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# Optimized Corrosion Performance of AISI 1345 Steel in Hydrochloric Acid Through Thermo-Mechanical Cyclic Annealing Processes

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Abstract: The thermo-mechanical treatments and cyclic annealing processes have the potential of optimizing the corrosion performance of carbon steels in corrosive environments. Herein, an attempt has been made to optimize the corrosion performance of AISI 1345 steel in hydrochloric acid by thermo-mechanical cyclic annealing treatments. AISI 1345 steel was produced and cast in the laboratory and subjected to three types of thermo-mechanical cyclic annealing treatments (TMCA). The first TMCA treatment comprised hot rolling at 1050 °C followed by oil quenching and single austenitizing at 900 °C followed by furnace cooling (TMSA). The second and the third TMCA treatments involved similar hot rolling processes with double austenitizing and furnace cooling (TMDA) and triple austenitizing and furnace cooling (TMTA) processes. Microstructure analysis showed that dual-phase (retained austenite + pearlite) microstructure was achieved after all TMCA treatments with an exception of secondary phase particles precipitation after TMSA treatment. Maximum fractions of retained austenite and minimum fractions of pearlite were achieved after TMTA treatment. Highly refined microstructure of size 26.7 µm was achieved after TMDA treatment whereas; TMSA treatment offered coarse grained microstructure of size 254 µm. Electrochemical analysis was performed in 5 vol% HCl solution using Tafel scan technique. Results revealed that both TMDA and TMTA treatments caused three-fold reduction in corrosion rates (3.025, 2.771 mpy) compared to non-treated steel sample. After 168 h of immersion corrosion analysis in 5 vol% HCl solution, the surface of TMTA treated sample was observed to be partially covered with a very thin, crack-free oxide layer exhibiting minimum oxygen (8.16%) percentage. These features indicated that the TMTA treated sample underwent a very low-intensity minor corrosion attack of HCl solution and exhibited the best immersion corrosion performance among all samples. Electrochemical and immersion corrosion analysis results were in good agreement.

**Keywords:** thermo-mechanical treatment; cyclic annealing; microstructure; corrosion performance; hydrochloric acid



### 1. Introduction

Due to excellent mechanical properties, exceptionally low cost, and availability [1], carbon steels are widely used in industrial applications i.e., construction, automobile, power production [2], oil and gas refineries [3], chemical, petroleum, mining [4], and allied industries. Carbon steels are used in manufacturing storage tanks for storing acids, alkali, and salt solutions [5]. Acidic solutions particularly HCl, H<sub>2</sub>SO<sub>4</sub>, and HNO<sub>3</sub> are commonly employed in the cleaning process of steel boilers, pickling of industrial steel circulating cooling system, ores processing, ion exchangers recovery, oil wells acidizing, and acid descaling [6,7]. In all these applications, carbon steels remain in contact with acids for a long time.

The carbon steels are highly susceptible to natural corrosion in acidic environments and cause considerable economic losses, hazard problems at the workplace, and waste of manpower and materials [8]. Therefore, it is mandatory to find quick, easy, and economical methods of minimizing these adverse corrosion effects on carbon steels [9,10]. Several strategies i.e., inhibitors [1–8,10], materials selection, cathodic protection, coatings [2], and heat-treatment [11], have been reported to control these undesirable mechanisms of degradation.

Various heat-treatment processes i.e., solution treatments [12,13], annealing [14], normalizing, hardening [15], oil quenching [16] and tempering [17,18], water quenching [16], stabilization and sensitization [19], aging [14,20–22], intercritical heat-treatment [23], thermo-mechanical treatment (TMT) [24], and cyclic heat-treatment [25], have been employed to optimize the corrosion performance of carbon steels.

TMT treatment is a very well-known, efficient, and cost-effective way of optimizing the corrosion performance of carbon steels [24]. TMT treatment causes grain refinement and bulk recrystallization of nano-scale particles and nano-sized subgrain boundaries in the matrix [26,27]. TMT treatment involves heating of steel to a sufficiently high temperature about 1100 °C, soaking at this temperature for sufficient time, hot rolling to the required shape, and quenching to room temperature [28]. On the other hand, cyclic heat-treatments are also well-recognized processes involving the repetition of austenitization [29–32], quenching [33], tempering [34], and annealing [35–38] processes. Cyclic annealing is one of the best methods of achieving ultra-fine microstructure and considerably improved properties of steel. It involves austenitizing near upper critical (AC<sub>3</sub>) temperature, furnace cooling to room temperature [35]. TMT and cyclic annealing treatments have been applied on various carbon steels but AISI 1345 steel has not been investigated in this era. AISI 1345 is one of the widely used steels in manufacturing bolts and wear-rings for various applications [39].

In the present work, AISI 1345 steel was produced and cast in the laboratory and subjected to various thermo-mechanical cyclic annealing (TMCA) processes. Microstructure analysis and micro Vickers hardness test results were obtained to evaluate the impact of TMCA treatments on steel's microstructure. To evaluate the corrosion performance of TMCA treated steel, the electrochemical and immersion corrosion analysis were performed in 5 vol% HCl solutions. Corrosion products properties were also evaluated by SEM, EDS spot analysis, and elemental mapping techniques after immersion corrosion analysis.

## 2. Materials and Methods

## 2.1. Materials

The experimental AISI 1345 steel was produced in the laboratory using mild steel scrap, ferro-silicon, ferro-manganese, and carburizer in the pure as-received forms. The required quantities of raw materials were weighed to produce a melt of 52.1 kg, melted in an induction furnace, and cast in the sand molds into ingots of dimensions  $50 \times 50 \times 300$  mm<sup>3</sup>. The chemical compositions of raw materials and produced AISI 1345 steel were measured by combustion analysis and wet analysis.

The weights and the chemical compositions of raw materials and produced AISI 1345 steel are given in Table 1.

<b>Raw Materials</b>	Wt. (kg)	С	Si	Mn	S	Р	Fe
Mild Steel Scrap	50.0	0.10	0.30	0.25	0.002	0.002	Bal.
Carburizer	0.08	98.0	-	-	0.015	-	-
Ferro-Silicon	0.11	-	70.0	-	-	-	Bal.
Ferro-Manganese	1.92	6.00	0.97	60.0	-	-	Bal.
AISI 1345	52.1	0.45	0.37	1.78	0.017	0.002	Bal.

Table 1. Weights and chemical compositions (wt. %) of raw materials and produced AISI 1345 steel.

#### 2.2. Thermo-Mechanical Cyclic Annealing Processes

The TMCA processes are schematically presented in Figure 1. In TMCA processes, firstly steel was subjected to TMT treatment by heating to 1050 °C, soaking at this temperature for 120 min, and hot rolling into sheets of 5 mm thickness through multiple passes followed by oil quenching to room temperature. After hot rolling, steel plate samples were subjected to three types of cyclic annealing processes. The first cyclic annealing process was designated as a thermo-mechanical single annealing process (TMSA), second as double annealing (TMDA) process, and third as a triple annealing (TMTA) process. TMSA process comprised of austenitizing at 900 °C for 30 min followed by very slow furnace cooling to room temperature. The TMDA process was performed by repeating the annealing process twice. The first annealing process was identical to the TMSA process whereas, in second annealing experimental steel was austenitized at a relatively lower temperature of 830 °C for 30 min followed by repeating the annealing the annealing process whereas, in third annealing the annealing process was austenitized at a relatively lower temperature of 830 °C for 30 min followed by repeating the annealing the annealing process was carried out by repeating the annealing process whereas, in third annealing process steel was austenitized at a relatively low temperature of 760 °C for 30 min followed by slow furnace cooling to room temperature.



**Figure 1.** Schematic diagram of thermo-mechanical cyclic annealing processes; (**a**) thermo-mechanical single annealing (TMSA), (**b**) thermo-mechanical double annealing TMDA, (**c**) thermo-mechanical triple annealing (TMTA) applied to AISI 1345 steel.

#### 2.3. Microstructure Analysis

The TMCA treated AISI 1345 steel samples were wetly sectioned into dimensions of  $10 \times 10 \times 5 \text{ mm}^3$  using a wire cut machine for microstructure analysis. All the samples were metallographically prepared by manual grinding on Federation of European Producers of abrasives (FEPA) grades P100, P200, P400, P600, P800, and P1000 grinding papers followed by automatic polishing on diamond pastes (6 µm, 3 µm, 1 µm and 0.25 µm) coated nylon and velvet cloths using an automatic polisher (StruersTegrapol–15 Grinder/Polisher USA). After etching in 4 vol% nital solution (4 vol% nitric achid + 96 vol% ethanol), the microstructure was analyzed under an optical microscope (Leica Model DM-15000M Germany) at 1000× magnification and then by scanning electron microscope (FEI brand model Inspect S50 USA SEM) at 2000× magnification. Elemental compositions of both phases (Retained austenite, Pearlite) of dual-phase microstructure were analyzed using an energy dispersive spectrometer (EDS) equipped with new generation silicon drift detector (SDD) attached with SEM. For this purpose, spot analysis technique was applied by selecting two spots; one on pearlite and the other on retained austenite. Three measurements were taken for each sample to check the reproducibility of results.

#### 2.4. Vickers Hardness Testing

Vickers hardness testing was performed on ground and polished steel samples of dimensions  $10 \times 10 \times 5$  mm<sup>3</sup> using Vickers hardness tester (Shimadzu Model HMV Japan) equipped with diamond indenter; under the load of 1000 g. Five readings were noted and averaged to get the final hardness value for each sample.

#### 2.5. Electrochemical Analysis

For electrochemical analysis, the steel samples of dimensions  $10 \times 10 \times 5 \text{ mm}^3$  were soldered with single copper wire and cold mounted in polyester resin with  $10 \times 10 \text{ mm}^2$  exposed surface area. All the samples were metallographically prepared by the aforementioned procedure. Polarization potential and corrosion kinetics of TMCA treated steel were evaluated in 5 vol% HCl solution by the Tafel scan technique on Autolab potentiostat (Metrohm PG30, Netherlands). In the three-electrode system, a saturated calomel electrode was used as a reference electrode, platinum wire as an auxiliary electrode, and experimental steel sample as a working electrode. To validate the reproducibility, three tests were performed for each sample.

#### 2.6. Immersion Corrosion Analysis

Immersion corrosion performance of TMCA treated AISI 1345 steel was evaluated in 5 vol% HCl solution at room temperature. The samples of dimensions  $10 \times 10 \times 5$  mm<sup>3</sup> were metallographically prepared by the aforementioned procedure from all sides and then vertically immersed in the HCl solution for 168 h. After this immersion time, the samples were taken out of the solution and washed with distilled water. The corrosion morphologies, elemental compositions, and distributions of corrosion products were analyzed using SEM (FEI brand model Inspect S50 USA SEM), EDS spot analysis, and elemental mapping technique.

#### 3. Results and Discussion

## 3.1. Microstructure Evolution

Microstructural features of TMCA treated steel samples were found very sensitive to the applied treatments. Optical and SEM micrographs of non-treated and TMCA treated steel samples are illustrated in Figures 2 and 3, whereas, the corresponding EDS spectra are presented in Figure 4. Variations in C and Mn percentages in retained austenite and pearlite phases and average grain size of microstructure with TMCA treatments are plotted in Figure 5.



**Figure 2.** Microstructure of non-treated; (**a**) optical micrograph, (**b**) SEM micrograph, and TMSA treated; (**c**) optical micrograph, and (**d**) SEM micrograph AISI 1345 steel, containing pearlite (P) and retained austenite (RA) phases.



**Figure 3.** Microstructure of TMDA treated; (**a**) optical micrograph, (**b**) SEM micrograph, and TMTA treated; (**c**) optical micrograph, and (**d**) SEM micrograph experimental AISI 1345 steel containing pearlite (P) and retained austenite (RA) phases.



Figure 4. EDS spectra of pearlite phase in (a) non-treated, (b) TMSA, (c) TMDA, and (d) TMTA treated AISI 1345 steel.



**Figure 5.** Plot of (**a**) carbon, (**b**) manganese percentages in retained austenite and pearlite phases, and (**c**) average grain size of microstructure achieved after various TMCA processes.

In non-treated form, AISI steel exhibited coarse grained (179.6  $\mu$ m) microstructure having low fraction of retained austenite phase randomly distributed along the grain boundaries of the lamellar pearlitic matrix as illustrated in Figure 2a,b. EDS spectra demonstrated that the retained austenite phase contains 1.36% C and 9.91% Mn whereas, the pearlite phase contains 2.14% C and 9.65% Mn as illustrated in (Figures 4a and 5). It is a well-known fact that TMT treatment including hot rolling and oil quenching produces lath martensite and retained austenite phases in microstructure [40],

but subsequent annealing process involving very slow furnace cooling transforms these phases into ferrite, pearlite, or retained austenite phases depending upon the chemical composition of steel [35,37,38]. Therefore, in this work, TMSA treatment transformed the martensite phase obtained after TMT treatment into pearlite phase whereas, retained austenite remains stable due to presence of Mn (1.78 wt%) in the steel. The microstructure obtained after TMSA treatment comprised of relatively coarse grained ( $254 \mu m$ ) microstructure with improved fractions of the retained austenite phase and reduced fractions of pearlite phase (Figure 2c,d) compared to non-treated steel. The TMSA treatment also reduced the C percentages (0.99% and 1.68%) in both retained austenite and pearlite phases as illustrated in Figures 4b and 5. Reduction in C percentage in both phases might be attributed to the precipitation of secondary phase particles along the grain boundaries. It has been reported that precipitation of secondary phase particles always causes diffusion of C from other phases to these particles [41–44].

On the other hand, TMDA treatment offered maximum refined grained (26.7  $\mu$ m) microstructure having relatively reduced fractions of pearlite and increased fractions of retained austenite (Figure 3a,b) compared to non-treated steel. Due to prolonged and double furnace cooling, most of the C content (3.82%) diffused into the pearlite phase whereas, minor C content (1.83%) lied in the retained austenite phase as illustrated in Figures 4c and 5.

As mentioned earlier, TMTA treatment involved three times repetition of austenitizing and slow furnace cooling, therefore TMTA treatment resulted in well-refined (37.8 µm) microstructure compared to non-treated steel but coarser than TMDA treated steel with maximum retained austenite fractions and minor fractions of pearlite at the great boundaries as illustrated in Figure 3c,d. Pearlite formed after TMTA treatment possessed the highest C (3.96%) and Mn (14.38%) contents among all samples (Figures 4d and 5).

#### 3.2. Vickers Hardness

Micro Vickers hardness values of non-treated and TMCA treated AISI 1345 steel are plotted in Figure 6. In non-treated form, experimental steel exhibited the highest hardness value (248 VHN) among all samples attributed to the highest fractions of the pearlite phase and lowest fractions of the randomly distributed retained austenite phase. As mentioned above, TMSA treatment produced a coarse-grained microstructure with improved fractions of retained austenite phase, reduced fractions of pearlite phase, and diffused the C of both phases to the secondary phase particles. Therefore, considerably reduced (19%) Vickers hardness (200 VHN) was achieved after TMSA treatment compared to non-treated steel.



Figure 6. Plot of micro Vickers hardness values of non-treated and TMCA treated AISI 1345 steel.

TMDA treatment offered almost identical Vickers hardness value to non-treated steel. This is because TMDA treatment produced a well-refined microstructure containing further improved fractions of retained austenite phase and reduced fractions of the pearlite phase present at the grain boundaries.

The lowest Vickers hardness value (168 VHN) was achieved after TMTA treatment. This is because a moderately grained microstructure was formed after TMTA treatment having negligible fractions of pearlite and almost maximum fractions of retained austenite.

#### 3.3. Electrochemical Properties

Tafel scans of non-treated and TMCA treated AISI 1345 steel are plotted in Figure 7. The polarization behavior of all steel samples was explored in -0.25 V to 0.25 V potential range with reference to open circuit potential at a scanning rate of 1 mV/s. The Tafel extrapolation method with NOVA 2.1 software was utilized to calculate the kinetic parameters given in Table 2. According to the Butler–Volmer relation: [45]

$$i_{net} = i_o \left\{ \exp\left(\beta_a \frac{nF}{RT} \eta_a\right) - exp\left[-(1 - \beta_c) \frac{nF}{RT} \eta_c\right] \right\}$$
(1)

where ' $\beta'_a$  anodic Tafel constant, ' $\beta'_c$  the cathodic Tafel constant, ' $\eta'_a$  is anodic polarization and ' $\eta'_c$  is cathodic polarization of the metal surfaces during Tafel extrapolation.



Figure 7. Tafel scans of non-treated and TMCA treated experimental AISI 1345 steel.

r Corrosion Rate ) (mpy)
9.143
) 16.27
7 3.025
2 2.771

Table 2. Kinetic parameters of non-treated and TMCA treated AISI 1345 steel.

The AISI 1345 steel demonstrated varying values of corrosion current density ( $I_{corr}$ ), potential ( $E_{corr}$ ),  $\beta_a$ ,  $\beta_c$ , and corrosion rate under varying TMCA treatments. In non-treated form, AISI 1345 steel exhibited moderate  $I_{corr}$  (20  $\mu$ A.cm<sup>-2</sup>) and moderate corrosion rate (9.143 mpy) whereas, after TMSA treatment, the value of  $I_{corr}$  significantly (78%) increased to 35.60  $\mu$ A.cm<sup>-2</sup>. According to the Butler–Volmer relation, the value of  $I_{corr}$  is directly proportional to the corrosion rate of steel [45]. Therefore, the corrosion rate of TMSA treated steel was also increased (78%) to 16.27 mpy compared to non-treated steel. This increment in  $I_{corr}$  and corrosion rate might be attributed to several factors i.e., coarsening of grain size, uniform distribution of retained austenite and pearlite phases, reduction of C percentages in retained austenite and pearlite phases, and precipitation of secondary phase particles. Although the retained austenite phase is reported to be less susceptible to corrosion attack, but corrosion cells formed in the presence of pearlite phase [46,47]. Therefore, the decrease in the pearlite phase after TMDA treatment reduced the number of corrosion cells and caused a three-fold decrease in corrosion rate (3.025 mpy) compared to non-treated AISI 1345 steel.

As mentioned earlier, fractions of the pearlite phase decreased and that of retained austenite increased to maximum level after TMTA treatment and offered the minimum number of corrosion cells. Due to this fact, the TMTA treated sample demonstrated the lowest corrosion rate (2.771 mpy) among all samples.

## 3.4. Immersion Corrosion Properties

One of the toughest challenges in corrosion science is the prediction of corrosion susceptibility of steels in a specific environment [48]. Corroded surfaces of non-treated and TMCA treated AISI 1345 steel obtained after immersion test in 5 vol% HCl solution for 168 h were analyzed by SEM, EDS spot analysis, and EDS mapping technique. SEM micrographs, EDS spectra, and elemental maps of corroded surfaces of all samples are illustrated in Figure 8. It has been reported that immersion of a steel in a specific corrosive environment can produce various corrosion products i.e., hematite (Fe<sub>2</sub>O<sub>3</sub>), magnetite (Fe<sub>3</sub>O<sub>4</sub>), lepidocrocite ( $\gamma$ -FeOOH), goethite ( $\alpha$ -FeOOH), calcite (CaCO<sub>3</sub>), and green rusts (hydrated ferrous–ferric compounds) [48–53]. As the HCl is an aggressive environment, the surface of non-treated steel was observed to be fully covered with thick crack-free corrosion layers (Figure 8a). It has been reported that a high percentage of oxygen in the corrosion product presents a high corrosion rate [54–58]. Here, the intensity of corrosion attack at non-treated steel surface can be judged from the oxygen percentage in the EDS spectrum and elemental map which are highest (57.5% and 71%) among all samples. High percentages of oxygen in the corrosion product indicated that non-treated steel sample underwent intense corrosion attack. On the other hand, the surface of the TMSA treated steel was also fully covered with the thick corrosion layer but this layer was observed to be spongy and has micro-cracks as can be seen in Figure 8b. Oxygen percentages were observed to be slightly lower (50.51% and 68%) than non-treated steel indicating an almost identical intensity of the corrosion attack at the TMSA treated sample surface.

The surfaces of both TMDA and TMTA treated samples were observed to be partially covered with thin, crack-free corrosion layers as illustrated in Figure 8c,d. The oxygen percentage (42% and 61%) in EDS spectrum and elemental map of TMDA treated sample exhibited that this sample underwent moderate corrosion attack of HCl. The intensity of this attack was relatively lower than non-treated and TMSA treated samples. TMTA sample demonstrated minimum oxygen percentage (8.16% and 42%) presenting highest corrosion resistance among all samples. The results of immersion corrosion analysis were observed to be in good agreement with electrochemical analysis results.

## 4. Conclusions

The following were the extracted conclusions:

- i. Dual-phase microstructures comprising retained austenite and pearlite phases were achieved after all TMCA treatments of AISI 1345 steel. Secondary phase particles precipitation was observed after TMSA treatment. Variations in grain size, fractions of both phases, and C and Mn percentages in both phases were observed with varying TMCA treatments. TMTA treated sample exhibited maximum fractions of retained austenite and minimum fractions of pearlite along grain boundaries. This pearlite contained the highest percentages of C (3.96%) and Mn (14.38%) among all samples. TMDA treatment provided maximum (26.7  $\mu$ m) whereas, TMSA minimum (254  $\mu$ m) grain refinement of microstructure.
- ii. TMDA and TMTA treatments significantly improved the electrochemical properties of AISI 1345 steel in 5 vol% HCl solution compared to non-treated steel. Both treatments caused a three-fold reduction in  $I_{corr}$  (6.610, 6.060  $\mu$ A.cm<sup>-2</sup>) as well as in corrosion rate (3.025, 2.771 mpy) attributed to the reduced fractions of pearlite and in turn number of corrosion cells. On the other hand, TMSA treatment exhibited very poor electrochemical properties among all samples.
- iii. The surface of the TMTA treated sample was observed to be partially covered with a very thin crack-free oxide layer having the lowest oxygen percentages (8.16% and 42%) in the EDS spectrum

and EDS map among all samples. These features proved that TMTA treated sample underwent a minimum corrosion attack of the HCl solution. On the other hand, the surface of TMSA treated sample was fully covered with thick, spongy oxide layer having clear micro-cracks and highest oxygen percentages (50.51% and 68%) in the EDS spectrum and EDS map among all treated samples. These features proved that TMSA treated sample underwent intense corrosion attack of the HCl solution. Results of electrochemical analysis and immersion corrosion analysis were in good agreement.



**Figure 8.** SEM micrographs, EDS spectra, and EDS maps of corroded surfaces of (**a**) non-treated, (**b**) TMSA, (**c**) TMDA, and (**d**) TMTA treated AISI 1345 steel in 5 vol% HCl solution.

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## References

- Merimi, I.; Benkaddour, R.; Lgaz, H.; Rezki, N.; Messali, M.; Jeffali, F.; Oudda, H.; Hammouti, B. Insights into corrosion inhibition behavior of a triazole derivative for mild steel in hydrochloric acid solution. *Mater. Today-Proc.* 2019, 13, 1008–1022. [CrossRef]
- Boutouil, A.; Laamari, M.R.; Elazhary, I.; Bahsis, L.; Anane, H.; Stiriba, S.E. Towards a deeper understanding of the inhibition mechanism of a new 1,2,3-triazole derivative for mild steel corrosion in the hydrochloric acid solution using coupled experimental and theoretical methods. *Mater. Chem. Phys.* 2020, 241, 122420. [CrossRef]
- 3. Nadi, I.; Belattmania, Z.; Sabour, B.; Reani, A.; Sahibed-dine, A.; Jama, C.; Bentiss, F. Sargassum muticum extract based on alginate biopolymer as a newefficient biological corrosion inhibitor for carbon steel in hydrochloric acid pickling environment: Gravimetric, electrochemical and surface studies. *Int. J. Biol. Macromol.* **2019**, *141*, 137–149. [CrossRef]
- 4. Bouhlal, F.; Labjar, N.; Abdoun, F.; Mazkour, A.; Idrissi, M.S.; Mahi, M.E.; Lotfi, E.M.; Skalli, A.; Hajjaji, S.E. Chemical and electrochemical studies of the inhibition performance of hydro-alcoholic extract of used coffee grounds (HECG) for the corrosion of C38 steel in 1 M hydrochloric acid. *Egypt. J. Pet.* **2019**. [CrossRef]
- 5. Ahmed, M.H.O.; Amiery, A.A.A.; Majedy, Y.K.A.; Kadhum, A.A.H.; Mohamad, A.B.; Gaaz, T.S. Synthesis and characterization of a novel organic corrosion inhibitor for mild steel in 1 M hydrochloric acid. *Results Phys.* **2018**, *8*, 728–733. [CrossRef]
- Ye, Y.; Yang, D.; Chen, H.; Guo, S.; Yang, Q.; Chen, L.; Zhao, H.; Wang, L. A high-efficiency corrosion inhibitor of N-doped citric acid-based carbon dots for mild steel in hydrochloric acid environment. *J. Hazard. Mater.* 2020, *381*, 121019. [CrossRef] [PubMed]
- Lgaz, H.; Saha, S.K.; Chaouiki, A.; Bhat, K.S.; Salghi, R.; Shubhalaxmi; Banerjee, P.; Ali, I.H.; Khan, M.I.; Chung, I.M. Exploring the potential role of pyrazoline derivatives in corrosion inhibition of mild steel in hydrochloric acid solution: Insights from experimental and computational studies. *Constr. Build. Mater.* 2020, 233, 117320. [CrossRef]
- El-Etre, A.Y.; Ali, A.I. A novel green inhibitor for C-steel corrosion in 2.0 mol·L<sup>-1</sup> hydrochloric acid solution. *Chin. J. Chem. Eng.* 2017, 25, 373–380. [CrossRef]
- 9. Devika, B.G.; Doreswamy, B.H.; Tandon, H.C. Corrosion behaviour of metal complexes of antipyrine based azo dye ligand for soft-cast steel in 1 M hydrochloric acid. *J. King Saud Univ. Sci.* **2020**, *32*, 881–890. [CrossRef]
- 10. Ishak, A.; Adams, F.V.; Madu, J.O.; Joseph, I.V.; Olubambi, P.A. corrosion inhibition of mild steel in 1M hydrochloric acid using haematostaphisbarteri leaves extract. *Procedia Manuf.* **2019**, *35*, 1279–1285. [CrossRef]
- 11. Sarvghad, M.; Aguila, D.D.; Will, G. Optimized corrosion performance of a carbon steel in dilute sulfuric acid through heat treatment. *Appl. Surf. Sci.* **2019**, *491*, 460–468. [CrossRef]
- 12. Zhao, B.; Zhao, W.; Shi, H.; Li, G.; Ding, Y. The effects of stabilizing treatment on microstructure and corrosion resistance of 316Ti stainless steel. *Eng. Fail. Anal.* **2019**, *105*, 961–969. [CrossRef]
- Kong, D.; Dong, C.; Ni, X.; Zhang, L.; Yao, J.; Man, C.; Cheng, X.; Xiao, K.; Li, X. Mechanical properties and corrosion behavior of selective laser melted 316L stainless steel after different heat treatment processes. *J. Mater. Sci. Technol.* 2019, *35*, 1499–1507. [CrossRef]
- 14. Sarkar, S.; Mukherjee, S.; Kumar, C.S.; Nath, A.K. Effects of heat treatment on microstructure, mechanical and corrosion properties of 15-5 PH stainless steel parts built by selective laser melting process. *J. Manuf. Process.* **2020**, *50*, 279–294. [CrossRef]
- 15. Sudhakar, I.; Vardhan, Y.V.; Sruthi, P.; Harshit, P.V.S.; Kumar, S.S.; Prakash, V.V. Study on Corrosion Behavior of Heat Treated High Carbon Low Alloy Steel. *Mater. Today-Proc.* **2018**, *5*, 3919–3925. [CrossRef]
- Chen, X.; Li, J.; Cheng, X.; Wang, H.; Huang, Z. Effect of heat treatment on microstructure, mechanical and corrosion properties of austenitic stainless steel 316 L using arc additive manufacturing. *Mater. Sci. Eng. A* 2018, 715, 307–314. [CrossRef]
- 17. Sharma, L.; Chhibber, R. Microstructure evolution and electrochemical corrosion behaviour of API X70 linepipe steel in different environments. *Int. J. Press. Vessel. Pip.* **2019**, *171*, 51–59. [CrossRef]
- Cerdán, C.E.; Ooi, S.W.; Joshi, G.R.; Morana, R.; Bhadeshia, H.K.D.H.; Akid, R. Effect of tempering heat treatment on the CO<sub>2</sub> corrosion resistance of quench-hardened Cr-Mo low-alloy steels for oil and gas applications. *Corros. Sci.* 2019, 154, 36–48. [CrossRef]

- 19. Behbahani, K.M.; Najafisayar, P.; Pakshir, M.; Shahsavari, M. An electrochemical study on the effect of stabilization and sensitization heat treatments on the intergranular corrosion behaviour of AISI 321H austenitic stainless steel. *Corros. Sci.* **2018**, *138*, 28–41. [CrossRef]
- Luo, H.; Yu, Q.; Dong, C.; Sha, G.; Liu, Z.; Liang, J.; Wang, L.; Han, G.; Li, X. Influence of the aging time on the microstructure and electrochemical behaviour of a 15-5PH ultra-high strength stainless steel. *Corros. Sci.* 2018, 139, 185–196. [CrossRef]
- 21. Wang, L.; Dong, C.; Man, C.; Kong, D.; Xiao, K.; Li, X. Enhancing the corrosion resistance of selective laser melted 15-5PH martensite stainless steel via heat treatment. *Corros. Sci.* **2020**, *166*, 108427. [CrossRef]
- 22. Chandra, K.; Kain, V.; Tewari, R. Microstructural and electrochemical characterisation of heat-treated 347 stainless steel with different phases. *Corros. Sci.* **2013**, *67*, 118–129. [CrossRef]
- 23. Kadowaki, M.; Muto, I.; Katayama, H.; Masuda, H.; Sugawara, Y.; Hara, N. Effectiveness of an intercritical heat-treatment on localized corrosion resistance at the microstructural boundaries of medium-carbon steel. *Corros. Sci.* **2019**, *154*, 159–177. [CrossRef]
- 24. Ganesh, P.; Kumar, A.V.; Thinaharan, C.; Krishna, N.G.; George, R.P.; Parvathavarthini, N.; Rai, S.K.; Kaul, R.; Mudali, U.K.; Kukreja, L.M. Enhancement of intergranular corrosion resistance of type 304 stainless steel through a novel surface thermo-mechanical treatment. *Surf. Coat. Technol.* **2013**, *232*, 920–927. [CrossRef]
- 25. Hafeez, M.A.; Inam, A.; Farooq, A. Mechanical and corrosion properties of medium carbon low alloy steel after cyclic quenching and tempering heat–treatments. *Mater. Res. Express* **2020**, *7*, 016553. [CrossRef]
- Prakash, P.; Vanaja, J.; Srinivasan, N.; Parameswaran, P.; Rao, G.V.S.N.; Laha, K. Effect of thermo-mechanical treatment on tensile properties of reduced activation ferritic-martensitic steel. *Mater. Sci. Eng. A* 2018, 724, 171–180. [CrossRef]
- Li, S.; Eliniyaz, Z.; Sun, F.; Shen, Y.; Zhang, L.A.; Shan, A. Effect of thermo-mechanical treatment on microstructure and mechanical properties of P92 heat resistant steel. *Mater. Sci. Eng. A* 2013, 559, 882–888. [CrossRef]
- 28. Ghosh, A.; Ghosh, M. Tensile and impact behaviour of thermo mechanically treated and micro-alloyed medium carbon steel bar. *Constr. Build. Mater.* **2018**, *192*, 657–670. [CrossRef]
- Balan, K.P.; Reddy, A.V.; Sarma, D.S. Effect of single and double austenitization treatments on the microstructure and mechanical properties of 16Cr-2Ni Steel. *J. Mater. Eng. Perform.* 1999, *8*, 385–393. [CrossRef]
- 30. Chang, E.; Chang, C.Y.; Liu, C.D. The effects of double austenitization on the mechanical properties of a 0.34C containing low-alloy Ni-Cr-Mo-V steel. *Metall. Mater. Trans. A* **1991**, 25, 545–555. [CrossRef]
- Pandey, C.; Mahapatra, M.M.; Kumar, P.; Kumar, P.; Saini, N.; Thakare, J.G.; Kumar, S. Study on effect of double austenitization treatment on fracture morphology tensile tested nuclear grade P92 steel. *Eng. Fail. Anal.* 2019, *96*, 158–167. [CrossRef]
- 32. Salunkhe, S.; Fabijanic, D.; Nayak, J.; Hodgson, P. Effect of single and double austenitization treatments on the microstructure and hardness of AISI D2 tool steel. *Mater. Today-Proc.* **2015**, *2*, 1901–1906. [CrossRef]
- 33. Cao, Z.; Shi, Z.; Yu, F.; Sugimoto, K.; Cao, W.; Weng, Y. Effects of double quenching on fatigue properties of high carbon bearing steel with extra-high purity. *Int. J. Fatigue* **2019**, *128*, 105176. [CrossRef]
- 34. Zhang, Y.; Yu, C.; Zhou, T.; Liu, D.; Fang, X.; Li, H.; Suo, J. Effects of Ti and a twice-quenching treatment on the microstructure and ductile brittle transition temperature of 9CrWVTiN steels. *Mater. Des.* **2015**, *88*, 675–682. [CrossRef]
- Arlazarov, A.; Gouné, M.; Bouaziz, O.; Hazotte, A.; Petitgand, G.; Barges, P. Evolution of microstructure and mechanical properties of medium Mn steels during double annealing. *Mater. Sci. Eng. A* 2012, 542, 31–39. [CrossRef]
- Feng, Y.Q.; Wang, F.M.; Tao, S.F.; Zhao, D.Q.; Zhang, X.Y. Structure and texture formation during single and double cold rolling and annealing processes for Nb+Ti added interstitial-free steels. *J. Iron Steel Res. Int.* 2013, 20, 42–49. [CrossRef]
- 37. Han, J.; Kang, S.H.; Lee, S.J.; Lee, Y.K. Fabrication of bimodal-grained Al-free medium Mn steel by double intercritical annealing and its tensile properties. *J. Alloys Compd.* **2016**, *681*, 580–588. [CrossRef]
- 38. Saha, R.; Ray, R.K.; Bhattacharjee, D. Attaining deep drawability and non-earing properties in Ti + Nb interstitial-free steels through double cold rolling and annealing. *Scr. Mater.* **2007**, *57*, 257–260. [CrossRef]
- 39. Eskandari, M.; Kermanpur, A.; Najafizadeh, A. Formation of nano-grained structure in a 301 stainless steel using a repetitive thermo-mechanical treatment. *Mater. Lett.* **2009**, *63*, 1442–1444. [CrossRef]

- 40. Prakash, P.; Vanaja, J.; Palaparti, D.P.R.; Reddy, G.V.P.; Laha, K.; Rao, G.V.S.N. Tensile flow and work hardening behavior of reduced activation ferritic martensitic steel subjected to thermo-mechanical treatment. *J. Nucl. Mater.* **2019**, 520, 19–26. [CrossRef]
- 41. Hafeez, M.A.; Farooq, A. Microstructural, mechanical and tribological investigation of 30CrMnSiNi2A ultra-high strength steel under various tempering temperatures. *Mater. Res. Express* **2018**, *5*, 016505. [CrossRef]
- 42. Hafeez, M.A. Effect of microstructural transformation during tempering on mechanical properties of quenched and tempered 38CrSi steel. *Mater. Res. Express.* **2019**, *6*, 086552. [CrossRef]
- 43. Hafeez, M.A.; Inam, A.; Arshad, M.A. Investigation on microstructural, mechanical, and electrochemical properties of water, brine quenched and tempered low carbon steel. *Mater. Res. Express* **2019**, *6*, 096524. [CrossRef]
- 44. Inam, A.; Imtiaz, Y.; Hafeez, M.A.; Munir, S.; Ali, Z.; Ishtiaq, M.; Hassan, M.H.; Maqbool, A.; Haider, W. Effect of tempering time on microstructure, mechanical, and electrochemical properties of quenched–partitioned–tempered Advanced High Strength Steel (AHSS). *Mater. Res. Express* **2019**, *6*, 126509. [CrossRef]
- 45. Roberge, P.R. Handbook of Corrosion Engineering; McGraw-Hill: New York, NY, USA, 2000.
- 46. Revie, R.W.; Uhlig, H.H. *Corrosion and Corrosion Control*, 4th ed.; John Wiley and Sons Inc.: New York, NY, USA, 2008.
- 47. Hafeez, M.A.; Farooq, A. Effect of heat treatments on the mechanical and electrochemical behavior of 38CrSi and AISI 4140 steels. *Metallogr. Microstruct. Anal.* **2019**, *8*, 479–487. [CrossRef]
- Hafeez, M.A. Mechanical investigation on mechanical properties and immersion corrosion performance of 0.35%C–10.5%Mn steel processed by austenite reverted transformation (ART) annealing process. *Metallogr. Microstruct. Anal.* 2020, 1–10. [CrossRef]
- 49. Lytle, D.A.; Sorg, T.J.; Frietch, C. Accumulation of arsenic in drinking water distribution systems. *Environ. Sci. Technol.* **2004**, *38*, 5365–5372. [CrossRef]
- Sarin, P.; Snoeyink, V.L.; Bebee, J.; Jim, K.K.; Beckett, M.A.; Kriven, W.M.; Clement, J.A. Iron release from corroded iron pipes in drinking water distribution systems: Effect of dissolved oxygen. *Water Res.* 2004, *38*, 1259–1269. [CrossRef]
- 51. Gerke, T.L.; Maynard, J.B.; Schock, M.R.; Lytle, D.L. Physiochemical characterization of five iron tubercles from a single drinking water distribution system: Possible new insights on their formation and growth. *Corros. Sci.* **2008**, *50*, 2030–2039. [CrossRef]
- 52. Peng, C.Y.; Korshin, G.V.; Valentine, R.L.; Hill, A.S.; Friedman, M.J.; Reiber, S.H. Characterization of elemental and structural composition of corrosion scales and deposits formed in drinking water distribution systems. *Water Res.* **2010**, *44*, 4570–4580. [CrossRef]
- Li, M.J.; Liu, Z.W.; Chen, Y.C.; Hai, Y. Characteristics of iron corrosion scales and water quality variations in drinking water distribution systems of different pipe materials. *Water Res.* 2016, 106, 593–603. [CrossRef] [PubMed]
- 54. Sarin, P.; Snoeyink, V.L.; Bebee, J.; Kriven, W.M.; Clement, J.A. Physico-chemical characteristics of corrosion scales in old iron pipes. *Water Res.* 2001, *35*, 2961–2969. [CrossRef]
- 55. Antunes, R.A.; Costa, I.; Faria, D.L. Characterization of corrosion products formed on steels in the first months of atmospheric exposure. *Mater. Res.* **2003**, *6*, 403–408. [CrossRef]
- 56. Gheno, T.; Monceau, D.; Zhang, J.; Young, D.J. Carburisation of ferritic Fe-Cralloys by low carbon activity gases. *Corros. Sci.* **2011**, *53*, 2767–2777. [CrossRef]
- Maslar, J.E.; Hurst, W.S.; Bowers, W.J.; Hendricks, J.H.; Aquino, M.I.; Levin, I. In situ Raman spectroscopic investigation of chromium surfaces under hydrothermal conditions. *Appl. Surf. Sci.* 2001, 180, 102–118. [CrossRef]
- Yang, F.; Shi, B.Y.; Gu, J.N.; Wang, D.; Yang, M. Morphological and physicochemical characteristics of iron corrosion scales formed under different water source histories in a drinking water distribution system. *Water Res.* 2012, 46, 5423–5433. [CrossRef]



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