

Effect of Step Quenching and Tempering on the Corrosion Behaviour of a Low Carbon Steel in HCl Solution

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Abstract: The trust of this research is to critically examine the effect of step quenching and tempering on the corrosion performance of a low carbon steel in 0.1M HCl aqueous solution. The steel was first normalized at 850°C for 1 hour. This was followed by step quenching heat treatment, which involved austenitizing at 850°C followed by slow cooling in the furnace to and soaking at various temperatures in the ($\alpha + \gamma$) region of 730°C, 750°C and 770°C for 30 minutes and then quenched in water. Some set of the samples were tempered for 1 hour at 320°C and air cooled. Samples were prepared for microscopic examination and corrosion performance evaluation from all the heat treatment procedures. The weight loss method was used to evaluate the corrosion rate. Volume fraction of martensite was measured for the as-quenched step quenched samples. From the results, it was observed that martensite volume fraction increased with increase in soaking temperature. The results also revealed that step quenching increases the susceptibility of the investigated steel to corrosion, while tempering the as-quenched step quenched steel reduces corrosion susceptibility. Hence, it was recommended that for applications of this material in chloride environment, tempering should always proceed step quenching.

Keywords: step quenching, tempering, martensite volume fraction, corrosion rate.

I. INTRODUCTION

Steel, over the years has remained the most versatile and most widely used engineering material. The quest for very high reliability and efficiency of engineering systems and applications had led to the search for ways of improving the properties of this vital material (steel) in order to enhance the quality of life and improve industrial safety, at a reasonably competitive cost.

Step quenching is one of the methods that are being employed to improve the mechanical properties of steel. Ikpeseni et al (2015) [1] reported an improvement in tensile strength and hardness with reduction in ductility and impact toughness after step quenching a normalized steel sample. It was further reported by same authors of improvement on all the properties mentioned above after tempering the step quenched samples [1 – 2].

Step quenching is one of the intercritical annealing heat treatment routes for developing dual phase (DP) steels [2]. Many researches [2 – 10] had been reported with encouraging results on mechanical property enhancement of DP steels. However, the corrosion behavior of these materials (DP steels) with improved mechanical properties have not attracted much research as in the case of mechanical properties. Yet the corrosion behavior of any material is critical to its functionality, efficiency, durability and integrity. Nevertheless, few researches have been conducted on the corrosion behavior of DP steels. For example, [11- 12] worked on DP steels in NaCl environment, Alaneme and Momoh [13] worked on H₂SO₄ and NaOH solutions, while [14 – 15] worked on H₂SO₄ and NaOH/H₂SO₄ environments. Most of their results are to some extent conflicting and contradictory. Hence, this present research is poised to examine the influence of SQ and tempering on the corrosion susceptibility of carbon steel in HCl environment.

II. MATERIALS AND METHOD

A 16mm diameter ribbed rod was used for this investigation. Spark spectrometer (NCS Labspark 750B) metal analyzer was used to determine its chemical composition shown in Table 1. The as-received material was subjected to turning on lathe to remove the ribs.

Thereafter, the steel was normalized at 850°C (30°C above its AC₃) for 1hour in order to annul the previous thermal, mechanical or thermo-mechanical treatment it would have undergone. 850°C was arrived at using Andrews's equation [16]. Furthermore, AC₁, AC₃ and M_s were evaluated to be 710°C, 819°C and 404°C respectively using the same Andrew's formula. This was followed by step quenching heat treatment which involved austenitizing at 850°C for 1 hour and furnace cooling to 730°C, 750°C and 770°C respectively (i.e. between AC₁ and AC₃) and soaking at each temperature for 30 minutes, followed by quenching in water – Fig. 1. Some of the step quenched samples were tempered for 1 hour at 320°C and air cooled.

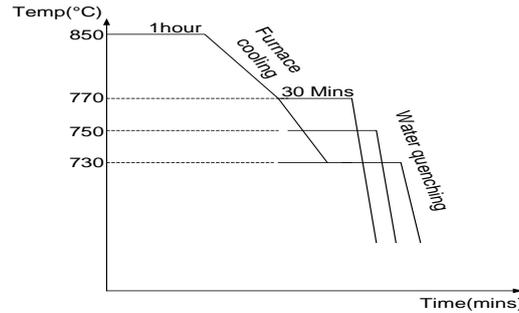


Fig. 1a: Schematic Illustration of Heat Treatment Schedule for SQ.

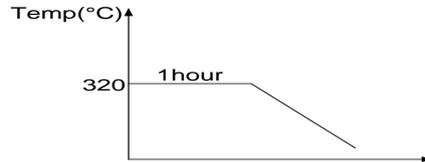


Fig. 1b: Schematic Illustration of the Tempering Heat Treatment Schedule for SQT

Samples were prepared according to standard procedures [17] for metallographic examination to characterize their microstructures. 2% NITAL was used as etchant for about 20s. The point count method using stereology was adopted to evaluate volume fraction of the phases present. Nikon Eclipse (ME 600) optical microscope fitted with photographic devices and computer accessories was used to examine the microstructures. Samples in the form of coupons were cut from the heat treated materials. The surfaces were cleaned by using various grits of emery paper to polish the surface. Thereafter, the samples were washed in water, rinsed in acetone, dried and stored under the desiccator. The samples were weighed before complete immersion in 0.1M HCl aqueous solution. The weight loss method was adopted in evaluating corrosion rate. Monitoring lasted for eighty days at eight days intervals. On each monitoring day samples are withdrawn from the environment, washed thoroughly using distilled water, rinsed in acetone and dried. A digital chemical weighing balance was used to weigh the samples before and after immersion. Corrosion rate was evaluated using equation (1)[18 – 20].

$$CR= W/At \tag{3.4}$$

- Where CR = Corrosion rate (mg/cm²/year)
- W = W₀ - W_n
- W =weight loss (mg)
- W₀ = initial weight
- W_n = final weight on each monitoring day
- A = total surface area of sample (cm²)
- t = exposure time in days converted to year

And

$$A = 2\pi r (r+L) \text{ because the samples are cylindrical in shape}$$

Where

- r = radius of sample (cm)
- L = Length of sample (cm)

III. RESULTS AND DISCUSSION

Table 3: Analysis of Volume Fraction Measurement

Sample	Intercritical Annealing Temperature (°C)	Sample Number	Martensite Volume Fraction [MVF] (%)				Total Average	Total Standard Deviation
			MVF per Sample	Average	Standard Deviation			
SQ730	730	1	45.8,37.5,33.3,33.3,41.7	38.3	±4.7	46	±4.9	
		2	37.5,54.2,45.8,41.7,45.8	45	±5.5			
		3	54.2,62.5,50.5,42.50	54.2	±4.6			
SQ750	750	1	50.5,8.3,58.3,50.5,42	54.2	±3.7	54	±4.8	
		2	41.7,50.5,42.5,42.50	50	±4.6			
		3	48.5,66.7,58.3,62.5,58.3	58.9	±6			
SQ770	770	1	58.3,66.7,66.7,54.2,66.7	62.5	±5.3	63	±5	
		2	54.2,62.5,50.5,42.50	54.2	±4.6			
		3	79.2,66.7,70.8,79.2,70.8	73.3	±5			

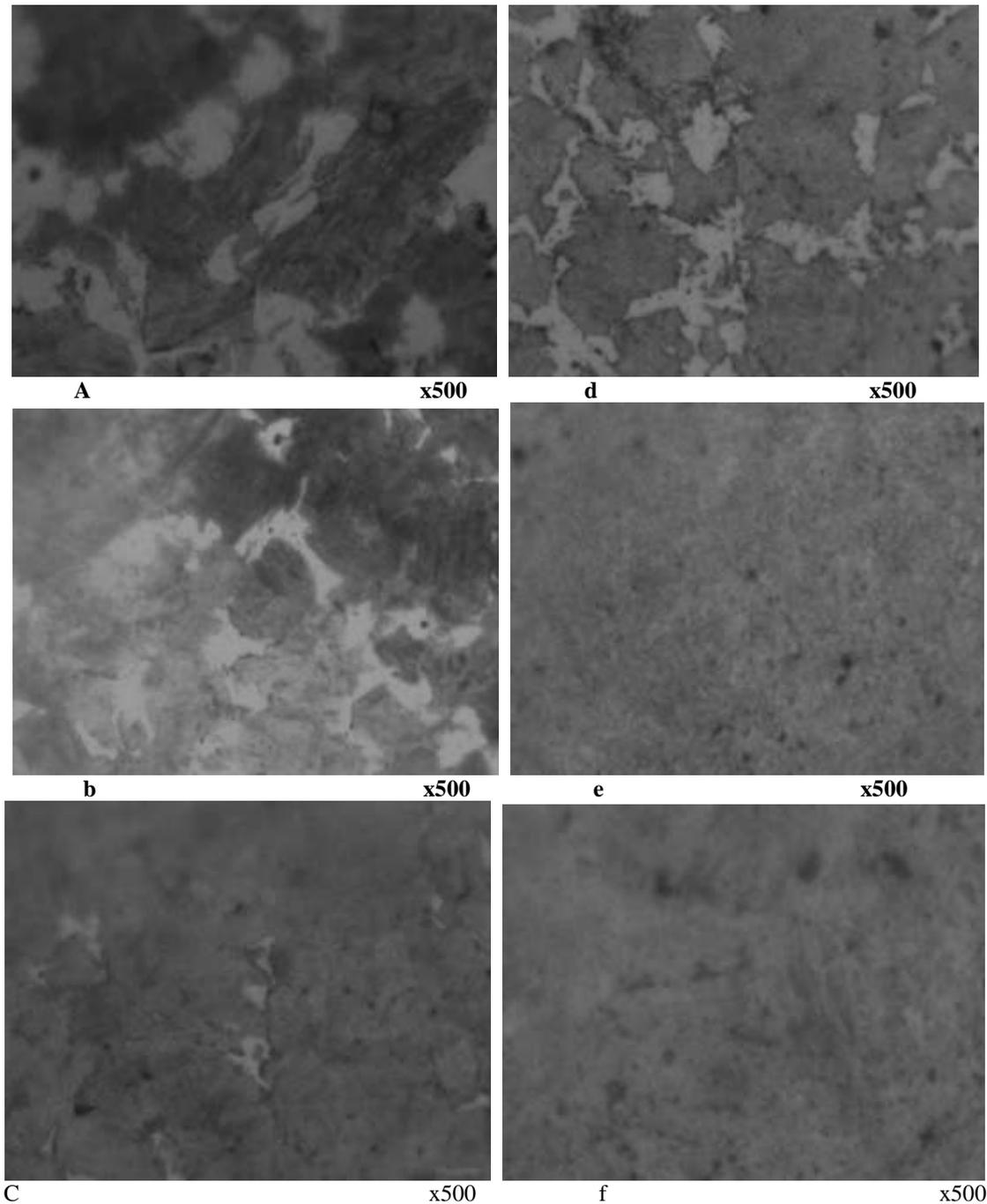


Fig. 3: Photomicrograph of as-quenched step quenched samples at: (a) 730°C (b) 750°C (c) 770°C
Photomicrograph of tempered step quenched samples at: (d) 730°C (e) 750°C (f) 770°C

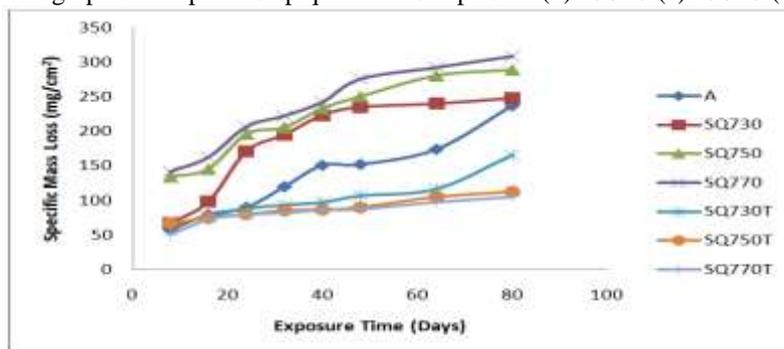


Fig. 4: Specific Mass Loss versus Exposure Time

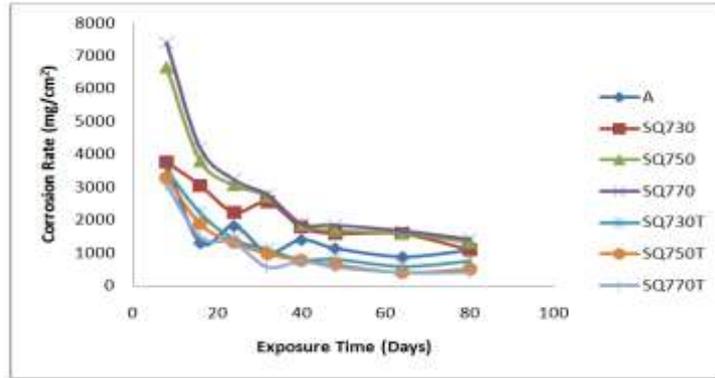


Fig. 5: Corrosion Rate versus Exposure Time

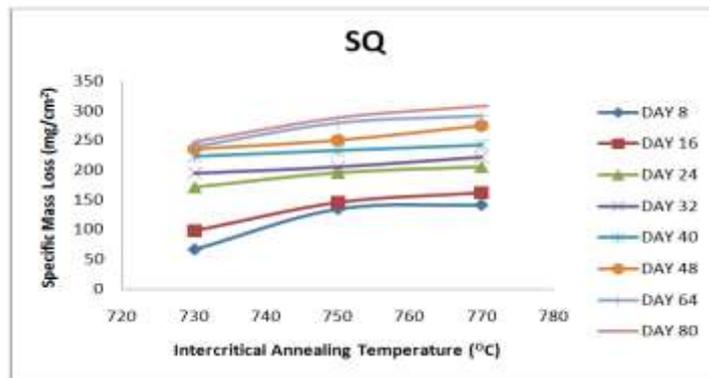


Fig. 6a: Specific Mass Loss versus Intercritical Annealing Temperature for as-quenched Step Quenched samples.

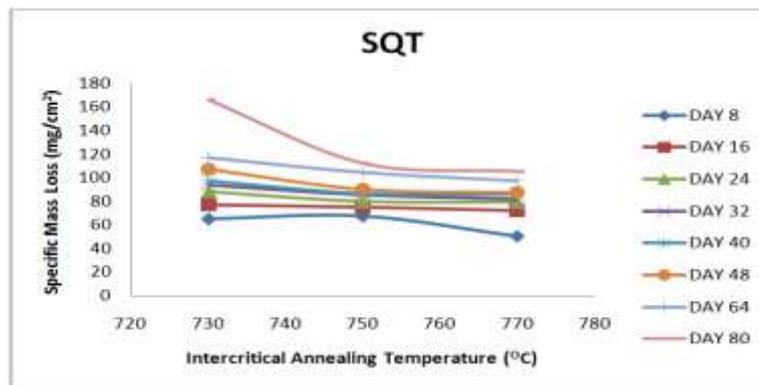


Fig. 6b: Specific Mass Loss versus Intercritical Annealing Temperature for Tempered Step Quenched Samples

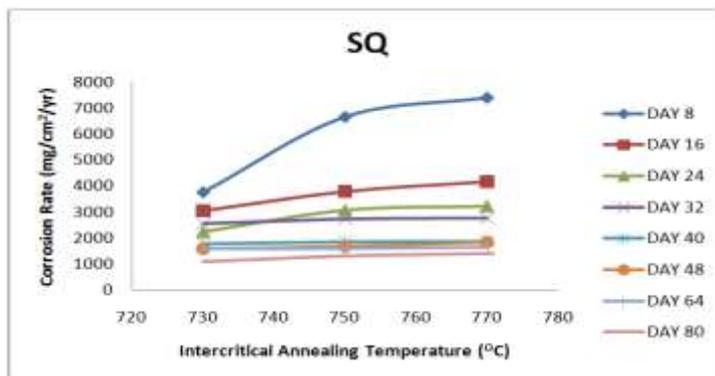


Fig. 7a: Corrosion Rate versus Intercritical Annealing Temperature for as-quenched Step Quenched samples.

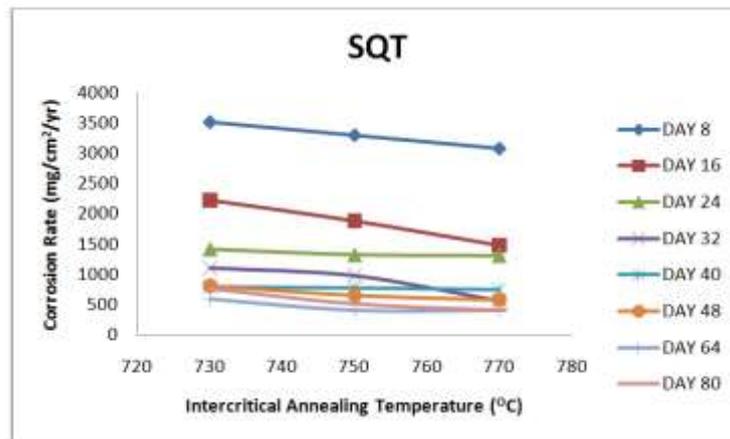


Fig. 7b: Corrosion Rate versus Intercritical Annealing Temperature for Tempered Step Quenched samples.

3.1 Effect of Step Quenching and Tempering on Microstructure

Fig. 3a-f show the microstructures developed after step quenching heat treatment and subsequent tempering at 320°C for 1 hour. The microstructure transformed from normalized structure (mixture of ferrite and pearlite) to irregularly shaped coarse ferrite matrix with martensite dispersed in it upon step quenching – Fig. 3a-c. The microstructure of the tempered samples showed grain refinement (Fig. 3d-e) when compared with their as-quenched step quenched counterparts.

Table 3 shows the martensite volume fraction in the microstructure of the as-quenched samples. It revealed that martensite volume fraction increased with increase in the intercritical annealing temperature.

3.2 Effect of Step Quenching on Corrosion Rate

Fig. 5 shows the corrosion rate of normalized (A), as-quenched step quench (SQ) and tempered step quenched (SQT) samples against exposure time. It revealed that step quenching increases the rate of corrosion, because corrosion rate of all the as-quenched samples are higher than that of the normalized sample. Corrosion rates increased with increase in soaking temperature for as-quenched samples – Fig. 7a. This could be attributed to increasing martensite volume fraction at higher temperature as reflected in Table 3.

Specific mass loss increased with increase in exposure time and soaking temperature at the ($\alpha + \gamma$) region; while corrosion rate decreased with increase in exposure time in the 0.1M HCl environment as shown in Figs. 4 and 5. Corrosion rates decreased from 7403.02mg/cm² to 1406.05mg/cm² for SQ770 sample on the 8th day and 80th day respectively; from 6667.13mg/cm² to 1316.94mg/cm² and from 3768.83mg/cm² to 1094.71mg/cm² for SQ750 and SQ730 samples respectively on days 8 and 80 respectively.

3.3 Effect of Tempering on Corrosion Rate

Figure 5 again shows that corrosion rate of samples after tempering at 320°C for 1 hour is lower than those of the as-quenched samples and also slightly lower than the corrosion rate of the normalized sample. The specific mass loss of the tempered step quenched samples is lower than those of as-quenched and normalized samples – Fig. 4 and Fig. 6. Generally, corrosion rates decreased with increase in exposure time while as expected the specific mass loss increased progressively as a function of time. The decrease in corrosion rate upon tempering could be attributed to increase in the relative amount of ferrite in the microstructure as can be observed from comparing the micrographs of the as-quenched samples with the tempered samples micrographs. For example, comparing Fig. 3a and 3d; Fig. 3b and 3e and Fig. 3c and 3f, an increase in the amount (qualitatively) of ferrite for each pair of micrograph can be observed (i.e. increase in the presence of light regions within the micrograph) after tempering. This is linked to the fact that on tempering at 320°C for 1 hour more ferrites are nucleated from martensite and already existing ferrite, thereby leaving the microstructure with a mixture of ferrite and tempered martensite after tempering. This increased amount of ferrite in the microstructure tends to reduce the cathode to anode (i.e. martensite to ferrite) area ratio and thus reduce corrosion rates, because on exposure to corrosion environment martensite becomes cathodic while ferrite anodic to the micro-galvanic cells that are setup [21]. Stress relieving upon tempering could also account for the decrease in corrosion rate of the tempered samples.

IV. CONCLUSION

From analysis in this research it can be concluded for 0.23%C steel in 0.1M HCl aqueous solution that: step quenching intercritical annealing heat treatment increases corrosion rate; increased soaking temperature within the intercritical annealing (i.e. $\alpha + \gamma$) region increases corrosion rate; and tempering as-quenched step

quenched samples reduces corrosion susceptibility. Therefore, for applications where this material will be exposed to chloride environment, tempering after step quenching is strongly recommended.

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