Casting properties of ASTM A128 Gr. E1 steel modified with Mn-alloying and titanium ladle treatment

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Abstract: This work aims to produce a high manganese steel with more refined austenite grains and better wear resistance without sacrificing the toughness and tensile properties by Mn alloying and Ti ladle treatment in comparison to ASTM A128 Gr. E1 steel (1.0C-13Mn) that is mostly used in the mining industry. The 1.0C-17Mn-xTi alloys (x=0, 0.05 and 0.1, in wt.%) were prepared. A relationship was established between the microstructures and mechanical properties of the as-cast and solution annealed alloys. Increasing Ti content increases the stable Ti(CN) phase on and beside the grain boundaries and decreases up to 37% the austenite grain size of the as-cast alloy with 0.10wt.% Ti. Correspondingly, after solution annealed, optimized titanium content (0.05wt.%) results in significant improvements in wear resistance, hardness, elongation, yield and tensile strengths by 44%, 31%, 30%, 8% and 12%, respectively, except 9% decrease in impact toughness compared to ASTM A 128 Gr. E1 steel without modification. These results show that 1.0C-17Mn-0.05Ti alloy can be used for parts exposed to high load wear and applied in conditions where relatively high tensile properties with sufficient ductility is needed.

Key words: high manganese steel; hadfield steel; ladle treatment; grain refinement; microstructure; mechanical properties

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1 Introduction

High manganese austenitic steels which are also called Hadfield steels are most commonly used as wear materials in the mining industry due to their excellent wear resistance [1]. Improvement of wear resistance of these steels without decreasing other mechanical properties is very important. The basic chemical composition of Hadfield steel includes 1.0wt.%–1.4wt.% carbon and 10wt.%–14wt.% manganese in a 1:10 ratio [2]. These steels are classified within different groups in ASTM A128 Standard (Specification for Steel Castings, Austenitic Manganese) and mostly Mo-containing grade (ASTM A128 Gr. E1) is used for the heavy section casting parts due to its high resistance to carbide

brittleness during reheating of the casting parts. In this way, the possibility of cracking during cutting and welding repair is decreased and consequently providing easier flame cutting of gates and feeders in foundry conditions [3]. These steels must be suitably heat treated to achieve high toughness and ductility. This heat treatment process must consist of uniformly heating the castings to a temperature applicable for the grade of steel produced, at least 1,000 °C, holding until the temperature is uniform throughout, and quenching in water.

Titanium, niobium, vanadium and aluminium are among the common alloying additions for grain refining in the steels [4–8]. Among them, titanium nitride (TiN), characterized as the most effective grain growth inhibiting microalloy, precipitates at temperatures above 1,323 K (1,050 °C) [9]. This assertion is accepted as a consequence of the higher volume fraction provided by the lower solubility of TiN in austenite compared to other carbides and nitrides. High-temperature precipitation of TiN, however, can also be harmful to its grain refining potential due to precipitate growth

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and coarsening at elevated temperatures^[10].

Zhuang et al.^[4] studied the effect of various Ti content on the microstructure and mechanical properties of chrome-containing high manganese steel in as-cast condition. They reported that the Ti alloying which refined the as-cast grain structure also resulted in the formation of inclusion and a lot of dispersed particles that enhanced hardness and wear resistance. However, as mentioned in our previous study, high manganese austenitic steels are too brittle in the as-cast condition due to their secondary precipitations, such as carbide and phosphide eutectic along the grain boundaries, which limit their application in the mining industry which requires high toughness and ductility with the combination of high wear resistance^[11]. In addition, the titanium content was limited to 0.017wt.%.

Najafabadi et al.^[12] also investigated the effect of titanium addition (up to 0.6wt.%) on the wear resistance of Hadfield steel and they found that 0.6wt.% Ti addition significantly increased the wear resistance. However, they didn't study the effect of various loads during wear testing. It is very significant to investigate work hardening and wear rate according to load in order to integrate the experimental behavior of Hadfield steel to industrial use. In addition, there is no extensive research on thick section cast parts in the literature. Fully

austenite microstructure without undissolved or precipitated carbides in thick-walled casting parts cannot be produced easily^[13]. Therefore, the current work aims to produce a high manganese steel (wall thickness $t > 50$ mm) with more refined grains and better wear resistance in comparison to reference steel ASTM A128 Grade E1 without sacrificing the toughness and tensile properties by Mn alloying and Ti ladle treatment.

2 Materials and methods

The alloys were obtained with an industrial-scale induction furnace that had a maximum capacity of 3,000 kg in a steel foundry located in Istanbul, Turkey. The corundum-based neutral lining was used in a melting furnace to prevent the attacking of manganese oxides. The required molten steel was prepared with classified steel scraps, standard silicon, low-carbon ferro-manganese, high-carbon ferro-molibdenum and recycled manganese steel products. When the raw material was fully melted, the molten steel was transferred to the pouring ladle which was pre-heated at 900 °C. Before the tapping, the Fe-Ti particles covered with steel scraps were placed at the bottom of the ladle. The compositions of the alloys are given in Table 1.

Table 1: Chemical compositions of alloys (wt.%)

Alloy	C	Mn	Si	Mo	P	S	Ti	Fe
ASTM 128 Gr. E1	1.00	13.11	0.53	1.15	0.035	0.003	0.006	Bal.
1.0C-17Mn	1.00	17.11	0.53	1.16	0.037	0.002	0.008	Bal.
1.0C-17Mn-0.05Ti	1.01	17.18	0.58	1.17	0.036	0.004	0.046	Bal.
1.0C-17Mn-0.10Ti	1.01	17.09	0.55	1.16	0.037	0.004	0.094	Bal.

The chemical analysis was performed by the ARL optical emission spectrometer according to the ASTM E2209 standard from chilled samples taken from the inside of the transferred ladle. Finally, test coupons with dimensions of 60 mm × 60 mm × 300 mm, as shown in Fig. 1, were cast into a chromite sand mold at the pouring temperatures of 1,440 °C and 1,450 °C, respectively. The alloys were solution annealed in an oxygen-controlled custom-built automatic heat treatment furnace with a specific heat treatment process which included heating the castings up to 650 °C and holding for 3 h. This mid-process was performed to prevent internal cracking formation and decrease in the length and amount of the carbide plates in the as-cast structure^[14]. Then, the samples were heated to 1,100 °C at the rate of 1.67 °C·min⁻¹ and held for 3 h. Finally, they were quenched in an industrial quenching tank with 90 t of water at 27 °C. It is strongly recommended that the water temperature before quenching be around 25 °C–28 °C and the final water temperature after solution annealing should never be above 35 °C in order to prevent carbide precipitation through all thick-walled sections^[15]. During the quenching, compressed air was fed into the quenching tank for improving the circulation of the water. As a result, the final water temperature was kept steady at 27 °C

in this study.

Metallographic examination was performed according to ASTM E3 (metallographic sample preparation) and ASTM E407 (metal micro-etching) standards. The sectioned specimens for metallography which were taken from the middle of the casting samples as shown in Fig. 1(c) were ground, polished and etched with 4wt.% nital solution for 10 s. The microstructures of the as-cast and solution annealed specimens were characterized using an optical microscope (OM, Olympus PME Tokyo) and a scanning electron microscope (SEM, FEI Quanta FEG 450) equipped with energy dispersive spectrometry (EDS, EDAX Octane Plus). The average grain size of each alloy was measured using the intercept method according to the ASTM standard E112 (standard test methods for determining average grain size) by using Image-J software (version 1.51 k), which was also used to determine the carbide volume fraction of the alloys. The amount of the precipitated phases was calculated using Thermo-Calc simulation software.

The surface hardness of the samples was measured using a Brinell hardness tester at room temperature in compliance with ASTM E10 standard. Tensile testing was performed with an Instron test machine, Model 300 LX, employing an average

Fig. 1: Chromite sand mold before casting (a), test coupons after casting and shake out (b), and location of test specimens (c)

of three specimens for each sample with respect to ASTM E8/E8M. The impact testing was carried out on a specimen of 10 mm×10 mm×55 mm at room temperature, using a 300 J capacity test machine, with the Charpy V-notch test method according to ASTM E23. The ball-on-disc wear testing was carried out in accordance with ASTM G99 standard. The tests were performed at room temperature with a 10 mm diameter alumina ball as the counter body. The weight loss of each sample was measured under 2.5 N, 5 N, 10 N and 15 N loads at 0.2 m·s⁻¹ sliding speed with 500 m sliding distance.

3 Results and discussion

In order to see the phases and the amounts of precipitates which could appear during solidification of alloys between 400–1,800 °C, Thermo-Calc software (TCFE7 database) was used and the results are given in Fig. 2, and the precipitation temperatures of these phases in 1.0C-17Mn-0.05Ti alloy are given in Table 2. It can be seen that with the decrease in temperature, MnS, M₇C₃ and M₃C (cementite) phases are precipitated successively. After the formation of austenite, firstly, MnS starts to form at 1,266 °C, followed by precipitation of partial primary carbides among the austenite phase at 868 °C.

Immediately afterwards, cementite precipitates at 776 °C and perlite transformation (BCC_A2) occurs at 568 °C.

Compared with the 1.0C-17Mn alloy [Fig. 2(a)], with the addition of titanium, stable Ti(CN) phase is formed above the solidification temperature (1,495 °C) [Fig. 2(b)] because titanium has higher nitrogen affinity than the other elements. The amount of the Ti(CN) precipitate keeps increasing up to the temperature (1,255 °C) at which solidification is completed. With the further decrease of temperature, the amount of precipitation remains constant as the diffusion of the atoms in the Ti(CN) particles is blocked by the solidified austenite phase.

3.1 Microstructural characterization of as-cast alloys

Figure 3 illustrates the effect of manganese and titanium content on the as-cast microstructure of the alloys. It shows that all the as-cast alloys have an austenite matrix surrounded by alloyed cementite (FeMn)₃C in the lamellar form, and molybdenum carbides (MoC) distribute in elongated or spherical form in the center of lamellar cementite [11]. The volume fractions of the carbides developed in the as-cast alloys were measured by Image-J software and are listed in Table 3. There is no significant difference in the amount of carbide in the four

Fig. 2: Predicted phase precipitation diagram of alloys in a temperature range of 400–1,800 °C: (a) 1.0C-17Mn and (b) 1.0C-17Mn-0.05Ti

Table 2: Phase precipitation temperatures of 1.0C-17Mn-0.05Ti alloy in a temperature range of 400–1,800 °C

Ti(CN)	Liquidus	Solidus	MnS	M ₇ C ₃	Cementite	BCC-A2
1,495 °C	1,380 °C	1,255 °C	1,266 °C	868 °C	776 °C	568 °C

Fig. 3: Optical micrographs showing the as-cast microstructure of alloys: (a) ASTM A128 Gr. E1, (b) 1.0C-17Mn, (c) 1.0C-17Mn-0.05Ti, (d) 1.0C-17Mn-0.10Ti

Table 3: Volume fraction of carbides and austenite in as-cast alloys (%)

	ASTM A128 Gr. E1	1.0C-17Mn	1.0C-17Mn-0.05Ti	1.0C-17Mn-0.10Ti
Carbides	7.5 ± 1.7	6.1 ± 2.5	7.48 ± 3.3	9.06 ± 2.5
Austenite	Bal.	Bal.	Bal.	Bal.

as-cast alloys. The SEM microstructures shown in Fig. 4 reveal that Ti(CN) precipitation ranging 2–5 μm occurs on or beside the grain boundaries, indicating the predicted equilibrium composition in Fig. 2 is in good agreement with the experimental measurements at ambient temperature. The Ti(CN) precipitation in the alloy with 0.05% Ti addition is randomly dispersed, however increasing Ti content to 0.10% results in the segregation of Ti(CN) beside the grain boundaries. Additionally, EDS results given in Figs. 4(b) and (d) show that phosphorus is concentrated at austenite grain boundaries and forms (FeMn)₃P eutectic^[16]. The new Ti(CN) interfaces acted as the nuclei sites for phosphorus segregation^[17].

Carbon is an element that diffuses rapidly at high temperatures, so when the liquid metal contacts with the ferrotitanium (FeTi) additives, carbon tends to diffuse rapidly towards the low carbon areas of FeTi. Likewise, the added ferro-titaniums (FeTi) begin to dissolve in the molten metal, allowing the titaniums to diffuse freely. Free titanium and carbon atoms react on the surface of FeTi particles to

form primary TiC particles. Immediately afterwards, due to thermal convection, primary TiC particles diffuse through the molten steel where the temperature is high, allowing diffusion of nitrogen atoms. Meanwhile, the transformation of TiC particles into Ti(CN) particles takes place^[18]. The lattice type of titanium is hexagonal closed packed and can form stable compounds with nitrogen, oxygen and carbon^[4,19]. According to the heterogeneous nucleation theory proposed by Turnbull et al^[20], a phase with a high melting point can be used as a heterogeneous nucleation when the incompatibility between two phases is less than 12%. In addition, the degree of incompatibility of titanium nitride with γ-Fe is known to be 10.61%^[4]. For all these reasons, the high melting point compounds formed by Ti, C, and N act as heterogeneous nuclei during the solidification of the molten metal. As it can be seen in Table 4, compared to the 1.0C-17Mn sample, the addition of 0.05Ti and 0.10 Ti resulted in a reduction of 26% and 37%, respectively, in the grain size of the as-cast alloys.

Fig. 4: SEM micrograph (BSE mode) and EDS results of as-cast alloys: (a, b) 1.0C-17Mn-0.05Ti and (c, d) 1.0C-17Mn-0.10Ti

Table 4: Grain size of alloys in as-cast and solution annealed condition

Alloys	As-cast	Solution annealed	Grain coarsening (%)
ASTM A128 Gr. E1	172 ± 11 μm	215 ± 10 μm	~25
1.0C-17Mn	243 ± 20 μm	338 ± 26 μm	~39
1.0C-17Mn-0.05Ti	179 ± 10 μm	187 ± 5 μm	~4
1.0C-17Mn-0.10Ti	152 ± 8 μm	161 ± 5 μm	~5

3.2 Microstructural characterization of solution annealed alloys

Figures 5 and 6 show the microstructures of the solution annealed alloys. Ti-free alloys reveals a fully austenitic microstructure while Ti-containing alloys show an austenitic structure including Ti(CN) particles, especially on and beside the grain boundaries. This indicates that except stable Ti(CN) particles, all the secondary precipitations such as lamellar carbides and phosphide eutectics are successfully dissolved into the austenite matrix with solution annealing heat treatment and rapid quenching. Additionally, solution annealing resulted in the grain coarsening of the alloys as can be seen in Table 4. However, Ti-containing alloys show a very small degree of grain coarsening compared to Ti-free alloys, because the precipitates act as pinning particles and prevent grain growth.

3.3 Mechanical properties

Figure 7 shows the mechanical properties of as-cast and solution annealed alloys. All the as-cast alloys show poor ductility and ultimate tensile strength due to the carbide and Ti(CN) precipitations along the grain boundaries [Fig. 7(a, c)]. These secondary precipitations cause stress occurrence and crack initiation, which resulting in brittle fracture. The degree of brittleness in austenitic manganese steels depends on the degree of grain boundary coverage by the secondary phases. In this respect, intergranular carbides play a key role but other embrittling phases such as phosphide eutectic, Ti(CN) precipitates and microporosity also are involved^[21]. Therefore, the highest ductility and tensile properties are obtained with 1.0C-17Mn alloy in as-cast condition. Moreover, increased manganese and titanium increases the hardness of all as-cast alloys [Fig. 7(c)]. The highest hardness and yield strength

Fig. 5: Optical micrographs showing the solution annealed microstructure of alloys: (a) ASTM A128 Gr. E1; (b) 1.0C-17Mn; (c) 1.0C-17Mn-0.05Ti; (d) 1.0C-17Mn-0.10Ti

Fig. 6: SEM micrograph (BSE mode) and EDS results of alloys in solution annealed condition: (a, b) 1.0C-17Mn-0.05Ti, (c, d) 1.0C-17Mn-0.10Ti

are obtained with 1.0C-17Mn-0.10Ti alloy due to the smaller grain size in comparison to other as-cast alloys. An increase of 4% in the yield strength of as-cast alloy is obtained when the austenite grain size is reduced from $243 \pm 20 \mu\text{m}$ without Ti to $152 \pm 8 \mu\text{m}$ with 0.10wt.% Ti inoculation (Table 4), which is in

good agreement with the grain boundary strengthening theory described by the Hall-Petch effect^[22].

The hardness of solution annealed alloys show a similar trend with their as-cast states, but with higher hardness values and excellent plastic deformation above 50%. This can be

Fig. 7: Comparison of mechanical properties of as-cast (a, c) and solution annealed (b, d) alloys

well explained with solid solution strengthening caused by the carbide resolution at the grain boundaries. In addition, increased Mn content from 13wt.% to 17wt.% in solution annealed condition increases the stacking fault energy and results in increment of shear stress needed for sliding [23]. This leads to the increase of ultimate tensile strength, elongation and Charpy impact energy, in 1.0C-17Mn alloy by 5%, 20% and 21%, respectively, compared to ASTM A128 Gr. E1 steel. On the other hand, formation of well-dispersed Ti(CN) particles with 0.05Ti addition can refine the austenite grain size and increase the dislocation density and hereby strongly hinder the movement of dislocations and work hardening ability. The combination of grain boundary strengthening, solid solution strengthening and dispersion strengthening results in an increase in elongation, hardness, yield and tensile strengths, in as-cast 1.0C-17Mn-0.05Ti alloy by 30%, 31%, 8% and 12%, respectively, in comparison to ASTM A128 Gr. E1 steel. However, the Charpy impact energy linearly decreases with Ti addition. Smaller grains are usually considered to be a significant approach to attain better toughness in body-centered cubic (BCC) steels. Plastic deformation mechanism is only dislocation slip in BCC steels. However, twinning, which greatly depends on grain size, is the main secondary deformation mechanism in face-centered cubic (FCC) austenitic manganese steels besides dislocation slip [24]. Due to the twinning effect being more active in greater grain size [18,25], Charpy impact energy for the alloy with 0.05wt.% Ti decreases 9% in comparison to ASTM A128 Gr. E1 steel. However, Ti(CN) particles that formed with 0.10wt.% Ti [shown in Figs. 5(d) and 6(d)] and that aggregate along the grain boundaries, mostly have sharp edges and corners which cause high stress

concentration. Therefore, for the alloy with 0.10wt.% Ti, a sharp decrease in ultimate tensile strength, elongation and Charpy impact properties of 9%, 30% and 43%, respectively, in comparison to ASTM A128 Gr. E1 steel is observed. There is no significant difference in yield strength and hardness with increase of Ti from 0.05% to 0.10%.

One of the most significant results in this study is that 0.05% Ti addition strikingly decreases the wear rates of solution annealed alloys, especially for high stress wear conditions as shown in Fig. 8. One reason is that Ti(CN) precipitates provide heterogeneous nuclei during solidification [4,20]. This results in grain refinement in general, and also prohibits grain growth during solution annealing. Also, Ti(CN) particles provide nucleation sites for phosphorus eutectics as can be seen in Fig. 4, which decreases the phosphorus eutectics on

Fig. 8: Wear rate of solution annealed alloys as a function of various loads

grain boundaries and around carbides^[19]. The other reason is that Ti(CN) particles are hard, which increase the surface hardness. However, as can be seen in Figs. 6(c) and (d), when the titanium content increases from 0.05% to 0.10%, Ti(CN) particles segregate and cluster. Instead of homogeneously dispersed hard particles, clustered particles cause increase in wear rates.

4 Conclusions

Microstructural modification and, as a consequence, improvement of mechanical properties of high Mn steels, which have a very wide application fields, is very important. In this study, the effects of Mn and Ti addition on microstructure and mechanical properties of as-cast and solution annealed ASTM A128 Gr. E1 steel were investigated. Especially as a result of the addition of titanium at an optimized rate, it was determined that:

(1) The combination of fine-grain strengthening, solid solution strengthening and dispersion strengthening results in a significant increase in elongation, hardness, yield and tensile strengths of 30%, 31%, 8% and 12%, respectively, beside 9% decrease in impact toughness in annealed 1.0C-17Mn-0.05Ti alloy in comparison to ASTM A128 Gr. E1 steel without Mn and Ti modification.

(2) High friction load wear resistance of solution annealed 1.0C-17Mn-0.05Ti alloy increases up to 44% in comparison to ASTM A128 Gr. E1 steel.

When improving the wear resistance of high manganese austenitic steels, it is also very important to increase the tensile strength and elongation without causing a significant decrease in impact strength. In this study, the 1.0C-17Mn-0.05Ti alloy can be used for materials exposed to high load wear and applied in the conditions where relatively higher tensile properties with sufficient ductility are needed.

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