Observation of Inhomogeneous Deformation in a Cold-Rolled Ti-added Ultra-Low Carbon Steel using High-Precision Markers Drawn by Focused Ion Beam

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Abstract. The development of deformation inhomogeneities in ultra-low carbon steel due to cold rolling has been investigated by observation of the longitudinal plane at chosen sites of a rolled sheet using SEM and SEM-EBSD techniques. Particular attention has been paid to the development of strain distribution in the crystals. The microstructures and the orientation distribution in the longitudinal plane of a sheet rolled to a 60% reduction in thickness were observed, and some grains with preferred orientation such as \( \alpha \)-fiber and \( \gamma \)-fiber were selected. In order to examine the local strain distribution of these grains due to cold rolling, high-precision dot markers, 0.3 \( \mu \)m in diameter, were drawn using a focused ion beam. Then, before being subjected to additional rolling, the sheet was fitted into a frame made of the same steel under a plane strain condition. Using the marker method, the local displacement of the grains due to cold rolling has been directly measured.

1. Introduction

The development of preferential orientation distribution in steel sheets significantly influences its mechanical and electrical properties. It is possible to measure the crystal orientation of a steel plate through diffraction methods such as X-ray diffraction and SEM-EBSD, and much is known about the conditions that cause the formation of the macroscopic and average orientation distribution on such plates. Dislocation substructures are often observed in rolled plates, and the crystal orientation varies locally with the formation of individual substructures [1-3]. It is well known that these micro-regions have a relatively large orientation difference than in the surrounding regions and are the initiation sites for recrystallization by heat treatment after rolling. To elucidate the formation mechanism of these recrystallized microstructures, it is important to understand the occurrence of inhomogeneous deformation in the cold-rolled plates, especially during early stage of deformation.

In our laboratory, to investigate the formation process of inhomogeneous deformation structures, a method to follow the crystal orientation distribution of a particular region before and after the rolling process was developed. So far, we have studied how the local orientation changes across the entire length of the longitudinal plane on a rolled plate with intermediate thickness reduction (approximately 70%) [4-5]. It was found that in addition to grains whose orientation changed almost simultaneously throughout the grain and developed a preferred orientation, there were grains whose interior was divided
into several regions, each of which developed its own different orientation. In some of the grains of this type, submicron-sized fine structures were observed to be formed on the newly generated misorientation boundaries. These grains are expected to become the initiation sites for recrystallization during heat treatment.

In this study, to understand the inhomogeneous deformation state caused by cold rolling, we constructed nano-order markers regularly along the longitudinal plane of the rolled plate using a focused ion beam (FIB), measured the displacement of those markers by observing the same area, and estimated the strain distribution. In particular, the changes in orientation and strain distribution of grains with a preferential orientation in the rolling process and grains with a certain degree of grain subdivision in the middle of the process were traced.

2. Experimental Procedure

Ti-added ultra-low carbon steel was employed; it had an initial grain size of approximately 50 μm after heat treatment. The plate was cold-rolled without lubrication until its thickness had reduced by 60%, and was then cut out. The longitudinal plane was polished for observation; the deformation structure was observed by SEM, and the crystal orientation distribution was identified by SEM-EBSD.

Several preferential orientation grains (α-fiber: rolling direction(RD)//<110> in the rolling direction, γ-fiber: normal direction(ND)//<111> in the normal direction of the plate, Cube: parallel to <100> in both RD and ND) were selected, and carbon dots with a diameter of 0.3 μm were deposited by focused ion beam (FIB) to be used as markers. The markers, which were arranged in a square structure, had intervals of 0.7 μm. To re-inspect the marked longitudinal plane after additional rolling, the specimen was inserted into a mold frame, which has same thickness as the specimen, before being rolled again, to protect the observation surface. Additional rolling was performed to attain a 70% thickness reduction, and plastic strain distributions were obtained by tracing the displacement of the markers, as well as capturing the changes in crystal orientation in chosen regions.

3. Results and discussion

Figure 1(a) is an SEM image of the longitudinal plane of a rolled plate at a 60% reduction in thickness, which was marked by carbon deposition using FIB. Figure 1(b) is an SEM image of the same area after

![SEM images](image-url)
additional rolling has been applied to deform the plate to a 70% reduction. After rolling, the markers remained attached to the longitudinal plane, although their positions changed due to deformation. From the displacement of the markers, the equivalent plastic strains in the squares formed by these points were estimated.

Equivalent plastic strain distributions were also calculated for the grains with the major preferred orientations grain. Figure 2 shows the equivalent plastic strain distributions within the five preferred orientation grains at a 70% reduction in thickness, which are cumulative for zero strain at 60% reduction in thickness. The bars in the figure correspond to the widths of the maximum and minimum values, and the black squares represent the average values in each grain. The mean strain value of each orientation grain is almost the same regardless of the orientation, while the variation of strain for γ-fiber grains is approximately twice as large as those for the other orientations. These results indicate that the inhomogeneity of deformation in grains of γ-fiber is more pronounced than in other orientations, and inhomogeneous structures such as shear bands were noticeably developed in the SEM images of the grains.

Figure 2 Equivalent strain distribution of grains with preferred orientation.

| Grain | A | B | C | D | E | F | G |
|-------|---|---|---|---|---|---|---|
| Cube  | 0.438 | 0.502 | 0.408 | 0.420 | 0.394 | 0.394 | 0.362 |

Figure 3 OIM images (ND) obtained from same area in longitudinal plane, (a) 60% reduction in thickness, (b) 70% reduction in thickness, (c) equivalent strain distribution at 70% reduction in thickness, (d) average equivalent strain of the areas A-G in the figures.
Figures 3(a) and (b) show the crystal orientation images parallel to the ND at 60% and 70% thickness reduction. The horizontal direction corresponds to RD. The orientation of the top and bottom regions inside the central grain in figure 3(a) differs slightly, and the color of the lower area of the grain changes more across the images, indicating higher misorientation between the top and bottom regions. Figure 3(c) shows the equivalent plastic strain distribution calculated from marker displacement at 70% thickness reduction. A region of strain concentration inclined toward the rolling direction can be seen. In figure 3(b), the top of the grain is red and the bottom is blue, and the orientation parallel to the normal direction of the plate changes gradually near the boundary. Despite the occurrence of such a change in orientation, the strain distribution shown in figure 3(c) is relatively uniform, with no significant variation between the top and bottom regions of the grains.

We investigated the relationship between the observed strain distribution and the change in orientation of the grains due to cold rolling. As shown in figures 3(a) and (b), seven locations (A-G) were selected, including a region with a significant change in orientation along the ND, and the average crystal orientation at each of these locations was calculated from the OIMs. In addition, these areas are indicated in figure 3(c). The average strains at these locations are summarized in figure 3(d). The areas where crystal orientation changes significantly with increasing thickness reduction correspond to the areas E-G; however, the average strains in these areas (<0.4) are less than the average strain at locations A-D (0.4-0.5), where the ND orientation in the grain is nearly maintained at almost <100>. Although the area G is very close to the grain boundary, the crystallographic orientation change within the region is small, and the measured strain is not large.

Based on the average strains obtained at 70% thickness reduction and the average crystal orientations at 60% thickness reduction at each locations A-G (figure 3), the crystal orientations at a thickness reduction of 70% were predicted using the Taylor model assuming a pencil glide. The predicted orientations were compared with the results obtained experimentally. As a method of orientation prediction, the Taylor model gives a unique orientation for any given strain, assuming a deformation that minimizes the sum of shear strains due to slip in the four <111> directions. The results are summarized in the 100 standard stereo projections shown in figure 4. Figures 4(a)-(g) show the average crystal orientation, ND, and RD for each crystal orientation at both 60% and 70% of thickness reductions at locations A-G (figure 3), as well as those at 70% thickness reduction, predicted by the Taylor model. Average crystal orientations of RD predictions at each location agree well with results. On the other hand, although average crystal orientations of ND are similar for locations A-D, the measured ND at a 70% reduction is rotated by approximately 15 degrees than that at 60% reduction, while the Taylor model gives a different orientation.
model predicts only a few degrees of change in orientation for E-G. As shown in figure 3(d), the equivalent plastic strains across locations of E-G are smaller than those across A-D. Therefore, it can be assumed that the crystal rotation caused by the large deviation from the Taylor model occurred in these areas, suggesting that <111> slips were activated inhomogeneously in the locations of E-G.

4. Summary
Carbon deposition markings were applied to the longitudinal plane of a rolled sheet using a focused ion beam system, and the equivalent plastic strain distributions of the grains with the preferred orientation were investigated after reducing thickness from 60% to 70% through cold rolling. The inhomogeneity of strain in the grain oriented γ-fiber was more pronounced than in the oriented α and Cube fibers. The division of grains inside the initial grains during additional rolling was observed, and the orientations of these grains after additional rolling were predicted from based on the strains generated and the orientations before rolling. A comparison of the estimated and measured orientations after additional rolling suggests that inhomogeneous activation of slip that deviates from the Taylor model occurs in regions where significant orientation changes occur.

Acknowledgment
This work was supported by JSPS KAKENHI Grant Number JP18K04751.

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