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Wear and corrosion behavior of selected up-quenched and step-quenched CuZnSn shape memory alloys

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Abstract. The effect of thermal treatment on the wear and corrosion behavior of three categories of Cu-Zn-Sn-Fe SMAs designated A, B, and C is studied. Wear properties were investigated using a tribometer whilst corrosion in 0.3 M H₂SO₄ and 3.5%NaCl media was studied using the potentiodynamic polarization method. The microstructure of the alloys mainly consists of FCC Cu-rich phase and Cu₅Zn₈ phase. The up-quenched A alloys show the highest hardness and lowest wear rate values of 72.1 HRB and 0.143 mm³/N/m respectively. Average COF was higher for the samples subjected to direct-quenching (0.35–0.12) than the up-quenched (0.16–0.12) and step-quenched (0.2–0.08) samples. Wear occurred by mixed mode mechanisms of abrasion and adhesion evident by grooves and wear particles on the surface. In 0.3 M H₂SO₄ medium, step-quenched alloys had corrosion rates in the range of 0.1022 to 1.1705 mm/yr, which is lower than the range of 0.1466 to 0.2855 mm/yr, and 0.1730 to 0.6027 mm/yr obtained for direct quenched and up-quenched samples respectively. In 3.5% NaCl solution, step-quenched alloys had the lowest corrosion rates 0.0251 mm/yr relative to samples subjected to up-quenching and direct quenching treatment. Generally, step-quenching treatment effectively improved the corrosion resistance of alloys in both media.

Keywords: Cu-Zn-Sn alloys / thermal treatment / structural characterization / corrosion properties wear properties

1 Introduction

Cu-based SMAs are increasingly being utilized in marine, structural, and industrial applications due to their desirable combination of properties [1–7]. In the marine sector, for instance, Cu-based SMAs are now being considered for heat exchanger tubings and piping systems [8,9]. In the deep sea, Cu-based SMAs can be utilized for connecting tubes because of their superelasticity and corrosion resistance properties [10]. The corrosion behavior of marine structures is affected by sea waves, wind, temperature, pH, the conductivity of seawater, and salinity among other environmental conditions [10]. It is observed that the corrosion density of metal structures in the austenitic state is higher compared to that of SMAs in the martensitic state and this makes SMAs better candidates

for marine applications [11]. In the industrial sector, Cu-Zn-Sn SMAs have gained acceptance in vibration dampers, bearing materials, worm gear, and applications involving hydraulic coupling systems such as connector pipes, valves, and pumps [12,13]. Most of these emerging areas of their applications involve solid surfaces in sliding contact which makes it very interesting to study their wear characteristics. Surface wear is a serious issue that alters the structural dimensions, causes surface deformation, lowers the materials properties, and results in the ultimate loss of their functional properties [14]. Wear properties of Cu-based SMAs therefore, play a key role in their selection for various industrial applications such as bearings, gears, valves, and pumps [15,16]. For these reasons, there are many studies on wear mechanisms seeking to improve the wear and surface performance of Cu-based SMAs. Haleem et al. [17] studied the wear and corrosion behavior of Cu-Zn-Al-based SMAs. It is observed that the Cu-Zn-Al-based SMA modified with

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B₂O₃ up to 3 wt.% showed a considerable improvement in the wear resistance of the unmodified Cu-Zn-Al-based SMA. The wear mechanism involves the loss of material from the surfaces due to friction caused by relative interface motions. Prashantha et al. [18] showed that the wear behavior of Cu-Al-Be SMA is significantly influenced by their surface properties such as their abrasive, adhesive, and surface fatigue characteristics. Moskvichev et al. [19] studied the wear behavior of Cu-Al-Mn SMA. The coefficient of friction is observed to be sensitive to the amount of soft α -phase which is dependent on the alloy composition, particularly on Mn concentration. Ercan [13] studied the wear characteristics of Cu-Al-Ta SMA and observed that the worn surfaces are characterized by grooves, ridges, debris materials, and surface delamination. Worn tracks revealed debris of softer materials such as Cu and Al atoms without Ta. The wear behavior of Cu-Al-Ni-based SMAs [3,11,20], Cu-Zn-Al [21], Cu-Al-Mn [22], Cu-Al-Be-based SMAs [18,23,24], and Cu-Al-Fe-Ni [25] have been well studied. These studies have shown that compositional design is a key factor in determining the wear mechanism of these Cu-based SMAs. There is a paucity of literature on the wear properties of Cu-Zn-Sn-based SMAs. On the other hand, corrosion is another problem that leads to material failure and significant financial losses with an estimated global direct annual cost exceeding US\$ 1.8 trillion [26]. The utilization of Cu-Zn-Sn SMAs in marine structures and hydraulic fittings exposes them to corrosive attack in various aqueous sulfate and chloride solutions. A few authors including Miura et al. [27], Yu et al. [28], Hutchison et al. [29], Karthik et al. [30], and Kaiser et al. [31] have reported the corrosion behavior of the alloys in Cu-Zn-Sn system in various media. These studies agree that corrosion products such as SnO₂ and Cu₂O are crucial to the formation of a protective layer during the corrosion of Cu-Zn-Sn alloys. It is also noted that a Cu-rich layer is created when zinc is selectively dissolved, which increases the corrosion resistance of Cu-Zn-Sn alloys in chloride solution [28]. Papadopoulou et al. [32] reported the corrosion behavior of Cu-Zn-Sn alloy containing 0.5 wt.% Pb in various concentrations of NaCl and sulfate aqueous solutions. It is observed that corrosion begins at the Cu-rich zones which act as anodic sites while the Sn-rich phases are cathodic and experience lesser corrosion attacks. The variation in the composition of the grain structures is observed to set up localized galvanic cells which lead to the formation of corrosion products and often results in passivation. Most of the studies reported on the corrosion behavior of Cu-Zn-Sn-based alloys focused on the effect of the concentration of the corrosive media on their microstructural susceptibility to corrosion. Although the studies showed that Cu-Zn-Sn-based SMAs have good corrosion resistance, there are reports of some incidences of leakage accidents due to pitting and crevice corrosion and the issue of stress corrosion cracking which have resulted in serious economic losses [7]. Therefore their corrosion and tribological properties need to be improved and to achieve this, heat treatment procedures are an essential option.

Heat treatment is applied to SMAs to change their thermal and structural properties to make them adequately meet the demands of specific properties required in certain applications [33]. According to reports, martensite formation due to the heat treatment process enhances the corrosion resistance properties of Cu-based SMAs [9,34]. Quenching thermal procedures results in a large volume of randomly distributed vacancies in the microstructure which affects the properties of these Cu-based SMAs [35]. The effect of direct-quenching, up-quenching, and step-quenching treatments on the damping properties of Cu-Al-Mn-Ni SMAs [36], shape memory properties of Cu-Zn-Al SMAs [37], and Cu-Al-Mn SMAs [38], have been studied. Also, the effect of quenching treatment on the transformation characteristics of Cu-Al-Fe-based SMAs has been reported [33,39]. These studies have shown that up/step-quenching treatments create defects (such as dislocations and vacancies) in the microstructure and affect the orientation and mobility of martensite interfaces which in turn show diverse effects on their functional properties. Sadawy [25] studied the effect of quenching and aging treatments on the tribological characteristics of Cu-Al-Fe-Ni SMA. The findings showed that aging the alloy at 350 °C led to the production of fine structures (α and k phases) which resulted in better wear properties. The study, however, noted that the aging treatment conducted above 350 °C resulted in a decrease in the wear resistance property of the SMA. Hsu et al. [40] studied the effect of aging heat treatment on the shape memory properties of Cu-Zn-Sn SMA. It is observed that the aging treatment conducted above 285 °C considerably reduced the shape memory capacity of Cu-Zn-Sn SMA due to the precipitation of γ (Cu₅Zn₈) phase in the parent matrix. It is interesting to note that little attention has been given to the influence of up/step-quenching treatments on the corrosion susceptibility of various ranges of compositions of Cu-Zn-Sn-based alloys in diverse corrosive media. Also, the scarcity of literature that deals directly with the wear properties of Cu-Zn-Sn-based SMAs makes it indispensable to carry out this study. It is crucial to study the wear behavior of Cu-Zn-Sn-based SMAs in relation to the environmental conditions to comprehend the circumstances that may arise in service due to abrasion and prevent unexpected functional failure. As a result, the goal of this paper is to assess the effect of various thermal quenching procedures on the wear behavior and corrosion properties of Cu-Zn-Sn-based alloys in NaCl and H₂SO₄ solutions.

2 Materials and methods

2.1 Materials

The samples of Cu-Zn-Sn-Fe shape memory alloy (SMA) were prepared from pure copper (purity 99.9 wt.%), zinc (purity 99.8 wt.%), tin (purity 99.8 wt.%) and iron (purity 99.2 wt.%). These raw materials were melted in a crucible furnace and the melts were cast into stainless steel split molds to produce the Cu-Zn-Sn-based SMA ingots. The composition of the three alloy categories designated A, B,

Table 1. Composition of the alloys.

Alloy ID	Chemical composition (wt.%)			
	Cu	Zn	Sn	Fe
A	Bal.	15.64	12.12	1.05
B	Bal.	26.11	9.34	0.82
C	Bal.	29.55	8.92	0.70

and C produced is presented in Table 1. After casting, the ingots were homogenized at a temperature of 550 °C for 5 h in a muffle furnace and then water quenched. This was carried out to eliminate chemical and structural inhomogeneity in the cast samples of the alloy. The machined samples were subjected to thermal treatments to remove stresses induced in them during the machining operation. The heat treatments involved the solution treatment of the alloy samples at a temperature of 550 °C for 5 h, followed by direct quenching (DQ) in water at room temperature (designated DQ samples). Some of the quenched samples were up-quenched in hot water at 100 °C for 15 min and thereafter quenched in water at room temperature (designated UQ samples). Another set of the samples was step-quenched in water at 100 °C after solution treatment at 550 °C for 5 h and thereafter quenched in water at room temperature (designated SQ samples).

2.2 Microstructural analysis

The alloy samples were prepared to a mirror surface following a series of grinding and polishing operations. Abrasive papers made of silicon carbide with grades ranging from 220 to 2000 grits were used to accomplish the grinding operation by, following the procedures stipulated in Alaneme et al. [41]. The samples were then polished using Al₂O₃ powder and etched by swabbing for 20 s using a solution made up of 5 g ferric chloride, 10 ml HCl, and 95 ml ethanol. Scanning electron microscope (SEM) was used for microstructural analysis and the micrographs were taken in backscattered electron (BSE) mode using a Thermo Scientific HeliosG4 PFIB UXe DualBeam FIB/SEM with fixtures for energy dispersive spectroscopy (EDS). SEM-EDS measurements were done in the same microscope with an EDAX EDS system and used to analyze the composition of the samples. The grain size and volume fraction of the phases were determined using the IMAGE-J software.

2.3 Wear test

The wear properties of the Cu-Zn-Sn-based SMAs were determined using a CETR UMT-2 model Anton Paar Tribometer. Before each wear test, the wear samples were polished, washed with acetone, and dried. Each of the test samples having a diameter of 16 mm and a length of 6 mm was held in position while the Tribometer arm that grips the counter face material (acting as the ball) was lowered onto the test sample (acting as a disc) to create friction. The counter face material is an EN-32 case-hardened steel with a hardness of 65HRC and a diameter of 165 mm. The

wear test was carried out following the procedure and standard stipulated by ASTM G99-17 (2017) Standard [42]. The wear test conditions were set to a nominal load of 4 N at a linear speed of 10 cm/sec and a sliding distance of 40 m. The readings of the wear rate and coefficient of friction (COF) with time are recorded for each test sample. To ensure reproducibility, the test was run at least three times on each specimen under the same testing conditions. The wear volume was determined using Archard's equation as stipulated by Sadawy [25]:

$$V = \frac{K.W.S}{H}, \quad (1)$$

where V is the wear volume, K is the wear coefficient, W is the applied normal load, S is the sliding distance and H is the hardness of the abraded material. The images of the worn tracks of the Cu-Zn-Sn-Fe samples were analyzed using a Carl Zeiss Sigma field emission scanning electron microscope.

2.4 Hardness test

Hardness was determined using the LEEB Rockwell hardness test apparatus (model SKD-552) following the procedure and standard stipulated by ASTM E18-20 Standard [43]. Before each hardness test, the samples were polished to obtain a smooth and plane parallel surface following standard procedures. The hardness test was carried out by applying a load of 100 kgf for 10 s. Six repeat test measurements were carried out on each sample and the average hardness value was used as the final result.

2.5 Corrosion test

Autolab Nova 2.1 Potentiostat was used for the corrosion analysis of the Cu-Zn-Sn-based alloys and the test was conducted by, following the ASTM G59-97 [44] standard. The corrosion test was conducted at room temperature (25 °C) with saturated silver/silver chloride serving as the reference electrode while the test sample serves as the working electrode. The Platinum electrode was used as the counter electrode for the corrosion cell. The surface of each corrosion test sample was ground using silicon carbide abrasive papers of 220, 800, 1000, 1200, and 2000 grits, polished with Al₂O₃ powder suspension, washed in ethanol solution, and dried [45]. The prepared surfaces of mounted samples were immersed in the corrosion cell which contains the corrosive solutions. The specimens were scanned with the Versastat equipment in potentiostatic mode at a scan rate of 1.0 mV/s while the potentiodynamic polarization measurements were carried out at a potential initiated at −200 mV to +250 mV. The test was carried out in two different media (0.3M H₂SO₄ and 3.5% NaCl solutions) serving as electrolytes in each case. Polarization curves were generated from the Autolab Nova 2.1 Potentiostat from which the corrosion potential (E_{corr}) and corrosion current density (i_{corr}) were determined through the analysis of the Tafel extrapolation of the anodic and cathodic curves. Each experiment was repeated twice to check the reproducibility of the results and there were no

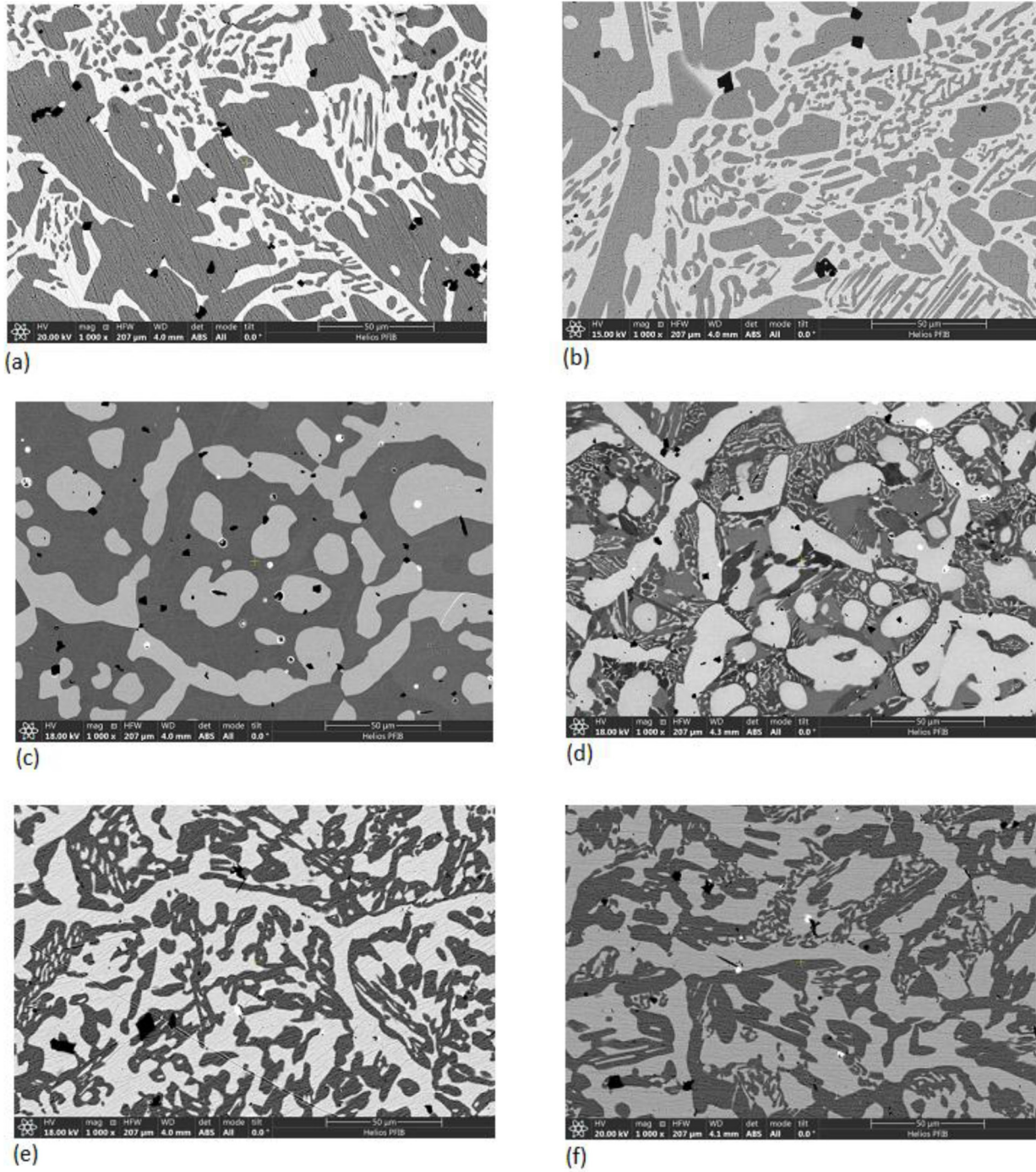


Fig. 1. Representative SEM micrographs of the: (a) up-quenched A alloy (b) step-quenched A alloy (c) up-quenched B alloy (d) step-quenched B alloy (e) up-quenched C alloy (f) step quenched C alloy.

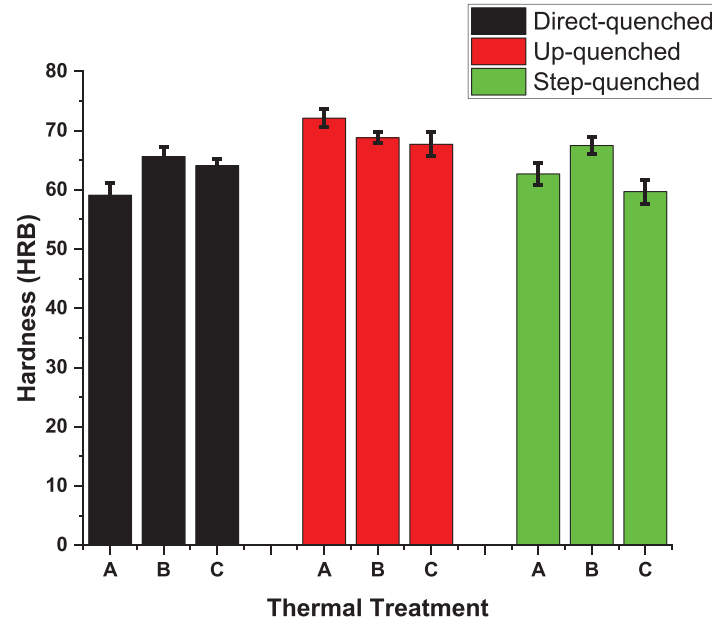


Fig. 2. The effect of thermal treatment on the hardness of Cu-Zn-Sn-based alloys.

significant variances in the repeat test results. The corrosion rates (CR) and the polarization resistance was determined using the ASTM G59-97 equation [44]:

$$CR = 3.27 \times 10^{-3} \cdot i_{corr} \cdot \frac{E_w}{\rho}, \quad (2)$$

where i_{corr} is the corrosion current density in $\mu\text{A}/\text{cm}^2$, E_w is the equivalent weight of the corroding sample in grams, and ρ is the density of the corroding sample in g/cm^3 .

3 Results and discussion

3.1 Microstructural characterization of Cu-Zn-Sn-based alloys

Figure 1 shows representative SEM micrographs of Cu-Zn-Sn-based alloys at various thermal treatment conditions. The microstructures consist of two different phase regions. The composition of the dark phase is FCC Cu while the bright phase is determined to be $\gamma\text{-Cu}_5\text{Zn}_8$. Varying degree of dispersed precipitate phases is also observed having white patches of Sn-rich precipitates and black layers of Fe-rich precipitates. Figure 1a shows the BSE micrographs of the up-quenched A alloy sample. The microstructure has a dendritic character with some Sn-rich (Cu_3Sn) and Fe-rich (Fe_4) phases which precipitate from the parent phase during up-quenching treatment. Figure 1b represents the BSE image of the step-quenched A alloy. The microstructure is similar to those of the up-quenched A alloy (Fig. 1a) except that the grains had dendritic and lamellar character. The average grain size of the step-quenched A sample is determined to be $17.06 \mu\text{m}$ which is smaller compared to $24.08 \mu\text{m}$ obtained for the up-quenched A samples. The BSE

micrograph of the up-quenched B alloy is shown in Figure 1c. The microstructure consists of globular grains with an average grain size of $66.94 \mu\text{m}$ which is greater compared to $34.20 \mu\text{m}$ obtained for step-quenched B samples. Some Cu_3Sn and Fe_7Zn_3 precipitates were observed. The microstructure of the step-quenched B alloy presented in Figure 1d showed similarity with those of the up-quenched B samples but the grains are elongated and contain fine dendritic structures with Cu_3Sn and Fe_4Zn_9 intermetallic second phases. Figures 1e and 1f represent the up-quenched C and step-quenched C samples respectively. They show similar features with fine dendritic structures but their grain morphologies are quite different. The grains in the up-quenched C alloy are well-delineated and contain some Cu_3Sn and Fe_7Zn_3 precipitates. On the other hand, the step-quenched C samples contain Cu_3Sn and Fe_4Zn_9 precipitates within and around the grain boundaries. The average grain size of the up-quenched and step-quenched C samples is determined to be $16.78 \mu\text{m}$ and $18.15 \mu\text{m}$ respectively.

3.2 Hardness test results of Cu-Zn-Sn-based SMAs

The hardness test results of Cu-Zn-Sn-based SMAs subjected to various thermal treatment procedures are presented in Figure 2. It is observed that up-quenched alloys showed higher hardness values compared to the direct-quenched and the step-quenched samples for all alloy categories. This observation is true as it is known from the literature that up-quenching treatments induce precipitation of the second phase in the parent matrix [38,46]. These precipitates (which act as barriers to dislocation movement) account for the increased hardness of the up-quenched samples. The direct-quenched and step-quenched samples were observed to exhibit similar

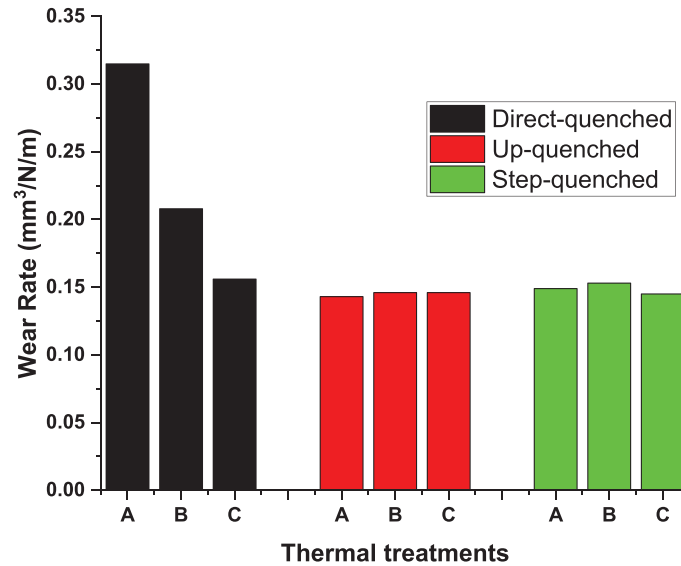


Fig. 3. Effect of the thermal treatments on the wear rate of the various Cu-Zn-Sn-based alloys.

Table 2. Wear test results of the various alloy categories at various thermal treatments.

Thermal treatments/ Alloy categories	Direct quenching (DQ)			Up-quenching (UQ)			Step-quenching (SQ)		
	A _{DQ}	B _{DQ}	C _{DQ}	A _{UQ}	B _{UQ}	C _{UQ}	A _{SQ}	B _{SQ}	C _{SQ}
Wear volume (mm ⁻³)	50.40	33.28	24.96	22.88	23.36	23.36	23.84	24.48	23.20

hardness behavior. As can be seen from Figure 2, there is a marginal difference in hardness values for the step-quenched and direct-quenched samples except for C alloys. Among all the three alloy categories tested, the up-quenched A alloy samples show the highest hardness value of 72.1HRB. This is attributed to the presence of Fe₄ phase and relatively finer grain structures in line with the Hall-Petch relation [47].

3.3 Wear behavior of Cu-Zn-Sn-based SMAs

The effect of the various quenching treatment processes on the wear rate of Cu-Zn-Sn-based SMAs is presented in Figure 3. The up-quenched and step-quenched samples of A, B, and C alloys exhibited similar wear properties with wear rates in the range of 0.143 to 0.153 mm³/N/m which are considerably lower when compared to 0.315, 0.208, and 0.156 mm³/N/m obtained for direct-quenched A, B, and C alloys respectively. The result showed that there is a marginal difference in wear resistance for the up-quenched and step-quenched samples. The alloy samples that were subjected to up-quenching (UQ) and step-quenching (SQ) treatments had better wear resistance properties compared to the direct-quenched (DQ) samples. Thermal treatments such as up-quenching and step-quenching have been reported to reduce the quench-in vacancies of Cu-based SMAs [38,48]. This suggests that up/step quenched alloys developed similar microstructural characteristics which are

more resistant to wear than the direct quenched alloys. This assertion is in line with the reports of many authors including [25,36–38] who agreed that up/step quenching treatments alter the microstructures (by affecting their vacancy concentration, grain morphology, martensite orientations, and precipitation behavior) of Cu-based SMAs. The wear test results presented in Table 2 show the wear volume of A, B, and C alloys at various heat treatment conditions. It is observed that the volume loss of material due to wear for the up/step-quenched samples is lesser compared to those of the direct-quenched samples. For instance, the wear volume of the alloy A sample decreased from 50.40 mm³ (obtained for the direct-quenched samples) to 22.88 mm³ and 23.84 mm³ obtained for the up-quenched and step-quenched A samples respectively. The main mechanism of material loss during the sliding motion at interfaces is abrasion. In comparison to B and C alloy samples, the direct-quenched A samples showed the greatest volume of wear. The reason is due to a lesser resistance to plastic deformation during interface motion which is created by the weak cohesive bond in their crystalline structures [17]. Also, the relatively greater average grain size of the direct-quenched samples validates the reason for their greater loss of material by abrasion mechanisms. This result is in accordance with the observations of Sadawy [25] where it was confirmed that fine grain structures improve the wear properties of a Cu-based SMA. The variations of coefficient of friction

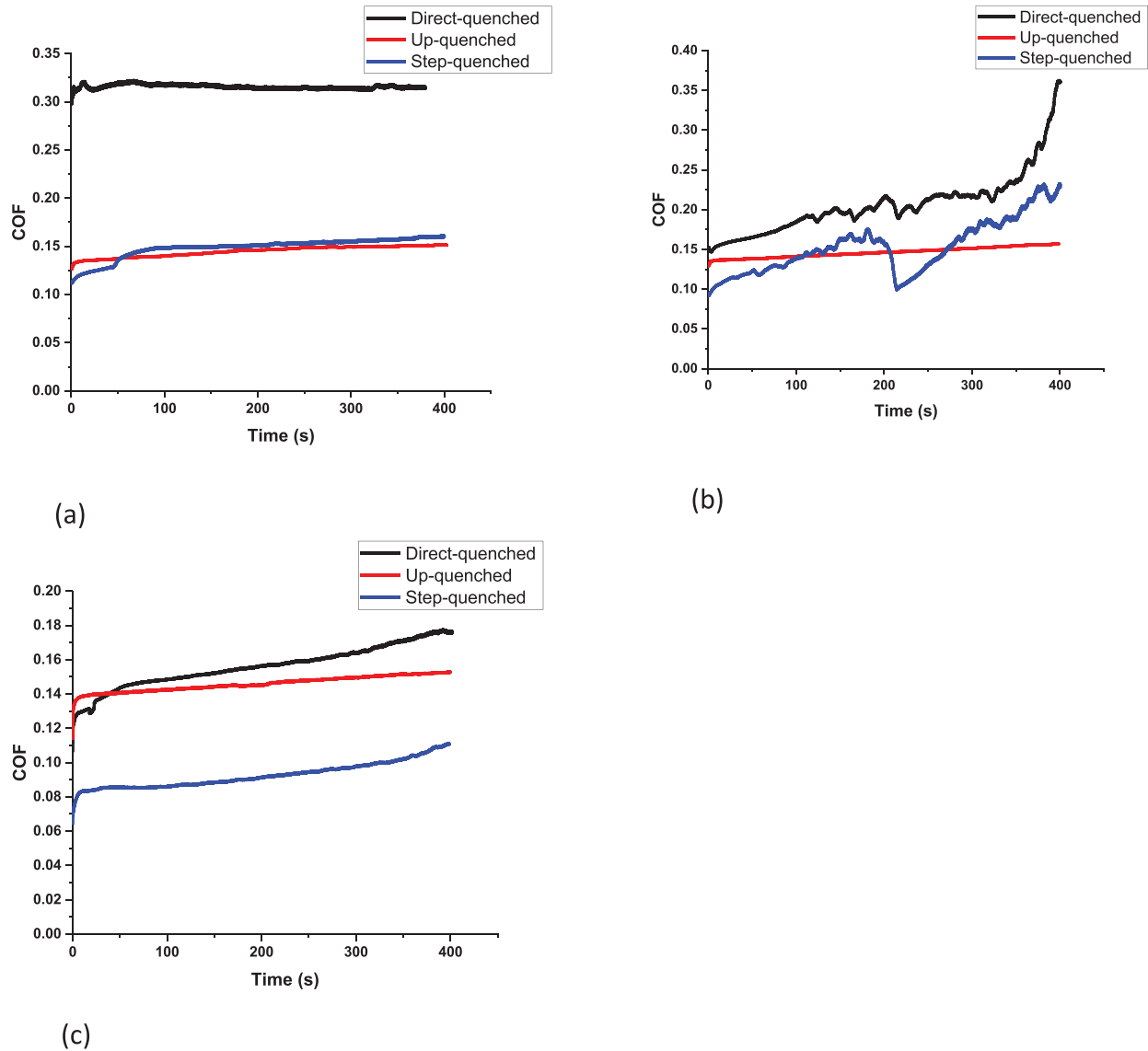


Fig. 4. The variations of COF as a function of time for Cu-Zn-Sn-based SMAs for: (a) A alloy category (b) B alloy category (c) C alloy category.

(COF) as a function of time for the Cu-Zn-Sn-based SMAs are presented in Figure 4. Each alloy exhibits a behavior that is different from the other. It is observed from Figure 4a that the wear trend for A alloys showed an initial sharp increase in COF, followed by a gradual but progressive rise as sliding time further increases. Although the direct-quenched samples exhibited very high COF values in the order of 0.30 to 0.33 which is far greater than the range of 0.10 to 0.15 obtained for the step-quenched and direct-quenched samples, the same trend is observed in the direct-quenched, up-quenched, and step-quenched samples. The COF–time plots show a trend of serrated wear behavior at the initial wear stage followed by a regime of stable behavior. The instability of the wear behavior is attributed to the acclimation that occurs as the material starts to wear [13]. The COF–time curves for B alloy samples are presented in Figure 4b. The direct-quenched B samples show the greatest COF values in the

range of 0.15 to 0.35 which is higher than the range of 0.08 to 0.20 and 0.12 to 0.16 obtained for the step-quenched and up-quenched samples respectively. The COF wear trend for the direct-quenched and step-quenched samples of B alloy is characterized by an initial sharp increase in COF, followed by a regime of irregular peaks and wear fluctuations as sliding time further increases (Fig. 4b). The initial sharp rise in COF with little contact time is due to the stick-slip wear behavior which occurs at the initial wear stage [13]. The fluctuations and irregular peaks in COF values with contact time during friction are attributed to the presence of bumps, cracks, and interfacial precipitates [13], and this account for the varied friction behavior in each of the alloy categories. The up-quenched B samples exhibit a trend of an initial sharp rise in COF, followed by a regime of slow but steady rise with the increase in sliding time. A similar trend is observed in C alloy samples as seen in Figure 4c. The

direct-quenched samples showed the highest COF values (0.12 to 0.18) compared to the up-quenched (0.12 to 0.15) and step-quenched (0.07 to 0.10) samples. The higher COF exhibited by the direct-quenched alloys could be attributed to a high volume of residual quenched-in vacancies which can prevent the movement of martensitic interfaces and create friction [38]. There is a marginal difference in the COF values for the step-quenched and up-quenched samples except for C alloy where the step-quenched samples showed very low COF values. The reason for this is not clear but may be linked to the alloy composition (higher Zn concentration). The wear behavior of the direct-quenched and step-quenched C samples exhibits a similar trend that is characterized by an initial notable rise in COF, followed by a regime of gradual increase with sliding time. In the up-quenched samples, the notable rise in COF observed at the initial stage is marked with instability which is followed by a stable region as the sliding time further increases. Based on the composition of the alloys, A samples exhibit the highest values of COF, followed by B alloys while C alloy categories had the lowest COF values. The high COF values observed for the A alloy may be linked to the presence of hard Fe_4 particles which were not found in B and C alloys. C alloys had the least amount of the precipitate phases and relatively finer average grain structures and this may be responsible for their lower COF values. This result is by those reported by Haleem et al. [17], Moskvichev et al. [19], and Mohan et al. [22] where it was established that wear properties of Cu-based SMAs are sensitive to the alloy composition and microstructures. The study thus concludes that the type of quenching treatment and alloy composition had an impact on wear performance since samples with a soft phase had a lower CoF.

3.4 Discussion on wear mechanisms

Representative SEM images of the worn surfaces of Cu-Zn-Sn-based SMAs at various heat treatment conditions are presented in Figure 5. Critical examinations of the worn surfaces showed that wear occurred in the test samples by mixing wear modes of abrasive and adhesive mechanisms, with abrasive wear being the dominant wear mechanisms. In literature, abrasive wear is known to occur when hard particles slide under load against a relatively softer surface leading to the formation of various dimensions of wear grooves on the test sample [49]. Figure 5a represents the wear tracks of the direct-quenched A alloy sample. Heavy wear is observed in this sample with features of various degrees of rough and deep wear grooves showing the dominance of the abrasive wear mechanism. Some features of adhered shear particles were also observed which indicate the occurrence of adhesive wear mechanism. The worn surface of the A alloy samples subjected to up-quenching treatment is presented in Figure 5b. The material exhibited high wear resistance as the worn surfaces were characterized by shallow fine grooves indicating the occurrence of an abrasive wear mechanism. An adhesive wear mechanism is also observed due to the

presence of some adhering debris of the soft Cu phase which acts as lubricant thereby obstructing further wearing of the surfaces. Figure 5d shows that wear occurred in the up-quenched B alloy samples via an abrasive mechanism. The worn surface is characterized by wear grooves which were rougher compared to those of the up-quenched A alloy samples. The worn surfaces shown in Figures 5c and 5e represent the wear tracks for the step-quenched A alloy and step-quenched B alloy samples respectively. Wear occurred in these alloys by mixing modes of abrasive and adhesive wear mechanisms. The worn surfaces of these alloys exhibit shallow but rough wear grooves with the dispersion of adhering brittle Cu_5Zn_8 particles which scratch the surface and aid further wearing and thus show slightly less resistance to wear compared to the up-quenched B alloy samples. Figure 5f shows the micrograph of the worn surfaces of the direct-quenched C alloy sample. The ploughing movement created grooves and scratches on the surface, which can be observed. The grooves are rough but not as deep as those found in direct-quenched A samples. The grooves are indicative of the dominance of the abrasive wear mechanism.

3.5 Corrosion properties of Cu-Zn-Sn-based SMAs

An electrochemical test for the alloy samples was carried out to determine the corrosion rate (mmpy) and to evaluate the effect of heat treatment on the corrosion resistance of Cu-Zn-Sn-based SMAs. The polarization curves obtained from the potentiodynamic tests for A, B, and C alloy categories in 0.3M H_2SO_4 and 3.5%NaCl media are presented in Figures 6 and 7 respectively. The polarization curves presented in Figure 6a for A alloys subjected to various quenching procedures showed similarities in polarization behavior. It is observed that the corrosion density of the up-quenched samples (143 $\mu\text{A}/\text{cm}^2$) is the highest which is followed by that of the direct-quenched samples (24.6 $\mu\text{A}/\text{cm}^2$) while the step-quenched samples had the least corrosion density (10.2 $\mu\text{A}/\text{cm}^2$). Consequently, the step-quenched samples exhibited better corrosion resistance properties with lower corrosion rates of 0.1181 mm/yr compared to 0.6027 mm/yr and 0.2855 mm/yr obtained for the up-quenched and direct-quenched samples respectively. The high corrosion rates observed for the up-quenched samples are attributed to their higher volume fraction of the precipitate phases which set up galvanic cells and accelerate the rate of corrosion. This result agrees with the observations reported by Papadopoulou et al. [32] where it was stated that variation in the composition of the grain structures promotes galvanic corrosion of Cu-Zn-Sn SMAs. The corrosion properties of Cu-Zn-Sn-based alloys in 0.3H $_2$ SO $_4$ corrosive medium are presented in Table 3. It can be seen that the direct-quenched and particularly step-quenched A samples show a greater tendency for thermodynamic stability due to their higher polarization resistance of 657.5 Ω and 1857.2 Ω respectively, compared to 144 Ω obtained for the up-quenched A samples. Similar trends are observed in Figure 6b which represent the polarization curves for B alloys subjected to direct-quenching, up-quenching, and

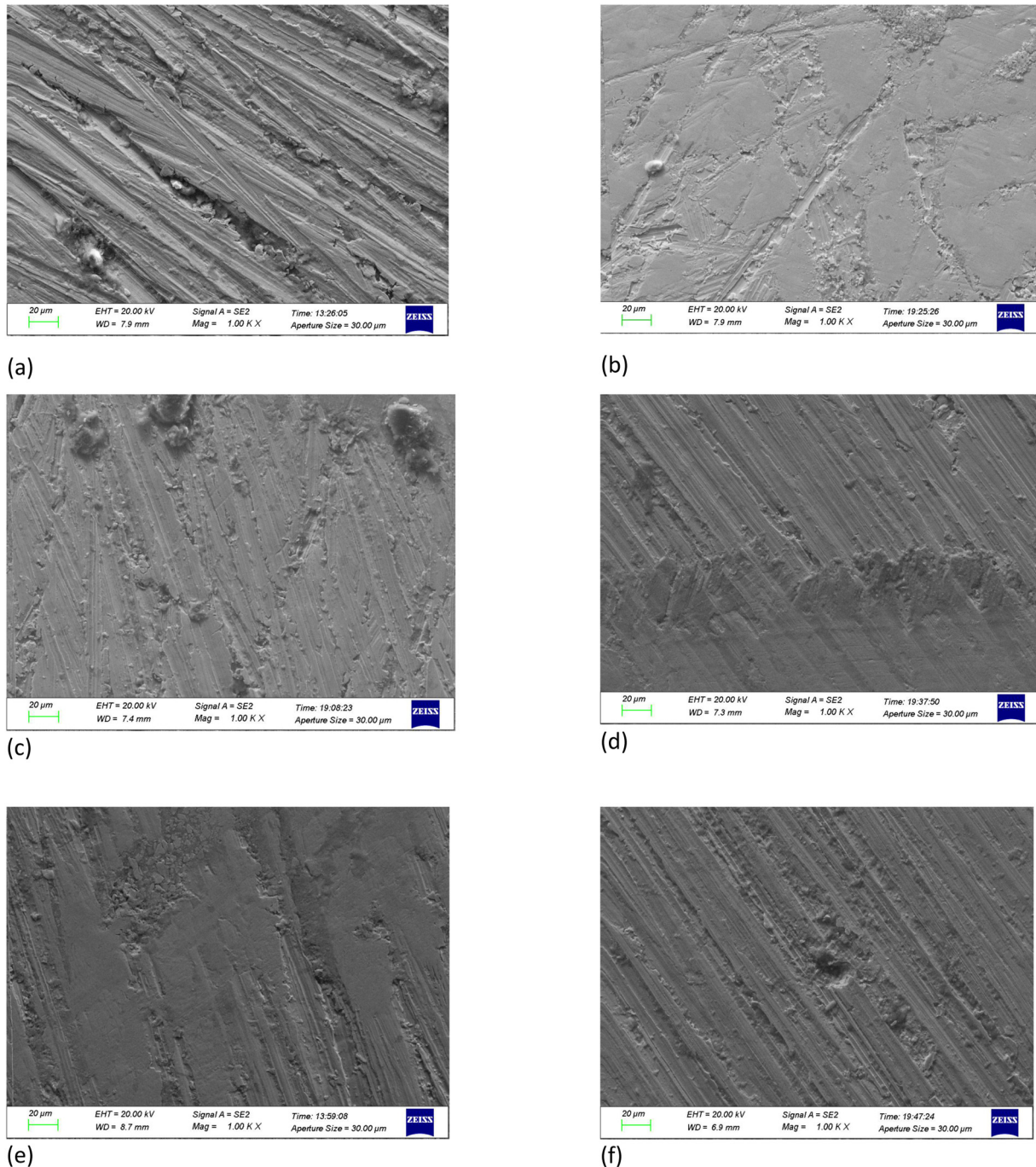


Fig. 5. Representative SEM images of the worn surfaces of the Cu-Zn-Sn-based SMAAs (a) Direct-quenched A alloy sample (b) Up-quenched A alloy sample, (c) Step-quenched A alloy sample (d) Up-quenched B alloy sample (e) Step-quenched B alloy sample (f) Direct-quenched C alloy.

step-quenching treatments. The corrosion rate of the step-quenched samples of B alloys is 0.1022 mm/yr against 0.1730 mm/yr and 0.1466 mm/yr obtained for up-quenched and direct-quenched samples respectively. The reason is due to the lower current density of $8.8 \mu\text{A}/\text{cm}^2$ obtained for the step-quenched sample compared to $12.6 \mu\text{A}/\text{cm}^2$ and $14.9 \mu\text{A}/\text{cm}^2$ obtained for the direct-quenched and up-quenched samples (Tab. 3). The polarization curves of C alloys presented in Figure 6c are

similar to those of A and B alloys. The up-quenched samples exhibited the highest current density of $28.2 \mu\text{A}/\text{cm}^2$, followed by the direct-quenched samples ($19.3 \mu\text{A}/\text{cm}^2$) while the step-quenched samples which had the least current density of $14.7 \mu\text{A}/\text{cm}^2$ exhibited superior corrosion properties. The result suggests that the intensity of current density and polarization potential are associated with the corrosion rate and passivation process respectively. The corrosion density is observed to increase with the

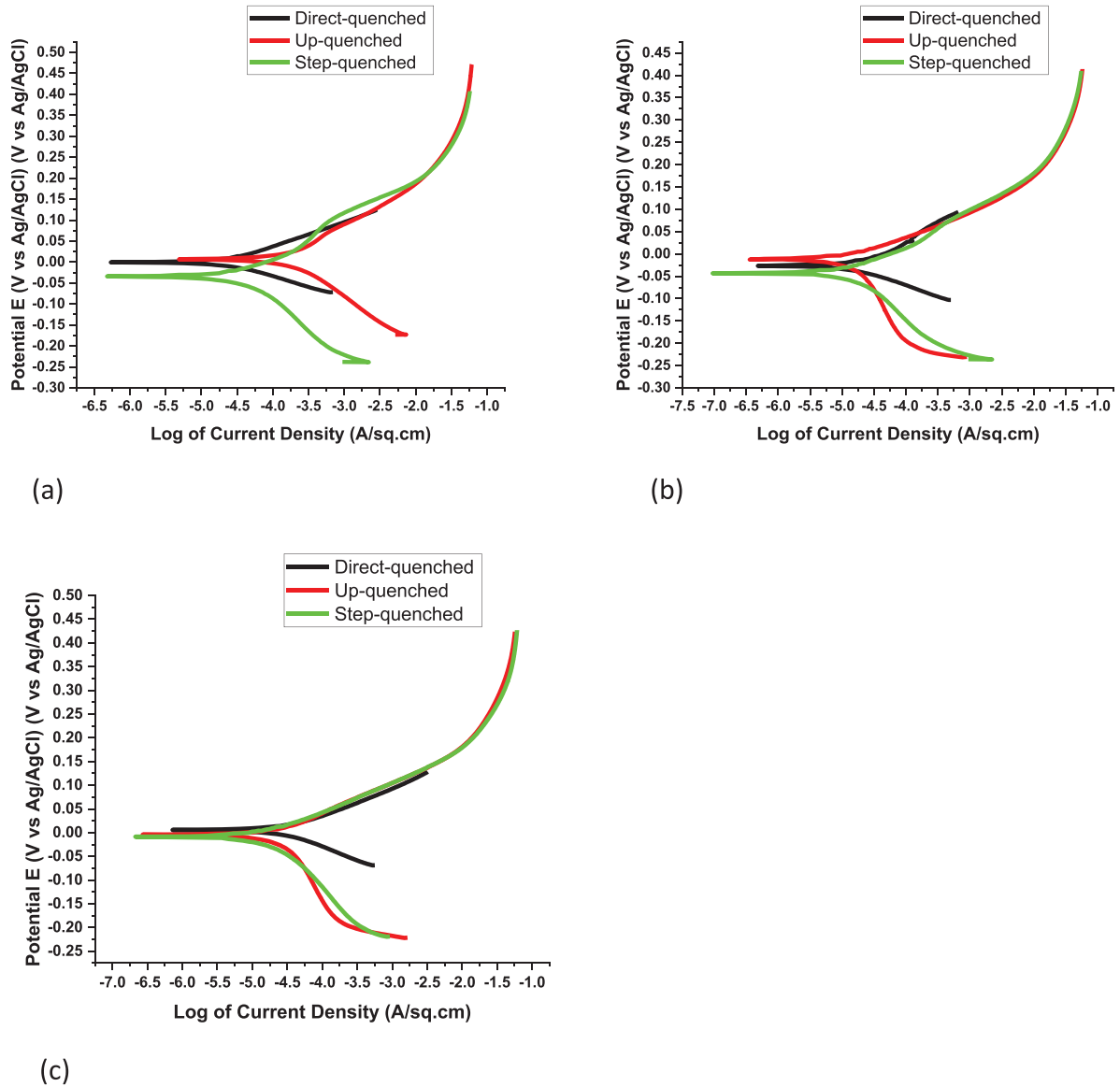


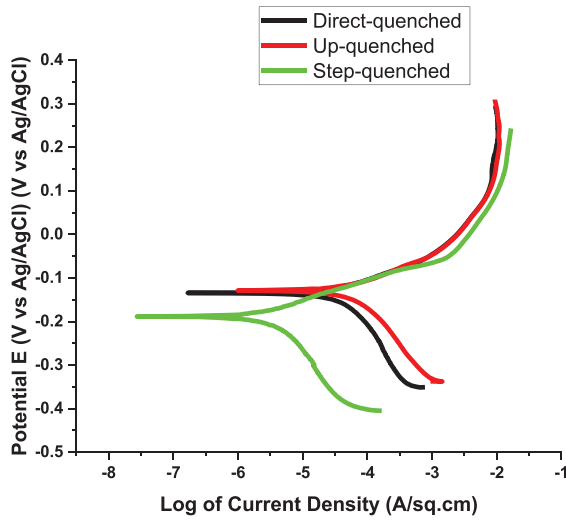
Fig. 6. Polarization curves of the various alloy compositions in 0.3M H₂SO₄ medium for: (a) A alloys (b) B alloys and (c) C alloys.

corrosion rate. The passivation effect, on the other hand, is attributed to the formation of a protective layer generated by corrosion products mainly SnO₂ and Cu₂O compounds in the corrosive medium [28,50]. Step-quenched C samples exhibited very high polarization resistance (1190.6 Ω) relative to the direct (1009.1 Ω) and up-quenched (717.6 Ω) samples. The corrosion rates irrespective of the alloy composition follow a trend of step-quenched samples showing more corrosion resistance properties followed by the direct-quenched samples while the up-quenched samples exhibited the least corrosion resistance properties. The high corrosion resistance observed in the step-quenched samples may be linked to their relatively finer grain structures when compared with those of the up-quenched and direct-quenched samples. This observation is confirmed in the report presented by Adnan et al. [51], where it was stated that the corrosion resistance of Cu-based SMAs increases considerably by reducing their grain structures. It can thus

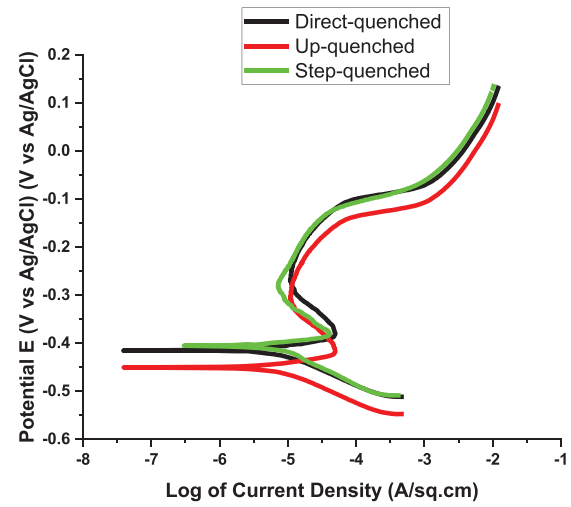
be inferred that the samples that were subjected to step-quenching developed microstructural characteristics that are not susceptible to corrosion in H₂SO₄ solution because they retain martensitic structures have lesser quenched-in vacancies and possess fine grain structures [9,38]. Figures 7a–7c shows the corrosion polarization curves in 3.5% NaCl medium for A, B, and C alloys. The E_{corr} versus I_{corr} curves for A alloys are presented in Figure 7a. Regarding the step-quenched and direct-quenched samples, it is observed that the up-quenched samples had the highest current density and consequently a greater tendency to corrode in 3.5% NaCl with a corrosion rate of 0.4268 mm/yr. The step-quenched and direct-quenched samples showed higher thermodynamic stability and lesser tendency to corrode in 3.5% NaCl solution with corrosion rates of 0.0251 mm/yr and 0.2682 mm/yr respectively. Table 3 shows the corrosion properties of Cu-Zn-Sn-based alloys in 3.5% NaCl medium. It is observed that the current density of the step-quenched A

Table 3. Corrosion properties of Cu-Zn-Sn-based alloys in 0.3H₂SO₄ and 3.5% NaCl media.

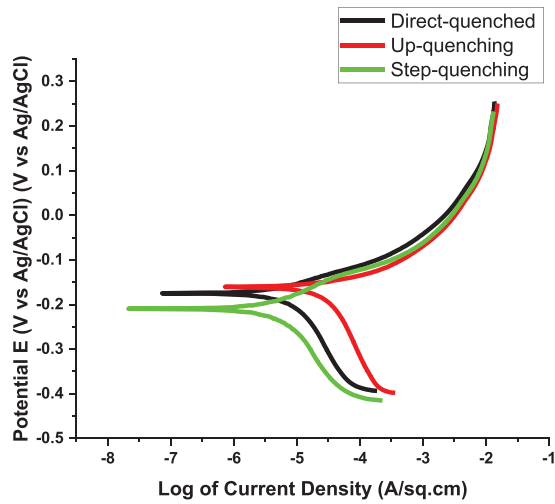
Alloy samples / Thermal treatments	Current density ($\mu\text{A}/\text{cm}^2$)		Polarization resistance (Ω)	
	0.3H ₂ SO ₄	3.5% NaCl	0.3H ₂ SO ₄	3.5% NaCl
Direct-quenched A alloy	24.6	23.1	657.5	606.2
Up-quenched A alloy	143	36.7	144.9	401.5
Step-quenched A alloy	10.2	2.16	1857.2	608.5
Direct-quenched B alloy	12.6	3.98	1501.2	1444.6
Up-quenched B alloy	14.9	4.18	1310.5	1163.3
Step-quenched B alloy	8.79	3.04	2102.9	1575.7
Direct-quenched C alloy	19.3	2.25	1009.1	1942.7
Up-quenched C alloy	28.2	30.6	717.6	630.6
Step-quenched C alloy	14.6	2.25	1190.6	1792.2



(a)



(b)



(c)

Fig. 7. Polarization curves of the various alloy compositions in 3.5% NaCl medium for: (a) A alloys (b) B alloys and (c) C alloys.

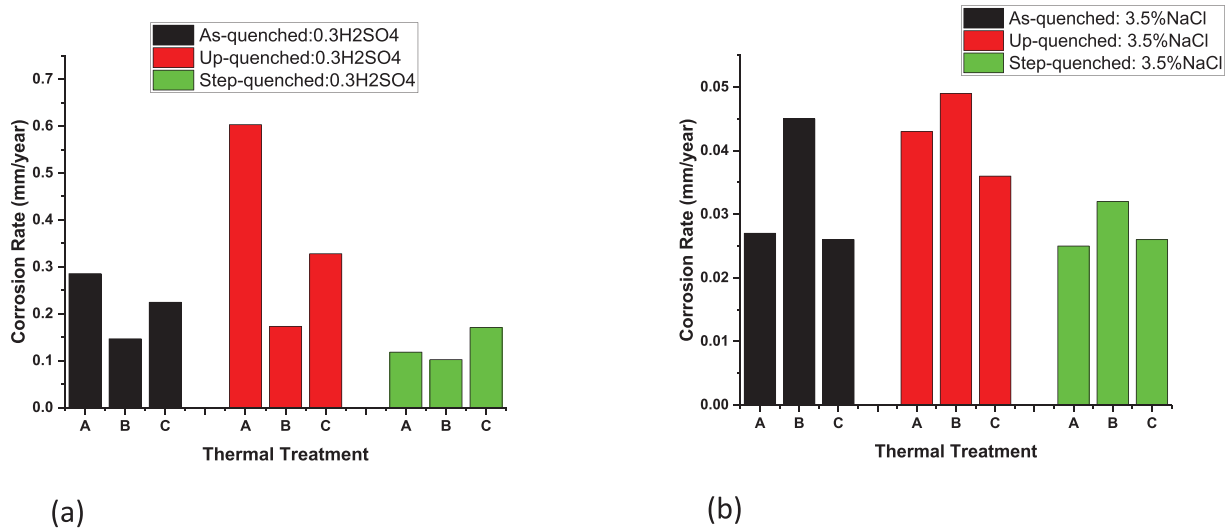


Fig. 8. Effect of heat treatments on the corrosion rate of various compositions of Cu-Zn-Sn-based SMAs in (a) 0.3M H₂SO₄ solution (b) 3.5% NaCl solution.

sample is $2.16 \mu\text{A}/\text{cm}^2$ which is lesser compared with $36.7 \mu\text{A}/\text{cm}^2$ and $23.1 \mu\text{A}/\text{cm}^2$ obtained for the up-quenched and direct-quenched samples. The polarization curves of B alloys presented in Figure 7b are dissimilar to those of A alloys in terms of their polarization behavior. The pseudo-passive zone, which follows the Tafel region of the anodic branches of polarization curves, is where the anodic current densities show a slight decrease due to the formation of soluble corrosion products on the surface of the samples [45]. In corrosion studies of Cu-Al-Mn SMAs by Vrsalović et al., [45] and Cu-Zn-Ni SMAs by [34], similar anodic behavior has been observed. There is a marginal difference in corrosion density for the up-quenched ($4.18 \mu\text{A}/\text{cm}^2$) and direct-quenched ($3.98 \mu\text{A}/\text{cm}^2$) samples while the step-quenched samples had the least corrosion density ($3.04 \mu\text{A}/\text{cm}^2$). It is observed that B alloys subjected to direct-quenching and up-quenching treatments exhibited similar corrosion rates of 0.046 mm/yr and 0.048 mm/yr respectively, while the step-quenched samples exhibited the least corrosion rate of 0.033 mm/yr . The polarization curves of C alloys presented in Figure 7c are similar to those of A alloys in terms of their polarization behavior. The step-quenched and direct-quenched samples exhibited excellent corrosion resistance properties with lower corrosion rates of 0.0261 mm/yr compared to 0.0356 mm/yr obtained for the up-quenched samples. The reason is due to the higher current density of the up-quenched C alloy ($30.6 \mu\text{A}/\text{cm}^2$) compared to $2.25 \mu\text{A}/\text{cm}^2$ obtained for the step-quenched and direct-quenched samples. This implies that the up-quenched samples developed a microstructure that is susceptible to corrosion in 3.5% NaCl whereas the step-quenched samples have lower reaction kinetics and therefore lesser propensity to corrode in 3.5% NaCl solution.

Figure 8 summarizes the corrosion behavior of the alloys in 0.3M H₂SO₄ and 3.5% NaCl solutions. The variation in corrosion rates showed that the corrosion behavior of Cu-Zn-Sn-based SMAs is sensitive to thermal treatments and the composition of the alloys. It observed

that the alloys are more susceptible to corrosion in 0.3M H₂SO₄ medium than in 3.5% NaCl solutions. It is observed from Figure 8a that step-quenching treatments are effective means of tailoring the corrosion resistance properties of Cu-Zn-Sn-based SMAs in 0.3M H₂SO₄ solutions. It is observed that the trend with regard to increasing corrosion resistance is given as A, C, and B. The results suggest that B alloys exhibited the best corrosion properties in 0.3M H₂SO₄ solutions and this may be related to the passivity nature of their corrosion products. Figure 8b represents the corrosion properties of the alloys in 3.5% NaCl solutions. It is observed that the trend with regard to increasing corrosion resistance is given as B, A, and C. The results suggest that C alloys exhibited the best corrosion properties in 3.5% NaCl solutions. The study noted that heat treatment affects the microstructural features of the alloys which in turn show diverse effects on their susceptibility to corrosion in 0.3M H₂SO₄ and 3.5% NaCl solutions. The results presented in this study confirmed the reports by Canbay et al. [33], Cai et al. [36], and Stosic et al. [37] who also found that up/step-quenching treatments create defects (such as dislocations and vacancies) in the microstructure and affect the orientation and mobility of martensite interfaces in Cu-based SMAs which in turn show significant effects on their properties.

4 Conclusion

The effect of various heat treatment procedures on the corrosion and wear behavior of varied compositions Cu-Zn-Sn-based SMAs of varied compositions is reported. The following conclusions were drawn:

- The microstructures consist of FCC Cu-rich-phase and Cu₅Zn₈ phase with varying degrees of dispersed precipitate phases. The step-quenched samples had finer average grain sizes and lesser volume fraction of precipitate phases compared to the up-quenched alloy samples.

- Up-quenching treatment effectively improved the hardness property of Cu-Zn-Sn-based SMAs by precipitation hardening mechanism. Among the three alloy categories tested, alloy A which contains quaternary 1.05 wt.%Fe addition yielded the best resistance to surface indentation.
- Up-quenching and step-quenching treatments equally improved the Wear properties of Cu-Zn-Sn-based alloys. Wear properties to some extent correlate with hardness properties. Samples from A alloys gave optimum wear resistance 0.143 mm³/N/m and hardness values (72.1HRB) for any given thermal procedure. Wear occurs mainly by the mechanism of abrasion. Step-quenching and up-quenching treatments reduce the COF of the alloys. The average values for COF are higher for A alloy samples than for B and C alloys.
- Cu-Zn-Sn-based SMAs are more susceptible to corrosion in 0.3M H₂SO₄ medium than in 3.5% NaCl solutions. The thermal treatments alter the microstructural features of the alloys which in turn show diverse effects on their susceptibility to corrosion in 0.3M H₂SO₄ and 3.5% NaCl media. Therefore, the corrosion susceptibility of Cu-Zn-Sn-Fe SMAs is sensitive to their microstructure and alloy composition. Generally, step-quenching treatment effectively improved the corrosion resistance of alloys in both media.

Conflict of interest

The authors declare no conflict of interest.

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