Unit Cell Definition of Polycrystal Sheet Material Based on SEM-EBSD Analyses

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Abstract. Recently, the multi scale analysis technology, by using the crystallographic homogenization based dynamic explicit finite element code, has been developed to assess macro continuum sheet material properties and further study the deformation and straining in the actual sheet forming. This homogenization finite element code employs the two scale hierarchical structure, which consists of a polycrystal microstructure and a macro continuum. In this study, for the reality, we focus to discuss “How to define a proper microstructure for the two scale finite element analysis, by employing the real measurement base polycrystal aggregation, which has been obtained by SEM-EBSD” observations. For scaling up from the micro polycrystal structure to the macro continuum, we define a unit cell by looking at the periodicity of crystal orientation, which is one of morphological factors to feature the polycrystal structure, which is named as “texture.” Through a statistical study of these measured polycrystal morphologies, finally we found a realistic polycrystal unit cell of the microstructure, which can be adopted for our multi scale finite element analyses.

INTRODUCTION

Recently, a high formability and a high strength have been required in automobile materials. Accordingly, the development of technologies to control crystals, including miniaturization of crystal grains and use of deposition effects, to increase their strength, while maintaining a high formability, has been promoted. In this study, we aim to develop a technique to evaluate macroscopic material properties, such as the strength and the formability, based on microcrystal structural analyses, by using the scanning electron microscopy and the electron backscattering diffraction (SEM-EBSD) analyses. That is, through obtaining a representative unit cell of three dimensional microcrystal structure, defined by using the crystal orientation data, which is used for our multi scale finite element analyses.

CRYSTALLOGRAPHIC HOMOGENIZATION METHOD

Basiclly, the periodicity of crystal morphologies, such as the grain shape and the orientation, should be satisfied in a unit cell of microcrystal structure as shown in Figure 1, which is used in the multi scale finite element analyses to predict macroscopic material properties. In this study, we try to define a unit cell from 3-dimensional (3D) SEM/EBSD measurements of pure-iron crystals and high-tensile-strength steels by looking at the periodicity of the crystal orientation.

FIGURE 1 Micro crystal and macro structures.
Designating the coordinate of macro continuum as $x$, and that of micro crystal structure as $y$, the relationship between the two coordinates is expressed using a scale ratio $\lambda$. A micro crystal structure of metal has a feature of inhomogeneity. This microscopic property can be reflected in the prediction of macroscopic properties by using the crystallographic homogenization method. In this method, the homogenization of properties of a micro crystal unit cell is executed. To define the unit cell, we have used a SEM-EBSD measured polycrystal morphological data.

**SEM-EBSD MEASUREMENTS OF CRYSTAL MORPHOLOGIES**

At first, we have observed morphologies of polycrystal of pure iron, at the initial and 30% tensile deformation (RD) states. Figure 2 shows the results of SEM-EBSD measurements with the highest accuracy, 0.1µm resolution. The orientation color maps in ND and RD directions show clearly the difference between the initial and 30% straining states. At the initial state, a maximum deviation of angle 3 degree is observed by looking at each crystal grain. It suggests that each grain has almost one crystal orientation. On the other hand, at 30 % tensile strain state, 10 degree maximum deviation angle was obtained by investigating each grain. It is concluded that the plastic strain induced sub-graining, the miniaturization, was occurred. These results suggest that a large number of finite element integration points inside one crystal is required to detect the plastic strain induced crystal rotation.

**Pure Iron: 3D micro crystal structures of pure iron (purity: 99.9%) plates at the initial and 30% tensile strain states were obtained by SEM-EBSD observations.**

Figure 3 shows a specimen at 30 % strain, which has a parallelepiped region of 5 mm width and 17 mm length and 0.5mm thickness. The wire-cut was executed to get a smaller size of rectangular plate (3mm x 1mm) as shown in Figure 4. The etching treatment was employed for SEM-EBSD measurement, and the continuous etching has been done in a laminar manner. Finally, we obtained the distribution of crystal orientation in a 3D parallelepiped region, 210.0 µm x 156.0 µm x 200.0 µm. At the initial state, the interval of measurement in plane in both directions, RD and TD, was 3.81 µm, and that in the thickness direction was 5.0 µm; measurements were performed for 40 layers. The number of crystal grains contained in the measurement region was 454, with a mean grain size of 27 µm. Figure 5 shows 3D orientation color map at described depth layers(Z: the height from the bottom). Figure 6 shows {111}, {100} and {110} pole figures and inverse pole figures(ND,RD and TD). It shows clearly the preferred orientations and $\gamma$ fiber texture.

**FIGURE 2.** Orientation color maps at the initial and 30% tensile strain states in the case of pure iron plate.

**3D SEM-EBSD MEASURED MICRO CRYSTAL STRUCTURE**

(a) Size of specimen.

(b) Cut out for SEM-EBSD measurement.

**FIGURE 3.** Tensile specimen size and wire cutting Configuration.

**FIGURE 4.** The etching apparatus and the specimen.
FIGURE 5. 3D orientation (RD) color maps of the pure iron at the initial state.

FIGURE 6. Pole figures and inverse pole figures of the pure iron at the initial state.

FIGURE 7. 3D orientation (RD) color map of the pure iron at 30% strain state.

FIGURE 8. Pole figures and inverse pole figures of the pure iron at 30% strain state.
Similarly, 3D orientation color map, pole figures and inverse pole figures, in the case of 30% strain state, were obtained as shown in Figures 7 and 8. The size of measured rectangular parallelepiped was 209.6 \( \mu m \) x 156.2 \( \mu m \) x 177.6 \( \mu m \). The interval of measurement in the plane in both directions, RD and TD, was 3.8 \( \mu m \) and that in the thickness direction was 4.8 \( \mu m \), and the measurement was performed for 37 layers in the thickness direction. The number of crystal grains contained in the measurement region was 1450, with a mean grain size of 16.0 \( \mu m \). From this, it was found that the average size of crystal grain is reduced by subgraining due to the plastic deformation. Further, it shows clearly the plastic strain induced texture evolution, rotated toward [101] and [111] directions.

**High Tensile Strength Steel (HSLA):**

Similarly, SEM-EBSD measurements were executed for a high tensile strength steel (HSLA) polycrystal sheet material. Figures 9 and 10 show a 3D orientation color map, pole figures and inverse pole figures. The size of measured rectangular parallelepiped was 229.0 \( \mu m \) x 149.0 \( \mu m \) x 172.0 \( \mu m \). The interval of measurement in the plane in both directions, RD and TD, was 3.8 \( \mu m \) and that in the thickness direction was 4.3 \( \mu m \), and the measurement was performed for 40 layers in the thickness direction. The number of crystal grains contained in the measurement region of top layer was 240, with a mean grain size of 11.0 \( \mu m \). It shows a different texture, such as \( \alpha \) fiber, and a smaller grain size than ones of the pure iron.

**DERIVATION OF MICRO CRYSTAL STRUCTURE “UNIT CELL”**

We try to find unit cells that satisfy periodicity of the crystal orientation, which was measured by SEM-EBSD apparatus. We cut off candidate cubes.
arbitrarily from the maximum measured regions, as shown in Figs. 5, 7 and 9. The frequency of crystal orientation angles of candidate cube were examined by comparison with ones of maximum measured regions, such as 90200 orientations in the case of the pure iron at the initial state, 83435 the pure iron at 30% strain and 93600 the HSLA. We employed these numbers as the populations. Two angles between (111) of crystal and ND and RD as shown in Figure 11, α and β, were selected to define the crystal orientation uniquely. Figures 12(a) and (b) show probability histograms of angles with the standard deviation bars in cases of 60µm and 100µm cubes. The total number of candidate cubes was calculated by employing the intervals in each direction, as 3.81µm in RD and TD directions, and 4.0µm and 3.7µm in ND direction. These probability histograms show that the smaller the side length of cube, the larger the fluctuation from the population, which is obtained in the maximum measured region. The error sums of squares in each candidate side length were determined as follows. At first, the frequency distribution of the angle n in the candidate cube as $x_n$, and one in the population as $\bar{x}_n$ were calculated. Second, the error sum of square of the difference between frequency distributions of one candidate cube and the population as follow.

\[ S = \sum_{n=0}^{180} (x_n - \bar{x}_n)^2. \]  

This error sum calculation was repeated for every cube, which had the same side length, and picked up over all in the maximum measured region. Repeating “m” times to evaluate the error sum $S_n$, finally, the error sum of squares $S$ can be determined by averaging as follow.

\[ \overline{S} = \frac{S_1 + S_2 + \cdots + S_{m-1} + S_m}{m} \]  

For the judgment of convergence, a threshold value of the error sum of squares, $1.0 \times 10^{-6}$, is employed. It means that a cube, whose crystal orientation distribution is almost same as the maximum measured region, can be assumed a unit cell. It has been confirmed that the probability histogram of crystal
orientation in each maximum measured region rigorously coincides with one of very large area, such as more than 1000 μm square region on the specimen surface, whose total number of orientation is 700,000.

Unit Cells of Pure Iron

A unit cell was derived by using the judgment of the error sum of squares with a threshold value of $10^6$. Figure 13(a) shows the relationships the side length of candidate cube and the error sum of squares in cases of angle $\alpha$ and $\beta$, defined in Fig. 11. The error sum of squares of a 100 μm cube was obtained as a value $2.83 \times 10^6$ for angle $\alpha$, and one as $3.37 \times 10^6$ for angle $\beta$. Accordingly, we determined a 100 μm cube to be the unit cell satisfying periodicity. The number of this unit cell of 100 μm cube contains 50 crystal grains.

In the case of 30% tensile strain state, Figure 13(b) shows similar results with ones at the initial state. Again a unit cell for this deformed pure iron plate was determined as 100 μm cube. These error sums of squares were $2.83 \times 10^6$ in the case of angle $\alpha$, and $1.97 \times 10^6$ for angle $\beta$. It contains 240 crystal grains. The plastic strain induced crystal miniaturization increases the number of crystal. It usually reduces the size of unit cell, but one grain has a large deviation of angle. Consequently, the size of unit cell is not reduced.

Unit Cells of HSLA

Similarly as the case of pure iron, a unit cell of HSLA was determined as 100 μm cube by employing the threshold value of $10^6$. The relationship between the error sum of squares and the side length of cube is shown in Figure 14. These values of error sums of the unit cell were $1.97 \times 10^6$ for angle $\alpha$ and $1.91 \times 10^6$ for angle $\beta$, respectively. It might have 730 grains. It seems very large, but the reason could be the scattering of the orientation distribution. The more scattering the crystal orientation, requires the more the crystal.

Figures 15(a) and (b) show unit cells of pure iron and HSLA with the orientation color map at the initial state.

**CONCLUSION**

In this study, below described results were obtained. (1) 3D micro crystal structures of pure iron sheet material, at the initial and 30% tensile strain states, and HSLA at the initial state were determined by using SEM-EBSD apparatus and the etching machine. (2) Unit cells, which satisfy the periodicity of crystal orientation, were determined by using the error sum of squares of orientation probability histogram. Three unit cells have the same size of cube, such as 100 μm, but the difference grain numbers, such as 50, 240 and 730, and textures. It means that the plastic strain induced subgraining and texture evolution have been detected accurately by our SEM-EBSD measurements.

**REFERENCES**