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Effect of Heat Treatment on Corrosion Resistance of Mild Steel

Feyisayo V. Adams✉, Toyyibat O. Badmus, Eugene Uwiringiyimana and Odonnell P. Sylvester

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Author affiliations:

Department of Petroleum Chemistry, School of Arts and Sciences, American University of Nigeria, Lamido Zubairu Way, Yola Township by-Pass, PMB, 2250 Yola, Nigeria

✉To whom correspondence to be addressed:

E-mail: feyikayo@gmail.com

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ABSTRACT

The corrosion behavior of heat treated mild steel in 0.1 M citric acid was studied using weight loss technique. Mild steel samples were heated at 650 and 950 ºC for 30, 60 and 90 min. The effect of heat treatment time and methods of cooling (normalizing, quenching and stress relief) on the corrosion resistance of mild steel in 0.1 M citric acid solution was also studied. The experimental results showed that the corrosion rates of the heat treated mild steel increased with time of heating irrespective of the cooling method used. The corrosion rates of samples treated at 650 ºC and 950 ºC at 30 and 90 min, respectively decreased with increase in weight loss. Also, the corrosion rate of mild steel treated at 950 ºC (normalizing and quenching) for 30 min were higher than those treated at 650 ºC (normalizing and quenching) for 30 min. However, the samples cooled using stress relief method at 650 ºC showed higher corrosion rates compared to that at 950 ºC. The corrosion rates of mild steel treated at 650 ºC for 90 min, exhibited lower corrosion rate than those treated at 950 ºC for 90 min. In general, mild steels heat treated at 650 ºC using normalized method showed lowest corrosion rates compared to the other cooling methods used. Also, heat treated at 950 ºC using stress relief showed lowest corrosion rate compared to the other cooling methods used.

KEYWORDS

Corrosion, Heat treatment, Mild steel, Organic acid.

INTRODUCTION

Heat treatment is a technique used in changing the physical and chemical properties of materials. The most common heat treatment methods used for carbon steels are normalizing, tempering, annealing and hardening [1,2]. These methods have varying effects on the properties of carbon steel such as ductility, strength, microstructure and hardness [3,4]. The temperature at which the alloy is treated determines its hardness after treatment which also as a result affects its corrosion resistance [5]. Heat treatment generally involves a constant heating of the steel to a temperature less than the critical temperature of the metal [1,333 ºF (about 723 ºC)] for carbon steel [6].

Heat treating the mild steel and then cooling it in the air is known as normalization [7]. The machinability, ductility as well as the strength of mild steel strength increase by normalizing the mild steel after heating. The brittleness of ferrous alloys reduces by normalization and a uniform fine-grained structure is also obtained.

Stress relieving, on the other hand, is a heat treatment of alloys that is carried out to improve the ductility and decrease the hardness of alloys. A full stress relieving process involves heat treating the alloy at severe temperatures for about 30 to 120 min and then allowing it to cool gradually to ambient temperature in the oven [8]. Heating is done to allow the alloy reach its austenite form, while the cooling process allows the alloy to transform from austenite form to either ferrite or cementite. This whole process is done to reduce the strength of the material (tensile and yield strength) while increasing its ductility.

Heat treatment by quenching is the process in which metal parts are cooled from the austenitizing treating temperature within the range of 815 to 870 ºC (1500 to 1600 ºF) for steel [9]. Carbon steels are quenched to control the amounts of mart-ensite in their microstructure. This process results in required hard-ness, toughness or strength and then minimizes distortion, residual stress and cracking [9]. The quench medium can either be liquid or gas depending on hardenability of the alloy to be tested, shape and section thickness are also considered.

Organic acids are often used as reagents in manufacturing. They also act as a good solvent in various industrial processes and as precursors in the production of other chemicals. Citric acid is known to be the most common of all the low-carbon organic acids [10]. It is mostly in contact with mild steels than any other steel due to the wide applications of mild steels.

In the past, many researchers had investigated the corrosion behaviour of heat treated carbon steel in an acidic environment [3,11-13]. It is known that mild steel despite of its strength is a very weak corrosion resistance metal. This weakness is as a result of the non-protective nature of the corrosion products that are formed when it corrodes. A study carried out by Loto and Matanmi [11] showed that untreated NST 37-2 steel samples were less corrosion resistance in hydrochloric acid compared to heat treated (hardened, normalized annealed and tempered) NST 37-2 steel samples. Afolabi and Peleowo [12] investigated the effect of heat treatment temperature and time on the corrosion resistance of austenitic stainless steel in oxalic acid. The report by the authors showed that the corrosion rate of austenitic stainless steel increased with increase in heat treatment time and temperature [13]. Most of the work done on the corrosion resistance of heat treated mild steels were in HCl, H_2SO_4, cassava fluids, juices and other organic acids or their mixture. Also, none has considered the corrosion resistance of heat treatment of mild steels at temperatures below and above the critical temperature of mild steel. It is therefore, necessary to study the effect of the temperatures which are below and above the critical temperature of mild steel and methods of heat treatment on the corrosion resistance of mild steel. In this study, the effect of the heat treatment temperatures (below and above the critical temperature of mild steel), methods of heat treatment and time of heating on the corrosion resistance of mild steel in citric acid was investigated.

EXPERIMENTAL

Mild steel sample with the compositions presented in Table-1 was used for the studies. The dimension of the mild steel sample used was 1.8 cm × 0.9 cm and thickness of 0.9 cm. The mild steel samples were heat treated at 650 and 950 ºC for a varying period of 30, 60 and 90 min. The heated samples were normalized, stress relieved and quenched in water. The corrosion study of the heat treated samples was carried out in 0.1 M citric acid for a period of 21 days using weight loss technique. The samples were checked for a change in weight at an interval of 3 days.

TABLE-1
CHEMICAL COMPOSITIONS OF MILD STEEL

Elements	Fe	C	Mn	P	S	Si
Weight (%)	Balance	0.15	1.00	0.035	0.035	0.30

The weight change values were used to calculate corrosion rate (CR) using eqn 1:

$$\text{Corrosion rate} = \frac{87.6w}{A\rho t} \quad (1)$$

where w is the change in weight (mg), A is the surface area of mild steel used (cm^2), ρ is the density (g/cm^3) and t is time in h.

RESULTS AND DISCUSSION

Corrosion kinetics of heat treated mild steel in citric acid: Figs. 1-3 showed the samples weight change (weight loss) against time of exposure in 0.1 M citric acid solution. The results showed that heat treated mild steel samples loose weight with the increase in exposure time, which has a linear relationship, the similar observation made by Osarolube *et al.* [14]. The trend of weight loss is seen to be quenching > normalized > stress relief at both temperature (650 and 950 ºC) and 30 min treatment time. However, the trend changes at treatment time of 60 and 90 min. At 60 and 90 min of treatment, stress relief samples showed the highest weight loss, followed by

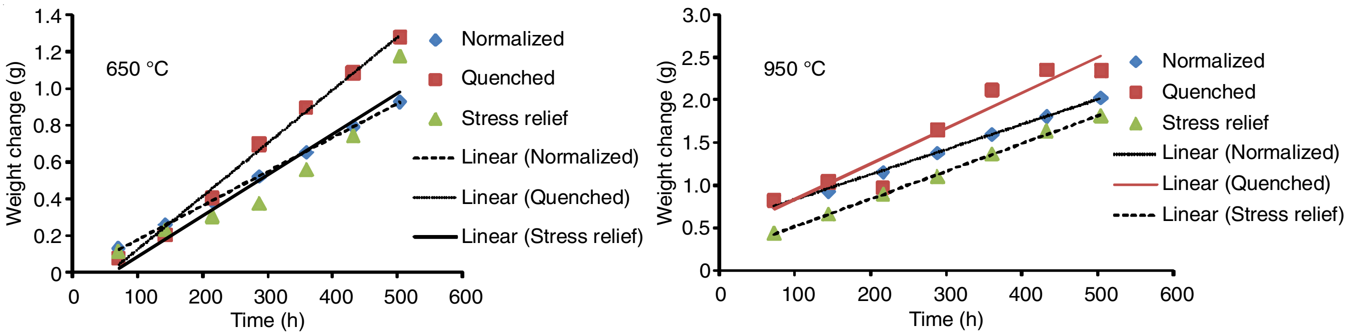

Fig. 1. Weight loss of heat treated samples for 30 min at 650 and 950 ºC

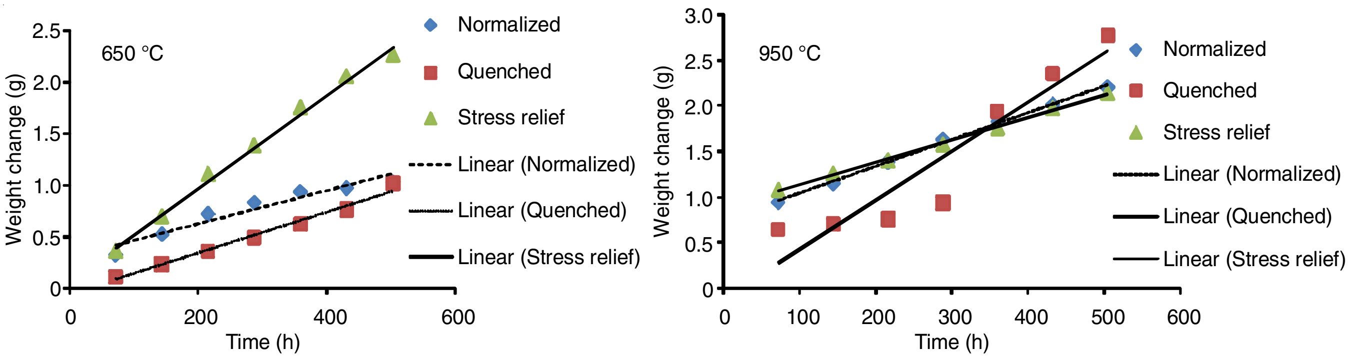

Fig. 2. Weight loss of heat treated samples for 60 min at 650 and 950 °C

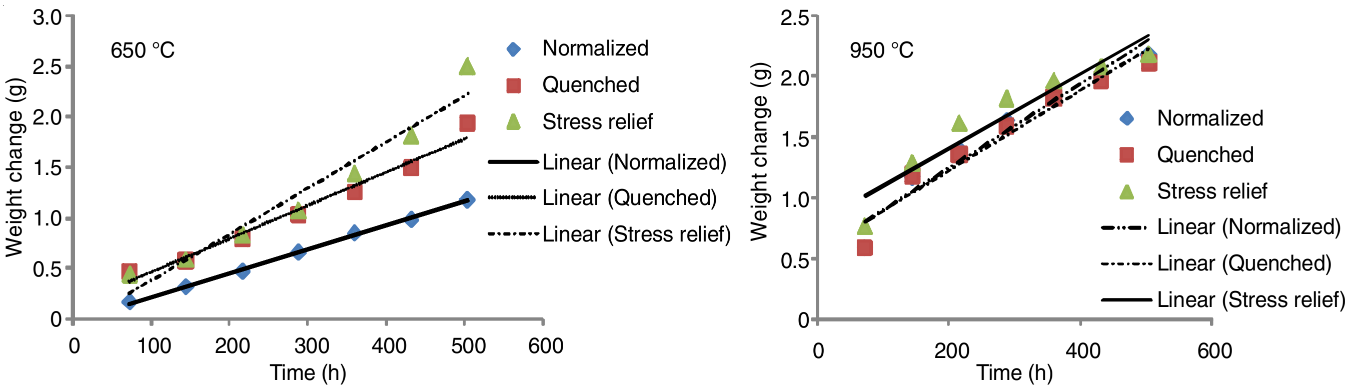

Fig. 3. Weight loss of heat treated samples for 90 min at 650 and 950 °C

normalized samples and least quenched samples, though the normalized samples and quenched samples tend to lose weight almost at the same rate.

The weight loss against time of exposure graphs were used to determine the corrosion kinetics of the heat treated mild steel samples in 0.1 M citric acid. There are three basic kinetics principles or laws that characterize the oxidation rates of pure metals; parabolic rate law (eqn. 2), logarithmic rate law (eqn. 3) and linear rate law (eqn. 4). These laws are modeled, respectively [15-18] as follows:

$$x^2 = K_p t + x_o \quad (2)$$

$$x = K_p \log (ct + b) \quad (3)$$

$$x = K_L t + x_o \quad (4)$$

where x = oxide thickness or change in weight, t = time, K_p = diffusion rate constant (directly proportional to diffusivity of ionic species that is the rate controlling step) and x_o = constant.

The kinetic studies of the heat treated mild steel samples shows a linear relationship between the changes in weight with time which indicates a first order reaction (Figs. 1-3). The linear rate law is related to the formation of non-protective oxide layers [19]. The results obtained from the weight loss tests show an increase in weight loss with time which is an indication of the formation of non-protective oxide films on the surface of the samples. This is shown from the obtained data in Table-2. The half-life ($t_{1/2}$) values were obtained [20] using the relationship given in eqn. 5:

$$t_{1/2} = \frac{0.693}{k} \quad (5)$$

TABLE-2
AVERAGE CORROSION RATE, DIFFUSION RATE CONSTANT AND HALF-LIFE ($t_{1/2}$) OF HEAT TREATED MILD STEEL SAMPLES TESTED IN 0.1 M CITRIC ACID

Temp. (°C)	Heating time (min)	Methods	K_p	$t_{1/2}$ (h)	Corrosion rate (mm/yr)
650	30	Normalized	0.001	693.0	1.3184
		Quenched	0.002	346.5	1.4892
		Stress relief	0.002	346.5	2.1930
	60	Normalized	0.001	693.0	2.2227
		Quenched	0.002	346.5	2.2613
		Stress relief	0.004	173.3	3.5137
	90	Normalized	0.002	346.5	1.6641
		Quenched	0.003	231.0	2.9686
		Stress relief	0.004	173.3	3.2026
950	30	Normalized	0.002	346.5	4.1855
		Quenched	0.004	173.3	4.6583
		Stress relief	0.003	231.0	3.1022
	60	Normalized	0.002	346.5	4.9078
		Quenched	0.005	138.6	3.8444
		Stress relief	0.002	346.5	5.1222
	90	Normalized	0.003	231.0	6.6534
		Quenched	0.003	231.0	6.5263
		Stress relief	0.003	231.0	4.9732

The values of diffusion rate constant and half-life ($t_{1/2}$) obtained from the above relations are summarized in Table-2.

According to parabolic rate law, the concentrations of diffusing species at the oxide-metal and oxide-gas interfaces are assumed to be constant with uniform and continuous oxide layer having a single phase type [19]. The linear rate and the logarithmic laws both exhibit empirical relationships, meanwhile metals with linear oxidation kinetics at a certain temperature have a propensity to undergo drastic oxidation [19].

The diffusion rate constant (K_p) generally increased with increase in time of heat treatment and temperature of treatment and is seen to lead to increase in corrosion rate of some of the heat treated mild steel samples; at higher K_p values, high corrosion rates were observed, but depending on the temperature and time.

Corrosion rates of heat treated mild steel samples in 0.1 M citric acid: The corrosion rates of heat treated mild steel samples in 0.1 M citric acid over the exposure time is shown in Fig. 4. An inverse relationship in the weight loss and corrosion rate was observed for samples heat treated for 30 min at 650 °C and 90 min at 950 °C. The corrosion rates decreased with increase in samples weight loss. Similar observation was made by Oparaodu and Okpokwasili [21].

The corrosion rate of normalized sample treated at 650 °C for 30 min was noticed to initially increased and later gradually decreased until stabilized. However, a steady increase in corrosion rate was observed for the quenched samples, which later stabilized with time. A gradual increase with an irregular behaviour was seen in the stress relieved samples.

The behaviour of heat treated mild steel samples at 650 °C for 60 min showed an initial increase followed by a gradual decrease in corrosion rate with time for normalized and stress relieved samples, while a gradual increase after an initial decrease was observed for the quenched samples. A similar behaviour was exhibited by the quenched samples at 60 and 90 min of heat treatment. The normalized samples showed more stability compared to other samples as the stress relieved samples showed similar behaviour with quenched samples at 90 min of heat treatment.

Generally, after an initial increase a rapid decrease in corrosion rate is seen in all the heating time and for all the methods of heat treatment at 950 °C. At 60 min of heat treatment, the normalized and stress relieved samples showed similar behaviour and stability, while the corrosion rate of the quenched samples decreased and later increase before stable at 360 h similar to its behaviour at 30 min of heat treatment. Meanwhile, the stress relieved samples showed more stability at 30 min of heat treatment. All the samples heat treated for 90 min exhibited similar behaviour in 0.1 M citric acid irrespective of their heat treatment method with quenched and normalized samples showing close corrosion rate.

The results showed the samples treated at 650 and 950 °C to exhibit corrosion resistance in 0.1 M citric acid in the order quenching > normalized > stress relief and stress relief > normalized > quenching, respectively for the first 100 h. Loto

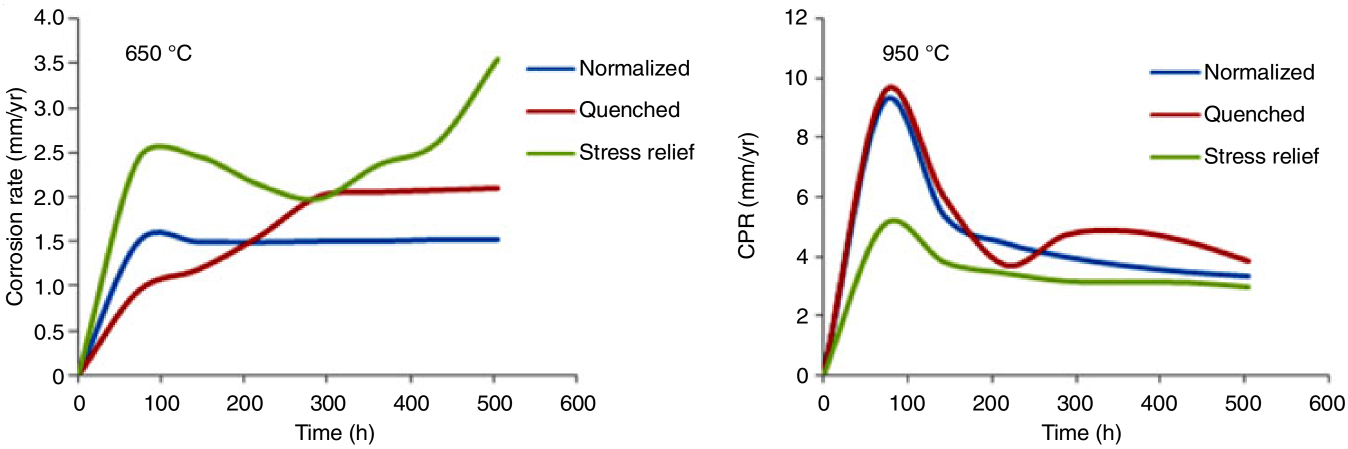

Fig. 4. Corrosion rates of heat treated samples for 30 min at 650 and 950 °C

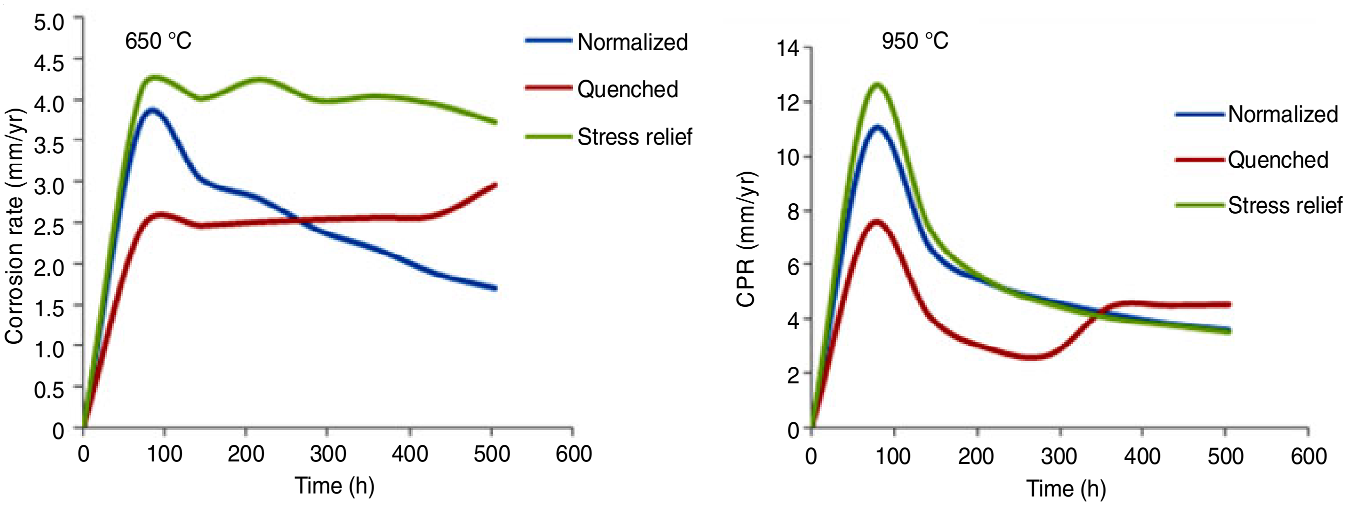

Fig. 5. Corrosion rate of heat treated samples for 60 min at 650 and 950 °C

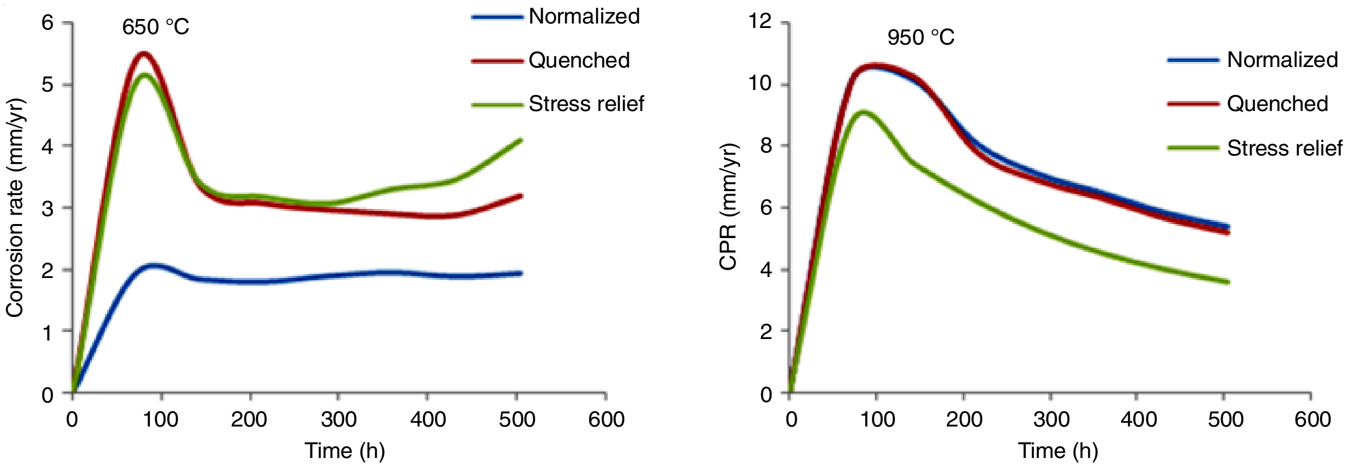

Fig. 6. Corrosion rates of heat treated samples for 90 min at 650 and 950 ºC treatment temperatures

and Matanmi [11] observed the corrosion resistance of mild steel in cassava juice to be in order of normalized > tempered > quenching, while Seidu and Kutelu [22] reported heat treated mild steel to more corrosion resitance when treated using annealing method followed by normalized and least quenching method. The normalized method of cooling found to be intermediate in corrosion resistance.

Conclusions

The corrosion behaviour of heat treated mild steel in 0.1 M citric acid was studied using weight loss technique. From the results obtained, the following conclusions are drawn:

- The corrosion rates of the heated mild steel samples generally decreased with increase in exposure time irrespective of the method of cooling used and increased with increase in heat treatment time.
- An inverse relationship was observed in samples heat treated at 650 ºC (30 min) and 950 ºC (90 min) as the corrosion rates decreased with increase in weight loss.
- The trend of corrosion rates was stress relief > quenching > normalized for all the samples treated at 650 ºC.
- The trend of corrosion rates was quenching > normalized > stress relief, stress relief > normalized > quenching and normalized > quenching > stress relief for samples treated at 950 ºC for 30, 60 and 90 min, respectively.
- At 30 min heat treatment time, the trend of weight loss was quenching > normalized > stress relief, irrespective of the heat treatment temperature. Meanwhile, the trends at 60 and 90 min were stress relief > normalized > quenching and stress relief > quenching > normalized, respectively.

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