meter and microscope. A shield-tube x-ray source of Cr-Kα radiation with a low power of 30 KV and 10 mA was used in this study. The diffraction intensity of a single crystal is very high and so the power of x-ray source should be tuned down in order to avoid the saturation of PSPC. The diameter of x-ray collimator is typically 0.3 mm, which can be selected to suite the experimental needs.

We will show the experimental result for a laser-irradiated Fe single crystal sheet of 0.23 mm thick. The orientation of the sample is about {110}<001>, which is known as the Goss orientation. Nd-YAG pulse laser was irradiated with the energy of 3.3 mJ/pulse along <1 $\bar{1}$ 0> direction (named 2-direction) at 0.3 mm intervals. The shape of each cavity is about 100 μm in diameter and about 4 μm in depth [6]. This kind

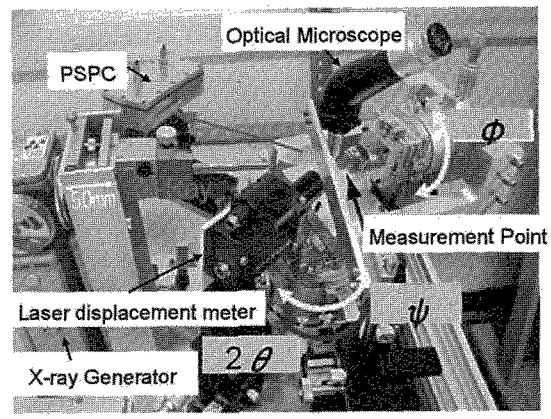

Fig.2 The x-ray stress measurement apparatus for a single crystal.

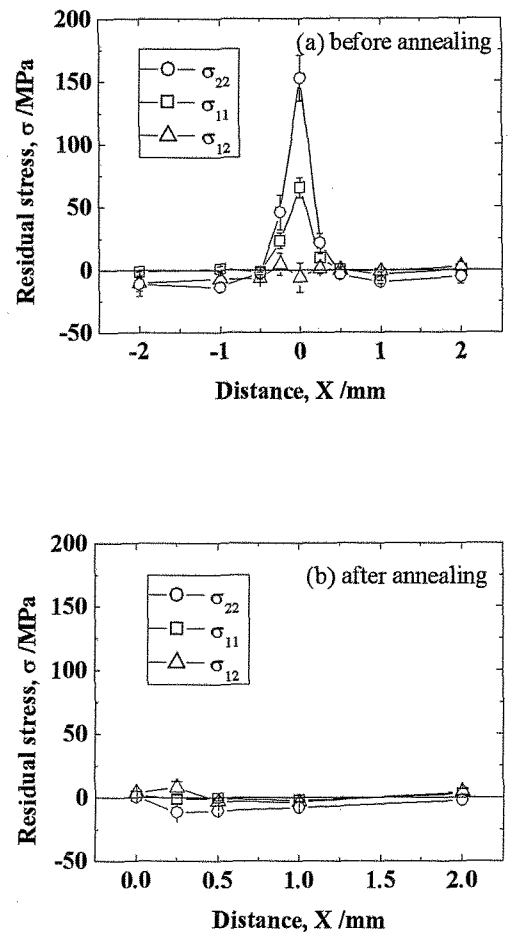

Fig.3 The residual stress distributions in laser-irradiated Fe single crystal (a) before and (b) after annealing.

of laser irradiation process is known as the magnetic domain refinement process for producing high performance electrical steels [7].

Six equivalent 211 diffraction planes of α-Fe, 211, 112, 121, $12\bar{1}$, $11\bar{2}$, and $21\bar{1}$, satisfied reflection condition and were chosen for the measurement. Figures 3(a) and (b) show the stress components σ_{11}, σ_{12} and σ_{22} around the laser-irradiated cavity line along <001> direction (named 1-direction) before and after annealing at 800 °C for 2 hours in H_2 atmosphere, respectively. We can see from Fig. 3(a) that the tensile stresses of about up to 70 MPa in 1-direction and 160 MPa in 2-direction were induced just around the laser-irradiated cavity line. The value of σ_{22} was almost two times larger than that of σ_{11} since the difference of elastic constant between 1- and 2-directions (E_1=132 GPa nad E2=220 GPa) [8]. The stress induced area was less than 0.5 mm, that is to say, the very limited area. σ_{11} and σ_{22} are considered to be the principle stresses in the surface plane because the shear stress, σ_{12} is negligible as is seen in this figure. After annealing, the residual stresses are completely relieved as is shown Fig. 3(b).

2.2 METHOD-2

The second method is devised for investigating the residual stress state in an inner part of a single crystal, which is not anymore a plane stress state. The stress-strain equation in three-dimensional stress state is expressed as follows:

$$\varepsilon_{33}^{L} = S_0 \sum_i \sum_j \sigma_{ij}^{P} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) + S_{12} \left(\sigma_{11}^{P} + \sigma_{22}^{P} + \sigma_{33}^{P} \right) + \frac{1}{2} S_{44} \sigma_{33}^{P}$$
$$+ \frac{1}{2} S_{44} \left(\sigma_{11}^{P} \sin^2 \phi - \sigma_{12}^{P} \sin^2 2\phi + \sigma_{22}^{P} \cos^2 \phi - \sigma_{33}^{P} \right) \sin^2 \psi$$
$$+ \frac{1}{2} S_{44} \left(\sigma_{31}^{P} \sin \phi - \sigma_{23}^{P} \cos \phi \right) \sin 2\psi \qquad (3)$$

where the suffixes of 1, 2 and 3 mean P_1, P_2 and P_3 directions in Fig. 1, respectively.

We can transcribe this equation for three orthogonal directions; 1, 2 and 3. These are shown as follows:

$$\varepsilon_{33}^{L} = S_0 \sum_i \sum_j \sigma_{ij}^{P} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) +$$
$$S_{12} \left(\sigma_{11}^{P} + \sigma_{22}^{P} + \sigma_{33}^{P} \right) + \frac{1}{2} S_{44} \sigma_{33}^{P} + \frac{1}{2} S_{44} \left(\sigma_{11}^{P} - \sigma_{33}^{P} \right) \qquad (4-1)$$

$$\varepsilon_{33}^{L} = S_0 \sum_i \sum_j \sigma_{ij}^{P} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) +$$
$$S_{12} \left(\sigma_{11}^{P} + \sigma_{22}^{P} + \sigma_{33}^{P} \right) + \frac{1}{2} S_{44} \sigma_{33}^{P} + \frac{1}{2} S_{44} \left(\sigma_{22}^{P} - \sigma_{33}^{P} \right) \qquad (4-2)$$

$$\varepsilon_{33}^{L} = S_0 \sum_i \sum_j \sigma_{ij}^{P} \left(\sum_k \gamma_{3k}^2 \pi_{ik} \pi_{jk} \right) +$$
$$S_{12} \left(\sigma_{11}^{P} + \sigma_{22}^{P} + \sigma_{33}^{P} \right) + \frac{1}{2} S_{44} \sigma_{33}^{P} \qquad (4-3)$$

where equations (4-1), (4-2) and (4-3) correspond to the stress-strain equations in 1, 2 and 3 directions, respectively. We can solve the simultaneous liner equations directly if the strain data along the 1, 2 and 3 direction are obtained.

This method is suitable for high energy synchrotron radiation facility. By using high energy x-ray (typically 30-70 keV), the x-ray beam can reach deep inside of the material. Furthermore, synchrotron radiation x-ray beam can be easily collimated to be a very small size less than 0.1 mm. Consequently, we can measure the x-ray diffraction profiles from the micro part area into a bulk material along three orthogonal directions.

We performed synchrotron radiation x-ray diffraction measurement at the BL22XU of JAEA in the SPring-8. Figure 4 shows the experimental setup for this experiment. The sample is also the laser-irradiated {110}<001> Fe single crystal. The diffraction indices are Fe 006 for 1-direcction (<001>), Fe $4\bar{4}0$ for 2- direction (<1 $\bar{1}$ 0 >) and Fe 440 for 3- direction (<110>), respectively. For these experimental conditions, eqs. (4-1), (4-2) and (4-3) can be transformed to eqs. (5-1), (5-2) and (5-3).

$$\varepsilon_{33}^{L} = (S_0 + S_{12} + \frac{1}{2} S_{44}) \sigma_{11}^{P} + S_{12} \sigma_{22}^{P} + S_{12} \sigma_{33}^{P} \qquad (5-1)$$

$$\varepsilon_{33}^{L} = S_{12} \sigma_{11}^{P} + (\frac{1}{2} S_0 + S_{12} + \frac{1}{2} S_{44}) \sigma_{22}^{P} + (\frac{1}{2} S_0 + S_{12}) \sigma_{33}^{P} \qquad (5-2)$$

$$\varepsilon_{33}^{L} = S_{12} \sigma_{11}^{P} + (\frac{1}{2} S_0 + S_{12}) \sigma_{22}^{P} + (\frac{1}{2} S_0 + S_{12} + \frac{1}{2} S_{44}) \sigma_{33}^{P} \qquad (5-3)$$

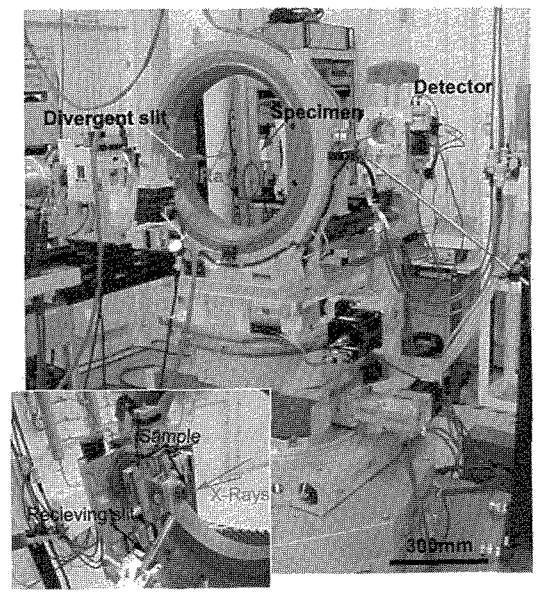

Fig.4 Experimental setup at the BL22XU of JAEA in the SPring-8.

The energy and the size of x-ray beam were 30 keV and 50 μm X 50μm, respectively. Figure 5 illustrates the diffracted area, which is called the gauge volume. The values of W and H are 118 μm

and 55 μm, respectively for 2θ=50° . The depth profile of the strain data were obtained just below the laser-irradiated spot up to 0.18 mm at 0.03 mm intervals. Figure 6 shows the depth profiles of strain values along the three directions. From this figure, it is clear that the extremely anisotropic strain state is introduces by laser irradiation. Especially, relatively large compressive strains are introduced along 1-direction. Figure 7 shows the derived depth profiles of stress values calculated by solving simultaneous linear eqs. (5-1), (5-2) and (5-3). We could firstly know that the anisotropic three-axis compressive residual stress state just below the surface.

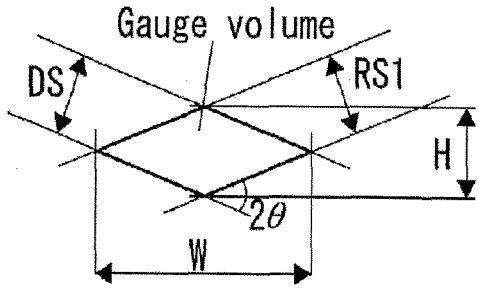

Fig.5 Illustration of gauge volume for the x-ray diffraction experiment.

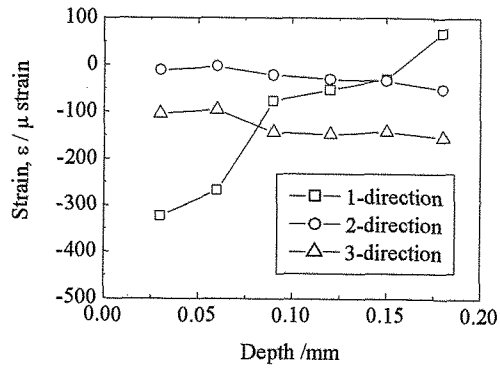

Fig. 6 Depth profiles of strains along the three orthogonal directions (1-, 2- and 3-directions).

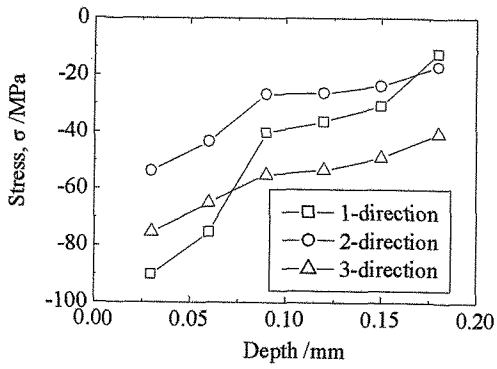

Fig. 7 Depth profiles of residual stresses along the three orthogonal directions (1-, 2- and 3-directions).

As the main subject of this paper is focused on the x-ray measuring techniques, the physical origin of the stress state in the present demonstration sample will be discussed in a separate paper.

3. CONCLUDING REMARKS

We have developed two types of x-ray diffraction methods for measuring stress state in a single crystal.

The first one is tuned to the laboratory x-ray diffraction system for measuring the plane residual stress components at the surface. The second method is devised for investigating the residual stress state in an inner part of the crystal. This method is suitable for high energy x-ray diffraction measurement with synchrotron radiation source. By using these methods, we could successfully analyze the residual stress state for a laser-irradiated Fe single crystal.

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