Grain refinement of 2Mn-0.1C steel by repetitive heat treatment and recrystallization

M H Park¹, A Shibata¹², N Tsuji¹²

¹ Department of Materials Science and Engineering, Kyoto University, Sakyo-ku, Kyoto, 606-8501, Japan
² Elements Strategy Initiative for Structural Materials (ESISM), Kyoto University, Sakyo-ku, Kyoto, 606-8501, Japan

Email: park.heom.25v@st.kyoto-u.ac.jp

Abstract. Grain refinement in metals is well-known as one of the most effective methods to enhance their strength without addition of other elements. In this study, repetitive heat treatment combined with subsequent cold-rolling and recrystallization were investigated to obtain ultrafine-grained ferrite. Ultrafine-grained (UFG) ferritic structure having a mean grain size smaller than 1 μm was fabricated by repetitive heat treatment at 810 °C for 180 s and cold rolling by 90 % plus a recrystallization heat treatment at 600 °C. Starting from this UFG ferrite, fine-grained dual phase (DP) steel composed of ferrite and martensite phases with grain sizes smaller than 3 μm could be also obtained by intercritical heat treatment at 740 °C for 30 minutes followed by water-quenching. The mechanical properties of the ultrafine-grained ferritic and DP structured specimens were evaluated by tensile test. Results of the tensile test showed that fine-grained ferritic and DP structures had higher yield and tensile strength than the coarse-grained ferritic and DP structures of the same steel.

1. Introduction

Dual phase (DP) steels composed of ferrite and martensite phases are widely used for sheet materials in the automotive industry due to their good mechanical properties, such as high strength, adequate ductility and good formability. It is generally known that several factors, such as grain size of each phase, volume fraction and distribution of martensite, change the mechanical properties of DP steels. Among them, it is expected that grain refinement is an effective way to further improve the mechanical properties of DP steels. Recently, several studies reported that fine-grained DP steels showed excellent mechanical properties compared with DP steels with conventionally coarse grain sizes [1-5]. In order to fabricate bulky metals with ultrafine-grained structures, severe plastic deformation (SPD) processes, such as Equal Channel Angular Pressing, Accumulative Roll-Bonding and High Pressure Torsion, have been widely used [6-9]. The ultrafine-grained DP steels have also been obtained successfully using SPD processes [10, 11]. However, it seems difficult to apply SPD processes to mass production of steels because of the limited dimensions of the SPD processed materials. Several alternative processes for refining grain size other than SPD have been also proposed, for example, repetitive heat treatment [12, 13] and recrystallization of cold-rolled DP steel [14]. In the present study, grain refinement of ferrite and DP structures through repetitive heat treatment and subsequent cold-rolling and recrystallization was investigated.
2. Experimental Methods

The material used in the present study is a 2Mn-0.1C (wt. %) steel. The detailed chemical composition of the steel studied is shown in Table 1. The heat treatment pattern conducted is shown in Fig. 1. Firstly, the specimen was austenitized at 830 °C for 6 h and then water-quenched (W.Q.) or furnace-cooled (F.C.) to obtain a coarse martensitic structure ((1) in Fig. 1) or a coarse ferrite-pearlite structure ((2) in Fig. 1), respectively. The austenite grain size of the specimen was refined through repetitive heat treatment of austenitizing at 810 °C for 180 s and water-quenching. In the last cycle of the repetitive heat treatment, the specimen was water-quenched to obtain a fine martensitic structure ((3) in Fig. 1) or air-cooled (A.C.) to obtain a fine ferritic structure ((4) in Fig. 1). After the repetitive heat treatment, the specimen was annealed at 740 °C (between \( A_{e1} \) and \( A_{e3} \), ferrite + austenite two-phase region) for 30 minutes and water-quenched to obtain a DP structure composed of ferrite and martensite ((5) in Fig. 1). In order to further refine the grain size of DP structure, the specimen was cold rolled by 90 % and annealed at 600 °C (below \( A_{c1} \)) for different holding times ranging from 1 minute to 20 minutes to obtain a fine recrystallized ferrite structure ((6) in Fig. 1). Finally, the specimen was heat-treated at 740 °C for 30 minutes to obtain a fine DP structure ((7) in Fig. 1).

The microstructure of the specimen at each stage of the heat treatment shown in Fig. 1 was observed by scanning electron microscopy (SEM) and electron backscattering diffraction patterns (EBSD) analysis under an accelerated voltage of 15 kV. For the SEM and EBSD observations, the specimens were polished mechanically and then electrolytically in a solution of 10 % HClO₄ and 90 % CH₃COOH.

The mechanical properties of the specimens were examined by a uniaxial tensile test at a strain rate of \( 8.3 \times 10^{-4} \) s⁻¹ at room temperature (RT).

### Table 1 Chemical composition of the steel studied (wt. %).

|   | C   | Mn  | Si  | P   | S   | Fe   |
|---|-----|-----|-----|-----|-----|------|
|   | 0.103 | 2.00 | 0.01 | <0.002 | 0.0008 | Balance |

**Figure 1** Schematic representation of the heat treatment route for the 2Mn-0.1C steel.

3. Results and Discussion

3.1. Grain refinement of austenite and ferrite by repetitive heat treatment

Figure 2 shows EBSD orientation color maps of (a, b) the specimens after austenitization ((a): W.Q., (b): F.C., corresponding to the stages (1) and (2) in Fig. 1, respectively), and (c, d) the specimens after the repetitive heat treatments (4-cycles) ((c): W.Q., (d): A.C., the stages (3) and (4) in Fig. 1). In Fig. 2 (a) and (c), the prior austenite grain boundaries determined through orientation analysis were drawn in black dashed lines. The average prior austenite grain size was decreased from 28.6 μm to 6.1 μm by the
repetitive heat treatment. The martensite structure contains a high density of lattice defects, i.e., dislocations and high angle boundaries. These lattice defects would act as nucleation sites for reverse transformation to austenite, resulting in the refinement of austenite grains by the repetitive heat treatment. However, further repetition of the heat treatment above 4-cycles did not change the austenite grain size. The grain size of ferrite in Fig. 2 (d) was 4.5 \( \mu \text{m} \), which was much smaller than that in Fig. 2 (b) (10.2 \( \mu \text{m} \)). This indicates that the grain size of ferrite depends on the prior austenite grain size.

Figure 2 EBSD orientation color maps of the specimens heat-treated at 830 °C for 6 h and then (a) water-quenched (stage (1) in Fig.1), or (b) furnace-cooled (stage (2)), repetitive heat-treated at 810 °C for 180 s for 4-cycles and then (c) water-quenched (stage (3)) or (d) air-cooled (stage (4)).

3.2. Grain refinement of ferrite by combination of cold rolling and recrystallization

As reported by Okitsu et al. [14], deformation and recrystallization of a DP structure can effectively refine finally obtained ferrite. The specimen with a DP structure fabricated by the repetitive heat treatment followed by the intercritical annealing at 740 °C was cold-rolled by 90 % and then heat-treated at 600 °C (below \( A_{e1} \) temperature) for different periods of holding.

The EBSD orientation color map of the 90% cold-rolled specimen with DP structure is shown in Fig. 3 (a). The colors correspond to the crystallographic orientation parallel to the transverse direction (TD). The high angle boundaries with a misorientation above 15 degrees and low angle boundaries with misorientation ranging from 2 degrees to 15 degrees are drawn in black and white lines, respectively. The observation direction of Fig. 3 was parallel to the transverse direction (TD) of the sheet specimen. Almost all grains (including the martensite phase) in Fig. 3 (a) were elongated along the rolling direction (RD). After the heat treatment at 600 °C for 1 minute, 2 minutes, 5 minutes, 10 minutes and 20 minutes, corresponding to the stage (6) in Fig.1, (Fig. 3 (b), (c), (d), (e) and (f), respectively), on the other hand, ferritic structures with equiaxed grains were observed. The average ferrite grain sizes of these recrystallized specimens (b), (c), (d), (e) and (f) are 0.7 \( \mu \text{m} \), 0.9 \( \mu \text{m} \), 1.2 \( \mu \text{m} \), 1.6 \( \mu \text{m} \) and 2.0 \( \mu \text{m} \), respectively. The fraction of high angle grain boundaries (HAGBs) of those specimens (b-f) was 73.6 %, 62.6 %, 67.3 %, 88.7 % and 85.6 %, respectively. This indicates that the grain size and fraction of HAGBs in the ferrite increase with increasing holding time at 600 °C.
3.3. Fabrication of dual-phase steel with different ferrite grain sizes

In order to fabricate fine-grained DP steels, the ferrite structures obtained in Fig. 3 (stage (6) in Fig. 1) were intercritically annealed in the ferrite + austenite two-phase region and then water-quenched. Figure 4 displays EBSD Image Quality (IQ) maps of the DP structures obtained by the intercritical annealing at 740 °C for 30 minutes followed by water-quenching of the specimens (a) at the stage (2) in Fig. 1, (b) at the stage (4) and (c) at the stage (6), respectively. The grain size of each ferritic structure was 10.2 μm, 4.5 μm and 1.2 μm, respectively. IQ map expresses the quality of Kikuchi-lines obtained at each scanning spot. Thus, the martensite phase including many lattice defects is expressed in darker contrast, while the ferrite phase in lighter contrast. It was found that almost all the martensite in Fig. 4 formed along grain boundaries of ferrite. The fraction of martensite in Fig. 4 (a), (b) and (c) was 44.4 %, 43.8 % and 42.2 %, respectively. The grain sizes of ferrite in the DP structured specimen of (a), (b) and (c), on the other hand, were 8.1 μm, 4.1 μm and 2.6 μm, respectively. This result indicates that the grain size of the ferrite phase in DP steels strongly depends on the grain size of the ferritic structure prior to the intercritical annealing.
3.4. Mechanical properties of ferritic and DP structures with different grain sizes

In order to evaluate the mechanical properties of the ferritic structures obtained and DP structures with different grain sizes, tensile tests were carried out at a strain rate of $8.3 \times 10^{-4}$ s$^{-1}$ and room temperature. Figure 5 shows obtained stress-strain curves of (a) the ferritic structures and (b) the DP structures with different ferrite grain sizes. The grain size of ferrite is indicated on each curve in Fig. 5.

In Fig. 5 (a), it was found that the yield strength as well as the tensile strength increased with decreasing the ferrite grain size, and the yield-drop and Lüders deformation were obviously recognized in the specimens. The uniform elongations of the specimens with ferrite grain sizes of 1.2 μm, 4.5 μm and 10.2 μm are 0.008, 0.195 and 0.220, respectively. The result indicates that the UFG (1.2μm) ferritic structure has limited uniform elongation compared with the specimens of 4.5 μm and 10.2 μm grain sizes, while the UFG specimen shows a high tensile strength. The UFG (1.2μm) ferritic structure showed early plastic instability, as has been reported in various UFG materials [15-17].

In Fig. 5 (b), on the other hand, the DP structure with a smaller grain size exhibits a higher elongation as well as higher strength. The uniform elongation of the specimens having the ferrite grain sizes of 8.1 μm, 4.1 μm, and 2.6 μm are 0.09, 0.12 and 0.12, respectively, which are not different to each other. On the other hand, the total elongation significantly increased from 0.15 to 0.23 with decreasing the grain size. From this result, it can be concluded that the fine-grained DP specimen with a ferrite grain size of 4.1 μm shows very good performance in both ductility (especially total elongation) and strength, compared with other specimens.
4. Conclusions
Grain refinement of a 2Mn-0.1C steel was tried by a repetitive heat treatment combined with cold rolling and recrystallization. SEM / EBSD analysis and tensile tests were carried out for the specimens obtained with various structures. The main results obtained in this study are as follows.

The grain size of austenite decreased down to about 6 μm by 4-cycles of the repetitive heat treatment (810 °C for 180 s plus water-quenching) without deformation. By using this process, the ferrite grain size also decreased down to about 4 μm by air-cooling after the 4-cycle repetitive heat treatment.

90% cold rolling of the fine DP structure followed by annealing at 600 °C (below A_{c1}) was effective to obtain ultrafine-grained (about 1-2 μm) ferritic structures. Recrystallized ferrite became coarse with increasing the holding time at 600 °C.

Fine-grained DP structures were obtained by intercritical annealing at 740 °C of the UFG ferrite fabricated through the repetitive heat treatment and 90% cold-rolling + annealing. The grain size of the ferrite phase in the DP steel depended on the grain size of ferrite prior to the intercritical annealing.

In the specimens with ferritic structures, the yield and tensile strengths increased with decreasing the ferrite grain size. However, early plastic instability happened in the UFG ferrite with a grain size of 1.2 μm. On the other hand, the DP specimens having smaller grain sizes showed larger tensile elongation as well as higher strength. The DP specimen with 4.1 μm ferrite grain size showed very good performance in ductility (especially total elongation) as well as strength.

Acknowledgements
This study was financially supported by the Grant-in-Aid for Scientific Research on Innovative Areas, “Bulk Nanostructured Metals” (area No.2201), and the Elements Strategy Initiative for Structural Materials (ESISM) all through the Ministry of Education, Culture, Sports, Science and Technology (MEXT), Japan. M.P. was supported by the Japanese Government Scholarship. All the supports are gratefully appreciated.

References
[1] Saeidi N, Ashrafizadeh F and Niroumand B 2014 Mater. Sci & Eng. A. 599 148
[2] Delince´ M, Jacques P J and Pardoen T 2006 Acta Mater. 54 3395
[3] Calcagnotto M, Pndge D and Raabe D 2010 Mater. Sci & Eng. A. 527 7832
[4] Calcagnotto M, Adachi Y. Ponge D and Raabe D 2011 Acta Mater. 59 662
[5] Okayasu M, Sato K, Mizuno M, Hwang D Y and Shin D H 2008 Int’l J Fatigue. 30 1360
[6] Azushima A, Kopp R, Korhonen A, Yang D Y, Micari F, Lahoti G D, Groche P, Yanagimoto J, Tsuji N, Rosochowski A and Yanagida A 2008 Manufacturing Tech. 57 716
[7] Stolyarov V, Zhu T, Lowe C and Valiev Z 2001 Mater. Sci & Eng. A. 303 82
[8] Tsuji N, Ueji R and Minamino Y 2002 Scripta Mater. 47 69
[9] Mohamed I F, Yonenaga Y, Lee S, Edalati K and Horita Z 2015 Mater. Sci & Eng. A. 627 111
[10] Son Y I, Lee Y K, Park K T, Lee C S and Shin D H 2005 Acta Mater. 53 3125
[11] Tsuji N, Kamikawa N, Ueji R, Takata N, Koyama H and Terada D 2008 ISIJ Int’T. 48 1119
[12] Furuhara T, Kikumoto K, Tsuji N, Rochowski A and Yanagida A 2008 ISIJ Int’T. 48 1040
[13] Shibata A, Daido S, Terada D and Tsuji N 2013 Mater. Trans. 54 1571
[14] Okitsu Y, Takata N, Tsuji N 2009 Scripta Mater. 60 78
[15] Ding Y, Jiang J and Shan A 2009 J Alloy Compd., 487 520
[16] Sun C, Ma J, Yang Y, Hartwig K T, Maloy S A, Wang H and Zhang X 2014 Mater. Sci & Eng. A. 597 416
[17] Ma E, 2003 Scripta Mater. 49 664