Heterogeneous Nano-structure and its Evolution in Heavily Cold-rolled SUS316LN Stainless Steels

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Evolution of heterogeneous nano-structure in heavily cold-rolled SUS316LN stainless steels was investigated in detail. Transmission electron microscopic observations from the transverse direction (TD) of the 92% rolled specimen revealed the formation of a typical hetero-nano structure composed of ultra-fine lamellar grains embedded with deformation twin domains. The twin domains had prolate ellipsoidal shape elongated parallel to TD. Two types of twin domains with different crystallographical orientations to matrices could be identified, i.e., (i) <211> // rolling direction (RD) and <110> // TD or (ii) <110> // RD and <211> // TD, although all the {111} twinning planes of both twin domains were oriented nearly parallel to the rolling planes. The ultra-fine lamellar grains were elongated along <100> direction and nearly parallel to RD. Deformation twins with a few nano-meter spacing were also frequently observed to develop in the lamellar grains. Evolution sequence of the hetero-nano structure during cold rolling was also investigated. At an early stage of rolling, deformation twins were gradually formed in the whole grains. Then, the regions fragmented grains by twins were further subdivided by a numerous number of shear bands inclined at about 20–45° from the RD, resulting in the formation of “eye-shaped” twin domains surrounded by shear bands and their crystallographical rotation. Cold rolling up to 50% caused a considerable increase in strength and decrease in ductility. While the strength was raised more with increasing reduction up to 92%, both the strength and ductility eventually slightly decreased by further rolling.

KEY WORDS: heavy cold rolling; heterogeneous nano-structure; austenitic stainless steels; microstructure.

1. Introduction

There is an increasing need to improve mechanical properties of metallic materials while reducing the additives used in the process, in order to enhance their recyclability and reduce their cost. A simple method to achieve this is the grain refinement of metallic materials, which results in superior balance of mechanical properties. A well-known example is strengthening by grain refinement, which can be well described by the Hall-Petch relation. Therefore, in the last two decades, several studies have investigated grain refinement of metallic materials. One of the breakthroughs in this field was using severe plastic deformation (SPD) to achieve grain refinement. This method is adopted worldwide since the 1990s.1–7 The SPDs have been applied also to steels. Tsuji et al. applied accumulative roll bonding on interstitial-free (IF) steel plates and successfully fabricated ultrafine-grained (UFGed) structure that had an average grain size of about 200 nm and it exhibited a tensile strength of 820 MPa and an elongation of 7%.4 Park et al. utilized an equal channel angular pressing on an Mn steel to obtain UFGed structure with an average size of about 500 nm and achieved a tensile strength of 945 MPa and an elongation of 10%.5 Nakao and Miura6) and Miura et al.8) studied UFGed SUS316L austenitic stainless steel produced by multi-directional forging (MDFing) with an average grain size of 10 nm or less. They reported that it showed an ultra-high strength of 2.2 GPa with an elongation of 10%. However, these SPDs were batch processes, which have low manufacturing efficiency and are not suitable for industrial mass production.

On the other hand, Miura et al. recently showed that heavy cold rolling of stable austenitic steels introduced “heterogeneous nano (HN) structure,” which is composed of nanoscale deformation-induced structures, such as deformation twin domains, shear bands and ultrafine low-angle lamellar grains.9) According to this study, the SUS316LN
steel, which was subjected to 92% cold rolling and a subsequent aging, exhibited a tensile strength of 2.7 GPa and an elongation of 5%. Although it has a slightly lower elongation compared to that of the SUS316L steels fabricated by MDFing,6,8 its strength was notably higher. This HN structure introduced by simple heavy cold rolling would be suitable for industrial mass production. In addition, it exhibited excellent mechanical properties comparable or superior to those of the UFGed structure fabricated by SPDs. However, few studies examined the microstructural evolution in SUS310S austenitic steels during cold rolling up to 95%.10 Morikawa and Higashida investigated the microstructural evolution in SUS310S austenitic steels during simple heavy cold rolling up to 95%.11 They obtained a UFGed structure consisting of deformation twins and shear bands formed with increasing rolling reduction. The area fraction of the UFGed structure reached 90% at 95% rolling. While, the coarse grain structure remained even after the 95% heavy cold rolling and the Brass texture was developed in the coarse grain region. However, in these studies, the relationship between the microstructure introduced by heavy cold rolling and the mechanical properties has not been investigated in detail.

In the present study, the microstructural evolution in a SUS316LN steel during simple heavy cold rolling has been investigated in detail. In addition, the sequence of HN structural evolution was analyzed. Furthermore, the microstructural factors that influence mechanical properties were also investigated and discussed.

2. Experimental Procedure

Table 1 shows the chemical composition of the hot-rolled SUS316LN stainless steel used in this study. The hot-rolled plates were solution treated at 1523 K for 3.6 ks and immediately quenched into water. They were then subjected to cold rolling up to 95% reduction in thickness. An optical microscopy (Keyence, VHX-5000) and a scanning electron microscopy (SEM/JEOL, JSM-7100F) equipped with an electron backscattered diffraction (EBSD) camera were used to observe the surface of the sheets. Substructures were observed using transmission electron microscopy (TEM/JEOL, JEM-2010 FEF and FEI TECNAI G30). The microstructure of the rolled sheets was mainly performed from their transverse direction (TD), but some specimens were also observed from the rolling direction (RD). The specimens for optical microscopic observations were mechanically ground and then electro-polished at 223 K and 20 V using a solution of 2-n butoxy-ethanol and perchloric acid (at a volume ratio of 95:5). The electro-polished specimens were then etched using an oxalic acid solution under a current density of 1 A/mm² for about 90 s at room temperature. The SEM-EBSD observations were performed after the electro-polishing. Thin foils for TEM observation were prepared using an ion-milling apparatus (JEOL, EM-09100IS). TEM observations were performed using JEM-2010 FEF and TECNAI G30 microscopes at accelerating voltages of 200 and 300 kV, respectively.

Tensile tests were carried out at an initial strain rate of 10⁻³ s⁻¹ at room temperature using an Instron-type universal testing machine. Dog-bone specimens of 5² × 2₀ × 0.5² mm³ with tensile axis parallel to the RD of the rolled sheets were cut out using a wire electric-discharge machine.

3. Results and Discussions

3.1. HN Structure in 92% Rolled Specimen

The macrostructure evolved in the solutionized specimens was observed with an optical microscope. The component grains were almost equiaxed and the average size was about 70 μm. Annealing twins were frequently observed inside the grain. The grains of the 92%-rolled specimen were elongated along RD.

Figure 1(a) shows a typical bright-field TEM image observed from TD of a specimen cold rolled to 92% reduction. The microstructure appears inhomogeneous and complicated, which is similar to the literature by Miura et al.9 who observed an “eye-shaped” twin domain surrounded by shear bands embedded in ultra-fine lamellar grains. Figure 1(b) depicts the selected-area diffraction pattern (SADP) of the eye-shaped twin domain. Analysis of SADP revealed that the {111} twin-boundary plane was almost parallel to RD. The average interspacing of twin boundaries was found

| Table 1. Chemical composition in mass% of a SUS316LN stainless steel used in the present study. |
|-----------------|--------|--------|--------|--------|--------|--------|--------|--------|--------|
| C    | Si    | Mn    | P     | S     | Al    | Ni    | Cr    | Mo    | N     | Fe    |
| 0.020 | 0.49  | 0.86  | 0.021 | 0.0004| 0.010 | 11.05 | 17.81 | 2.52  | 0.183 | Bal.  |

Fig. 1. (a) TEM micrograph showing a twin domain in a SUS316LN stainless steel cold rolled to 92% reduction in thickness. Incident beam direction is parallel to Transverse Direction (TD). (b) Corresponding Selected-Area Diffraction Pattern (SADP) to (a).
to be about 30 nm. The twins shown in Fig. 1(a) are of mechanical ones introduced during cold rolling and will be discussed in a later. The crystallographical orientation of the twin domain in Fig. 1(a) is defined as the twin boundary normal of \(<111> // ND\), shear direction of \(<211> // TD\), \(<110> // RD\). The twins with the \(<110> // TD\) orientation were often observed. Whereas some eye-shaped twin domains were revealed to have different crystallographical orientation from the analysis of Fig. 2, i.e., the twin boundary normal of \(<111> // ND\), shear direction of \(<211> // TD\), \(<110> // RD\). Thus, there was a 90° difference in the shear direction of the latter twin domain around the \([111]\) axis compared to that in Fig. 1(a). Figure 3(a) shows a bright-field TEM micrograph of a twin domain observed from ND. Here, there were differences in the observations compared to those from TD. A long spheroidal shape (cigar-shaped) twin domain elongated along TD was revealed. Figure 3(b) shows the SADP of the same sample. The twin domain with cigar shape was found to have the same crystallographical orientation as that shown in Fig. 1(a). Similarly, it was confirmed that there were also cigar-shaped twin domains with the same crystallographical orientation as that shown in Fig. 2. This indicates that the cross section of the twin domains with a cigar-shape elongated along TD is recognized as the “eye shape” when observed from TD.

Figure 4(a) shows a bright-field TEM image of the lamellar region. The lamellar grains were found to have a longitudinal direction parallel to RD and an average lamellar boundary spacing of about 70 nm. Figure 4(b) depicts a SADP obtained using a selected-area aperture of 2.5 µm. The SADP exhibits a net pattern indicating that the misorientation among lamellar grains was rather small, in other words, the lamellar boundaries are small angle ones. The SADP analysis confirmed that the longitudinal direction of the lamellar grains was nearly parallel to the \(<100>\) direction. An enlarged image of the area indicated by the broken line in Fig. 4(a) is shown in the inset in the upper right corner of Fig. 4(a). In the lamellar grains, an evolution of ultra-fine deformation twins with inter-boundary spacing of several nano-meters was identified. Figure 4(c) shows the SADP of the area indicated by the broken line in Fig. 4(a). The angle between the ultra-fine deformation twins and ND or RD was approximately 45°. Therefore, this strongly suggested that the twins concerned were formed after the development of lamellar grains.

Figures 5(a) and 5(b) illustrate inverse pole figure (IPF) maps obtained by SEM-EBSD observations from TD. The maps were drawn with confidence index values of more than 0.1 upon the EBSD analysis. Figure 5(a) shows a typical map, in which the color is decoded parallel to the incident beam direction (// TD). The figure shows many green colored regions, which are regions that have a crystallographical orientation of \(<110> // TD\). In addition, there were also blue colored regions, which indicates a \(<211> // TD\) orientation. The two types of eye-shaped twin domains with crystallographical orientations of \(<110> // TD\) or \(<211> // TD\) mentioned in Figs. 1 and 2 were again confirmed here. The size of these twin domains was about 1 µm (Figs. 1(a) and 2(a)). Thus, the green and blue colored regions observed in Fig. 5(a) can be corresponded to the two different types of twin domains. Figure 5(b) is a IPF map redrawn from Fig. 5(a) by color decoding parallel to RD. Compared to Fig. 5(a), the green colored regions having \(<110> // TD\) changed into the blue color that represents \(<211> // RD\), and inversely the blue colored region having the \(<211> // RD\) direction turned into green color. Yamasaki et al. conducted more detailed SEM-EBSD analysis, which confirmed that these green and blue colored regions in the IPF maps were
twin domains. This confirms that the green or blue colored regions on the IPF maps represent the deformation twin domains. However, the lamellar regions and shear bands were not identified, due to the limiting resolution of the SEM-EBSD employed. Thus, these regions are reflected as random noise in Fig. 5.

The area fraction of twin domains, i.e., the green and blue colored regions in Fig. 5(a), was estimated to be about 57% distributed over the whole observed area. This value is considerably high compared to that of about 10% in a 92% cold-rolled SUS316LN steel reported by Miura et al. They suggested that this low area fraction of the twin domains caused the relatively low tensile strength (less than 2 GPa) of the heavily cold-rolled SUS316LN steel. This was confirmed in the study by Aoyagi et al., in which they revealed that tensile strength increases with the area fraction of twin domain through multi-scale crystal-plastic simulation using crystal information of the HN structures. The SUS316LN steel employed in the present study was fabricated by the same thermo-mechanical treatment that the tensile strength of 2.7 GPa was achieved by Miura et al. Therefore, it can be confirmed that the area fraction of twin domains strongly affects the strength of HN structured steels.

3.2. Evolution of HN Structure

TEM observation was systematically carried out to study the evolution process of the HN structure using specimens cold rolled to 50%, 80%, 92% and 95% reduction.

TEM observation of the 50% cold-rolled sample revealed subdivision of coarse initial grains by dense evolution of mechanical twins (Fig. 6(a)). However, the development of deformation twins still appeared inhomogeneous. In some grains, there were only substructural evolution with high density of dislocations and stacking faults (Fig. 6(b)). This inhomogeneous evolution of deformation twins was also confirmed by the optical microscopic observations. The inhomogeneous microstructure differed from grain to grain would be affected by the initial crystallographical orientation.

Figure 7 shows a typical bright-field TEM micrograph of the cold-rolled 80% specimen. Here, the deformation twins were well developed throughout the grains. In addition, shear bands were formed to cross the deformation twins. These results were macroscopically confirmed by means of optical microscope. The SADP indicates that only a slight misorientation (up to a few degrees) developed among the twin regions separated by shear bands, while the misorientations among the twin regions and the shear bands were considerably large (between 20° and 40°). Therefore, twin domains were formed through the subdivision of twinned regions by shear bands. Moreover, the shear bands were
formed not only within the grain but sometimes traversed entire specimen. The formation of these macroscopic shear bands was often observed in the nano-layered materials stacked with different metals and alloys. Thus, it is particularly difficult to achieve slip deformation by dislocation glide across the layers, due to the extremely fine inter-layer spacing. Therefore, macroscopic shear banding takes place as a kind of avalanche phenomenon in order to relieve the applied stress. This is consistent with the inter-spacing between the developed twin boundaries in the present study, which was less than 100 nm (Figs. 6, 7, 8, 9 and 10). Furthermore, an extremely large amount of work hardening within the twin regions must take place during heavy cold rolling. To continue plastic deformation, shear bands were

![Fig. 6](image1.png)

**Fig. 6.** TEM micrographs showing (a) deformation twin and (b) high density of dislocations and stacking faults in a SUS316LN stainless steel cold rolled to 50% reduction in thickness. Incident beam direction is parallel to TD.

![Fig. 7](image2.png)

**Fig. 7.** TEM micrograph showing deformation twin divided by shear bands in a SUS316LN stainless steel cold rolled to 80% reduction in thickness. Incident beam direction is parallel to TD.

![Fig. 8](image3.png)

**Fig. 8.** TEM micrograph showing twin interfaces continuously rotated in a twin domain in a SUS316LN stainless steel cold rolled to 80% reduction in thickness. Incident beam direction is parallel to TD.

![Fig. 9](image4.png)

**Fig. 9.** TEM micrograph showing transition from a twin domain into low-angle lamellar grains in a SUS316LN stainless steel cold rolled to 95% reduction in thickness. Incident beam direction is parallel to TD. The inset is enlarged image of the portion indicated by dashed-line.

![Fig. 10](image5.png)

**Fig. 10.** Changes in average inter-spacings of twin boundaries \( t \), low-angle lamellar boundaries \( \lambda \), and angle \( \theta \) between twin boundary normal and RD as a function of reduction in thickness by cold rolling.
inevitably formed across the twin boundaries and sometimes across the entire specimen. In the twin region displayed in Fig. 8, the twin boundaries within a twin domain were continuously rotating toward the RD. This indicates that shear banding causes simultaneous crystallographical rotation of twin domains during the subdivision of deformation twin region into twin domains.

The microstructure evolution of the specimens rolled to 92% reduction is already described in detail in section 3.1. Further cold rolling up to 95% did not lead to a drastic change in the morphology of the HN structure. However, transitions from twin domains to low-angle lamellar structure were often observed as shown in Fig. 9. The feature in the center of Fig. 9 resembles the eye-shaped twin domain, but the SADP revealed that the crystallographic orientation was completely different from that of twin domain. The inset in the upper right of Fig. 9 exhibits an enlarged view of the area indicated by the dotted line. It indicates that the deformation twin region remained only in a small portion of what was previously an eye-shaped domain, while the rest changed into low-angle lamellar structure.

The changes in the microstructural parameters (twin-boundary spacing $t$, low-angle lamellar-boundary spacing $\lambda$, angle $\theta$ between twin boundaries and RD) depending on reduction on cold rolling are summarized in Fig. 10. As the rolling reduction increased, the twin-boundary spacing decreased. The low-angle lamellar regions began to develop at around 80% reduction. In addition, both the twin boundary spacing and the inter-spacing among the lamellar boundaries decreased with the increase in the rolling reduction. Moreover, the twin boundaries within the twin domains continuously rotated with the increase in the reduction until they eventually became parallel to the ND plane.

### 3.3. Mechanical Properties

The tensile tests were conducted using specimens cold rolled to different reductions (50%, 80%, 92%, 95%) with the tensile axis parallel to the RD. The obtained nominal stress-nominal strain curves are shown in Fig. 11. Dependencies of 0.2% proof stress $\sigma_{0.2}$, tensile strength $\sigma_{UTS}$ and elongation $\varepsilon_t$ on the reduction are depicted in Fig. 12. The strength was significantly increased by 50% cold rolling while the elongation was drastically reduced. Nevertheless, increasing the rolling reduction up to 92%, no significant change was observed in the elongation although the strength constantly increased. According to the Hall-Petch relation, the strength of metallic materials $\sigma$ increases with the decrease in the grain size $d$ as described by $\sigma \propto d^{-\frac{1}{2}}$.

However, in materials is composed of nano-lamellar structure with lamellar spacing $L < 100$ nm, the strength is expressed as follows: $\sigma \propto L^{-1.18,19}$ As shown in Fig. 10, the twin-boundary spacing $t$ and the lamellar-boundary spacing $\lambda$ at 80% reduction are already less than 100 nm and they further decreased with the increasing reduction. Therefore, the increase in the strength by cold rolling up to 92% could be attributed to the decrease in $t$ and $\lambda$.

It is known that sharp [011] <112> texture develops in heavily cold-rolled austenitic steels. This development of strong texture causes the ductility of metallic materials to decrease. On the other hand, the formation of eye-shaped twin domains bearing the [111] <211> orientation was reported to suppress the development of the [011] <112> sharp rolling texture in the HN structured austenitic steel. Therefore, even after heavy cold rolling, the attained moderate ductility should be affected by the change in the rolling texture, due to the formation of HN structure. Moreover, it should be remarkable that the amount of work hardening ($\sigma_{UTS} - \sigma_{0.2}$) during tensile tests (the inset in Fig. 11) became more pronounced while the uniform elongation decreased with the increasing reduction. Therefore, the almost constant elongation at higher reductions could be attributed to the post-uniform deformation during tensile tests. However, the mechanisms of these specific experimental results have not been clarified yet, and hence further study is necessary.

Both strength and ductility slightly decreased after cold rolling to 95%. The values of $t$ and $\lambda$ at 95% reduction were lower than those at 92% reduction (Fig. 10). Therefore, the decrease in $t$ and $\lambda$ is expected to cause an increase in the strength. However, the strength was found to decrease at 95% reduction (Fig. 12). As described in section 3.2, the transition of the eye-shaped twin domain to the low-angle lamellar structure was observed in the 95% specimen.

The texture evolved at different reductions (40%, 50%, 60%, 70%, 80%, 92% and 95%) was measured using X-ray diffraction technique and the key results are exhibited in

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**Fig. 11.** Nominal stress - nominal strain curves of SUS316LN stainless steel after cold rolling to various reduction ratio. The tests were performed at RT at a strain-rate of $10^{-3}$ s$^{-1}$. The inset showing the enlarged curves of the steels after cold rolled to 50% and 92% reduction, respectively.

**Fig. 12.** Change in 0.2% proof stress $\sigma_{0.2}$, tensile strength $\sigma_{UTS}$, amount of work hardening $\sigma_{UTS} - \sigma_{0.2}$ and elongation to failure $\varepsilon_t$ for SUS316LN stainless steel after cold rolling to various reduction ratio on cold rolling.
Fig. 13. The relative intensity of the ND // {011} component was about 4 in the initial stage of rolling and no significant change was observed until it reached 60% reduction. However, the relative intensity rapidly increased to about 6.4 at 80% reduction, at which the HN structure began to develop (section 3.2), and it further increased to about 7.1 and 10.4 at 92% and 95% reductions, respectively. The increase in the relative intensity caused by the change in reduction from 92% to 95% (difference in equivalent strain ~0.47) is large compared to that from 80% to 92% (difference in equivalent strain ~0.92). On the other hand, the relative intensity of the ND // {111} component decreased corresponding to the increase in the ND // {011} component with increasing rolling reduction. These results indicate (in a semi-quantitatively way) that the volume fraction of the eye-shaped twin domains with the ND // {111} component decreased with the increase in the rolling reduction. Aoyagi et al. have shown from the multi-scale crystal-plastic simulation using crystal information of HN structures that the strength decreases with the decrease in volume fraction of the eye-shaped twin domains. Therefore, in spite of the decrease in $t$ and $\lambda$ (Fig. 10), the decrease observed in strength at 95% reduction could be ascribed to the decrease in the volume fraction of the twin domains. According to this hypothesis, it can be concluded that the effect of volume fraction of the eye-shaped twin domains on the strengthening is more notable compared to that of the decrease in $t$ and $\lambda$. Moreover, the decrease in ductility might be attributed to the rapid development of much sharper rolling texture after the increase in the subsequent rolling reduction from 92% to 95%.

This analysis and arguments suggest that the optimum strength-ductility balance can be achieved at a certain structure ratio (twin domain/low-angle lamellar region).

**Figure 14** exemplifies a typical bright-field TEM micrograph observed after the tensile failure of the 92% cold-rolled specimen. Subdivision of a twin domain by shear bands could be seen. This phenomenon was frequently observed after tensile tests. On the other hand, no significant change was distinguished in the low-angle lamellar regions. However, more detailed TEM observations revealed that ultra-fine deformation twins similar to those shown in Fig. 4(a) were developed in almost all low-angle lamellar grains. Therefore, they were most likely formed during tensile deformation. Thus, shear banding and mechanical twinning are considered the two dominant mechanisms for achieving moderate and almost constant ductility of the HN structure developed after heavy cold rolling (Fig. 12). However, quantitative data, such as the volume fraction of deformation twins formed in low-angle lamellar

![Fig. 14. TEM micrograph of a SUS316LN stainless steel cold rolled to 92% in thickness after tensile deformation to failure at RT.](image-url)
grains and the change in the texture due to the formation of ultra-fine deformation twins, should also be studied. In addition, further studies on the evolution of microstructure during deformation are essential to clarify the cause of the excellent balance between strength and ductility in the HN structured steels.

4. Summary

This study systematically investigated the microstructure and mechanical properties of SUS316LN austenitic stainless steels cold rolled to different rolling reductions. The results of the study are summarized below:

(1) Heavy cold rolling of SUS316LN steels introduced heterogeneous nano-structures consisting of deformation twin domains, shear bands and low-angle lamellar grains.

(2) The twin domains had a cigar-like shape elongated along the transverse direction (TD) and perpendicular to the rolling direction (RD). Two types of twin domains were observed. They had a crystallographic orientation bearing the shear direction <211> parallel to either RD or TD.

(3) The longitudinal direction of lamellar grains was parallel to RD and nearly parallel to the <100> direction. The lamellar boundaries were identified to be small-angle ones. Ultra-fine deformation twins were formed in some of the lamellar grains. The analysis of the crystallographical orientation and geometry revealed that they were developed after the formation of lamellar grains.

(4) Deformation twins were developed at the early stage of rolling (~50% reduction in thickness). Subsequent rolling caused subdivision and rotation of the deformation twins by shear bands. Eventually, eye-shaped twin domains with an unusual crystallographical orientation were developed.

(5) At the early stage of the rolling (~50%), the tensile strength of the steel increased but the ductility considerably decreased. However, at the medium and later stages of the rolling (up to 92%), the strength gradually increased, while the ductility remained constant. Nevertheless, further cold rolling to 95% led to a slight decrease in both strength and ductility. The decrease in the strength and ductility after the 95% reduction was attributed to the transition of twin domains into a low-angle lamellar structure.

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