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Mechanical and corrosion properties of medium carbon low alloy steel after cyclic quenching and tempering heat–treatments

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Abstract

Cyclic quenching and tempering (QT) heat–treatment processes have the potential to improve the mechanical properties of steel due to the formation of fine grain size microstructure. This work aims to investigate the mechanical and corrosion properties of experimental medium carbon low alloy steel under cyclic heat–treatment processes. Cyclic single quenching and tempering (SQT), double quenching and tempering (DQT), and triple quenching and tempering (TQT) heat–treatment processes were carried out on the medium carbon low alloy steel. X–ray diffraction analysis validated the formation of fine ε-carbide (Fe2.4C) after intermediate low–temperature tempering in DQT and TQT heat–treatments. These fine carbides provided preferential nucleation sites for the formation of new austenitic grain, resulting in a refined microstructure composed of supersaturated (carbon) lath martensite (α′) and retained austenite (γ). The DQT and TQT heat–treatments improved 19% tensile strength, 100% elongation, and 95% impact absorption energy in comparison to SQT heat–treatment. DQT and TQT heat–treatments also transformed the brittle behavior of SQT steel into a ductile behavior. It was also revealed by the small number and size of dimples along with a large number of short tear ridges in fractographs. Morphology of corrosion products of heat–treated experimental steel was also studied in a 5% NaCl solution. The SQT heat–treated sample showed porous morphology of corrosion products, while the TQT sample showed morphology with less porosity on the surface. The DQT sample showed solid corrosion morphology with little porosity. As the grain size went on decreasing in the cyclic heat–treatments the corrosion rate was also dropped.

1. Introduction

The demand for medium carbon low alloy steels is progressively increasing in automotive, aerospace, defense, and other industries. The reason behind this is their superior mechanical properties, excellent fracture toughness, and high fatigue resistance and wear resistance [1–3]. These properties are attributed to the lath α′ and retained γ phases in their microstructure developed by the heat–treatment process [4]. Quenching and tempering (QT) process is one of the widely used heat–treatment processes in the manufacturing industry. Quenching process develops carbon supersaturated α′ phase, while subsequent tempering diffuses excess carbon of α′ phase into retained γ phase [5–7]. Lath martensitic transformation during the QT process divides the prior γ grain into many packets and further subdivided each packet into blocks of parallel aligned laths. As both the packet and block exhibit high angle grain boundaries, these are considered to have a significant impact on the mechanical properties of steel [8].

It has been reported that properties achieved by QT processes can be further improved by grain size reduction [2, 9, 10], which is possible through microalloying [11], cyclic heat–treating [10, 12] and controlling starting microstructure during the manufacturing of steel [2]. Cyclic heat–treating is one of the most suitable, easier, and economical method involves the repetition of QT processes. Few investigations have been conducted on cyclic heat–treatments by repeating QT processes twice (DQT). The DQT cyclic heat–treatments involves austenitizing, oil quenching, intermediate low–temperature tempering, re–austenitizing, and second oil
quenching followed by moderate temperature tempering. The grain size of 9CrWVTiN steel containing coarse M23C6 carbides [12], 5Cr steel containing M3C3b carbides [8], AISI 4140 steel [2], and SCRAM steel [10] have been reported to be significantly refined by DQT treatment. This grain refinement results in a considerable increase in strength, impact absorption energy, and reduction in ductile–brittle transition temperature (DBTT) in comparison to conventional single (SQT) quenching and tempering heat–treatment [10].

On the other hand, corrosion resistance of carbon steels is an important property from the practical application point of view. The poor corrosion resistance of carbon steels causes their rapid destruction in various industrial environments i.e. mining, petrochemical, and chemical process industry. As a result carbon steels require continuous replacement after some time resulting in significantly increased maintenance cost [13–15].

Hence, medium carbon low alloy steel having a specific composition was produced and cast in the laboratory. This steel was subjected to cyclic heat–treatments including conventional single (SQT), double (DQT), and triple (TQT) quenching and tempering processes. Microstructure analysis and mechanical test results were obtained to validate the microstructure/mechanical properties relationship. Fractography was also performed to evaluate the fracture mechanism. Immersion corrosion analysis was carried out in 5 wt% NaCl solution and morphology, elemental composition and distribution of corrosion products were studied.

2. Experimental work

2.1. Materials

A melt of the experimental medium carbon low alloy steel was produced in the laboratory by using commercial grade raw materials i.e. mild steel strips, ferro–molybdenum, ferro–manganese, ferro–silicon, ferro–chrome, carburizer, and nickel. Charge sheet of raw materials for producing a melt of 50 kg of steel is given in Table 1.

The heat of the steel was prepared in a laboratory induction furnace and cast into ingots of size 75 × 75 × 450 mm in sand moulds. Chemical composition (wt%) of the produced experimental medium carbon low alloy steel is given in Table 2.

2.2. Cyclic heat–treatment

Heat–treatment cycles of cyclic QT processes applied to the experimental steel are presented in Figure 1. The steel samples were austenitized at 900 °C for 30 min followed by oil quenching. The aforementioned processes were performed once in single (SQT), twice in double (DQT), and thrice in triple (TQT) QT heat–treatments. A subsequent tempering process is mandatory to lower brittleness of steel and make it useable for industrial applications. Therefore, the SQT heat–treatment was accomplished by tempering at 450 °C for 60 min as revealed in the heat–treatment cycle as presented in Figure 1(a). The DQT heat–treatment was comprised of first low–temperature tempering process at 250 °C for 60 min after oil quenching and second quenching and tempering was carried out at 450 °C for 60 min (Figure 1(b)). While the TQT heat–treatment process was performed by doing two intermediate low–temperature tempering processes at 250 °C for 60 min and final tempering at 450 °C for 60 min after oil quenching from the austenitizing temperature of 900 °C (Figure 1(c)). For the quenching process, martensite start temperature (\(M_s\)) was determined by the Nehernberg equation [16].

\[
M_s = 498.9 - 300C - 33.3Mn - 22.2Cr - 16.7Ni - 11.1(Si + Mo)
\]
Where, C, Mn, Cr, Ni, Si, and Mo are the weight percentages of these alloying elements in prepared medium carbon low alloy steel given in table 2.

2.3. Microstructure analysis
For metallographic examination, the heat–treated samples were cut into a size of \(10 \times 10 \times 10\) mm. These samples were ground using FEPA grade SiC grinding papers, P100, P200, P400, P600, P800, and P1000. Polishing was performed on velvet and nylon clothes using a diamond paste of grade 6, 3, 1 and 0.25 \(\mu\)m on automatic polisher (Ecomet 250 Grinder/Polisher USA). To reveal the microstructure, etching was performed by 2% Nital solution. Light optical microscope (Leica Model DM–15000M Germany) and scanning electron microscope (FEI Inspect S50 SEM) were used for microstructural examination. Energy dispersive spectrometer (EDS) attached to SEM was used to characterize the elemental composition of different microstructural phases by spot analysis. Grain size analysis was performed according to the ASTM E112 standard. For DQT and TQT heat–treated samples, phase analysis was also performed to ensure the formation of fine carbides using x–ray diffractometer (XRD) (Bruker D8 advance Germany) equipped with CuK\(\alpha\) (\(\lambda = 1.5418\) Å) radiations using Ni filter and solid–state detector in the 2\(\theta\) range of 40 °C–86 °C.

2.4. Mechanical testing
Hardness testing was carried out on micro Vickers hardness tester (Shimadzu Model HMV Japan) using a diamond indenter under the load of 1000 g for 10 s. An average of five readings was taken as the hardness for each sample. Charpy V notch (CVN) impact test was performed on heat–treated samples of dimensions \(10 \times 10 \times 55\) mm with 2 mm V notch depth at room temperature. For this purpose, Charpy V notch impact tester (Avery Denison USA) equipped with 300 J hammer was used. Fractured surfaces of impact tested samples were analyzed by SEM to observe the fracture behavior of heat–treated experimental steel. The tensile testing samples were machined into the dimensions of gauge length 50 mm, gauge diameter 12.5 mm, fillet radius 10 mm, and reduced section length 56 mm according to the ASTM E8 standard. Tensile tests were carried out on computer–controlled (Kelson’s, India) universal testing machine having a capacity of 400 KN.

2.5. Immersion corrosion analysis
Immersion corrosion performance of heat–treated steel samples was studied in a 5% NaCl solution. Samples of dimensions \(10 \times 10 \times 10\) mm were ground and polished by the procedure mentioned in section 2.3. These samples were then immersed in a 5% NaCl solution having pH 6–6.5 at room temperature. After a period of

Figure 1. Heat–treatment cycles adopted for; (a) SQT, (b) DQT, and (c) TQT processes applied to medium carbon low alloy experimental steel.
168 h of immersion, samples were taken out of the solution, washed and dried. Corroded surfaces of all heat-treated samples were analyzed under SEM at various magnifications. Elemental compositions of corrosion products formed on the surfaces of the corrosion tested steel samples were analyzed by SEM–EDAX spot analysis and elemental mapping.

3. Results and discussion

3.1. Microstructure evolution

Microstructural features of medium carbon low alloy steel treated under cyclic heat–treatments are illustrated in figures 2–4. In quenching–treatment process, prior γ grain transforms into packets of lath α′ phase placed in different orientations. These packets are further subdivided into blocks of identical crystallographic orientations of laths. On the other hand, the subsequent tempering process diffuses out excess carbon from the lath α′ phase to γ phase resulting in the formation of low carbon α′ phase with retained γ phase [2].

Therefore, in current work, micrographs of the SQT, DQT, and TQT heat–treated samples are exhibiting packets and blocks of lath α′ phase with retained γ phase (figure 2). The α′ phase in microstructure is generally thought to create a strengthening effect in steels [17] by enhancing dislocation density [18]. SEM micrographs and corresponding EDS spectra of SQT, DQT and TQT heat–treated samples are illustrated in figure 3, which are in good agreement with optical micrographs.

After first austenitizing and quenching, the intermediate low–temperature tempering stage in DQT and TQT treatments is associated with nucleation of fine carbides. These carbides provide preferential nucleation and growth sites for γ grains during austenitization in the second stage resulting in a refined γ phase during austenitization. It has also been reported that the grain size of α′ phase achieved after quenching observed to be directly proportional to the prior γ grain size before quenching. Therefore, grain refinement of the γ phase occurred during re—austenitizing, which in turn refined the lath α′ grains after re—quenching in the second stage [2]. In this work, the nucleation of fine carbides after intermediate low–temperature tempering at 250°C in DQT and TQT treatments is validated by XRD analysis. XRD spectrums of both treated samples exhibited
intensity peaks with habit plan (110), (200), and (211) presenting the $\alpha'$ phase and intensity peak with habit planes (201) and (211) presenting the fine carbides in it as shown in figure 4.

Due to this fact, DQT treated sample in present work offered well refined (3.3 $\mu$m) microstructure having $\alpha'$ phase with retained $\gamma$ phase compared to SQT treated sample (15.9 $\mu$m) as presented in table 3. Although, the second stage of DQT treatment refined the grain size obtained after the first stage, while the third stage of TQT treatment did not do further grain refining. Hence, maximum grain refinement was resulted by the DQT treatment, while TQT treatment showed moderate grain refinement compared to SQT treatment.

3.2. Mechanical properties
The average grains size (AGS) in a polycrystalline metal significantly affects the mechanical properties [17]. Strengthening in quenched and tempered martensitic steel is possible by numerous strengthening mechanisms i.e. dislocation, solid solution, grain boundary, precipitation, and martensitic lath strengthening [9, 18, 19]. In current work, DQT and TQT heat–treatments not only strengthened the experimental steel but also enhanced the ductility (elongation and reduction in area) as given in table 3. Because of identical
tempering temperature and time, solid solution and dislocation strengthening should be the same and ineffective in this work [9]. A sthe DQT and TQT treatments resulted in grain size reduction, so grain boundary strengthening (grain size reduction strengthening) is possible here [18–22]. The results show that DQT heat–treatment not only caused an 18.69% improvement in strength ($R_m$) with a little reduction in hardness ($HV$). But also 100% improvement in ductility ($\varepsilon_T$ & $R_A$) as compared to the SQT heat–treated sample. Reduced AGS produced by DQT heat–treatment has different crystallographic orientations and a common grain boundary. Slip or dislocation moving across this grain boundary compels to alter the direction of motion which is tougher due to enhanced crystallographic mis–orientations resulting in strengthening of steel. Another reason associated with grain boundary strengthening is a discontinuity of slip planes from grain to grain created by the atomic disorder with a grain boundary [18, 19]. These results are in good agreement with the works of Sanij et al and Liu et al [2, 9].

On the other hand, TQT heat–treatment provided 11.94% improved $R_m$ and much–improved $\varepsilon_T$ at the little expense of HV compared with SQT treatment. The TQT heat–treatment produced significantly better results compared with SQT heat–treatment but relatively lower $R_m$ and higher $\varepsilon_T$ than DQT heat–treatment. This is because greater AGS was obtained by TQT heat–treatment than DQT heat–treatment. Another reason is associated with the depletion of carbon contents in the $\alpha'$ phase as illustrated in the EDS spectrum (figure 4(c)). TQT heat–treatment provided an optimum combination of mechanical properties.

### 3.3. Impact absorption energy and fracture mechanism

Critical fracture strain is the main factor of determining impact absorption energy that must be overreached by the applied extensional strain at the crack tip [2, 23, 24]. Fractured surfaces of SQT, DQT, and TQT heat–treated medium carbon low alloy steel samples are illustrated in figure 5, while the corresponding impact absorption energy values are given in table 3. From the impact absorption energy profile it is evident that DQT heat–treatment improved the impact absorption energy 66.7% as compared with SQT heat–treatment. While TQT heat–treatment further improves the impact absorption energy more than two times the SQT heat–treatment. It has been reported that improvement in impact absorption energy is attributed to reduced grain

### Table 3. Mechanical properties of cyclically heat treated medium carbon low alloy steel.

| Sample ID | $R_m$ (MPa) | $\varepsilon_T$ (%) | $R_A$ (%) | Hardness (VHN) | Impact Energy (J) | AGS ($\mu$m) |
|-----------|-------------|---------------------|-----------|----------------|------------------|--------------|
| SQT       | 1423        | 8                   | 24        | 351            | 10               | 15.9         |
| DQT       | 1689        | 14                  | 32        | 332            | 15               | 3.3          |
| TQT       | 1593        | 16                  | 35        | 303            | 20               | 5.7          |

Figure 4. XRD spectrum of DQT and TQT treated samples of medium carbon low alloy steel performed after intermediate low temperature tempering at 250 °C for 60 min.
size and the fraction of retained $\gamma$ phase in the microstructure. This improvement is also evident by the quantity and size of dimples present in fractographs. The quantity of tear ridges also demonstrates the impact absorption energy and fracture behavior of steel. Greater number of tear ridges indicates higher impact absorption energy [9].

It can be observed that SQT heat–treated fractograph contained large QCFs formed by coalescence of micro voids with a minor amount of dimples presenting purely brittle behavior as illustrated in figure 5(a). On the other hand, DQT heat–treated fractograph comprised of a great amount of large size dimples with few quasi–cleavage facets (QCF) indicating comparatively ductile behavior (figure 5(b)). Moreover, few long tear ridges were observed in the SQT treated fractograph presenting low impact absorption energy, while DQT heat–treated contained a great number of small tear ridges presenting better impact absorption energy. Amount of small size dimples as well as short tear ridges in TQT treated fractograph were much greater than SQT and DQT treated samples presenting the highest impact absorption energy and sufficient $\varepsilon_T$ as shown in figure 5(c). This ductile behavior of the TQT heat–treated sample may also be attributed to the reduced volume fraction of the lath $\alpha'$ phase in the microstructure. It has been pointed out that the smallest substructure unit for controlling impact absorption energy is martensitic block because of the same size of QCF and block.

### 3.4. Immersion corrosion properties

Prediction of steel dissolution in a specific environment is one of the toughest challenges in corrosion science and engineering [25]. Immersion corrosion properties of cyclic heat–treated medium carbon low alloy steel were evaluated in 5% NaCl solution by analyzing corrosion morphology. SEM micrographs, EDS spectrums, and EDS–maps of corroded surfaces are illustrated in figures 6–8. The surface of the SQT heat–treated sample was observed to be partially covered with an oxide layer having non–uniform thickness and micro–cracks. A thick, crack–free, oxide layer was observed on $\alpha'$ phase portions while a thin, unstable oxide layer with micro–cracks was formed on the $\gamma$ phase (figure 6(a)). This oxide layer has a partially spongy and partially massive morphology (figure 6(b)). A clear difference in corrosion morphologies, the thickness of the oxide layer and distribution was observed in two phases which are
attributed to two different corrosion mechanisms. It has been reported that anodic reaction causes oxidation of steel [26].

The attack of NaCl solution on the DQT heat–treated sample was similar to its attack on the SQT heat–treated sample with a difference of corrosion morphology (figure 6(c)). Corrosion morphology of the DQT heat–treated sample was observed to be porous structured (figure 6(d)). A thick, stable oxide layer with minor micro–cracks uniformly covered the surface of the TQT heat–treated sample as can be observed in figure 6(e). This layer has a throughout rod–shaped morphology with micro–cracks as shown in figure 6(f). It has been reported that the corrosion reaction cannot be further proceeded due to the formation of corrosion products on the steel surface. However, the morphology of corrosion products has a significant impact on the corrosion resisting mechanism. The porous structure and rod–shaped structure corrosion products have the opposite impact on the resisting mechanism. Furthermore, it has also been observed that porous–structured corrosion product resists corrosion reaction more significantly than rod–shaped corrosion product which is attributed to its high compactness than rod–shaped corrosion product [27].
SEM–EDS spectrums of corroded cyclic heat–treated experimental steel samples are illustrated in figure 7. SEM–EDS spectrums of SQT heat–treated sample showed the highest 33.76 wt% of O, while the lowest 31.46 wt% of O was revealed by DQT heat–treatment. The SEM–EDS spectrum from the corrosion product of the TQT heat–treated sample showed 32.97 wt% of O, resulting in corrosion rate between SQT and DQT heat–treatments (figure 7).

SEM–EDS elemental–maps of cyclic heat–treated medium carbon low alloy steel are illustrated in figure 8. SQT heat–treated sample exhibited localized corrosion attacks throughout the surface and demonstrated the uniform distribution of corrosion products having 67% O as shown in figure 8(a). High oxygen percentage in the corrosion product is presenting a high corrosion rate as previously reported in the literature [28–32]. On the other hand, the DQT heat–treated sample exhibited 58% O in EDS–map (figure 8(b)). This decrease in carbon percentage demonstrates the lower corrosion rate occurred on the surface of the DQT heat–treated sample. TQT heat–treated sample showed 56% O, and 6%Cl uniformly distributed on the surface (figure 8(c)). Precipitation of Cl indicated that TQT heat–treated sample underwent different corrosion mechanisms compared to other samples. Corrosion products formed on all samples may consist of magnetite (Fe₃O₄), goethite (α–FeOOH) and lepidocrocite (γ–FeOOH) while...
sometimes hematite (Fe₂O₃), calcite (CaCO₃) and green rusts (hydrated ferrous–ferric compounds containing CO₃²⁻, Cl⁻ or SO₄²⁻) [32–37].

4. Conclusions

Following conclusions are drawn from this study;

1. The microstructures of all SQT, DQT, and TQT heat–treated samples showed packets and blocks of tempered laths α′ phase along with retained γ–phase. The SQT heat–treated sample showed comparatively coarse grain–sized, TQT treatment provided moderate grain–sized and DQT offered fine grain–sized microstructure (15.9, 5.7 and 3.3 μm), respectively.
2. With a slight drop in hardness, DQT, and TQT heat–treatments resulted in improved $R_m$ (1689 MPa, 1593 MPa), $\varepsilon_T$ (14%, 16%), $R_A$ (32%, 35%), and impact absorption energy (15 J, 20 J) as compared to SQT sample.

3. Fractographs of SQT heat–treated sample showed wide quasi cleavage facets with minor small size dimples and long few tear ridges presenting purely brittle behavior. While DQT and TQT heat–treated fractographs contain a high quantity of large size dimples and short tear ridges presenting relatively less brittle behavior.

4. Spongy corrosion product morphology and high wt% Oxygen in the elemental map of the corrosion product formed on the surface of the SQT heat–treated sample revealed a high corrosion rate in comparison to DQT and TQT processes. TQT showed partially spongy corrosion morphology while DQT showed solid morphology with little spongy areas.

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