Effects of Heat Treatment and Si Addition on the Mechanical Properties of 0.1 wt% C TRIP-aided Cold-rolled Steels

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The purpose of the present study was understanding the effects of heat treatment and silicon content on the microstructure and mechanical properties of low-carbon TRIP-aided cold-rolled steel sheets. Two steels of the same base composition, 0.10%C–1.5%Mn–Si–0.5%Cu (hereafter all in weight percent), but containing 0.94 % Si and 1.48 % Si were cold rolled to 1 mm thick sheet. The sheets were intercritically annealed and isothermally treated in the temperature range of bainite reaction in order to vary the volume fraction of retained austenite and the mechanical properties. The fractions of retained austenite increased with decreasing intercritical annealing and isothermal treatment temperatures, resulting in the improvement in tensile strength, elongation, and the strength–ductility balance. In the steel having the higher silicon content, a higher fraction of retained austenite and better mechanical properties were achieved than in the steel having the lower silicon content. The findings indicate that partitioning of C and Mn to the austenite during intercritical annealing, together with a higher Si content, increase the stability of the austenite and affect the optimum intercritical and isothermal heat treatment temperatures.

KEY WORDS: TRIP-aided cold-rolled steels; low carbon; multiphase; Si effect; heat treatment; mechanical properties; retained austenite.

1. Introduction

Recently, demands for high-strength cold-rolled steel sheets from industries, automotive industry in particular, have been rapidly increasing in order to improve safety as well as to reduce weight. TRIP (transformation induced plasticity)-aided cold-rolled steel sheets have also received increasing attention as steels having both high strength and high ductility because of the martensitic transformation of retained austenite during plastic deformation.^{1–4)} Since the first TRIP-aided steels were developed by Zackay *et al.*,⁵⁾ many studies have been made in this area. A considerable amount of retained austenite can be obtained, together with excellent tensile strength and ductility, when cold-rolled C–Mn–Si steels are heated up to the $(\alpha+\gamma)$ two phase region and then isothermally treated for several minutes in the temperature range of bainite reaction. A conventional continuous annealing line (CAL) can be easily applied to the isothermal treatment.^{5–8)} Studies on TRIP-aided cold-rolled steels have mainly focused on the cases of the carbon content of 0.2–0.4 wt%. These medium-carbon TRIP-aided cold-rolled steels have many advantages in mechanical properties over conventional high-strength cold-rolled steels. However, weldability, which is an important property required for cold-rolled steels, can deteriorate because of the high carbon content. In order to overcome this shortcoming, more studies are required to lower the carbon content.

It is known that silicon, a major alloying element in

TRIP-aided cold-rolled steels, plays an important role,^{9–11)} and is expected to substantially contribute to the mechanical property improvement and to the higher volume fraction of retained austenite in cold-rolled steels containing lower carbon than conventional TRIP-aided ones. To assure the strength and ductility improvement from stabilized retained austenite in low-carbon C–Mn–Si TRIP-aided cold-rolled steels, an optimum combination of alloying elements and optimum conditions for intercritical annealing (annealing in the $(\alpha+\gamma)$ region) and isothermal treatment should be established so that the effect of alloying elements can be maximized.

Meanwhile, the addition of copper to C–Mn–Si TRIP-aided steels was confirmed to contribute to the enhancement of strength and elongation by the present authors,¹²⁾ although copper was known as a harmful element due to its hot shortness. Thus, in the present study, two compositions of cold-rolled steel sheets in which the carbon content was lowered to 0.10% and 0.5% of copper was also present were fabricated. Investigations were made into how variations in the intercritical annealing and isothermal treatment conditions and in the silicon content affected the mechanical properties and the volume fractions of retained austenite of cold-rolled steel sheets.

2. Experimental Procedure

2.1. Melting and Rolling

Two kinds of steel ingots, named as S1 and S2, were fab-

Table 1. Chemical compositions (wt%) and transformation temperatures (°C) measured using a dilatometer of the steels investigated.

Steel	C	Mn	Si	Cu	A _{C1}	A _{C3}	Ms
S1	0.10	1.51	0.94	0.49	738	882	445
S2	0.10	1.52	1.48	0.51	755	907	449

Table 2. Heat-treatment conditions of the cold-rolled steel sheets.

Steel	Intercritical Annealing	Intercritical Annealing	Isothermal Treatment	Isothermal Treatment
	Temperature (°C)	Time (min.)	Temperature (°C)	Time (min.)
S1	810, 780	5	450, 470	1, 3, 5, 10, 20
S2	830, 810			

ricated by vacuum induction melting. **Table 1** lists their chemical compositions, and A_{C1}, A_{C3}, and martensite transformation starting temperatures (Ms) measured using a dilatometer. Steel ingots were rough-rolled into slabs of 25 mm in thickness. The slabs were homogenized at 1250°C for two hours, hot-rolled into sheets of 3 mm in thickness. The finish rolling temperature was 900°C. The hot-rolled steel sheets were pickled by a 10% HCl solution of 80°C, and were cold-rolled into sheets of 1 mm in thickness.

2.2. Intercritical Annealing and Isothermal Treatment

The conditions of intercritical annealing and isothermal treatment are significantly affecting the formation of retained austenite and the subsequent mechanical properties in TRIP-aided cold-rolled steels.^{1,13–14} In this study, the intercritical annealing was conducted at the temperature range of 780–830°C for five minutes using a salt bath. The isothermal treatment was carried out in the temperature range of 450–470°C for 1–20 min, and then air-cooled. **Table 2** summarizes the heat-treatment conditions of the present study.

2.3. Evaluation of Mechanical Properties

Tensile specimens (longitudinal direction) with a gage length of 25.4 mm and a width of 6.3 mm were prepared from the cold-rolled steel sheets. The tensile specimens were deformed at room temperature at a crosshead speed of 2 mm/min using a tensile tester, and yield strength, tensile strength and elongation were measured. The lower yield point was adopted as yield strength in the case of yield point phenomenon, whereas 0.2% offset value in the case of continuous yielding.

2.4. Microstructural Observation

Since C–Mn–Si TRIP-aided steels have very complex microstructures composed of ferrite, bainite and/or martensite, and retained austenite, identification of each phase is unclear when etched in a nital solution. A sodium metabisulfite solution (Na₂S₂O₃·H₂O 10 g + H₂O 100 ml) was used in conjunction with the nital etching to identify the phases. When the specimens etched by the sodium metabisulfite solution are observed by an optical microscope, it is easier to define the phases since ferrite is displayed light or dark gray, bainite or martensite as black,

while retained austenite as white.¹²⁾

2.5. Volume Fraction Measurement of Retained Austenite

Volume fraction of the retained austenite was measured using an X-ray diffractometer (XRD). Mo-K_α characteristic X-ray was used, and the volume fraction of retained austenite, V_γ , was calculated from the integrated intensity of ferrite and austenite peaks using the equation, $V_\gamma = 1.4I_\gamma / (I_\alpha + 1.4I_\gamma)$.^{15,16} Here, I_γ is the average integrated intensity obtained at {220}_γ and {311}_γ peaks, and I_α is that obtained at {211}_α peak.

3. Results

3.1. Microstructural Variation vs. Heat Treatment

Figure 1 presents optical micrographs of the S1 steel after the isothermal treatment at 450°C, when the isothermal treatment time and the intercritical annealing temperature are varied. When the specimens are quenched into water after the intercritical annealing at a certain temperature between A_{C1} and A_{C3}, austenite formed during the intercritical annealing transforms into martensite. Thus, the volume fraction of ferrite and austenite can be determined through the image analyzing method after the intercritical annealing. The volume fraction ratio of ferrite and austenite formed at 810°C was about 40:60, while at 780°C about 50:50. In the case of the intercritical annealing temperature of 810°C, being (A_{C1} + A_{C3})/2, the ferrite fraction increases as the isothermal treatment time becomes longer, whereas the fraction of secondary phases displayed gray or black decreases. This tendency is relaxed more or less as the intercritical annealing temperature is lowered. When the S1 steel was intercritically annealed at 780°C, the fraction of secondary phases is reduced compared to the case of 810°C.

Figure 2 shows optical micrographs of the S2 steel isothermally treated at 450°C. The overall microstructural variation tends to be similar to that of the S1 steel. The ferrite fraction increases with increasing the isothermal treatment time, and this tendency is less visible as the intercritical annealing temperature drops from 830°C, being (A_{C1} + A_{C3})/2, to 810°C at which the fraction ratio of ferrite and austenite is 50:50.

Figures 3(a) and **3(b)** are optical micrographs of the S1 and S2 steels after they were etched by a sodium metabisulfite solution. **Figures 3(a)** and **3(b)** show the S1 steel intercritically annealed at 780°C and the S2 steel intercritically annealed at 810°C, respectively, followed by the isothermal treatment at 450°C for 5 min. The retained austenites are homogeneously distributed all over the microstructure, and most of them coexist with nearby ferrite, bainite or martensite, whereas some are isolated inside ferrite grains.

3.2. Mechanical Properties and Volume Fraction of Retained Austenite

Figure 4 summarizes the tensile properties and the fraction of retained austenite of the S1 steel. The heat-treatment conditions are shorthanded in **Fig. 4** with two numbers linked by a hyphen, such as 810-450, meaning that the specimen was intercritically annealed at 810°C and then isothermally treated at 450°C. Tensile strength and elonga-

Fig. 1. Optical micrographs of the S1 steel intercritically annealed at 810°C and 780°C and isothermally treated at 450°C for 1 and 10 min (etched by a 2% nital solution).

Fig. 2. Optical micrographs of the S2 steel intercritically annealed at 830°C and 810°C and isothermally treated at 450°C for 1 and 10 min (etched by a 2% nital solution).

tion decrease, but yield strength considerably increases with increasing the isothermal treatment time, irrespective of the intercritical annealing temperature. Comparing the mechanical properties with the intercritical annealing temperature, yield strength at 780°C is lower than that at 810°C by about 30 MPa, but tensile strength stays about the same level, while elongation and strength–ductility balance are high. Comparing the cases of isothermal treatment at 450°C and at 470°C following intercritical annealing at 780°C, the former shows higher tensile strength, elongation, and strength–ductility balance than the latter. Such variations in mechanical properties are closely related with the fraction

of retained austenite. The variations in the fraction of retained austenite as to the intercritical annealing temperature and the isothermal treatment temperature and time are found quite substantial. In the case of the intercritical annealing temperature of 810°C (810-450 specimens), the fraction of retained austenite is close to 10% when the isothermal treatment time is 1 min, but is reduced down to 2% when isothermally treated for 10 min. When intercritically annealed at 780°C (780-450 specimens), it is slightly over 10% at the initial stage of isothermal treatment, and then decreases to about 4% after 10-min isothermal treatment; this case thus shows the higher fraction of retained

Fig. 3. Optical micrographs of (a) the S1 steel intercritically annealed at 780°C and isothermally treated at 450°C for 5 min and (b) the S2 steel intercritically annealed at 810°C and isothermally treated at 450°C for 5 min (etched by a sodium metabisulfite solution).

austenite overall than the 810°C case. Isothermal treatment at 450°C (780-450 specimens) also shows the higher fraction of retained austenite in comparison with 470°C (780-470 specimens).

Figure 5 presents the mechanical properties and the fraction of retained austenite of the S2 steel, whose overall tendency is similar to the case of the S1 steel as observed in Fig. 4. However, the mechanical property variation as to the isothermal treatment time is smaller here. The fraction variation of retained austenite is also smaller, and shows a reduction of about 2–4% as a function of isothermal holding time when isothermally treated at 450°C (810-450 specimens). As in the case of the S1 steel of Fig. 4, the steel intercritically annealed at 810°C at which the fraction ratio of ferrite and austenite is 50:50 and isothermally treated at 450°C (810-450 specimens) has the higher retained austenite fraction, together with better mechanical properties overall. It is found from Figs. 4 and 5 that lowering intercritical annealing temperature and isothermal treatment temperature works more favorably for the increased fraction of retained austenite and the subsequent improvement in strength and elongation.

3.3. Effect of Silicon Addition on Mechanical Properties and Retained Austenite Fraction

Figure 6 shows the variations in the mechanical property and the fraction of retained austenite of the S1 and S2 steels intercritically annealed at the temperature, at which the fraction ratio of ferrite and austenite is 50:50 (780°C for the S1 steel and 810°C for the S2 steel) and then isothermally treated at 450°C, as a function of the isothermal

Fig. 4. Tensile properties and retained austenite volume fractions of the S1 steel intercritically annealed at 810°C and 780°C for 5 min and isothermally treated at 450°C and 470°C.

treatment time. The S2 steel having higher silicon content has much higher tensile strength and slightly higher elongation than the S1 steel, whereas its yield strength is similar or a bit lower, thereby showing more excellent strength–ductility balance than the S1 steel. This is because the S2 steel has the higher fraction of retained austenite than the S1 steel. Particularly because of the relatively smaller variation in the retained austenite fraction of the S2 steel as to the isothermal treatment time, mechanical properties also vary to a lesser degree. Thus, the mechanical properties of low-carbon cold-rolled steel sheets can be improved considerably by increasing the silicon content. It is also more beneficial to increase the silicon content for higher strength and ductility by TRIP effect.

4. Discussion

Microstructures and fractions of retained austenite of cold-rolled steels substantially vary with the intercritical annealing conditions due to the redistribution of solute ele-

Fig. 5. Tensile properties and retained austenite volume fractions of the S2 steel intercritically annealed at 830°C and 810°C for 5 min and isothermally treated at 450°C and 470°C.

ments, particularly carbon. At a higher intercritical annealing temperature, the austenite fraction increases, but a large amount of retained austenite cannot be obtained because the carbon content in austenite decreases. At too a low intercritical annealing temperature, the carbon content in austenite can increase, but the obtainable amount of retained austenite is also reduced because of the low austenite fraction. Many researchers have reported that the intercritical annealing temperature to obtain the higher fraction of retained austenite is $(A_{C1} + A_{C3})/2$.^{1,11,13,17)} In the cases of the S1 and S2 steels, when intercritically annealed at 810°C and 830°C, being $(A_{C1} + A_{C3})/2$, respectively, the austenite fractions were measured as about 60%. On the contrary, when intercritically annealed at 780°C and 810°C at which the austenite fraction was about 50%, both the S1 and S2 steels showed an increased fraction of retained austenite, together with improved properties. These results indicate that the intercritical annealing should be conducted at a temperature at which the stability of austenite can be raised by increased concentration of alloying elements in austenite by

Fig. 6. Tensile properties and retained austenite volume fractions of the S1 steel intercritically annealed at 780°C and isothermally treated at 450°C and the S2 steel intercritically annealed at 810°C and isothermally treated at 450°C.

lowering the fraction of austenite formed during the intercritical annealing. When the intercritical annealing temperature is set too low, the mechanical properties do not improve much because the fraction of retained austenite is reduced due to the reduction of the absolute amount of austenite and because retained austenite with higher stability than an appropriate stability level is formed.^{12,13,17)} According to the investigations into the effects of the intercritical annealing and the aforementioned results, the appropriate temperature is the one at which the fraction ratio of ferrite and austenite is 50:50. The authors confirmed similar results in cold-rolled 0.15%C–1.5%Mn–1.5%Si steels, which have slightly higher carbon content than the steels of the present study, but lower than the conventional TRIP-aided cold-rolled steels.¹³⁾

Mechanical properties of TRIP-aided cold-rolled steels also significantly vary with the isothermal treatment conditions. It was reported that the isothermal treatment at $M_s + (20\text{--}30^\circ\text{C})$ produced highest fraction of retained

austenite and excellent mechanical properties.^{17,18)} However, when the S1 and S2 steels were isothermally treated at 470°C ($M_s + 20^\circ\text{C}$), the fraction of retained austenite was low, and the mechanical properties were not good in comparison with the case of the isothermal treatment at 450°C (M_s). When isothermally treated at 450°C, the bainitic transformation can be more active than the case of 470°C in shorter time, and the formation of retained austenite can be accelerated.

In the isothermal treatment, the austenite formed during the intercritical annealing is transformed to bainite. Austenite stabilizers such as carbon and manganese are mostly soluble in the austenite during the intercritical annealing. Therefore, the chemical composition of the austenite formed during the intercritical annealing differs from the overall composition of the cold-rolled steel, and the transformation point of them, particularly M_s point, becomes lower than the M_s point of the steel. When the S1 or S2 steel is intercritically annealed at 780°C or 810°C, at which the fraction ratio of ferrite and austenite is 50:50, the manganese content in austenite becomes about 2% as that in ferrite is assumed to be 0.5% according to the Fe–Mn binary phase diagram.¹⁹⁾ Assuming that all the carbon is dissolved in austenite, disregarding the carbon solubility in ferrite, the carbon concentration in the intercritical austenite can be calculated as 0.2%. Thus, austenite having 0.2% carbon and 2.0% manganese is formed during the intercritical annealing. As the isothermal treatment time is increased, the carbon concentration in the remained austenite becomes higher, and the M_s temperature of the austenite gradually decreases, thereby forming metastable retained austenite at room temperature. In the TTT diagram of a 0.23%C–1.86%Mn steel,²⁰⁾ which has a composition similar to that of the austenite formed during the intercritical annealing in the S1 or S2 steel, the nose temperature of the bainite transformation curve is about 430°C, 30°C higher than the M_s point. Thus, the lower isothermal treatment temperature is more favorable for improved mechanical properties of the S1 and S2 steels. In this respect, it is possible to lower the isothermal treatment temperature below 450°C. Taking into consideration of the chemical composition of austenite formed during the intercritical annealing of the S1 and S2 steels and the isothermal transformation curve²⁰⁾ of a steel having a similar chemical composition, it can be expected that they have higher fraction of retained austenite and better mechanical properties when they are isothermally treated at 430°C than at 450°C.

Besides the establishment of the optimum heat-treatment conditions, also important is the selection of appropriate alloy composition. The S2 steel showed somewhat lower yield strength than the S1 steel, but much higher tensile strength and a similar elongation (Fig. 6). Since both steels were intercritically annealed under the same condition, *i.e.*, at the temperature at which the fraction ratio of ferrite and austenite is 50:50 and then isothermally treated at 450°C, the differences in the fractions of retained austenite and mechanical properties are attributed to the difference in the silicon content. According to Sawai *et al.*,²¹⁾ who studied the effects of alloying elements on the formation of retained austenite in C–Mn–Si TRIP-aided cold-rolled steels, silicon had stronger effect than manganese. Silicon contributes to

higher fraction of retained austenite and its ability to increase the carbon concentration in adjacent austenite since silicon prevents the carbide precipitation in bainite and increases carbon content as well during the isothermal treatment.^{9–11,21)} Thus, the S2 steel with higher silicon content can have higher retained austenite fraction and better mechanical properties than the S1 steel, when both were intercritically annealed and isothermally treated under the same conditions. It is confirmed that the addition of silicon works favorably for the formation of a larger amount of stabilized retained austenite, particularly in low-carbon TRIP-aided cold-rolled steels, since the decrease of the fraction of retained austenite due to the low carbon content can be compensated by the silicon addition, together with improvement in mechanical properties.

Significance of this study lies with the finding that cold-rolled steels having lower carbon content than conventional TRIP-aided steels can have high strength and ductility through appropriate alloy design and proper heat-treatment conditions.

5. Conclusions

Following conclusions were reached in this study after conducting intercritical annealing, isothermal treatment, microstructural observation, and evaluation of mechanical properties and retained austenite fraction on two low-carbon cold-rolled steel sheets specifically designed to improve the weldability.

- (1) 0.1% C low-carbon cold-rolled steels whose carbon content is lower than conventional high-strength TRIP-aided cold-rolled steels can also have high strength and ductility by establishing appropriate intercritical annealing and isothermal treatment conditions.
- (2) There exists an appropriate intercritical annealing temperature range according to alloying elements. When the S1 or S2 steel is intercritically annealed at the temperature range where the austenite fraction is about 50%, the highest retained austenite fraction and the best mechanical properties are obtained.
- (3) In order to insure high fraction of retained austenite and excellent mechanical properties in low-carbon TRIP-aided cold-rolled steels, the isothermal treatment temperature should be determined in consideration of the fraction and composition of austenite formed during the intercritical annealing.
- (4) Higher silicon content in low-carbon TRIP-aided cold-rolled steels favors higher fraction of retained austenite and leads to improved mechanical properties.

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