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Deformation mechanism of ultrafine-grained high-Mn TWIP steels

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Abstract: In our previous study, we have found that high-Mn austenitic TWIP steels with fully recrystallized ultrafine grained (UFG) structures having mean grain sizes smaller than 1 μm could be fabricated by simple cold rolling and subsequent annealing process. Although the UFG TWIP steels showed both high strength and large ductility, the reason for such outstanding mechanical properties was not understood yet. In addition, yield drop phenomenon was observed in the UFG specimens, which is not often seen in metals with FCC structure. In the present study, dislocation density during tensile tests was measured and the result showed that the dislocation density increased suddenly at the yield point in the UFG specimens. In addition, microstructure evolution of 31Mn-3Al-3Si TWIP steel during tensile deformation was also studied. Quantitative analysis of deformed microstructures was conducted by means of ECCI (electron channelling contrast imaging) technique in SEM. We found that larger amount of dislocations and stacking faults generated around the yield point in the UFG specimen, while only dislocation pile-up was confirmed in the coarse-grained specimens. Based on these results, a new deformation mechanism is proposed to explain the yield drop phenomenon in the UFG high-Mn TWIP steel.

1. INTRODUCTION

High-Mn steels exhibit a stable single austenite phase (FCC (Face-Centered Cubic) structure) at ambient temperature and a large amount of deformation twins generate when subjected to deformation, resulting in high strength and large ductility [1]. Because of the peculiar deformation mode, the high-Mn steels are also known as twinning-induced plasticity (TWIP) steels. Due to the good balance of high strength and outstanding elongation, high-Mn austenitic TWIP steels attract a great attention in automobile industries. However, the lower yield strength due to FCC structure of austenite is a weakness which hinders the practical application of TWIP steels. Grain refinement is a possible approach to improve the low yield strength of metallic materials without changing the chemical compositions according to the Hall-Petch relationship.

Nowadays, severe plastic deformation (SPD) processes, including equal-channel angular pressing (ECAP) [2], accumulated roll bonding (ARB) [3], and high pressure torsion (HPT) [4], are well investigated for fabricating bulk nanostructured materials. However, the size of the samples obtained by the above SPD methods is so limited that SPD is not realistic for practical application in large scale and continuous production of materials. In our previous study, a fully recrystallized ultrafine grained (UFG) 31Mn-3Al-3Si TWIP steel was fabricated simply by conventional cold rolling and subsequent annealing without SPD [5]. The UFG material had the minimum average grain size of 0.4 μm, and showed high yield strength. It is well known that a trade-off relationship exists between strength and ductility. However, the UFG TWIP steel still possessed a good balance of strength and ductility. In addition, a yield drop phenomenon, which is not seen in FCC materials, was observed in UFG TWIP steel. On this basis, we

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have investigated the microstructure evolution during deformation to clarify the deformation mechanisms of the yield drop phenomenon in the UFG 31Mn-3Al-3Si TWIP steel in the present study.

2. EXPERIMENTAL PROCEDURE

A 31Mn-3Al-3Si TWIP steel, one of the typical high-Mn TWIP steels, was used in the present study. The chemical composition is shown in Table 1. The starting material with a thickness of 12 mm was cold-rolled to 1 mm thickness with a total reduction of 92 % (the equivalent strain was 2.87). In order to fabricate fully recrystallized specimens with various mean grain sizes, the 1 mm thick cold-rolled sheet was annealed at temperatures varied from 700 °C to 1000 °C, and the annealing time was changed from 0.3 ks to 3.6 ks. After the annealing process, the specimens were subsequently water-quenched.

Table 1 Chemical composition of the 31Mn-3Al-3Si TWIP steel used in the present study (wt.%).

C	Mn	Si	Al	N	S	Fe
0.005	31	3.0	3.0	0.004	0.012	Bal.

Mechanical properties of the fully recrystallized specimens with various grain sizes were evaluated by tensile tests at an initial strain rate of 8.3×10^{-4} s^{-1} at room temperature using a uniaxial tensile testing machine. Tensile test specimens with a gauge length of 10 mm, width of 5 mm and thickness of 1 mm were cut from the cold-rolled and subsequently annealed sheets by electrical discharge machine. In order to measure the tensile strain of the specimen precisely, an extensometer was attached to the gauge part of the specimen to detect the displacement.

Microstructures of the obtained fully recrystallized specimens were characterized by a field emission-type scanning electron microscope (FE-SEM) equipped with an electron backscattering diffraction (EBSD) system. All the microstructures were observed on the longitudinal sections perpendicular to the transverse direction (TD) of the cold-rolled and annealed sheets. Some of the specimens tensile tested up to the yield point were observed by electron channelling contrast imaging (ECCI) [6] technique in FE-SEM to quantitatively analyse the deformed microstructure. The EBSD and ECCI specimens were prepared by electro-polishing in a solution of 100 ml $HClO_4$ + 900 ml C_2H_5OH at room temperature and buff-polishing using colloidal silica with a particle size of 0.02 μm as a polishing agent. For measuring dislocation densities, we conducted an X-ray diffraction (XRD) measurement for some of the specimens with different grain sizes, which were deformed by the tensile test. Dislocation densities were estimated by the conventional Williamson-Hall analysis [7].

3. RESULTS AND DISCUSSION

3.1. Microstructure observation

Fig. 1 shows EBSD grain boundary maps of the 31Mn-3Al-3Si TWIP steel after cold rolling and subsequent annealing at various temperatures for various time periods. High angle grain boundaries with misorientation (θ), $15^{\circ} \leq \theta$, low angle grain boundaries with $2^{\circ} \leq \theta < 15^{\circ}$ and annealing twin boundaries ($\Sigma 3$) are drawn by black lines, green lines and red lines, respectively. All the microstructures exhibited equiaxed grains with a large amount of annealing twins, indicating that they were fully recrystallized. The mean grain sizes were measured by using the linear interception method, including annealing twin boundaries on the EBSD grain boundary maps. Specimens with mean grain sizes ranging from 0.79 μm

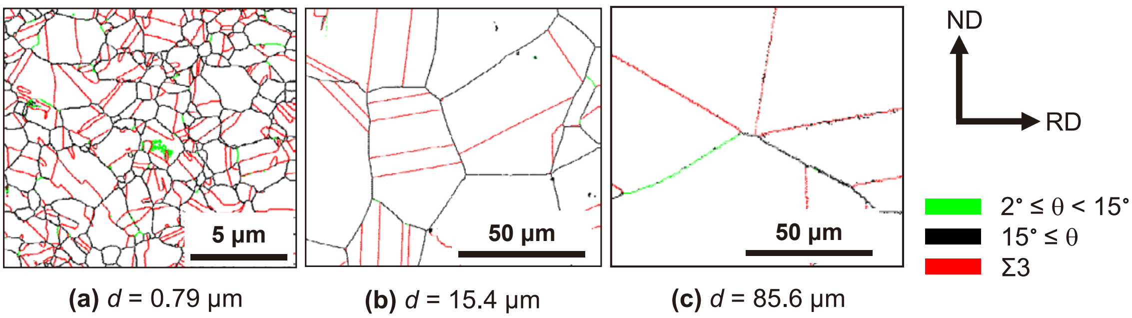

Fig. 1 Grain boundary maps of the cold rolled and subsequent annealed 31Mn-3Al-3Si TWIP steel: (a) 92 % cold-rolled and annealed at 700 °C for 0.3 ks, and (b) 92 % cold-rolled and annealed at 950 °C for 0.9 ks, and (c) 92 % cold-rolled and annealed at 1000 °C for 3.6 ks. The average grain sizes are also indicated.

to 85.6 μm were obtained in the present study. The grain sizes shown in Fig. 1 were (a) 0.79 μm (annealed at 700 °C for 0.3 ks), (b) 15.4 μm (annealed at 950 °C for 0.9 ks), and (c) 85.6 μm (annealed at 1000 °C for 3.6 ks), respectively.

3.2. Mechanical properties

Nominal stress-strain curves obtained from the tensile tests of the 31Mn-3Al-3Si TWIP steel with various mean grain sizes are shown in Fig. 2. The grain sizes are also indicated in the figure. The yield strength and ultimate tensile strength increased and tensile elongation decreased simultaneously with decreasing the mean grain size. However, the specimen with the finest grain size of 0.79 μm exhibited a high yield strength as well as an adequate uniform elongation of 68 %, which is a typical advantage of high-Mn austenitic TWIP steels. In addition, it is noteworthy that a yield drop phenomenon, which is often observed in ultrafine-grained pure Al, pure Ti and IF steels [8, 9], was also confirmed in the specimens with grain sizes smaller than 2.4 μm. With decreasing the mean grain size, the yield point phenomenon became more significant.

The yield strength and tensile strength obtained from the stress-strain curves are plotted as a function of inverse square root of the mean grain size in Fig. 3, which is known as a Hall-Petch plot. The 0.2 % offset proof stress was taken as the yield stress of the specimens exhibiting continuous yielding, while the upper yield stress was taken for those exhibiting the yield drop phenomenon. A typical Hall-Petch relationship stood over the entire range of the grain size from 85.6 μm to 0.79 μm for the tensile strength. However, positive deviation from Hall-Petch relationship of the yield strength occurred when the mean grain size was smaller than 2.4 μm, which coincided with the grain size that exhibited the yield drop phenomenon shown in Fig. 2.

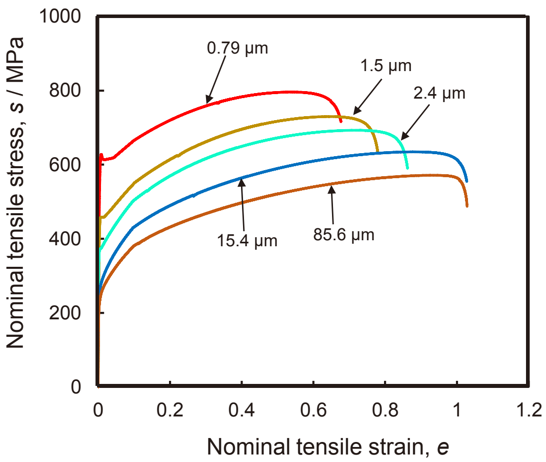

Fig. 2 Nominal stress-strain curves of the 31Mn-3Al-3Si TWIP steel with various grain sizes.

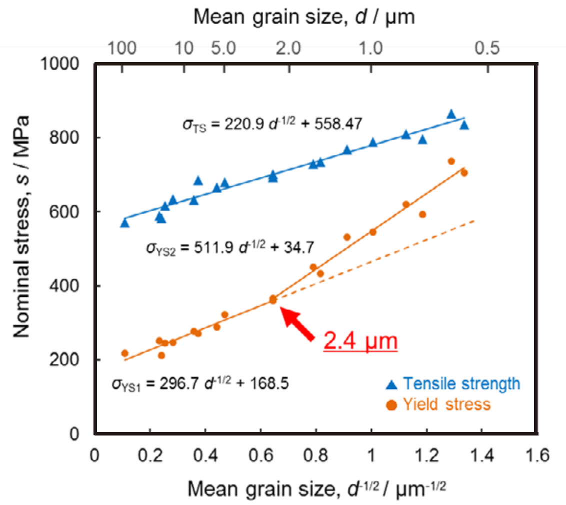

Fig. 3 Yield stress and tensile strength of the 31Mn-3Al-3Si TWIP steel plotted as a function of invers square root of the mean grain size (Hall-Petch plot).

3.3. Characterization of deformed microstructure

As shown in Fig.3, the yield strength of the fine grain-sized specimens (d < 2.4 μm) showed a different Hall-Petch relationship from that of the coarse grained specimens. In order to clarify the deformation mechanism of the UFG specimens, dislocation densities of the specimens with various grain sizes were measured by XRD method. The result showed that dislocation density suddenly increased at the yield point in the specimen with the grain size of 0.79 μm, while the dislocation density in the coarse-grained specimen (d = 15.4 μm) increased gradually over the entire range of the grain size.

In addition, deformation microstructures of the specimens tensile deformed to yield points were characterized by the SEM-ECCI technique. In the coarse-grained specimen with the mean grain size of 15.4 μm, only dislocation pile-up at the grain boundaries was observed. However, dislocations as well as stacking faults were observed in the UFG specimens with the grain size of 0.79 μm. It has been believed that deformation twinning suppressed with decreasing the grain size in TWIP steels. However, the results obtained in this study did not necessarily follow such a tendency. The change of the dislocation density and the deformation microstructure analysis suggested that the deformation mechanism in the UFG structures is different from that of the coarse grained structures at the yield point.

In order to explain the unique deformation behaviour around the yield point in the UFG specimens, we consider an emission of defects, dislocations and stacking faults, etc., from grain boundaries. The appearance of the yield drop phenomenon (discontinuous yielding) in the UFG specimens suggests a lack of mobile dislocations in the microstructures. Under such a situation, the emission of defects from grain boundaries is considered to be necessary to promote macroscopic plastic deformation. The rapid generation of dislocation density and increasing amount of stacking faults at the yield point in the UFG specimens support the emission of the defects from grain boundaries. Consequently, the yield drop in the UFG specimens can be explained by a stress relaxation caused by the rapid emission of defects from grain boundaries. From the results of stress-strain curves and Hall-Petch analysis, it is supposed that this mechanism becomes dominant when the grain size is smaller than 2.4 μm.

4. CONCLUSIONS

(1) In the present study, the mechanical properties and the deformation mechanism of the UFG 31Mn-3Al-3Si TWIP steel with various grain sizes were investigated. The fully recrystallized specimens with various grain sizes, including UFG specimens with the grain size of 0.79 μm were fabricated by conventional cold-rolling and annealing.

(2) The specimen with the ultrafine grains having mean grain sizes smaller than 1 μm exhibited a high yield stress and an adequate uniform elongation. A yield drop phenomenon was observed in the specimens with the mean grain sizes smaller than 2.4 μm. From the Hall-Petch analysis, the yield stress of the specimens having the mean grain sizes smaller than 2.4 μm exhibited a positive deviation from that expected from the extrapolation of the Hall-Petch curve for the coarse grain sized specimens.

(3) The dislocation density by XRD and microstructure observation by SEM-ECCI and the measurement of showed the increase of stacking faults and dislocations around the yield point in the UFG specimens, which suggested that the deformation mechanism changed in the UFG specimens. A modal that defects emit from grain boundary is proposed to explain the yield drop phenomenon in the UFG specimens.

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