Effect of Microstructure Variation on Differential Hardening Behavior of Steel Sheets under Biaxial Tensile State

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For further improvement of press formability of steel sheets, it is important to clarify the relation between macroscopic mechanical properties and microstructure under multi-axial deformation state. The objective of this work is to correlate the work hardening behavior with microstructure evolution under biaxial tensile state. The materials used in this study were interstitial-free (IF) steels with different grain sizes. First, the work hardening behaviors were examined by conventional uniaxial tensile and bulge tests, and the differential hardening behavior was measured. Next, in-situ observation of microstructure evolution was conducted for uniaxial and biaxial tensile deformation using the microscopic biaxial tensile system with electron back scatter diffraction patterns (EBSD) with scanning electron microscope (SEM) analysis. The texture development and inhomogeneous deformation were observed clearly under the biaxial tensile state. The effect of texture development on the yield locus was investigated using Taylor-Bishop-Hill (TBH) theory and the orientation distribution function (ODF). It was found that the differential hardening at the grain level was caused by the crystal rotation. Finally, the inhomogeneous deformation was analyzed. The material with fine grain exhibited relatively homogeneous deformation and high work hardening ratio even over a large strain range under biaxial tensile state. It was suggested that the texture development over a large strain range and the homogeneity of deformation resulted in the differential hardening behavior.

KEY WORDS: in-situ observation; interstitial-free steel; electron back scatter diffraction patterns; scanning electron microscope; biaxial tensile test; differential hardening.

1. Introduction

For weight reduction and improvement in collision safety thin steel sheets with high strength are being progressively applied to car components.1) Ductility is usually lower for higher strength steel sheets. Further, in recent years, the shape of car components tends to be complicated from the viewpoint of satisfying the elaborate car design. Therefore, the difficulty in press forming of car components is increasing. The press forming processes are examined in advance by numerical simulation using finite element methods (FEM). The accuracy of the numerical simulation is, however, still not sufficient to predict forming defects, such as surface roughness2) and distortion,3) of micron order. To improve the prediction accuracy, it is important to accurately model the deformation behaviors of materials in multi-axial tensile state. One of the important behaviors is work hardening.

Morrison4) investigated the effect of grain size on the work hardening exponent, $n$, in uniaxial tensile state, and proposed the relationship equation of $n = 5/(10 + d^{-1/2})$, where $d$ is the grain diameter. Masui et al.5) further investigated the effect of grain size separating from the other factor and demonstrated that the $n$-value increased with grain size. Leslie et al.6) investigated the effect of addition of alloy elements on the $n$-value. It is well known that the $n$-value decreases generally with increase in the addition of alloy elements, and the purification of steel is considered as a basic direction to increase the $n$-value. Uenishi et al.7) conducted the shear tests using the specimens composed of a single crystal for several representative orientations and reported that the work hardening behavior was notably different for each crystal orientation.

The work hardening properties in multi-axial stress state have also been studied.8,9) Abe et al.10) investigated the relation between work hardening rate and crystal orientation in multi-axial stress state using stretching test of single crystal sheet. The crystals with one of the {111} planes aligning in the normal direction (ND) showed the highest $n$-value, while those with the {901} plane orientation showed the lowest $n$-value. And they considered that the influence of orientations on the $n$-value was weakened by crystal boundaries for polycrystalline steels.

Kuwabara et al.11) developed the biaxial tensile test system, and measured the work hardening of various sheet
materials. They reported that the work hardening behavior in the biaxial tensile test was different from that in the uniaxial tensile test.\textsuperscript{12}) Namely, in the biaxial tensile state, the work hardening was more than that in the uniaxial state and the contour of plastic work expanded in the first quadrant at the initial stage of deformation. Further, the work hardening decreased and the contour shrank at the later stages of deformation. The difference was observed notably for interstitial-free (IF) steels. The change in the contour of plastic work is called the differential hardening.\textsuperscript{13,14}) They supposed that the effect of crystal texture on differential hardening was less because the crystal texture hardly changed at the initial biaxial deformation.

Ikeematsu et al.\textsuperscript{15}) observed the difference in the dislocation structure between uniaxial and biaxial tensile tests. They considered that the mutual interaction of dislocation and grain boundary affects differential hardening.

Referring to the above studies it is supposed that crystal orientation or grain boundary affects differential hardening.

Numerical studies have also been conducted actively in recent years using information of microstructure and crystal orientation. Van houtte\textsuperscript{16}) proposed a crystal plasticity analysis of polycrystalline material based on Taylor-Bishop-Hill (TBH) theory, and calculated yield locus by the orientation distribution function (ODF), referring to the study by Van houtte.\textsuperscript{16}) Furthermore, the effect of inhomogeneous microscopic deformation on macroscopic work hardening behavior is discussed by analyzing the observed phenomena in a microscopic biaxial tensile test.

2. Experimental Procedures

2.1. Materials

The test materials used in this study are three types of IF steel sheets with a single phase of ferrite. The materials A, B, and C exhibit average grain sizes of 29, 16, and 9.3 $\mu m$ in ND, respectively. Table 1 shows the mechanical properties of the materials obtained by the uniaxial tensile test in macroscopic scale. The gauge length and width were 50 and 25 mm, respectively, and the strain rate was $1.0 \times 10^{-3} s^{-1}$. The properties except for $r$-values are indicated with those in the rolling direction (RD). The $r$-values were measured at a plastic strain of 0.1. Note that the average $r$-values are greater than 1.5. The peculiar work hardening has been reported in an IF steel sheet,\textsuperscript{15)} which has $r$-value of larger than 1.5.

2.2. Biaxial Tensile Test in Macroscopic Scale

In addition to the uniaxial tensile test described above the equi-biaxial bulge test\textsuperscript{28)} shown in Fig. 1 was conducted using a die with a diameter of 100 mm and a rectangular

![Fig. 1. Experimental setup for bulge test.](image_url)

<table>
<thead>
<tr>
<th>Material</th>
<th>Initial thickness [mm]</th>
<th>YP* [MPa]</th>
<th>TS* [MPa]</th>
<th>u-EL* [%]</th>
<th>EL* [%]</th>
<th>$r$-value*\textsuperscript{2}</th>
<th>Average $r$-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1.6</td>
<td>150</td>
<td>286</td>
<td>29</td>
<td>54</td>
<td>1.7</td>
<td>1.5</td>
</tr>
<tr>
<td>B</td>
<td>0.75</td>
<td>185</td>
<td>340</td>
<td>25</td>
<td>44</td>
<td>1.2</td>
<td>1.8</td>
</tr>
<tr>
<td>C</td>
<td>0.75</td>
<td>270</td>
<td>447</td>
<td>20</td>
<td>34</td>
<td>1.1</td>
<td>1.7</td>
</tr>
</tbody>
</table>

* Measured at plastic strain of 0.10.
** Measured in rolling (0°) direction.
specimen with a side length of 150 mm. The sheets were deformed by hydrostatic pressure with a blank holder force of 100 kN. The strain rate was the same as that in the uniaxial tensile test. The biaxial tensile stress, \( \sigma_b \), was calculated by the following equation.

\[
\sigma_b = \frac{p \rho}{2t}
\]

where \( p \) is the inner pressure, \( \rho \) is the average curvature radius at thickness center, and \( t \) is the thickness at the center of specimen. \( t \) was calculated from strains on the sheet surface assuming volume constancy. The strain measurement system of AutoGrid® with four optical cameras was employed for analyzing strains on the surface.

2.3. Observation of Microstructural Evolution in Uniaxial and Biaxial Tensile Tests in Microscopic Scale

To observe the microstructural evolutions during deformation, uniaxial and equi-biaxial tensile tests in microscopic scale were conducted. Figure 2 shows the experimental system for the tests with in-situ SEM-EBSD analysis developed in our previous study.27) Here, we briefly explain the test system again. The size of the apparatus is 166 mm in length, 140 mm in width and 42 mm in height, owing to the limitation of space in the vacuum chamber of field emission SEM (FE-SEM). The two servomotors enable biaxial tension. The displacements of \( x \) and \( y \) crossheads are synchronized by software. In case of an uniaxial tensile test, only the \( y \)-axis direction in Fig. 2 is utilized. The displacement of each crosshead is measured using a step gauge on the back surface of crosshead. After providing predetermined displacements in both tensile directions using software control, the crossheads are held mechanically by using gears embedded in the system. Subsequently, in situ SEM-EBSD analyses are conducted under uniaxial and biaxial tension. The backward displacement during the holding state is approximately only 1 \( \mu \)m in each tensile direction. The crystal orientations on the top surface of the specimen are calculated by OIM collection v7.1. The SEM observations and EBSD analyses are performed at 500 magnification in the square visual field with a side length of 200 \( \mu \)m at the center of specimen. The step size of EBSD analysis is 1.0 \( \mu \)m. The strain during the test was calculated from the displacement of crosshead. And the thickness plastic strain was calculated assuming the volume constancy.

Figures 3 and 4 show the specimens for the microscopic uniaxial and equi-biaxial tensile tests, respectively. The shape and dimensions of specimen were determined from FEM.27) The specimens were cut from the sheets by a wire-electrical discharge machine. The X shaped reduction area (Fig. 4(d)) was fabricated by machining. The reduced thickness, \( t_r \), was approximately 30% of the initial thickness, \( t_i \). RD was set to the tensile direction in uniaxial test and one of two tensile directions in biaxial test. The top side surface was buffed and electrically polished for the SEM-EBSD analysis.

3. Results

3.1. Work Hardening Behaviors in Uniaxial and Equibiaxial Tensile States

Figure 5 shows the comparison of the work hardening behaviors between uniaxial and biaxial tensile tests in macroscopic scale for the three materials, namely, A, B,
and C. The absolute value of the plastic strain in thickness direction, $|\varepsilon^p_t|$, is used for the biaxial tensile test. In the early stage of deformation up to the strain of approximately 0.05, the work hardening rate is larger for the biaxial tensile test than for uniaxial one in all the materials. These are the so-called differential hardening behaviors, similar to those reported by Kuwabara et al. However, it is observed from Fig. 5 that in a strain range larger than 0.10, the work hardening rates differ depending on the materials. The work hardening rate in a biaxial test becomes the same level as or partially somewhat smaller than that in uniaxial test for the material A. In contrast, the work hardening rate in the biaxial test is still larger than that in the uniaxial test for the material B and, especially, for the material C. Although the bulge test was conducted as well as the uniaxial tensile test under a sufficiently static strain rate of $1.0 \times 10^{-3}$ s$^{-1}$, the fluctuation was observed in the hardening rate of the material A in bulge test. The tests were conducted twice for each material, and the similar results were obtained. The fluctuation is supposed to be peculiar to the material A.

In general, the differential hardening behavior needs to be evaluated using contours of plastic work. Here, we define the stress ratio, $X$, as an index to analyze the differential hardening behaviors as follows:

$$X = \frac{\sigma_b}{\sigma_u}$$

where $\sigma_b$ is the true stress at a certain plastic work in the uniaxial tensile test, and $\sigma_u$ is the true stress in the biaxial tensile test at the same plastic work as that in the uniaxial test. The value of $X$ is constant when the material exhibits the isotropic hardening behavior. In other words, the increase in $X$ means that the work hardening in the biaxial tensile state becomes greater than that in the uniaxial state. The differential hardening behaviors for the large deformation range are different depending on the material.

### 3.2. Microstructural and Textural Evolutions in Uniaxial and Equi-biaxial Tensile Deformation

From here, the mechanism of the abovementioned differential hardening is examined from the view point of the microstructural change in uniaxial and biaxial tensile states. Figures 7 and 8 show the microstructural evolutions during uniaxial and biaxial tensile tests in microscopic scale for the material A, respectively. In these figures four types of maps before deformation and at two levels of strain are illustrated. The maps are the image quality (IQ) map, the inverse pole figure (IPF) map, the grain average misorientation (GAM) map and the Taylor factor map. The IQ map is employed to simply analyze accumulated strain energy. The IPF maps observed in the ND are employed to indicate the distributions of crystal orientation. The GAM maps are used to indicate the degree of deformation of grains. The Taylor factor maps are used to relatively indicate the deformation resistance of grains. The Taylor factor maps are calculated for 40401 observation points by the OIM Analysis v7.2.1, assuming 24 slip systems of BCC structure of the families {110} $< 111 >$ and {211} $< 111 >$ and the same critical resolved shear stress (CRSS) for both families of slip systems. Uniform uniaxial tension in RD and equi-biaxial tension in RD were assumed respectively for Figs. 7 and 8.

First, during uniaxial tensile deformation, the following tendencies can be seen from Fig. 7. The value of IQ decreases with the progress of deformation in the entire area of field frame. The IPF maps show that the crystal orientation gradually varies with the progress of deformation depending on the grains and that the variation of orientation is relatively large near grain boundaries. Especially for the circled grains with near {111} plane, the crystal orientations tend to vary from the near {111} plane (blue color) in ND to the near {221} plane (sky blue color), and the {111} plane texture was weakened. The value of GAM increases
with the progress of deformation in the entire area of field frame. The value of Taylor factor in the area where it is comparatively small before deformation increases with the progress of deformation, and the distribution of the Taylor factor becomes more homogeneous with red color.

Next, during biaxial tensile deformation the following characteristics can be seen from Fig. 8 compared with Fig. 7. Similar to the result of the uniaxial test, the value of IQ decreases with the progress of deformation and the localized decrease appears at a large strain. The IPF maps show that the grains with near {111} plane orientations in ND tend to rotate to {111} plane orientations with the progress of deformation (e.g., circled grain) and the more obvious development of texture in ND occurs in biaxial deformation, while the development of the {111} plane texture is not seen in the uniaxial test. These results have been verified in our previous study by the measurement of texture with X-ray diffraction for the Marciniak-type macroscopic biaxial tensile test. The previous measurement showed that the maximum intensity of {111} orientation increased with strain. From the GAM map it is observed that the difference among grains increases in biaxial deformation. To be pre-
cise, the average and the maximum values are 0.5 and 1.6, respectively, before deformation. In biaxial deformation they become 1.5 and 4.8, respectively, at a strain of 0.21, while in uniaxial deformation they become 2.3 and 4.9, respectively, at a strain of 0.23. The tendency of the GAM value being large at the grains with orientations near the {001} plane orientation (cf. IPF map) can be observed. It is considered that the inhomogeneous deformation is derived from these grains.

From the Taylor factor map the tendency is observed that the grains are divided into two groups with the progress of deformation. One group of grains has smaller Taylor factors and their orientations are near the {001} plane orientation, and the other has larger Taylor factors and near the {111} plane orientation. The difference in Taylor factor, namely the difference in flow stress among grains, may cause the inhomogeneous deformation.

The materials B and C showed the same tendency as the material A mentioned above with respect to the texture development and the inhomogeneous deformation in the {001} orientation, except for the degree of distribution of crystal orientation. These results will be described in Figs. 9 and 11.

As shown in IPF of Fig. 8, the {111} and {001} planes tend to become perpendicular to the ND under biaxial deformation. To investigate quantitatively the accumulation degrees of the {111} and {001} texture due to biaxial deformation, Fig. 9 shows the evolutions of the intensity of {111} and {001} plane orientations during the biaxial test for three materials obtained by EBSD analysis. The intensity was obtained by calculating the ODF for each result of EBSD measurement. The intensity of crystal orientations whose {111} or {001} plane is perpendicular to the ND was exhibited in Fig. 9. The intensity at each strain level is normalized by the intensity before deformation. The intensities increase rapidly with strain at early stage for the material A and gradually for the materials B and C. Kato\(^{31}\) reported that the stabilized orientations under compressive deformation were {111} and {001} in loading direction. In case of biaxial tension in the RD and TD, the ND is compressive direction. Therefore, these results are consistent with the report by Kato.

According to Fig. 6, the \(X\) value of material A tended to increase at the small strain range, i.e., the differential hardening behavior was observed.

4. Discussion

The above results have shown that the development of texture occurs in the biaxial tensile state, while little development occurs in the uniaxial tensile state. Also, the develop-

![Fig. 9. Intensity variations of crystal orientation of (a) {111} and (b) {001} planes in IPF during biaxial tensile test.](image)

![Fig. 10. Yield locus calculated by TBH theory for each grain of each strain level of material A.](image)

<table>
<thead>
<tr>
<th>Material</th>
<th>A</th>
<th>B</th>
<th>C</th>
</tr>
</thead>
<tbody>
<tr>
<td>Max. misorientation in the grain</td>
<td>16°</td>
<td>14°</td>
<td>20°</td>
</tr>
<tr>
<td>Inverse pole figure (IPF)</td>
<td>![IPF image]</td>
<td>![IPF image]</td>
<td>![IPF image]</td>
</tr>
<tr>
<td>Kernel average misorientation (KAM)</td>
<td>![KAM image]</td>
<td>![KAM image]</td>
<td>![KAM image]</td>
</tr>
</tbody>
</table>

![Fig. 11. IPF and KAM maps during biaxial tensile test.](image)
opment of texture shown in Fig. 9 is qualitatively similar to the evolution of stress ratio shown in Fig. 6. Further, inhomogeneous deformation has been observed at the later stage for the material A, where the stress ratio decreases. The relation between the differential hardening and the two factors is examined further in this section.

4.1. Effect of Texture Development on Differential Hardening Behavior

As shown in Fig. 9, the texture development was characterized by the increase in the intensity of the \{111\} and \{001\} plane orientations. It is presumed that the texture development would yield differential hardening. It was reported the shape of yield locus changed when the differential hardening occurred. Therefore, to investigate roughly the influence of the lattice rotation on differential hardening, TBH model was used to calculate the change in yield locus (plastic contour). To simplify the problem, the analysis was conducted for two single grains that exhibit the notable variations in crystal orientation during deformation. Figure 10 shows the IPF and the kernal KAM value is used to represent the degree of inhomogeneity during biaxial tensile testing. Figure 11 shows the IPF and the KAM maps, it is observed that the KAM value is relatively high at grain boundaries. The latter is more pronounced in the material A, suggesting that the inhomogeneous deformation is more pronounced in material A than in materials B and C. From the IPF and the KAM maps, it is observed that the KAM value of larger than 3.0 tends to appear near the boundaries of the \{001\} grain. These results suggest that the degree of inhomogeneous deformation depends on the grain size.

To quantitatively analyze the occurrence of inhomogeneous deformation, the variation of average KAM values are evaluated. The average KAM value was calculated from KAM values over the entire measured area in each EBSD result. Figure 12 shows evolutions of the average KAM values during the biaxial tensile test for materials A, B and C. For the material A, the average KAM value increases continuously with plastic strain also over a large strain range, although it remains almost unchanged in the very small strain range under 0.02 or so. Namely, the material A deforms homogeneously only in the early stage of deformation and then deforms inhomogeneously at the large strain range. On the contrary, the average KAM values for the materials B and C remain almost unchanged at the large strain range, although they increase notably with plastic strain at the small strain range. Consequently, the inhomogeneity of deformation becomes larger for material A than materials B and C over a large strain range.

As mentioned earlier, the stabilized orientations under macroscopic biaxial tension are \{111\} and \{001\} plane orientations. In contrast, as clearly observed in the IPF and KAM value maps in Fig. 11, plane orientations other than \{111\} and \{001\} plane orientations are observed locally at the regions with relatively high KAM values. This result suggests that the deformation mode at the regions with high KAM values is different from that at the regions with low KAM values; thus, the work hardening at the regions with high KAM values would also be different from that at the regions with low KAM values because the evolution of crystal orientations as well as the active slip systems would also be different.

The correlation between the heterogeneity of deformation and the evolution of differential hardening is further discussed. From Figs. 6 and 12, the stress ratio $X$ in Fig. 6 maps also show that there is a difference in KAM values within a grain and that the value is relatively high at grain boundaries. The latter is more pronounced in the material A, suggesting that the inhomogeneous deformation is more pronounced in material A than in materials B and C. From the IPF and the KAM maps, it is observed that the KAM value of larger than 3.0 tends to appear near the boundaries of the \{001\} grain. These results suggest that the degree of inhomogeneous deformation depends on the grain size.
is high when the average KAM value in Fig. 12 is small. The stress ratio $X$ for the materials A increases only at the small strain range under 0.02 or so, and the values of $X$ for the materials B and C increase over a large strain range. Therefore, it is considered that the homogeneity of deformation is related with the differential hardening behavior in the biaxial tensile state. It remains at the same level of homogeneity in all the materials, indicating that the grain boundary region deforms more inhomogeneously than the center region for all the materials, indicating that the grain boundary region deforms more inhomogeneously than the center region.

$$
\text{Increment in average KAM value between at grain boundary and other region.}
$$

5. Conclusions

In the present study, the relation between work hardening behavior and microstructure evolution under biaxial tensile state was investigated in three types of IF steels with different grain sizes using both macroscopic and microscopic biaxial tensile tests. A crystal plasticity analysis using the TBH theory was also applied to investigate the effect of texture development on the differential hardening behavior. The results obtained in the present study are summarized as follows.

1. The IF steels show the differential hardening behavior in macroscopic scale, and the hardening behaviors at large strains notably depend on the material.
2. The texture development and inhomogeneous deformation are observed in the microscopic tensile tests. These are more pronounced in the biaxial tensile test than in the uniaxial tensile test. It is observed that the texture development had a strong correlation with the differential hardening behavior.
3. The crystal plasticity analysis depicts that the rotation of crystallographic orientations during the deformation somewhat affects differential hardening at grain level.

REFERENCES