Effect of Ti-microalloy Addition on the Formability and Mechanical Properties of a Low Carbon (ST14) Steel

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Abstract

A low carbon Al-killed Ti-added steel was produced in Mobarakeh Steel Complex in Iran. Mechanical properties and microstructural characterizations of steel in the as-cast, hot-rolled, cold-rolled and annealed conditions are presented. Dilatometery tests were carried out to measure the finishing and annealing temperatures of steel. It was found that the addition of small amounts of Ti increased the transformation temperatures. In addition, microalloying with titanium improved the ductility of the produced slabs and hot rolled steel sheet. However, low temperature annealing of Ti-added cold-rolled steel sheets led to a decrease in ductility. The results indicate that the full softening of the Ti-added cold-rolled steel is completed for an annealing time of 2hr at 800°C.

Keywords: Ti-microalloyed steel, Formability, Mechanical properties, Aging.

1- Introduction

Recently, application of Ultra-Low Carbon (ULC) and Interstitial–Free (IF) steels has been widely increased in the structural parts of automotive vehicles due to their excellent formability, extra deep drawability and moderate yield strength ^{1,2)}.

It was found that reduction of carbon and nitrogen contents of ferrite led to a better formability in cold rolled and annealed sheets. In order to meet this demand, vacuum-degassing facilities and continuous annealing lines are used ^{2,3)}.

ULC and IF steels contain small amounts of carbon and nitrogen, typically less than 50ppm. The level of interstitial elements in ferrite can be further reduced by adding strong carbide and nitride forming elements such as Ti and/or Nb ⁴⁻⁶).

The mechanical properties and final microstructure of Ti-Nb microalloyed steels depend on the slab reheating temperature, reduction in hot finishing rolling temperature, rolling. accelerated cooling temperature, amount deformation during cold rolling, annealing temperature and slab composition. Good combination of formability and strength of Ti and Nb containing ULC steels as hot rolled condition can be obtained by accurate choice of thermomechanically controlled processes 7-10). This paper compares the microstructure, mechanical properties formability of a low carbon steel (ST14) with a Tiadded low carbon steel. Furthermore, the effects of

Ti addition as well as annealing condition were investigated on recrystallization behavior of low carbon steel.

2- Experimental Procedure

2-1- Steel making and casting

Ti-added steel was made in Mobarakeh Steel Complex. The composition of final products is shown in Table 1. To compare the effect of Ti as a microalloving element on mechanical properties of steel, some melts with composition given in Table 1 (St14) were selected. St14 steel is one of the common products in M.S.Co. During the melt of both steels, CaO was added as a flux. Aluminum was added in the ladle. In addition, a DH vacuum degassing procedure was carried out for a period of 68 min followed by addition of CaF₂, CaO, and Fe-Mn (and Fe Ti in Ti-added steel) while Ar gas was fed. After compositional adjustment, continuous casting of steels was carried out with an initial temperature of 1585°C. The resulting slabs had a dimension of 0.2m in thickness, 1.2m in width and 8.0m in length.

2-2- Evaluation of slabs

To investigate the mechanical properties of the slabs in the as-cast condition and after the homogenizing heat treatment, specimens of $0.05\times0.01\times0.2\text{m}^3$ were cut in the thickness direction of slabs. Homogenizing treatment was performed in an electric resistance furnace.

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Table 1. Chemical composition (wt%) of steels processed at M.S.Co.

Steel	С	Si	Mn	P	S	Al	Ti	N(ppm)
Ti-added	0.012	0.008	0.073	0.004	0.010	0.037	0.055	40
St14	0.02	0.004	0.222	0.007	0.004	0.058	O	30

Table 2. Average mechanical properties of investigated slabs.

Steel	Yield strength (N/mm ²)	Tensile strength (N/mm ²)	Elongation at fracture (%)
Ti-added St14	154 ± 2	290 ± 1	43 ± 2
	139 ± 3	291 ± 2	38 ± 1

Table 3. Critical temperatures determined by dilatometer.

Steel	Heating rate (°C/s)	A _{c1} (°C)	Cooling rate (°C/s)	A_{r3} (°C)
Ti-added St14	5.1	870±3	1.4	808 ± 1
	5.1	838±3	1.4	772 ± 2

The specimens were heated at a rate of 50°C/min and then held at 1250°C for 20min. Afterwards, they were cooled in air.

For controlled hot rolling of the slabs and subsequent cold rolling and annealing, the transformation temperatures, $A_{\rm r3}$ and $A_{\rm c1}$ were determined for both steels by dilatometric technique.

2-3- Hot rolling and coiling processes

Slabs were hot rolled in ten passes (roughing and finishing passes) from an initial thickness of 200mm to 2.6mm after reheating at 1250°C for 2hr. The finishing rolling temperatures were about 870°C and 900°C for St14 and Ti-added steels, respectively. After hot rolling, the strips were cooled and coiled at 570°C. After acid pickling, specimens were examined using tensile test procedure (DIN 50125). The specimens were cut in the transverse (T) direction of rolling.

2-4- Cold rolling and annealing

After acid pickling of hot rolled strips, five passes cold rolling were conducted using a reduction of 75%, which reduced the thickness of strips to 0.6mm. Annealing was done in box annealing furnaces in an atmosphere of HN_x gas. The coils were heated up to the temperature of 670°C with a rate of 50°C/hr and then cooled at a similar rate. To study the mechanical properties, tensile specimens were prepared in the directions of 0°, 45° and 90° to the rolling direction of the sheets. Since the strain hardening exponent (n) and plastic strain ration (r) are closely related to the formability of the sheet material, their values were measured during tensile testing. The values of n for the samples were calculated from the slope of "Stress-Strain" curves according to Hollomon equation ($\sigma = k\epsilon^n$). The r-value for each direction was determined using a three axial extensometer and a computer analysis during the

tensile test. The results of tensile tests were averaged using the following formula 11):

$$X = \frac{(X_0 + 2X_{45} + X_{90})}{4} \tag{1}$$

where X is any tensile property at the 0, 45 and 90° to the rolling direction. Standard metallography procedures were used for optical microscopy of the cold rolled and annealed samples.

2-5- Subsequent annealing treatment

In order to improve the mechanical properties of Ti-added cold rolled and annealed steel, a secondary annealing was performed on selected specimens in a laboratory furnace with a rate of 50°C/hr at 650, 700, 750 and 800°C for 30min. One sample was annealed at 800°C for 2hr. After annealing, the mechanical properties and microstructure of samples were investigated.

3- Results and Discussion 3-1- Mechanical properties of slabs and dilatometric measurements

The mechanical properties of slabs are shown in Table 2. An increase in strength and elongation was observed for the Ti-added slab as a result of formation of Ti(C, N) precipitation ^{12,13}.

Despite the higher strength values, the Ti-added steel exhibited higher elongation due to its lower content of C and Mn. As expected, the steel with the lower C and Mn content showed a higher ductility for all processing conditions ¹³⁻¹⁵⁾.

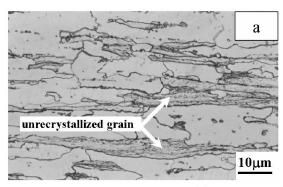
The results of dilatometric measurements are given in Table 3. According to these results, addition of Ti to the composition of slabs increases both A_{c1} and A_{r3} temperatures. It has been reported that microaddition of titanium and niobium has a remarkable retardation effect on recrystallization due to the suppression of grain boundary migration by formation of fine carbonitride particles.

Table 4. Average mechanical properties of investigated hot rolled steels.

Steel	Yield strength (N/mm ²)	Tensile strength (N/mm ²)	Elongation at fracture (%)
Ti-added St14	278 ± 3 342 ± 2	321 ± 1 448 ± 3	40 ± 2 30 ± 1

Table 5. Average mechanical properties of investigated cold rolled annealed steel sheets.

Steel	Yield strength	Tensile strength	Elongation at	Strain Hardening	Normal
Steel	(N/mm^2)	(N/mm^2)	Fracture (%)	Exponent (n)	Anisotropy (r _m)
Ti-added	310 ± 2	404 ± 2	27 ± 1	0.16 ± 0.01	1.1 ± 0.05
St14	179 ± 1	320 ± 1	39 ± 2	0.25 ± 0.01	1.7 ± 0.08



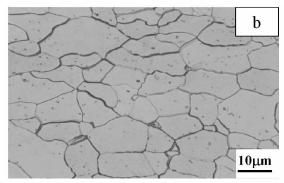


Fig. 1. Microstructure of the investigated steel sheets at cold-rolled and annealed condition, (a) Ti-added steel and (b) St14 steel.

3-2- Mechanical properties of hot rolled steels

The mechanical properties of hot rolled steels are given in Table 4. It can be seen that a higher elongation is obtained for the Ti- added hot rolled steel. By lowering concentrations of C and Mn, the ductility of steel increased, as mentioned in section 3.1

It is expected that the addition of Ti increases the strength, but hot rolled samples containing 0.055% Ti exhibited a lower yield and tensile strength compared to the St14 steel, which is in accordance with the work done by Kejian et al ¹⁷⁾. However, the loss of strength obtained in the present work is as severe as that mentioned previously.

3-3- Results of cold rolled batch annealed steels 3-3-1- Microstructure of the steels

The microstructures of the cold-rolled and subsequently annealed steels are presented in Fig. 1. In general, they have ferritic microstructure with different grain morphologies. The microstructure of Ti-added steel contains a mixture of recrystallized and unrecrystallized ferritic grains. However, fully recrystallized grains are observed in microstructure of St14 steel (Figure 1-b). It has been reported that the morphology of ferrite grains of cold-rolled and annealed steels is related to the chemical composition, hot rolling, cold-rolling and annealing conditions ¹⁸). As mentioned in section 3.1 and as

reported by Hayakawa et al ¹⁹, recrystallization temperature of steel increases with an increase in titanium content. Therefore, by addition of Ti, complete recrystallization occurs at higher temperatures.

3-3-2- Mechanical properties of steels

The average mechanical properties of steel sheets, cold-rolled and annealed, are given in Table 5. These results are in good agreement with microstructure observations. The fully recrystallized grains in St14 steel is the reason for higher ductility, n and r-values and lower strength than Ti-added steel. On the other hand, a lower r-value and a lower elongation at fracture were obtained for Ti-added steel annealed after cold rolling. This sample exhibited a duplex structure containing recrystallized and unrecrystallized ferritic grains. It has been demonstrated that materials with a strong {111} texture have high normal anisotropies or r_m values ²⁰⁾. Other texture components, such as the {001} have been found to be detrimental to the drawability. In practice, the intensity ratio of the above two $I_{\{111\}}/I_{\{001\}}$, was found to be components, approximately a linear function of $r_m^{\ 21)}$ Figure 2-a. On the other hand, according to Figure 2-b the {111} is the strongest component in the cold-rolled state, and usually remains so after annealing. However, its magnitude may either decrease or increase, and

commonly shows an initial decrease followed by an increase in the later stages of recrystallization. Figure 2–b also demonstrates that the {111} component continues to increase while the {100} component decreases during grain growth after recrystallization 20,22,23)

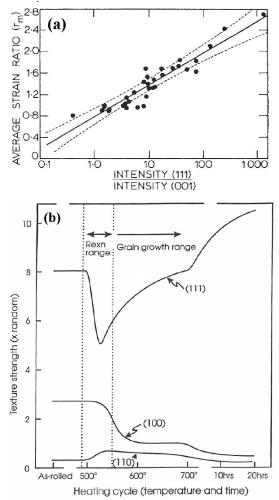


Fig. 2. a) Correlation between r_m and $I_{\{111\}}/I_{\{001\}}$ texture ratio in steels²¹, b) Variation of important texture components during box-annealing of cold rolled low carbon steel ²⁰.

3-4- Effects of subsequent annealing on microstructure and mechanical properties of Ti-added cold rolled and annealed steel sheet

The relation between annealing temperatures and microstructures of Ti-added cold-rolled steels was investigated in order to examine the effect of microalloying element addition on recrystallization behavior. Figure 3 illustrates the effect of annealing temperatures on microstructures of Ti-added cold-rolled steel. The microstructures indicate that some unrecrystallized grains remained unchanged after annealing at temperatures of 650, 700 and 750°C Figures. 3(a-c). However, the sample annealed at 800°C Figure 3d exhibits fully recrystallized ferritic grains.

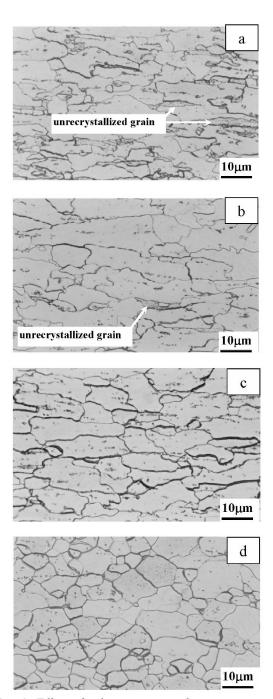


Fig. 3. Effect of subsequent annealing temperatures on microstructures of cold rolled Ti-added steel sheets, (a) 650°C, (b) 700°C, (c) 750°C and (d) 800°C (annealing time of 0.5hr).

The effect of laboratory annealing on microstructures of Ti-added cold-rolled samples shows that the industrial annealing temperature (670°C), which was employed for annealing treatment, was not enough to produce fully recrystallized ferritic grains. The effects of subsequent annealing temperatures on mechanical properties of Ti-added cold rolled steels are represented in Table 6. It is found that in annealing at low temperatures, the strength increased and the elongation decreased. However, by annealing at

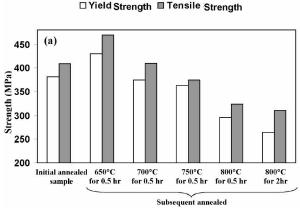
800°C, the yield and tensile strengths decreased while the ductility increased. The reason for these observations can be due to aging of samples and carbonitride precipitates formation during low temperature annealing. It is believed that in titaniumbearing steel, titanium nitride is formed during casting and titanium carbide is formed above 900°C. When the titanium content is above that in the titanium-to-nitrogen stoichiometric ratio (3.42), a significant amount of titanium can be dissolved into the austenite during reheating. Some of the dissolved titanium might precipitate in the ferrite phase and might introduce a hardening effect into steels ²⁴⁾. On the other hand, it should be noted that such carbideforming elements also accelerate the formation of ferrite phase. Thus, during rapid cooling of steel, the diffusion of these elements is prevented; as a result, they form ferrite solid solution. In this case, carbon forms iron carbide (Fe₃C) or is dissolved in ferrite solid

solution. During subsequent heating, carbon precipitates firstly as Fe₃C. Above 450°C, carbides of microalloying elements such as titanium, niobium and vanadium start to precipitate reducing the Fe₃C phase ^{16,25)}. Furthermore, uncompleted recrystallization due to the presence of Ti in ferrite also affects the formability of steels.

The effects of the annealing temperature (Table 6) and time on mechanical properties of Ti-added steel are compared in Figure 4. The yield and tensile strengths decrease with an increase in the annealing time and temperature Fig. 4-a, but elongation increases as shown in Figure 4-b. The microstructural studies showed that after annealing at 800°C for 2hr, the strength decreases, as a result of fully recrystallization and subsequent grains growth. Therefore, full softening of the Ti-added steel can be achieved for an annealing time of 2hr at 800°C.

Table 6. Mechanical properties of Ti-added cold-rolled steels after subsequent annealing at different temperatures for 0.5hr.

Annealing temperature (°C)		Yield strength (N/mm ²)	Tensile strength (N/mm ²)	Elongation at fracture (%)
Initial annealed sample		310 ± 2	404 ± 2	27 ± 2
Subsequent annealed samples	650 ± 5 700 ± 5 750 ± 5 800 ± 5	430 ± 2 374 ± 3 363 ± 1 295 ± 1	470 ± 2 410 ± 3 374 ± 2 324 ± 1	10 ± 1 15 ± 1 16 ± 1 38 ± 1



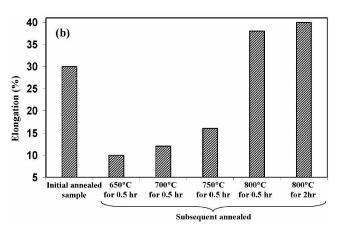


Fig. 4. Effect of subsequent annealing temperatures on (a) yield and tensile strengths and (b) elongation of Ti-added cold-rolled steel.

4- Conclusion

For the purpose of production of the Ti-added low carbon steel sheet with suitable formability, the effects of several important parameters on microstructure and mechanical properties of steel were examined in different conditions. It was found that:

1 - A small addition of Ti increases the strength and ductility of the slab.

- 2- The presence of Ti increases the A_{cl} , A_{r3} and recrystallization temperatures.
- 3- The results of mechanical tests showed that annealing at low temperature decreases the ductility of the Ti-added cold-rolled steel sheet.
- 4- The Ti-added cold-rolled steel sheet annealed at 800°C for 2hr exhibits the best ductility.

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6- References

- [1] R. Mendoza, J. Huante, M. Alanis, C. Gonzalez-Rivera and JA. Juarez-Islas: Mater. Sci. Eng. A, 276(2000), 203.
- [2] R. Yoda, I. Tsukatani, T. Inoue and T. Saito: ISIJ Int., 34(1994), 70.
- [3] R. Mendoza, J. Camacho, G. Lugo, C. Lopez, L. Herrera, J. Reyes, C. Gonzalez and JA. Juarez-Islas: ISIJ Int., 37(1997), 176.
- [4] S. Akamatsu, M. Hasebe, T. Senuma, Y. Matsumura and O. Akisue: ISIJ Int., 34(1994), 9.
- [5] S. Carabajar, J. Merlin, V. Massardier and S. Chabanet. Mater. Sci. Eng. A, 281(2000), 132.
- [6] S. Yim, P. Wray, KM. Tiitto, CI Garcia and AJ. DeArdo: ISIJ Int. 43(2003), 1615.
- [7] R. Lagneborg, T. Siweek, S. Zajac, B. Hutchinson: Scand. J. Metall., 28(1999), 250.
- [8] F. Leysen, J. Neutjens, K. Mols, S. Vandeputte and Y. Houbaert: Mater. Sci. Forum, Basque Country, Spain, 284-286(1998), 263.
- [9] D. Bai, J. Asante, M. Cooke and J. Dorricott: 43rd MWSP Conf. Proc., Vol XXXIX, ISS, Warrendale, PA; (2001), 543.
- [10] EJ. Palmiere, CI Garcia and AJ. DeArdo: Mater. Sci. Forum, Basque Country, Spain; 284-286(1998), 151.
- [11] M.A. Akoy, E.S. Kayali and H. Cimenoglu: ISIJ Int. 44(2004), 422.

- [12] B. Engle: Proc. of Inter. Sympo. Niobium, Florida, USA, (2000), 675.
- [13] V. Thillou, M. Hua, CI Garcia, C. Perdrix and AJ. DeArdo: Mater. Sci. Forum, Basque Country, Spain; 284-286(1998), 311.
- [14] I. Gupta and D. Battacharya: Pro. Int. Con. on Metallurgy of Vacuum-degassed Steel Products, TMS, Warrendale, PA, (1989), 43.
- [15] WB. Hutchinson, KI. Nilsson and J. Hirch Pro. Int. Con. on Metallurgy of Vacuum-degassed Steel Products, TMS, Warrendale, PA, (1990), 109.
- [16] I. Tamura, C. Quchi, T. Tanaka and H. Sekine: Thermomechanical Processing of High Strength Low Alloy Steels. Butterworth: Butterworth & Co. Ltd,.(1988), 19.
- [17] H. Kejian and T.N. Baker: Mater. Sci. Eng. A, 169(1993), 53.
- [18] DO. Wilshynsky, DK. Matlock and G. Krauss: Pro. Int. Con. on Metallurgy of Vacuum-degassed Steel Products, TMS, Warrendale, PA, (1989), 247.
- [19] H. Hayakawa, Y. Furuna, M. Shibata and N. Takahashi: Testu-to-Hagane; 69(1983), S594.
- [20] W. B. Hutchinson, Int. Metals Rev.; 29(1984), 25
- [21] R. K. Ray, J. J. Jonas, and R.E. Hook: Int. Mat. Rev.; 39(1994), 129.
- [22] K. Ushioda, N. Yoshinaga, K. Koyama and O. Akisue: Proc. Conf. on Physical Metallurgy of IF Steel ISIJ, Tokyo, (1994), 227.
- [23] KM. Titto, C. Jung, P. Wray, CI Garcia and AJ. DeArdo: ISIJ Int., 44(2004), 404.
- [24] S. Wang: Mater. Sci. Eng. A, 145(1991), 87.
- [25] M. R. Toroghinejad, A.O. Humphreys, D. Liu, F. Ashrafizadeh, A. Najafizadeh, J.J. Jonas: Metall. Mater. Trans.: 34(2003), 1163.