



Effect of Si Content on Phase Transition Temperature, Microstructure and Hardness in Medium Mn Steel

Ajit Panigrahi^{1*}, Gyana Ranjan Mishra^{2,3}, Ankit Kumar Sahoo^{1,4}, Madhusmita Behera¹, Pratima Kumari Mishra¹, Bhagyadhar Bhoi¹

¹*Advanced Materials Technology Department, CSIR-Institute of Minerals and Materials Technology (IMMT), Bhubaneswar 751013, India*

²*Research and Development Division, Tata Steel, Jamshedpur 83100, India*

³*Department of Mechanical Engineering, University of New Brunswick, Fredericton, NB E3b 5A3, Canada*

⁴*School of Minerals, Metallurgical and Materials Engineering, Indian Institute of Technology Bhubaneswar 752050, India*

*email: ajit@immt.res.in

Abstract – Medium Mn steels (C = 0.15–0.19 wt.%, Mn = 5.00–5.20 wt.%) with variation of Si content (1.9, 2.45, 3.4 wt.%) were prepared using vacuum induction melting furnace. The cast billets were forged and cooled to room temperature in air. Microstructural investigation of the forged alloys showed fully martensitic structure along with minor content of retained austenite. With Si content, the hardness of forged steel were found to be increasing. The transition temperatures (A_{c1} , A_{c3} , M_s , and M_f) were determined using differential scanning calorimetry (DSC) measurements. Increase of Si content raised A_{c1} and A_{c3} and caused reduction in M_s and M_f . The forged samples were austenized at 900 °C for 15 min, followed by isothermal holding at 700 °C for 1 and 4 h and water quenched. The microstructure consisted of ferrite, martensite, and retained austenite. Microhardness measurements of heat-treated steels also showed significant enhancement of mechanical strength with increase of Si content.

Keywords – Medium Mn steel; Forging; Transition temperature; Heat treatment; Hardness.

INTRODUCTION

Recently, advanced high-strength steels (AHSS) have shown greater demand in automotive applications because of combination of higher strength along with adequate ductility [1–3]. First generation (1st-G) AHSS [4,5] includes dual-phase (DP), martensitic (MART), transformation-induced plasticity (TRIP) steels, whereas twinning induced plasticity (TWIP) steel with Mn content of 15 to 25 wt.%, high Mn TRIP steels belong to the generation 2nd-G AHSS [1,6]. 1st-G AHSS exhibits relatively lower strength and ductility compared to 2nd-G AHSS. The remarkable combination

of strength (1.0 GPa) and ductility (> 50%) in 2nd-G AHSS is attributed to the formation of strain induced martensite or mechanical twinning during deformation. However, these steels suffer from high material cost and have lower productivity. Therefore, 3rd-G AHSS has been developed, involving lower material cost and adequate combination of mechanical properties. 3rd-G AHSS [7–9] includes medium Mn steel and quenching and partitioning processed steel.

The element Mn is an austenitic stabilizer. Therefore, to obtain a complete austenitic structure, 18–30 wt.% Mn is required. Medium Mn steel, whereas, contains Mn in the range of 3–10 wt.% and the microstructure consists of ferrite (α), martensite (α'), and retained austenite (γ_R). This steel was first developed by Miller [10], has been studied by many researchers because of the wider microstructural control. In order to get the microstructure of $\alpha + \alpha' + \gamma_R$, the steel should be soaked at intercritical region and annealed/quenched. The influence of Si in medium Mn TRIP and TWIP steels were reported [11,12]. Silicon is an active ferrite stabilizer and is established as an effective solid solution strengthening element. In the present work, we have investigated the influence of Si addition on the transition temperatures, evolution of microstructure and hardness. It is observed that increase of Si content causes increase of transition temperatures (A_{c1} and A_{c3}) and hardness of the alloy.

EXPERIMENTAL

A. Alloy Preparation



AISI 1021 steel, low Carbon (LC) grade ferromanganese, and 75 grade ferrosilicon were taken in required amount and melted in vacuum induction melting furnace (HHV, India) to get three medium manganese steels containing ~5.0 wt.% Mn. with varying Si content (S1: 1.90, S2: 2.45, and S3: 3.40 wt.%). The composition of these steels is enlisted in Table I. The as cast billet was homogenized at 1200 °C for 1 h, forged, and subsequently air cooled to room temperature. These samples were further subjected to austenization (at 900 °C for 15 min) followed by inter-critical annealing (between A_{c1} and A_{c3} : 700 °C for 1 h and 4 h) and water quenched. Schematic of the heat treatment is shown in Fig. 1.

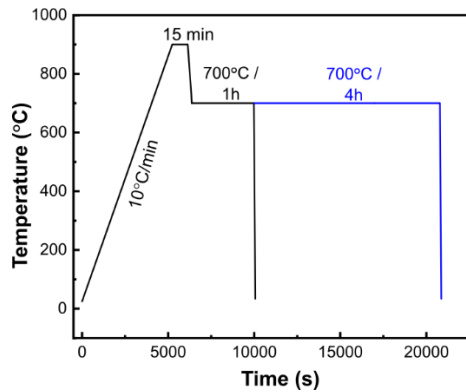


Fig. 1 Schematic diagram of heat treatment schedule followed for S1, S2, and S3 steels.

TABLE I
Composition of medium Mn steel.

Samples	C	Mn	Si	Al	Cr	P	S	Fe
S1	0.15	5.10	1.90	0.21	0.08	0.01	0.03	Bal.
S2	0.19	5.00	2.45	0.18	0.08	0.04	0.01	Bal.
S3	0.17	5.20	3.40	0.09	0.08	0.03	0.01	Bal.

B. Characterization

The microstructure was observed using field emission scanning electron microscope (FESEM, SUPRA, GEMINI55, Carl Zeiss, Germany) operated at accelerating voltage of 15 kV. The samples were mechanically polished and etched with 2% Nital solution. Structural analysis of forged samples was carried out using $Cu-K_{\alpha}$ radiation ($\lambda = 1.5406 \text{ \AA}$), performed in X-ray diffractometer (PAN analytical X'pert PRO, UK operated at 40 kV and 30 mA).

Differential scanning calorimetry (DSC, Setaram Setsys Evolution) experiments were done in the temperature range of 30–1000 °C with a heating rate of 5 °C/min and a cooling rate of 30 °C/min to determine the transition temperatures (A_{c1} and A_{c3}). The bulk hardness of forged samples was evaluated using Vickers hardness tester with a force of 3 kgf for a dwell time of 10 s. Heat treated samples were subjected to Vickers micro-hardness test (MMT-X, Matsuzawa, Japan) with a force of 0.2 kgf for 10 s.

RESULTS AND DISCUSSION

A. Microstructure and structural investigations

The FESEM images of S1, S2, and S3 samples are shown in Fig. 2 (a-c). The microstructure of these forged samples revealed lath martensitic structure along with retained austenite. XRD patterns of these samples show peaks characteristic to α' -martensite (see Fig. 2(d)). The peaks of austenite (γ) are not observed. This may be due to the lower content of austenitic phase below the detection limit of X-ray instrument.

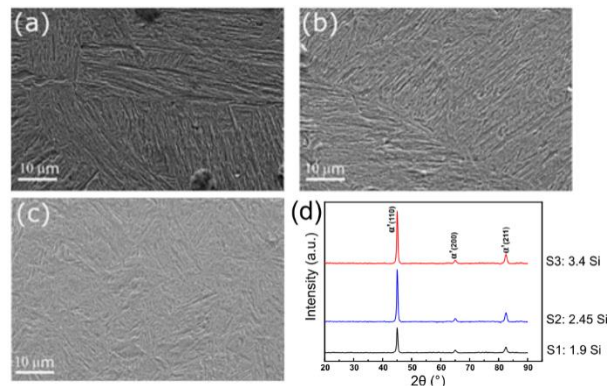


Fig. 2 (a-c) FESEM images, (d) XRD patterns of forged S1, S2, and S3, respectively.

B. Determination of transition temperatures by DSC

The heating and cooling curves of S1, S2, and S3 steels are shown in Fig. 3. With increase in the Si content, both A_{c1} and A_{c3} temperatures are found to be increasing. For S1 steel which contains 1.90 wt. % Si, A_{c1} and A_{c3} temperatures are 621 and 720°C, respectively. With increase of Si content to 2.45 wt.% (S2 steel), A_{c1} increases to 660°C and A_{c3} lies at 730°C. In the case of S3 steel, A_{c1} and A_{c3} temperatures further



increase to 687 and 760°C, respectively. Similar to Al, Si is expected to work as a ferrite stabilizer, increasing the austenite formation temperature and therefore, tends to enlarge the inter-critical region [13]. DICTRA package was utilized to calculate A_{c1} and A_{c3} temperature. For S1, A_{c1} and A_{c3} are 578 and 793 °C, respectively. Increase of Si content results in higher A_{c1} and A_{c3} . S2 steel containing 2.45 Si shows $A_{c1} = 592$ °C and $A_{c3} = 807$ °C. Furthermore, S3 steel exhibits higher transition temperatures ($A_{c1} = 632$ °C and $A_{c3} = 842$ °C). From cooling curves, martensitic transformation temperatures (M_s and M_f , where s and f stands for start and finish, respectively) were determined. It is observed that M_s temperature shows a decreasing trend with increase of Si content. The M_s temperature decreases from 357 to 348 and further to 302 °C with increase of Si from 1.90 to 2.45 and finally to 3.40 wt.%, respectively. The M_f temperature shows almost similar values (varies from 195 to 180 °C), irrespective of Si content. The M_s temperature was estimated using the following equation [14] and found to be 312, 293, and 286 °C for S1, S2, and S3 samples, respectively.

$$M_s = 539 - 423C - 30.4Mn - 7.5Si + 30Al \quad (1)$$

The observed M_s temperature is about 16-40 °C higher than the calculated values.

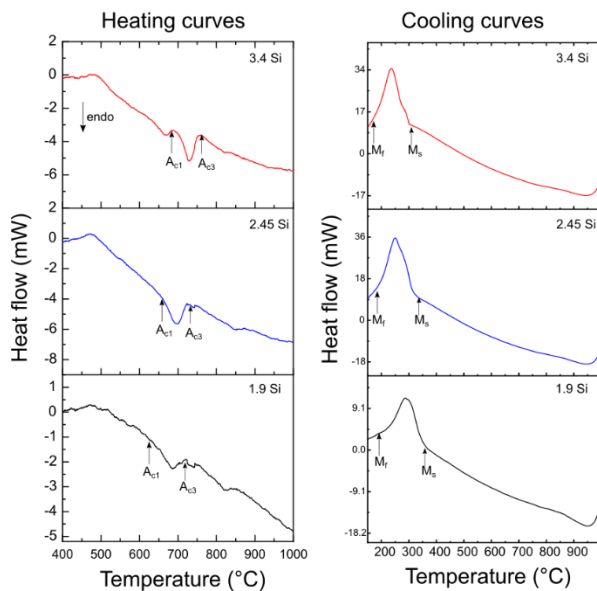


Fig. 3 Heating and cooling curves of S1, S2, and S3 steels showing transition temperatures (A_{c1} , A_{c3} , M_s and M_f).

It is noticed that A_{c3} temperature obtained by DSC experiments is lower than that of DICTRA calculation. To ascertain A_{c3} temperatures, thermal dilation measurements with respect to temperature were carried out in a push-rod type Horizontal Dilatometer (Linesis, Germany), maintaining the heating and cooling rate of 10 °C/min under argon atmosphere. A_{c3} temperatures for S1, S2, and S3 steels were found to be 741, 790, and 820 °C, respectively.

C. Hardness measurements

The bulk hardness and microhardness on forged S1, S2, and S3 steels are presented in Fig. 4 (a). The bulk hardness values are observed to be higher than those of microhardness. This is due to the fact that bulk hardness measurement is contributed by a larger amount of grains than that of microhardness measurement. Also, it is noted that with increase of Si content, the hardness increases. S3 steel shows the highest value of bulk hardness $HV_3 = 559 \pm 18$ VHN and microhardness $HV_{0.2} = 488 \pm 17$ VHN.

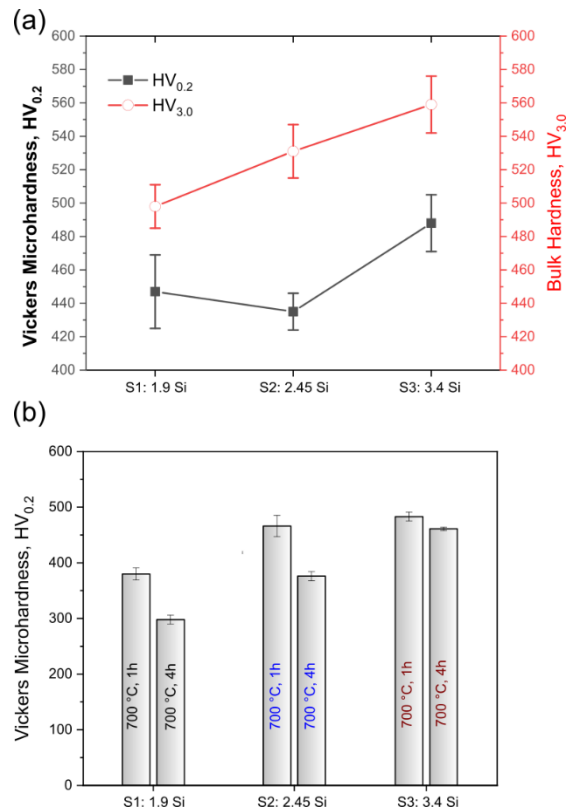




Fig. 4 (a) Bulk hardness and microhardness of forged S1, S2, and S3 steels using Vickers indenter, (b) Vickers microhardness results of S1, S2, and S3 steels after heat treatment, mentioned in Fig. 1.

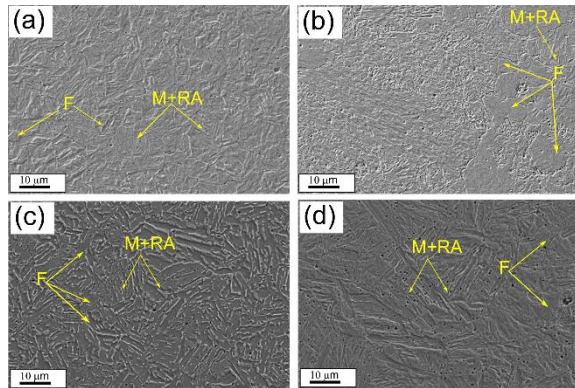


Fig. 5 Microstructure of steels after heat treatment (a) S1 and (b) S3 at 700 °C for 1 h, (c) S1 and (d) S3 at 700 °C for 4 h.

The microstructures of S1 and S3 steel samples after austenization at 900 °C for 15 min, followed by soaking at intercritical region of 700 °C for 1 and 4 h and water quenched to room temperature are shown in Fig. 5. The micrographs show ferrite (α) matrix including the martensite (α') and retained austenite (γ_R). The occurrence of similar microstructure has been reported in medium Mn steel [15]. Quenching from $\alpha + \gamma$ -region resulted in formation of hard α' from γ along with α . It is reported that Mn level of 3-13% is sufficient to obtain 15-45 wt.% retained austenite [8,16]. The selected temperature of 700 °C lies close to the A_{c1} temperature in the order S3→S2→S1. Therefore, during isothermal holding at intercritical region, it is expected that the formation of ferrite phase is more in S3 compared to S2 and S1. The same is evident from the microstructure (see Fig. 5). In other words, the formation of γ_R would be lower in S3 as compared to S1 and S2, which in turn, transforms to lesser amount of martensite upon quenching. Applying the rule of composite mixture, one could deduce that the hardness would increase in the order S1→S2→S3. However, a reverse trend is observed. In case of isothermal holding at 700 °C for 1 h, the hardness values are found to be 380 ± 11 , 466 ± 19 , and 483 ± 8 VHN for S1, S2, and S3, respectively. Recently, Sun et al. reported that strain partitioning between austenite

and ferrite is playing a major role in controlling the mechanical properties than the austenitic phase fraction and its stability [8]. It was reported that Si is an active solid solution strengthening element. In dual phase steel, Si enhances significantly the strength of material. Incorporation of Si increases C-activity coefficient in ferrite and austenite and decreases the solubility of C in ferrite [17–19]. This explains the reduction of hardness values in S1 compared to S3. With increase of holding time to 4 h, there is a considerable drop in hardness is observed. The hardness values are 298 ± 8 , 376 ± 9 , and 461 ± 3 VHN for S1, S2, and S3, respectively. This decrease can be due to the growth of ferrite and austenite grains during prolonged intercritical heating.

CONCLUSIONS

In the present investigation, medium Mn steels containing C = 0.15–0.19 wt.% and Mn = 5.00–5.20 wt.% with variation of Si content (1.90, 2.45, 3.45 wt.% Si) were prepared using vacuum induction melting and forging. These steels were subjected to intercritical heating at 700 °C for 1 and 4 h after austenization and water quenching. The transition temperatures (A_{c1} , A_{c3} , M_s , and M_f) were measured. Microstructures of the forged and heat treated samples were observed under FESEM and the hardness measurements were carried out. Based on the results obtained, following conclusions are drawn:

- (1) The microstructure of the forged steels consists of lath martensite along with retained austenite.
- (2) With increase of Si content, both A_{c1} and A_{c3} transition temperatures increase and M_s and M_f temperatures decrease.
- (3) The microstructure of heat treated steels consists of ferrite, martensite, and retained austenite.
- (4) Incorporation of Si improves the strength of forged steels. Also, heat treated forged steels show similar trend. Isothermal holding at intercritical temperature for longer duration decreases the strength.

ACKNOWLEDGMENT

The authors would like to acknowledge the Tata Steel Ltd. for funding this work through SSP project No. SSP-349. We would like to thank Central Characterization Department (CCD), CSIR-IMMT Bhubaneswar for carrying out FESEM and XRD.



REFERENCES

- [1] Y.K. Lee, J. Han, "Current opinion in medium manganese steel," *Mater. Sci. Technol.*, vol. 31, pp. 843–856, 2015.
- [2] J. Yang, Y.N. Wang, X.M. Ruan, R.Z. Wang, K. Zhu, Z.J. Fan, Y.C. Wang, C. Bin Li, X.F. Jiang, "Effects of manganese content on solidification structures, thermal properties, and phase transformation characteristics in Fe-Mn-Al-C Steels," *Metall. Mater. Trans. B.*, vol. 46, pp. 1365–1375, 2015.
- [3] N. Nakada, K. Mizutani, T. Tsuchiyama, S. Takaki, "Difference in transformation behavior between ferrite and austenite formations in medium manganese steel," *Acta Mater.*, vol. 65, pp. 251–258, 2014.
- [4] S. Zaefferer, J. Ohlert, W. Bleck, "A study of microstructure, transformation mechanisms and correlation between microstructure and mechanical properties of a low alloyed TRIP steel," *Acta Mater.*, vol. 52, pp. 2765–2778, 2004.
- [5] W. Jeong, "Effect of carbon on the plastic strain ratio of low carbon dual-phase steels," *Met. Mater. Int.*, vol. 20, pp. 49–53, 2014.
- [6] D.W. Suh, S.J. Kim, "Medium Mn transformation-induced plasticity steels: Recent progress and challenges," *Scr. Mater.*, vol. 126, pp. 63–67, 2017.
- [7] E.J. Seo, L. Cho, Y. Estrin, B.C. De Cooman, "Microstructure-mechanical properties relationships for quenching and partitioning (Q&P) processed steel," *Acta Mater.*, vol. 113, pp. 124–139, 2016.
- [8] B. Sun, F. Fazeli, C. Scott, N. Brodusch, R. Gauvin, S. Yue, "The influence of silicon additions on the deformation behavior of austenite-ferrite duplex medium manganese steels," *Acta Mater.*, vol. 148, pp. 249–262, 2018.
- [9] L. Liu, B. He, M. Huang, "The role of transformation-induced plasticity in the development of advanced high strength steels," *Adv. Eng. Mater.*, vol. 20, pp. 1701083, 2018.
- [10] R.L. Miller, "Ultrafine-grained microstructures and mechanical properties of alloy steels," *Metall. Transactions.*, vol. 3, pp. 905–912, 1972.
- [11] T. Furukawa, "Dependence of strength–ductility characteristics on thermal history in lowcarbon, 5 wt-%Mn steels," *Mater. Sci. Technol.*, vol. 5, pp. 465–470, 1989).
- [12] O. Grässel, L. Krüger, G. Frommeyer, L.W. Meyer, "High strength Fe-Mn-(Al, Si) TRIP/TWIP steels development - properties - application," *Int. J. Plast.*, vol. 16, pp. 1391–1409, 2000.
- [13] B. Sun, F. Fazeli, C. Scott, S. Yue, "Phase transformation behavior of medium manganese steels with 3 Wt pct aluminum and 3 Wt pct silicon during intercritical annealing," *Metall. Mater. Trans. A.*, vol. 47, pp. 4869–4882, 2016.
- [14] J. Mahieu, J. Maki, B.C. De Cooman, S. Claessens, "Phase transformation and mechanical properties of Si-free CMnAl transformation-induced plasticity-aided steel," *Metall. Mater. Trans. A.*, vol. 33, pp. 2573–2580, 2002.
- [15] H. Aydin, E. Essadiqi, I.H. Jung, S. Yue, "Development of 3rd generation AHSS with medium Mn content alloying compositions," *Mater. Sci. Eng. A.*, vol. 564, pp. 501–508, 2013.
- [16] G. Mishra, A.K. Chandan, S. Kundu, "Hot rolled and cold rolled medium manganese steel: Mechanical properties and microstructure," *Mater. Sci. Eng. A.*, vol. 701, pp. 319–327, 2017.
- [17] B.C. De Cooman, "Structure-properties relationship in TRIP steels containing carbide-free bainite," *Curr. Opin. Solid State Mater. Sci.*, vol. 8, pp. 285–303, 2004.
- [18] P. Jacques, Q. Furnémont, A. Mertens, F. Delannay, "On the sources of work hardening in multiphase steels assisted by transformation-induced plasticity," *Philos. Mag. A.*, vol. 81, pp. 1789–1812, 2001.
- [19] K. Jeong, J.E. Jin, Y.S. Jung, S. Kang, Y.K. Lee, "The effects of Si on the mechanical twinning and strain hardening of Fe-18Mn-0.6C twinning-induced plasticity steel," *Acta Mater.*, vol. 61, pp. 3399–3410, 2013.