Effects of Nb and Mo Addition to 0.2%C–1.5%Si–1.5%Mn Steel on Mechanical Properties of Hot Rolled TRIP-aided Steel Sheets

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The development of high strength hot rolled TRIP (Transformation induced plasticity)-aided steel sheets with 780 MPa was carried out by taking into account the addition of Nb and Mo to 0.2C–1.5Si–1.5 Mn mass% steel and coiling conditions after hot rolling. The results reveal that the addition of 0.05% Nb can attain higher elongation with higher strength compared with Nb-free steel. The obtained tensile strength in this steel was higher than 780 MPa. The multiple addition of 0.2% Mo with 0.05% Nb results in higher TS (Tensile Strength) by the large amount of fine NbMoC precipitates than Nb containing steel without the deterioration of TS–El (Elongation) balance under the possible hot rolling conditions. The good ductility in 0.05% Nb containing steel was mainly obtained by large volume fraction and high carbon concentration of retained austenite. In addition, finely dispersed retained austenite made some contribution to the improvement of ductility.

KEY WORDS: TRIP; high strength hot rolled steel sheet; elongation; Nb; Mo; coiling temperature.

1. Introduction

In order to meet the automobile industry’s need for weight reduction while still taking into consideration possible hazards to the environment and safety improvements, the application of advanced high strength steel sheets such as TRIP (Transformation induced plasticity)-aided steel and DP (Dual Phase) steel have been examined. Automobile makers have asked for formable high strength steel sheets of 780 and 980 MPa tensile strength (TS) grade steels for suspensions and structural parts.

It is well known that the higher the strength of steel sheets, the poorer the press formability. Conventional HSLA (High Strength Low Alloy) steels could not satisfy the press formability for such high strength applications, especially for ultra high strengths such as 780 and 980 MPa TS grade. TRIP effect is the most successful mechanisms for increasing the strength of steels without deteriorating the strength and elongation balance. The formability of TRIP-aided steel primarily depends on the amount and stability of retained austenite. However the steel sheets containing high carbon and high alloying elements such as Ni and Mn can not be applied for automobile parts due to their low weldability, low surface condition and high production cost. Hence, carbon content must be limited to less than 0.2% and Si, Mn and Al are limited to less than 2% respectively. It was difficult to produce 780 or 980 MPa steel in such a chemical composition. However, although a many research results on TRIP-aided steel produced by heat treatment after cold rolling have been reported,1) there were few reports on hot rolled TRIP-aided steel.2–7) In addition, those that published were limited to relatively low strength levels. The microallying elements such as Ti, Nb, V and Mo are known to increase strength through precipitation hardening. As many reports suggest the Nb or Nb and Mo addition to TRIP-aided steel resulted in higher strength and better ductility,6–8) the effect of those elements on TRIP-aided hot rolled steel was examined in this study.

Based on the above mentioned background, the development of 780 MPa grade TRIP-aided steel was carried out by taking into account the addition of Nb and Mo and coiling conditions after hot rolling.

2. Experimental Procedure

2.1. Chemical Compositions of Steels and Processing Conditions

Five kinds of 0.2C–1.5Si–1.5Mn–0.01P–0.035Al (mass%) steels with different amounts of Nb and Mo were induction melted as 40 kg in air. The chemical composi-

| Steel | C  | Si  | Mn  | P  | S  | Al  | Nb | Mo | N  |
|-------|----|-----|-----|----|----|-----|----|----|----|
| B    | 0.21| 1.49| 1.45| 0.005|0.003|0.032|   |    |    |
| LN   | 0.21| 1.48| 1.48| 0.005|0.003|0.021|0.016|    | 0.0088 |
| LNM  | 0.21| 1.49| 1.49| 0.005|0.002|0.028|0.017|0.10 | 0.0085 |
| HH   | 0.21| 1.60| 1.53| 0.005|0.003|0.031|0.048|    | 0.0073 |
| HNM  | 0.20| 1.47| 1.51| 0.004|0.003|0.028|0.047|0.20 | 0.0065 |
tions of steels are shown in Table 1. Steel B is the base steel without any addition of Nb and Mo. Low Nb of 0.02% and low Nb+low Mo of 0.1% are added to base steel B and designated steels LN and LNM. Steels HN and HNM contain high Nb of 0.05% and high Nb+high Mo of 0.2%.

As shown in Fig. 1, the rough rolled 30 mm in thickness slabs were hot rolled to 3 mm in thickness. The reheating temperature and finishing temperature were 1200°C and 850°C respectively. As a basic experimental condition, the hot rolled sheets were water sprayed by 70°C/s to 750°C and air cooled for 10 s and then water sprayed again to coiling temperature and kept for 10 min, and then air cooled. In order to study the effect of coiling temperature, coiling temperatures were varied from 300 to 550°C with 50°C intervals. In addition to the basic coiling conditions, the holding time at coiling temperature of 400°C and 450°C was varied for 5 min and 50 min.

2.2. Testing Procedure

The specimens for the tensile test were cut in a longitudinal direction and machined to JIS No 5 specimen with the thickness of 2.0 mm, whose gauge length and width are 50 mm and 25 mm respectively. The testing speed was 27 mm/min. Number of specimens for each tensile test was three.

The microstructure was observed by optical microscopy with LePera etching9) as well as SEM (Scanning Electron Microscopy) with Nital etching. TEM (Transmission Electron Microscopy) observations were carried out to observe the microstructure and precipitates.

Volume fraction of retained austenite (γR) was measured by saturation magnetization measurement.10) The carbon concentration in retained austenite (Cγ, mass%) was estimated by substituting the average lattice constant a0 into the following equation, (1).11) The average lattice constant a0 was determined by measuring the lattice constant of (200)γ, (220)γ, and (311)γ by Mo–Kα radiation.

\[ C_{\gamma} = (a_0 - 3.578)/0.033 \]

3. Experimental Results

3.1. Mechanical Properties

The bainite transformation which brings the carbon enrichment in retained austenite proceeds during the coiling process, in the case of hot rolled sheet. Therefore, the effect of coiling conditions is one of the most important process parameters in the hot rolling process.

The effects of alloying elements and coiling temperature (CT) on yield strength (YS) and tensile strength (TS) are shown in Fig. 2. Most of steel shows minimum YS at CT of 450°C. Nb or Nb and Mo added steels show increase in YS in high CT range, in contrast steel B shows nearly same TS with increase in CT in this range. Nb and Mo added steels show highest YS at CT of 350°C and 400°C. TS decreases remarkably with increase in CT from 300°C to 400°C in all of the steels and then a moderate decrease in TS with an increase in CT of 400°C is sensitive to alloying elements. At CT of 400°C, TS of steel B is 760 MPa, whereas TS of LN and HN are 830 MPa and Nb+Mo containing steels are 880 MPa. The steels with 0.02 or 0.05 Nb show nearly same TS in all of the CT ranges.

The effects of alloying elements and CT on total elongation (EI) is shown in Fig. 3. Except for steel LNM which showed peak EI at CT of 450°C, all of the steels show highest EI at CT of 400°C. Steels HN and HNM which contain 0.05% Nb show higher EI than that of steel B at CT of 400°C, in spite of their higher TS. The highest EI of 32% is obtained in steel HN. With a decrease in CT from the peak
CT, El decreased remarkably in all of the steels, and with increase in CT from the peak CT, El decreases slightly. In order to know TS and El balance, TS/H₁₁₀₀₃ El is calculated and shown in Fig. 4. The results exhibit a similar trend as El shown in Fig. 3. The highest value is obtained at CT of 400°C for steels HN and HNM. Above all, the steel B shows the lowest value. From these results it is clarified that Nb addition of 0.05 % brings higher TS with better El, and the steel with 0.2 % Mo together with 0.05 % Nb shows as high TS/H₁₁₀₀₃ El value as 0.05 % Nb containing steel, HN, with remarkable increase in TS.

3.2. Microstructures

The volume fraction and carbon concentration of retained austenite in the experiment of basic coiling conditions are shown in Fig. 5. The behavior of retained austenite with CT and alloying elements is almost accorded with the behavior of El or TS×El shown in Figs. 3 and 4. The retained austenite increases with an increase in CT and shows the highest value at CT of 400°C, and then decreases with increase in CT. In the high CT range of 500°C and 550°C, Mo added steels show higher retained austenite than other steels. The volume fraction of retained austenite of steel HN which showed the highest El is as high as 16%. This value is about 4% higher than other steels. Except Mo added steels, the volume fraction of retained austenite at CT higher than 500°C is almost 0%. Carbon concentration of the steel HN also showed the maximum value at CT of 400°C as 1.2%.

The effect of alloying elements on the microstructures of the steels coiled at 400°C are shown in Fig. 6. In the optical micrographs with LePera etching, the gray, black and white areas represent ferrite, bainite and retained austenite and/or martensite respectively. In SEM micrographs with Nital etching, the black area and white area represent ferrite and second phase composed of bainite, retained austenite and martensite respectively. Microstructure of steel B is composed of equiaxed ferrite and second phase. On the other hand, the Nb containing steels show elongated second phase. The proportion of ferrite is not affected by the addition of Nb, but is decreased by the addition of Mo. The addition of high Nb and Mo (steel HNM) brings about the refinement of ferrite grain.

The effect of CT on the microstructure of steel HN is shown in Fig. 7. The volume fraction of white area decreases remarkably with an increase in CT. In addition, the morphology of second phase changes with CT. The main structure of the second phase of the steels coiled at low temperature of 300°C must be martensite. Because although the white area in optical micrograph occupies about 40%, the volume fraction of retained austenite shown in Fig. 5 is only 7%. While in the steel coiled at higher than 500°C, cementite at the grain boundary and small amount of fine pearlite are observed by TEM. Small amount of white area is observed in the optical micrographs etched by LePera etchant. Judging from the fact the volume fraction of retained austenite was 0% in Fig. 5, all of the white portion must be martensite. The most of white area of the steel coiled at 400°C must be retained austenite, judging from the volume fraction of retained austenite was 16% in Fig. 5.

3.3. Effect of Coiling Conditions on Mechanical Properties

In the actual hot rolling process, the thermal history after coiling is different at the position of coil, i.e., inner or outer,
edge or center and coil weight, which results in the change in the mechanical properties. Therefore, the stable mechanical properties in all of the coils and in the wide range of production processes are required by the actual production site. In order to study the change in the mechanical properties with the changes in the coiling conditions, the holding time at CT of 400°C and 450°C is changed to 5 min and 50 min using steels B, HN and HNM. The effects of coiling condition on the mechanical properties and volume fraction of retained austenite are shown in Fig. 8.

TS decreases with increase in the holding time at CT of both 400°C and 450°C. Higher TS in steels HN and HNM is obtained in all of the CT and holding time conditions compared with Nb free steel B. Steel B shows 780 MPa only at a condition for 5 min at 450°C. Steel HN exhibits 780 MPa in all of the ranges except for CT of 450°C for 50 min. Steel HNM shows higher TS than 880 MPa in the most of the conditions.

Steel HN and HNM show the highest El for the steels coiled at CT of 400°C and held for 10 min. The lowest El is observed for the steels coiled at 450°C and held for 50 min in all of the steels.

Steel HN and HNM show higher TS×El values in the wide area of coiling conditions than steel B. The changes in TS×El with coiling condition shows nearly same trend with above mentioned El.

Figure 8 also shows the volume fraction of retained austenite. Steel HN shows high volume fraction of about 15% in wide area, which was in accordance with the results of El in Fig. 8. Steels B and HNM show high area of large volume fraction of retained austenite of 10 to 13% in the same coiling condition. The volume fraction of retained austenite at the holding time of 50 min is extremely low in all of the steels.
It was revealed that mechanical property and microstructure are largely affected by the coiling condition and alloying elements such as Nb and Mo. The metallurgical background for them are discussed in this section.

### 4.1. Volume Fraction and Carbon Enrichment for the Retained Austenite

El of TRIP-aided steel is governed mainly by the volume fraction and stability of retained austenite which is mainly controlled by carbon content in retained austenite. As shown in Fig. 3 and Fig. 5, the good correlation between El and retained austenite was observed also in this study. The reason why the highest volume fraction and carbon concentration was obtained for the steel coiled at 400°C is discussed in this section.

The carbon enrichment process after hot rolling is illustrated in the equilibrium diagram for 1.5% Si–1.5% Mn steel in Fig. 9. To line in this figure means the locus of the points at which the chemical free energy of ferrite is identical to that of austenite with the same chemical composition. Therefore, the maximum carbon concentration in austenite is determined by the carbon content at To line. The carbon enrichment in austenite progresses in a intercritical region through ferrite transformation at cooling process and in bainite transformation region at coiling process. If the keeping time at lower coiling temperature region is long enough to reach To line, high carbon concentration in austenite along To line should be obtained. However, since the holding time limited in this experiment, carbon concentration in austenite can not reach to To line in low CT range. Therefore, the carbon concentration and the volume fraction of retained austenite remain low in low CT range, and the volume fraction of martensite becomes high. Similarly, in high temperature CT range, carbide can form easily and the carbon in austenite decreases. As a result, the volume fraction of retained austenite becomes small due to the low carbon concentration in retained austenite. Thus, the highest volume fraction of retained austenite with high carbon concentration can be obtained for the steel coiled at 400°C as the result of the best combination of bainite transformation rate and carbon content at To line. The carbon concentration in the retained austenite is nearly consistent with the carbon content obtained by equilibrium diagram. The total carbon in retained austenite of steel HN coiled at 400°C is calculated as 0.19%. This value nearly corresponds to all of the carbon in the steel, indicating that carbon is efficiently enriched in the retained austenite.

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The high volume fraction of retained austenite in 0.05 Nb containing steel, HN, is explained by lowered Ms temperature through carbon enrichment of the austenite and grain refinement of austenite. Fine austenite tends to accelerate ferrite transformation starting at the austenite
grain boundary, resulting in the enrichment of carbon to austenite. The reason why Mo added steels show higher volume fraction of retained austenite in high CT range than other steels is thought to be brought about by the pearlite and bainite transformation retardation by Mo.

4.2. Effect of Precipitates on Strength

TS increased with the addition of Nb and Mo. However, the effect of these elements on TS is slightly complex. In order to know the effect of Nb and Mo on TS, the difference in TS, ΔTS, between Nb and/or Mo added steels and base steel, B, is shown in Fig. 10. If the microstructures of all of the steels are the same, the ΔTS is caused by only precipitation hardening. However, as the microstructure is not the same, the interpretation is rather complex, especially in the low coiling temperature range. Because the small amount of difference of martensite affects the TS remarkably. Therefore the discussion is focused on the high coiling temperature range than 400°C.

In the high coiling temperature range, higher than 400°C, ΔTS of Nb steels is about 75 MPa regardless of CT. It is assumed that NbC precipitates mainly during cooling after hot rolling. On the other hand, Nb–Mo steels show an increase in ΔTS with the increase in CT. The maximum ΔTS obtained at CT of 500°C is as high as 240 MPa in higher Mo containing steel, HNM. NbMoC must be precipitated during the coiling process, in addition to the precipitation during cooling.

In order to prove the above mentioned explanation, the precipitates were observed by TEM. The identification of precipitates was carried out by EDX. The results are given in Fig. 11. In steel HN, despite of the difference in CT, nearly the same precipitates such as nano-meter NbC and coarse NbC were observed. On the other hand, in steel HNM, a large amount of nano-meter NbMoC and coarse NbC were observed. Fine precipitates must be precipitated during the cooling and coiling process in ferrite region, and coarse NbC must be precipitated in austenite region or
undissolved precipitates. Although the quantitative analysis of the volume fraction with the difference of CT and chemical composition was not determined, the qualitative evidence was shown in these figures.

The quantitative estimation for precipitates in four kinds of steels is estimated by the equilibrium precipitation curves calculated by Thermo–Calc. shown in Fig. 12. The volume fraction of NbC shows the same value in the coiling temperature range below 550°C. According to Fig. 12, the volume fraction is different between steels LN and HN. However, ΔTS is same in two steels. These facts suggest that some amount of solid solute Nb is remaining in final product in high Nb containing steel HN. In contrast, NbMoC shows remarkable change in this range. This could be associated with the precipitation of NbMoC during the coiling process as well as the cooling process. The larger ΔTS in steel HNM must be given by the larger volume fraction of the precipitates in steel HNM.

4.3. Factors Affecting the Elongation

As mentioned in 4.1, elongation of TRIP-aided steel is determined mainly by the volume fraction and stability of retained austenite which is mainly controlled by carbon content in retained austenite. The change in volume fraction and carbon concentration of retained austenite with CT shown in Fig. 5 and the volume fraction of retained austenite in various holding times at CT of 400°C and 450°C shown in Fig. 8 are almost in accordance with the behavior of EL. These results mean that El is mainly controlled by the volume fraction and carbon concentration of retained austenite.

In addition, it is reported that several factors affect the elongation of TRIP-aided steels. These include the morphology of retained austenite, interparticle spacing of retained austenite and the morphology of the ferrite matrix.

The morphology of retained austenite is strongly affected by the addition of Nb as shown in Fig. 6. In some works on multiphase steels, the very important role of microstructural refinement in formability was pointed. Regsbee et al. clearly showed that a fine dispersion of retained austenite is advantageous in attaining high n-value (work hardening coefficient), which is accorded with uniform elongation, even at the constant amount of retained austenite. In order to investigate whether the results obtained in this experiment are...
explained by the above mentioned view, the morphology of retained austenite and n-value in this experiment are measured and discussed.

The morphology and dispersion of retained austenite were observed by EBSP for steels B and HN, and results were given in Fig. 13. Figures 13(a) and 13(c) show the image quality of steel B and HN respectively. Fig. 13(b) and 13(d) show \( \gamma \) phase indicated by red color region and \( \alpha \) phase indicated by green color region for steels B and HN respectively. The volume fractions of retained austenite observed in EBSP are 3.9% for steel B and 8.6% for steel HN. Although the values are fairly smaller than those observed by magnetic saturation method, in the case of EBSP analysis, the volume fraction of retained austenite tends to decrease due to the transformation by the release from static hydro pressure and by the strain during the preparation procedure of the specimen etc. The difference of the observed area may be one of the reasons of it. Therefore the EBSP analysis is effective only for the morphology observation. The results clearly show that the retained austenite in steel HN is small and finely distributed, while it is large and coarsely distributed in steel B. In addition, the retained austenite in steel HN exists mainly along the bainitic ferrite lath but mainly along the ferrite boundary in steel B. Although it was difficult to measure the exact interparticle spacing of retained austenite, Nb added steel HN showed the smaller interparticle spacing compared with Nb free steel B.

The effects of alloying elements and coiling temperature on n-value are shown in Fig. 14. The n-value in Nb added steels showed higher value in all of CT range. The n-value of steels HN and B coiled at 400°C, whose morphology of retained austenite is shown in Fig. 13, was 0.25 and 0.16 respectively.

From these results, the higher n-value in steel HN is explained by the finer dispersion of retained austenite as pointed out by Ruhlsbee. The higher elongation in steel HN is brought about by the higher n-value, which corresponds with uniform elongation. The fact that the finely dispersed retained austenite is obtained in 0.05% Nb steels demonstrates the importance of Nb addition as high as 0.05%. Judging from the same TS between steels LN and HN, a considerable amount of the added Nb in 0.05% steel must be in solid solution. The solid solution Nb efficiently brings about the grain refinement and small interparticle spacing of the retained austenite through the austenite grain refinement. This could be one of the responsible points for the higher ductility in steels with 0.05% Nb.

5. Conclusions

In order to develop high strength TRIP-aided hot rolled steel sheet, the effects of Nb and Mo additions to Fe–0.2%C–1.5%Si–1.5%Mn and coiling conditions after hot rolling on the mechanical properties were investigated and the following conclusions were obtained.

(1) 0.05%Nb containing steel showed higher El than Nb-free steel, regardless of the higher TS. The obtained TS and El at the best coiling temperature at 400°C were 822 MPa and 32%. Since the TS and El in Nb free steel is 762 MPa and 27% respectively under the same hot rolling condition, it is clear that the addition of Nb is effective in producing 780 MPa grade hot rolled TRIP-aided steel with excellent El.

(2) The multiple addition of 0.05%Nb and 0.2% Mo increases the TS of 120 MPa TS than that of Nb–Mo free steel at CT of 400°C. This result suggests that the addition of Mo with Nb is effective in producing higher TS grade hot rolled TRIP-aided steel.

(3) The highest El and TS×El were obtained when the steels were coiled at 400°C and kept for 10 min. This condition corresponds to the condition showing the highest volume fraction and carbon concentration of retained austenite.

(4) The higher strength in Nb containing steel is mainly attributed to the precipitation of fine NbC. The NbC precipitated mainly during the cooling process. While, the highest strength in Nb and Mo containing steel is brought about by larger amount of fine precipitation of NbMoC, which precipitated during both the cooling and cooling process.

(5) Good ductility in 0.05%Nb containing steel was mainly obtained by large volume fraction of retained austenite. In addition, fine dispersed morphology of retained austenite also contributed to the improvement of ductility through high n-value.

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