Microstructure Property Correlation of Low Alloy Steel Processed through Heat Treatment

Pankaj kumar¹, Md. M. Husain^{2*}, Sagram Hembrom^{1*}, B.N. Roy¹ ¹Department of Metallurgical Engineering, B.I.T, Sindri, Dhanbad – 828123, India ²MTE Division, CSIR-NML, Jamshedpur – 831007, India

¹pankajkumar28101998@gmail.com, ²murtuja@nmlindia.org, ¹shembrom.met@bitsindri.ac.in ¹bnroy.met@bitsindri.ac.in

Abstract:

In the current study, an effort has been made to establish a relationship between the low alloy steel's microstructure and properties. Austenitizing temperature of for this low steel was taken at 1150°C for 1-hour holding for annealing, normalizing, quenching in water and oil. Tempering was done (below A1) at 550° C for a two-hour holding period, and then quenched specimens were cooled by air. By using optical microscopy and SEM, on base specimen ferrite, carbides and early austenite grains (PAG). Microstructure of the annealing specimen was ferrite + pearlite. Microstructure of the normalizing specimen was proeutectoid ferrite and fine carbide with bainite. Ferrite + tempered martensite + carbide precipitates are observed in microstructure of quenched in water followed by tempering of low alloy steel. Ferrite + bainite + carbide precipitates are observed in microstructure of quenched in oil followed by tempering of low alloy steel. The mechanical properties of base alloy, annealing, normalizing, water and oil quenching followed by tempering low alloy specimens are was tested by Vickers macro hardness, Tensile test (UTS, Y.S, Elongation %) and Charpy impact test are 195.46 HV, 150.19 HV, 289.90 HV, 337.33 HV and 289.71 HV; (610.82 MPa, 444.99 MPa, 823.28 MPa, 954.89 MPa and 916.73 Mpa; 492 MPa, 241.41 MPa, 604.74 MPa, 837.04 MPa and 788.15 MPa; 22.07 %, 18.94 %, 12.17 %, 11.84 % and 12.63 %); 85.5 J, 20.67 J, 32.67 J, 25.33 J and 22.67 J respectively. Fractography of tensile specimens of base alloy and heat treated reveal ductile fracture.

Keywords: Low alloy steel; Heat treatment; Microstructure; Mechanical properties.

1. Introduction

Heat treatment, which entails timed heating and cooling, is applied to a particular metal or alloy in order to produce the appropriate microstructure and mechanical properties (hardness, toughness, yield strength, ultimate tensile strength, percentage elongation and percentage reduction). The four most important heat treatments, Q (water) & tempering, Q (oil) & followed by tempering, and annealing, are often used to change the microstructure and mechanical properties of engineering materials, particularly steels. Annealing is the type of heat treatment that is most frequently employed to soften iron or steel materials when elongations and a discernible level of tensile strength are required in engineering materials. Due to the microstructure of ferrite and pearlite, it refines its grains [1]. A material is heated to an austenitic temperature range and then cooled with air during the normalization process. The main aim of this treatment is to create a pearlite-dominated matrix, which improves the raw material's strength and hardness. It is also used to remove undesired free carbide that was present in the sample at the time

it was acquired. Additionally, it is utilized to get rid of unwanted free carbide that was in the sample when it was received [2]. In order to enhance a steel's mechanical qualities, particularly its strength and wear resistance, it is often quenched and tempered. When quenching, the steel or its alloy is heated to a temperature that promotes the formation of austenite, kept there until the required quantity of carbon has been dissolved, and then quenched in water or oil at the proper rate. Quenched steels are only used in a very limited number of technical applications due to their extreme brittleness and requirement for 100% martensite in the quenched stage of the steel in order to achieve its maximum yield strength. Tempering could be used to gradually improve ductility and impact strength while lowering hardness in quenched steel. The resulting microstructures are either a ferrite or a bainite precipitate in a matrix, depending on the tempering temperature. While steel is an iron alloy with a particular amount of carbon ranging from 0.15-1.5%, low alloy steels are those with 0.15-0.25% carbon [3].

Steel is used extensively due to two main reasons: (i) It is present in vast quantities as Fe_2O_3 in the earth's crust and requires little energy to convert to Fe. (ii) It can be made to exhibit a great variety of microstructures and mechanical properties. Its affordability, ductility, and suitable casting, working, and machining properties can be linked to its relevance. It is also amenable to simple heat treatments to provide a range of characteristics [2]. Increased operating parameters, i.e., operating at temperatures and pressures greater than 600°C and 27 MPa, respectively, [4–9] can be used in thermal power plants to increase efficiency and reduce carbon dioxide emissions [10–12]. They are used in advanced high-strength steel, railways lines, boiler tubes, and protective armor steel, among other things [13]. The heat treatment cycle needs to be optimized based on both the chemical content and thickness of the material, even though the guidelines only advocate basing it on chemical composition.

The present scope of work is built on top of this. Investigating the effects of various heat treatment methods on the microstructure and mechanical characteristics of low alloy steel is the main goal of the current study. In the current investigation, solutionizing at 1150°C and tempering at 550°C are two of the recommended heat treatment cycles. To determine the ideal mechanical qualities, a through investigation of the microstructure is conducted together with an evaluation of mechanical properties.

2. Experimental Procedure

Tuble 1. Chemical Composition of Low Anoy Steel									
Elements	С	Ni	Mn	Si	S	Р	Cr	Мо	Fe
Weight %	0.09	0.12	0.39	0.20	0.011	0.009	2.24	0.95	Bal.

 Table 1. Chemical Composition of Low Alloy Steel

Table 1 shows the chemical composition and mechanical characteristics respectively. Samples were prepared for heat treatment from a low steel plate. Heat treatment process were carried out by muffle furnace.

Condition	Annelaed	Normalized	Q (water) & T	Q (oil) & T			
Temperature, °C	1150	1150	1150 + 550	1150 + 550			
Holding time, hrs.	1	1	1 + 2	1 + 2			
Cooling medium	Furnace	Air	Water + Air	Oil + Air			

Table 2. Schematic Table Showing Different Heat Treatment of Low Alloy Steel

Table 2 depicts a schematic diagram of the heat treatment procedure. Heat treatment process, annealing, normalizing, quenching in water and oil followed by tempering was carried out. Austenitizing temperature of for this low steel was taken at 1150°C for 1 hrs. holding for annealing, normalizing, quenching in water and oil. Tempering was done on specimens that had been quenched in water and oil at 550°C for a two-hour holding period, then the specimens were air-cooled. Samples of tranverse section of heat treated specimens are prepared for metallography. The sample is hand polished using emery paper of grit size 80, 120, 220, 400, 600, 800, 1000, 1200, 1500, 2000 and 2500 to make polish surface. Alumina and colloidal solution was used for cloth polishing. Afterwards, etching was done on nital that contains 98 ml ethanol and 2 ml nitric acid [14].

The sample was prepared for microscopic analysis with the help of leveling hand press and slides for optical microscope. The microstructure of base alloy and other heat-treated low alloy steels was examined using optical microscopy (OM) and scanning electron microscopy (SEM-EDS).Vickers macro hardness (HV) of base alloy and heat treated alloy was determined with load 30 kg. Charpy impact v-notch specimens of base alloy and heat treated alloy were machined. Standard Charpy V-notch specimens were tested at room temperature in a pendulum impact test machine (ZWICK ROELL 300 J, Model: RKP 450). Tensile specimens in transverse direction of the plate were fabricated from base alloy and heat treated alloy. A servo-electric test apparatus (INSTRON, Model 8862) with a cross head speed of 0.5 mm/min was used to conduct the global tensile test. SEM-EDS was used to perform tensile fracture surface fractography.

3. Results and Discussion

3.1 Effect of Heat Treatment on Microstructure Properties

The evolution of the base alloy's microstructure and mechanical properties as well as the heat treatment of low alloy steels were studied in the current work. Microstructural characterizations of base alloy and heat treated alloy were carried out by using OM and SEM-EDS. For Mechanical properties, Tensile properties, Hardness and Charpy impact properties were evaluated. Fractography of tensile broken specimens were carried out by SEM.

3.1.1 Microstructure Evolution

Figures 1 (a-e) and 2 (a-e) show the optical and scanning electron microstructure of the base alloy and the heat-treated alloy, respectively. Grain size of base alloy ~ 30 μ m. Base alloy consists of ferrite with dispersed fine carbides at grain boundaries and inside the grain (Fig. 2a). M₂₃C₆ and M₇C carbides precipitates might be in low alloy steel (base alloy). Prior austenite grain (PAG) is also observed in base alloy (Fig. 1a). Annealed sample shows ferrite + pearlite microstructure, is shown in Fig. 1(b) and Fig. 2(b) [15].

However, pearlite is not regular (alternate layer ferrite and pearlite). Broken cementite are observed in pearlite colony in annealed sample. Some carbides are also observed in the microstructure. Microstructure of normalized sample is proeutectoid ferrite and bainite. Plate like carbides are observed within bainite. Ferrite + tempered martensite / bainite + fine carbide precipitates are observed in microstructure of quenched in water followed by tempering of low alloy steel. Ferrite + bainite / martensite + carbide precipitates are observed in microstructure of quenched in oil followed by tempering of low alloy steel. Carbides precipitates might be $M_{23}C_6$, M_7C , M_2C and MC formed in normalizing, quenching in water and oil followed by tempering. Phase transformation occurs due to diffusion and nucleation. The martensite change to ferrite by losing its carbon. Transformation of

cementite occurs only when the carbon combines with epsilon carbides and some amounts of carbides and tempered martensite precipites also occurred due to presence of alloying elements. Therefore quenched followed by tempered gives an excellent mechanical properties of steel [16].



Figure 1. Optical Microstructure of Specimens (a) Base alloy (b) Annealed (c) Normalized (d) Q (water) & T (e) Q (oil) & T



Figure 2. SEM Micrographs of Specimens (a) Base alloy (b) Annealed (c) Normalized (d) Q (water) & T (e) Q (oil) & T

3.2. Effect of Heat Treatment on Mechanical Properties

The effects of several heat treatments (annealing, normalizing, Q (water) and tempered, and Q (oil) tempered) on the mechanical properties of the heat treated and Base alloy samples (tensile strength, yield strength, hardness, toughness, percent elongation, and percentage reduction) are shown in Table 3. The base alloy sample had a tensile strength of 610.82 MPa, a yield strength of 492 MPa, hardness value of 195.46 HV, a toughness of 85.5 J, an elongation 22.07 %, and a reduction 32.33 %.

Mechanical properties								
Heat Treatment	Tensile Strength (MPa)	Yield Strength (MPa)	Hardness (HV)	Toughness (J)	Percentage Elongation (%)	Percentage Reduction (%)		
Base Alloy	610.82	492	195.46	85.5	22.07	32.33		
Annealed	444.99	241.41	150.19	20.67	18.94	59.96		
Normalized	823.28	604.74	289.90	32.67	12.17	57.91		
Q (water) & T	954.89	837.04	337.33	25.33	11.84	35.63		
Q (oil) & T	916.73	788.15	289.71	22.67	12.63	38.24		

 Table 3. Mechanical Properties of Heat Treated and Untreated Low alloy Steel

In comparison to the Base alloy sample, the annealed sample's mechanical properties showed reduced tensile strength (444.99 MPa), yield strength (241.41 MPa), hardness (150.19 HV), toughness (20.67 J), elongation (18.94 %), and an increase in reduction area (59.96 %) than the base alloy sample. It is possible to decreases in tensile strength, hardness, toughness, percentage elongation, and yield strength to the formation of ferrite + pearlite matrix, broken cementite, observed in pearlite colonies, and the presence of some carbides in the microstructure of the annealed sample by cooling.

The normalized sample has tensile strength values of 823.28 MPa, yield strength of 604.74 MPa, hardness of 289.90 HV, toughness of 32.67 J, percentage elongation of 12.17 %, and percentage reduction of 57.91 correspondingly. The optimal austenitizing temperature of 1150°C and a faster cooling rate resulted in an increase in tensile strength, toughness, and hardness relative to the untreated sample. The decrease in percentage elongation was also lower than that of the base alloy samples because the proeutectoid ferrite matrix structure produced during normalization of low alloy steel primarily contained fine carbide precipitates with little bainite.

According to its mechanical properties, the Q (water) and tempered sample exhibited the highest tensile strength (954.89 MPa), yield strength (837.04 MPa), and hardness (337.33 HV) values. The sample was heated for two hours at 550°C, let to cool to ambient temperature, and then austenized for one hour at 1150°C. The tensile strength and hardness are both increased by this treatment, but there was also significant toughness and percentage elongation, 25.33 J and 11.84 %, respectively.

Mechanical properties of the Q (oil) and tempered samples were 916.73 MPa, 788.15 MPa, 289.71 HV, 22.67 J, 12.63 % and 38.24 respectively, for tensile strength, yield strength, hardness, toughness

percentage of elongation and percentage of reduction. When the mechanical characteristics of Q (oil) and Q (water) samples were compared, it was found that a tempering temperature of 550°C resulted in a decrease in tensile strength, yield strength, toughness, and hardness while increasing percentage elongation and reduction, which is associated with the formation of bainite, martensite, ferrite, fine carbide, and prior austenite grain boundaries (PAGB). The test findings showed that no heat treatment, however, performed better in terms of elongation than the untreated sample. Figures 3 to 8 show that the tensile strength, hardness, toughness, percentage elongation and percentage reduction of treated and untreated low alloy steel samples vary.



Figure 3. Tensile Strength of Heat Treated and Base alloy Samples of Low Alloy Steel



Figure 4. Yield Strength of Heat Treated and Base alloy Samples of Low Alloy Steel



Figure 5. Hardness of Heat Treated and Base alloy Samples of Low Alloy Steel



Figure 6. Toughness of Heat Treated and Base alloy Samples of Low Alloy Steel



Figure 7. Percentage Elongation of Heat Treated and Base alloy Samples of Low Alloy Steel



Figure 8. Percentage Reduction of Heat Treated and Base alloy Samples of Low Alloy Steel

3.3 Fractography

To ascertain the mode of fracture, the tensile fracture surfaces of the samples were studied under the SEM. Fractographs of tensile broken specimens are shown in Fig. 9 (a-e). All tensile broken specimens reveal dimples, confirm ductile mode of fracture. Micro voids are also observed in all fractographs due to presence of precipitates.



Figure 9. SEM Fractogrphs of Tensile Broken Specimens of (a) Base alloy (b) Annealed (c) Normalized (d) Q (water) & T (e) Q (oil) & T

Base alloy shows more fines and almost homogenoius distribution of dimples than heat tread alloy. Base alloy displays the typical ductile characteristics of micro-dimples created by micro void nucleation from carbides. Annealed fractogrphy shows combination of coarse dimples and fine dimples as well as inhomogenoius distribution of dimples than others heat treated alloy. Coarse dimples are due to presence of higher grain size. The annealed sample has been annealed demonstrates the grain growth and coalescence of micro voids during fracture. Therefore, these specimens show more ductile than other heat treated alloy.

Normalized fractogrph shows fine dimples and almost homogenious distribution of dimples, like base alloy. Fine and coarse both dimples are observed. It shows significantly lower ductility than base alloy and annealed sample. The fracture surfaces are made up of a lot of irregularly sized, deep, and shaped dimples. Dimple size in quenched (water) & tempered shows slightly coarse than normalized sample. Microcracks are observed in quenched (water) & tempered sample due more presence more hard phase. It shows high tensile and yield strength and lower ductility as normalized sample. Microcracks are also observed in quenched (oil) & tempered sample and normalized sample. Similar un-uniform dimple size and inhomogenous distribution of dimples are observed in quenched (oil) & tempered sample and normalized sample. It also shows high tensile and yield strength and lower ductility. However, it shows lower tensile and yield strength than quenched (water) & tempered.

4. Conclusion

In the current study, low alloy steel's microstructure and mechanical properties were examined after undergoing various heat treatments such as annealing, normalizing, and quenching in water and oil, followed by tempering. Microstructure evolution of heat treated low alloy steel was carried out. Tensile properties, hardness and impact properties were also carried to establish the correlation of heat treatment of low alloy steel with its microstructure and mechanical properties. Based on results the following conclusions were made:

- Microstructure of base alloy was observed ferrite + carbides. The grain size of low alloy steel is ~30 μm. Annealed sample consists of ferrite + pearlite with few fine disperse carbide particles. Microstructure of normalized sample is proeutectoid ferrite and fine carbides with bainite. Quenched (water) & tempered consists of ferrite + tempered martensite + carbide precipitates. Quenched (oil) & tempered consists of ferrite + bainite + carbide precipitates. Carbides precipitates M₂₃C₆, M₇C, M₂C and MC might be formed during heat treatment.
- 2. Hardness of base alloy, annealed alloy, normalized alloy, quenching in water followed by tempering and quenching in oil followed by tempering are 195.46 HV, 150.19 HV, 289.90 HV, 337.33 HV and 289.71 HV respectively. Maximum hardness and minimum hardness are obtained 337.33 HV and 150.19 HV of quenched in water followed by tempering and annealed samples respectively due to phase transformation.
- 3. Tensile strength, yield strength, and percent total elongation of the base alloy and the heat treated alloy are, respectively, 610.82 MPa, 444.99 MPa, 823.28 MPa, 954.89 MPa, and 916.73 MPa; 492 MPa, 241.41 MPa, 604.74 MPa, 837.04 MPa, and 788.15 MPa; and 22.07 %, 18.94 %, 12.17 %, 11.84 %, and 12.63 %. Low alloy steel that has been quenched in water and then subjected to tempering has maximum tensile and yield strengths of 954.89 MPa and 837.04 MPa, respectively, with a 11.84 % elongation. The tensile strength and yield strength were significantly reduced as a result of the annealed sample elongation, which was discovered to be up to 18.94 %.

- 4. The charpy impact energy of base alloy, annealed alloy, normalized alloy, quenched in water followed by tempering and quenched in oil followed by tempering are 85.5 J, 20.67 J, 32.67, 25.33 J and 22.67 J respectively. Highest impact energy 32.67 J is obtained. However, charpy impact energy of all heat treated alloy was significantly reduced as compare to base alloy. Fractography of tensile specimens of base alloy and heat treated alloy reveal ductile fracture.
- 5. Quenched (water) & tempered gives maximum tensile strength and yield strength with adequate ductility. Despite having lower ductility than base alloy, quenched (water) & tempered alloy has much higher tensile and yield strengths than base alloy and other heat-treated alloy.

Reference

- D.A Fadare, T.G. Fadara and O.Y. Akanbi, "Effect of Heat Treatment on Mechanical Properties and Microstructure of NST 37-2 Steel," Journal of Minerals Characterization & Engineering, vol. 10, no. 3, (2011), pp. 299-308.
- [2] Bhaskar Chandra Kandpal, D.K. Gupta, Ashok Kumar, Ashish Kumar Jaisal, Atul Kumar Ranjan, Ankit Srivastava, Prashant Chaudhary, "Effect of Heat Treatment on Properties and Microstructure of Steels," Materials Today: Proceeding, vol.44, part 1, (2021), pp. 199-205.
- John, V.B., "Introduction to engineering materials," 2nd edition., Macmillan Publishing Company Ltd., (1980), pp. 321-324.
- [4] J. Cao, Y. Gong, K. Zhu, Z.G. Yang, X.M. Luo, and F.M. Luo and F.M. Gu, "Effect of Precipatates on Long Term Creep Deformation Properties of P92 and P122 Type Advanced Ferritic Steels for USC Power plant," Materials Science and Engineering: A, vol. 32, (2011), pp. 2763-2770.
- [5] P. J. Ennis and A. Czyrska Filemonowicz, "Recent Advances in Creep Resistant Steels for Power Plant Applications," Sadhana A, vol. 28, (2003), pp. 709-730.
- [6] Z. Zhang, G. Holloway and A. Marshall, "Properties of T/P92 Weld Metals for ULTRA Super Critical (USC) Power Plant," Int. J. Microstructure and Materials Properties, vol. 6, (2011), pp. 20-39.
- [7] M Abd El-Rahman Abd El-Salam, I El-Mahallawi and M R El-Koussy, "Influence of Heat Input and Post-Weld Heat Treatment on Boiler Steel P91 (9Cr - 1 Mo – V- Nb) Weld Joints Part 2 – Mechanical properties," International Heat Treatment Surface Engineering., vol. 7, (2013), pp. 32-37.
- [8] S A David, J.A Siefert and Z Feng, "Welding and Weldability of Candidate Ferric Alloys for Future Advanced ultrasupercritical Fossil Power Plants," Science Technology Weld. Joint, vol. 18, (2013), pp. 631-651.
- [9] P.J.Ennis, A. Zielinska-Lipiec and A. Czyrska Filemonowicz, "Influence of Heat Treatments on the Microstructural Parameters and Mechanical Properties of P92 Steel," Materials Science and Technology, vol. 16, (2000), pp. 1226-1233.
- [10] J. Hald, "Microstructure and Long- Term Creep Properties of 9 12 % Cr steels," International Journal of Pressure Vessels and Piping, vol. 85, (2018), pp. 30-37.
- [11] Wei Yan, Wei Wang, Yi-Yin Shan & Ke Yang, "Microstructural Stability of 9-12% Cr Ferritie/Martensite Heat Resistant Steels," Frontiers of Materials Science, vol. 7, (2013), pp. 1-27.
- M. Yoshizawa, M. Igarashi, K. Moriguchi, A. Iseda, H.G. Armaki and K. Maruyama, Effect of Precipitates on Long
 Term Creep Deformation Properties of P92 and P122 Type advanced Ferritic Steels for USC Power plants," Materials Science and Engineering: A, vol. 510 - 511, (2009), pp. 162-168.
- [13] A.J. Alawode, M.B. Adeyemi, "Effect of Degrees of Deformation and Stress Relief Temperatures on the Mechanical Properties and Residual Stresses of Cold Drawn Mild Steel Rods," Journal of Materials Processing Technology, vol.160, (2005), pp. 112–118.

- [14] Min Shan HTUN, Si Thu KYAW and Kay Thi LWIN, "Effect of Heat Treatment on Microstructures and Mechanical Properties of Spring Steel," Journal of Metals, Materials and Minerals, vol.18, (2008), pp.191-197.
- [15] Rahul George, Manoj Samson R, Keshav Ottoor, Geethapriyan T, "The Effects of Heat Treatments on the Microstructure and Mechanical Properties of EN 19 Steel Alloy, International Journal of Materials Science and Engineering, vol.6, no.2, (2018), pp. 56-66.
- [16] P.J Ennis and A Czyrska- filemonowicz, "Recent Advances in Creep-Resistance Steels for Power Plant Application, Sadhan A, vol.28, part 3 & 4, (2003) pp.709-730.