The role of Sn on microstructure, wear and corrosion properties of Al-5Zn-2.5Mg-1.6Cu-xSn alloy

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Abstract
In the present investigation, Al-5Zn-2.5Mg - 1.6Cu -xSn (x = 0.0, 0.2, 0.4, 0.6, 0.8, and 1.0 wt%) alloys were fabricated using melting and casting technique. The microstructures of the alloys were studied using optical, scanning electronic microscope/EDS and X-ray diffraction. The corrosion behaviour was performed using electrochemical measurements and immersion tests while the wear behaviour was carried out by pin-on-disc technique. The findings revealed that incorporating Sn to the Al-5Zn-2.5Mg alloy improved its corrosion and wear resistance due to refining the grains. The corrosion potentials shifted from −884 to −943, −955, −996, −1008 and −1012 mV (Ag / AgCl), while the coefficient of friction declined from 0.69 to 0.62, 0.51, 0.34, 0.29 and 0.22 with increment of Sn content from 0.0 to 0.2, 0.4, 0.6, 0.8 and 1.0 wt%, respectively. On the other hand, the results illustrated that the wear rate diminished from 4.42 * 10⁻³ to 1.47 * 10⁻³ (mm³/Nm) with increasing Sn from 0.0 to 1.0 wt%. Furthermore, the findings showed that increment of Sn content stimulated the uniform corrosion on the surface of alloys.

1. Introduction

Aluminum alloys are preferred engineering material for aerospace applications, automotive, mineral processing and marine industries due to excellent thermal conductivity, high strength/weight ratio, fatigue performance and corrosion resistance [1–4]. The hardness and strength of aluminum alloys may be enhanced by the formation of small uniformly dispersed particles of intermetallic phases within the Al- matrix. The demand of bearing materials with higher strength-to-weight ratio and better corrosion and tribological properties is necessary for bearing materials. Different techniques such as mechanical alloying, rapid solidification and thermal spraying were used to achieve these demands. High strength Al-alloys such as Al-Zn-Mg-Cu alloys are candidate materials. In this system, Mg combines with Zn and forms precipitates, such as MgZn2 and Mg3Zn8. The composition optimization of Al-Zn-Mg-Cu alloys plays a vital role in improving the properties of Al-Zn-Mg-Cu alloys. Zn and Mg as the main elements are the key factors in ageing precipitation, which can significantly affect the microstructure and strength of these alloys [8]. Increasing Mg content in Al-Zn-Mg-Cu alloys leads to higher hardness and strength. However, increasing Mg content may broaden the grain boundary precipitates, causing a reduction in toughness [8]. Many authors investigated the effect of magnesium on Al alloys [9]. Orozco et al [10] studied the impact of magnesium on the electrochemical properties of Al-Zn-Mg system. It was found that increasing magnesium leads to an enhancement in electrochemical behavior of aluminum. Yun et al [11] reported that increasing Zn causes an enhance in the strengthening, resulting in higher strength without reducing the elongation at break of Al-Mg alloy. Sadawy et al [12] found that the grain size of Al and the size of the precipitates decrease with decreasing Zn/Mg ratio in Al-Zn-Mg alloys. The pitting locates at the center of the intermetallic precipitates and the size of pit increases with increment of Zn/Mg mass ratio.

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Addition of soft alloying elements such as Sn, Pb, In and Bi to Al alloys can produce soft tribological alloys. The soft alloying elements spread over the contact surfaces forming a self-lubricating contact layer permitting the wear of the Al-rich matrix to be reduced [13]. It is known that Sn in Al-Sn system possesses very low solubility in liquid and solid states. The solubility of Sn is fewer than 0.01 wt% in Al-matrix at approximately temperature of 231.2 °C while this value increases to about 0.1% at temperature of 600°C and above [14, 15]. After solidification, its resulting structure consists of Al and Sn particles. The alloy components are homogeneously distributed at a high cooling rate. The mechanisms of trace Sn enhancing precipitation hardening behavior of Al-Mg-Si alloys was evaluated from the precipitate microstructure viewpoints by Liu et al [16]. The results displayed that the solute Sn stimulated the formation of fine precipitates instead of course, causing a considerable refinement of precipitate microstructure. Further, Sn acted as nucleation sites for precipitates. Understanding the relationship between wear, corrosion and microstructure is a vital requirement for the purpose of alloy design.

Liu et al [17] found that Sn particles are useful for increasing the wear resistance because Sn particles display a low shear strength which decreases the friction force and hence improves the anti-friction properties of the alloy. A similar results were obtained by Cruze et al [18] and Lu et al [19]. The addition of Sn increases the hydrogen overpotential of Al and reduces the self-corrosion rate of Al anodes [20]. Gudivic et al [21] found that Sn improves the corrosion properties of Al by impeding cathodic reaction of hydrogen evolution. However, He et al [22] examined the electrochemical properties of Al-Mg-Si alloys containing Sn ranged from 0.03 to 0.4 wt %. Their findings demonstrated that 0.03 wt% Sn can enhance corrosion resistance while higher addition (0.4 wt %) causes detrimental effect on corrosion resistance.

Few investigations have been highlighted on the microstructures, wear and corrosion resistance of Al-Zn-Mg alloys containing Sn. Therefore, the present study aims at studying whether Sn additions possesses detrimental or desirable effect on Al-Zn-Mg alloy alloys and understanding their performance with different Sn content.

### 2. Methods

The current investigation has performed using Al-5Zn-2.5Mg-1.6Cu-xSn alloys (x = 0.0, 0.2, 0.4, 0.6, 0.8, and 1.0 wt%). The alloys were fabricated by melting Al-5Zn-2.5Mg-1.6 Cu and high purity Sn. A measured weight of 5Zn-2.5Mg-1.6 Cu was placed into a graphite crucible and melted at 850 °C. The temperature of the furnaces was measured by digital thermometers UT320 Series controlled to ±1 °C of the set temperature. A measured content of Sn was introduced and mechanically agitated for 5 min. The molten alloy was poured into stainless steel mold of dimensions (200 × 30 × 10 mm) and then cooled naturally to room temperature. The obtained alloys have been analyzed by XRF as shown in table 1. The as-cast alloys were cut into 30 mm × 10 mm × 10 mm for different tests.

The microstructure of as-cast alloys was investigated using optical microscope, scanning electronic microscope (SEM, Joel-JXA-840A) coupled with energy dispersive x-ray (EDS) and x-ray diffraction analysis (XRD, Philips Analytical X-ray B.V. Machine). Different specimens have been prepared by grinding with 600, 800, 1000 and 1200 grit SiC paper respectively, and then cloth polished sequentially using 3 and 1 μm diamond paste according to (ASTM E3–95) [23]. The surfaces of samples were cleaned using acetone and dried in atmosphere. All specimens have been etched using a Keller solution of 150 ml distilled H2O + 25 ml HNO3 + 15 ml HCL +10 ml HF. The grain size measurement was performed with ImageJ software (version 1.52n).

The Brinell hardness test according (ASTM E10) was carried out on the all specimens to evaluate the mechanical behavior. Brinell hardness (HB) was measured in the current research using a Mettowa hardness tester 10 mm diameter steel ball indenter.

### Table 1. Chemical compositions of Al-5Zn-2.5Mg-1.5Cu–xSn alloys.

| Sample name | Elements wt% |
|-------------|--------------|
|             | Zn | Mg | Cu | Sn | Ni | Fe | Si | Al |
| a           | 5.21 | 2.63 | 1.66 | 0.0 | ≤0.10 | ≤0.011 | ≤0.007 | Bal. |
| b           | 5.25 | 2.42 | 1.57 | 0.22 | ≤0.11 | ≤0.012 | ≤0.008 | Bal. |
| c           | 5.02 | 2.56 | 1.61 | 0.41 | ≤0.10 | ≤0.010 | ≤0.005 | Bal. |
| d           | 5.12 | 2.44 | 1.65 | 0.65 | ≤0.19 | ≤0.021 | ≤0.008 | Bal. |
| e           | 5.08 | 2.58 | 1.56 | 0.83 | ≤0.12 | ≤0.011 | ≤0.005 | Bal. |
| f           | 5.05 | 2.53 | 1.53 | 1.01 | ≤0.12 | ≤0.012 | ≤0.007 | Bal. |
The Potentiostat / galvanostate (EG & G model-273) with M 352 a software supplied by (EG&G Princeton applied research) has been used for fitting the obtained data. A three-electrode system composed of as-cast Al-Zn-Mg alloy with surface area of 1.0 cm$^2$ as a working electrode, Pt-counter electrode, and Ag / AgCl-reference electrode have been utilized. The polarization technique was conducted with a scan rate of 0.5 mV/s. The PAR Calc Tafel Analysis has been used to fit the obtained data to the Stern-Geary model [24]. Pure reagent have been used to prepare all different solutions. Immersion corrosion tests were performed at room temperature in 3.5 wt% NaCl electrolyte according to ASTM G1 and G31 [25, 26]. The samples were ground as mentioned before. The corrosion product was removed from the corroded samples by putting in concentrated HNO$_3$ for 3 min The corrosion rate was determined using the following equation.

$$r = \frac{K \cdot \Delta W}{A \cdot D \cdot t}$$

(1)

Where $r$ signifies the corrosion rate (mm yr$^{-1}$), $K$ is a constant (8.76 $\times$ 10$^4$), $t$ refers to the time of soaking (h), $A$ signifies the area (cm$^2$), $\Delta W$ represent the weight loss in the nearest 1 mg and $D$ refers to the density of the material (g/cm$^3$).

A pin-on-disc machine was utilized to examine the dry sliding wear behavior of Al-Zn-Mg alloy in air. All sliding wear tests were performed at constant speed of 1.67 m s$^{-1}$ and applied load 12 N. The wear sliding time controlled at 30 min for all tests. An emery paper of 600 grit size fixed on rotating aluminum disc. Then the pin specimen pushed vertically into a rotating disc and the load was applied. The specimens were removed, cleaned with a soft brush after each test and weighed to find the weight loss. Schematic diagram of the sliding wear tester machine is shown in figure 1.

An electronic balance with an accuracy of 0.1 mg was used. The wear rate was determined from the volume loss using Archard equation [27]

$$V = \frac{K W}{H}$$

(2)

where $V$ is the volume loss of the abraded material per unit sliding distance, $K$ is the wear coefficient, $W$ is the applied normal load and $H$ is the hardness of the abraded material. The worn surface was identified by SEM to determine the wear mechanism present in the tribosystem.

3. Results and discussion

3.1. Microstructures

Figure 2 depicts the microstructures of the as-cast alloys containing different contents of Sn as an alloying element. The alloys possess grains microstructure. The size of grains declines as Sn content increases. This means that Sn solute atoms have the ability to restrict the growth of grains. Therefore, Sn acts as a grain refiner for Al-Zn-Mg alloys.

According to Lu et al [28] the refining effect of Sn addition on the precipitate microstructure derived from the increased homogenous precipitate nucleation rate. Furthermore, incorporating Sn into Al-Zn-Mg as a solute decreases the melting point of liquid phase at the interface, causing a reduction in the solidification rate. This leads to creating an extra-constitutional supercooled sites for nucleation [13]. Further, figure 2 shows that sample (a) possesses more uneven grain size distribution, while the other samples exhibit uniform grain size distribution. Further, it can be seen that along the grain boundaries black areas can be found, such areas might be
attributed to the formation of intermetallic precipitates (IMPs). More details on these particles can be found later in SEM/EDS section.

Figure 3 shows the grain size distribution of the alloys. It is clear that the grain size reduced by 56.4, 80.8, 81.3, 81.55, and 86.26% with increasing Sn content to 0.2, 0.4, 0.6, 0.8 and 1.0 wt%, respectively. SEM/EDS of as-cast Al-5Zn-2.5Mg-1.6Cu and Al-5Zn-2.5Mg-1.6Cu–1.0 Sn alloys is shown in figures 4, 5 and tables 2, 3. The alloys illustrate grain microstructures with intermetallic precipitates at grain boundaries. These IMPs form network shape along the grains.

The shapes of intermetallic precipitates are regular and irregular with different sizes in the as-cast alloys. However, Al-5Zn-2.5Mg-1.6Cu–1.0 Sn alloy characterizes by presence of Sn-rich particles and a new intermetallic precipitates composed of MgSn2. The chemical compositions of the marked precipitates can be summarized in tables 2, 3. Figures 6 and 7 illustrate the EDS mapping of alloys a and f. It is clear that all alloying elements exhibit uniform distribution.

The X-ray pattern of the cast alloy without Sn as shown in figure 8 certifies the presence of α-Al and different intermetallic phases composed of MgZn2, Mg2Zn3, and AlMg2Zn. The other alloys have the same phases, while the Sn peaks did not appear in the XRD pattern due to its low content.

The MgSn2 phase did not detected due to its low content in the matrix. The different phases were produced during the solidification process of the alloys. The crystallite size of α-Al was also calculated from X-ray diffraction by Debye–Scherrer equation [29]:

$$D = \frac{k\lambda}{\beta\theta}$$

where K is the Scherrer constant, λ represents the wavelength of light used for the diffraction, β is the ‘full width at half maximum’ of the sharp peaks, and θ refers to the angle measured. The Scherrer constant (K) in the above formula accounts for the shape of the particle and is commonly taken to have the value of 0.9.

The outcomes as shown in figure 9 illustrate that the crystallite size of α-Al is reduced by increment of Sn content. It declined from 604 to 232 Angstrom with an increase of Sn to 1.0%wt. This trend may be attributed to the increase of the specific surface area of Sn by increasing its content into the matrix, providing more positions for nucleation.

3.2. Hardness

The hardness findings of the as-cast Al-Zn-Mg alloy with varied Sn additions is presented in figure 10. Evidently, the hardness values of the Sn modified Al-Zn-Mg alloy augment with an increase of Sn additions.
from 0.2 to 1.0 wt%. A hardness value of 86.13 HB was obtained for Al-Zn-Mg without Sn. The hardness values increased by 10.3, 18.42, 24.32, 30.03 and 33.51%; with increment of Sn to 0.2, 0.4, 0.6, 0.8 and 1.0 wt%, respectively.
This behavior is due to refinements of grains that produce more grain boundaries by increasing Sn content. According to Hall mechanism [30] the grains boundaries serve as physical barriers to dislocation motion, preventing their movement and consequently, it improves the hardness. Further, increasing Sn content into the matrix leads to increasing Mg$_2$Sn phase which acts as a new hardener phase [31].

### Table 2. EDS spectroscopy analysis of intermetallic particles for the as cast alloy without Sn.

| Spot No. | Elements wt% |
|----------|--------------|
|          | Zn  | Mg  | Al  |
| 1        | 15.82 | 4.2 | 88.38 |
| 2        | 11.8 | 5.21 | 82.99 |
| 3        | 12.56 | 6.13 | 81.31 |

### Table 3. EDS spectroscopy analysis of intermetallic particles for the as cast alloy with 1.0 wt% Sn.

| Spot No. | Elements wt% |
|----------|--------------|
|          | Zn  | Mg  | Sn  | Al  |
| 1        | 9.88 | 4.66 | —   | 85.46 |
| 2        | 5.66 | 10  | 12.67 | 71.67 |
| 3        | 1.65 | 2.30 | 82.56 | 13.49 |

Figure 6. EDS mapping of alloy (a).
3.3. Corrosion behavior

The potentiodynamic polarization plots of the as-cast Al-5Zn-2.5Mg—xSn alloys are given in figure 11. To attain stable open circuit potential values, all specimens were soaked in 3.5 wt% NaCl for 30 min before tests.
Noticeably, the cathodic and anodic curves of all specimens give similar polarization behavior. The cathodic curves characterize the reduction of oxygen and hydrogen on the surface of cathodic sites of alloys according to equations (4) and (5) [32–34], while the anodic curves signify the dissolution of alloys.

\[
O_2 + 2H_2O + 4e^- = 4OH^- \tag{4}
\]

\[
2H_2O + 2e^- = H_2 + 2OH^- \tag{5}
\]

However, the outcomes show that the cathodic and anodic current densities gradually decrease with increment of Sn content. This means that Sn constrain the oxygen and hydrogen reaction kinetics. The corrosion parameters (corrosion potential (E_corr), corrosion current density (i_corr), anodic and cathodic slopes (β_a and β_c) deduced from polarization plots are given in table 4.

The results reveal that the corrosion current decreased from \(4.42 \times 10^{-6}\) to \(1.32 \times 10^{-7}\) (A cm\(^{-2}\)), this suggests that susceptibility to corrosion attack is reduced by increasing Sn. This behavior is due to two reasons; first, refining the grains decreases the concentration of impurities at grain boundaries. Similarly, it was reported that the refinement of Al-10wt%Sn alloy reduced its corrosion rate in 3.5 wt% NaCl [35]. Secondly, when the Sn associated with Al-Zn-Mg alloys dissolves into the aggressive solution, its cations cover the cathodic site on the surface of alloys and therefore decrease the corrosion current [19]. Further, the results illustrate that the
corrosion potential declines from $(-884$ to $-1012$ mV) due to dissolving the passive film and inhibiting further formation of the film on the surface of alloys in presence of Sn.

The corrosion rate obtained from immersion tests is shown in figure 12. A similar trend for electrochemical tests was also obtained in 3.5 wt% NaCl.

Table 4. Electrochemical parameters deduced from potentiodynamic polarization measurements for Al-5Zn2.5-Mg-1.5Cu-xSn alloys in 3.5 wt% NaCl solution.

| Sample | $i_{corr}$ (A/cm$^2$) | $E_{corr}$ (mV) | Corrosion rate (mm/y) | $-\beta_c$ (mV/dec) | $\beta_a$ (mV/dec) |
|--------|------------------------|-----------------|-----------------------|---------------------|-------------------|
| a      | $44.24 \times 10^{-6}$ | -884            | 0.49                  | 118.22              | 218.12            |
| b      | $14.32 \times 10^{-6}$ | -943            | 0.15                  | 125.45              | 221.68            |
| c      | $8.11 \times 10^{-6}$  | -955            | 0.09                  | 127.55              | 222.89            |
| d      | $3.90 \times 10^{-6}$  | -996            | 0.04                  | 131.26              | 224.24            |
| e      | $1.91 \times 10^{-7}$  | -1008           | 0.002                 | 132.76              | 226.11            |
| f      | $1.32 \times 10^{-7}$  | -1012           | 0.001                 | 133.55              | 226.87            |
3.4. Wear behavior

The variation of wear rate with respect to Sn content is illustrated in figure 13. Undoubtedly, the wear rate decreases with increment of Sn content from 0.2 to 1.0 wt%. The wear rate decreased by 7.31, 14.63, 20.73, and 30.49% with increment of Sn to 0.2, 0.4, 0.6, 0.8 and 1.0 wt%, respectively. The behavior is ascribed to Sn particles which spread over the Al-matrix and work as internal solid lubricant. Further, increasing Sn particles along the matrix causing a softening effect. This obviously proves that increment of Sn-rich particles increases the load-carrying capacity of the alloys. Similar results were obtained for Al-Bi-Sn by Costa et al [36]. They reported that presence of Sn acts as a solid lubricant when spread over the tough Al-matrix and a good tribological behavior is obtained.

The difference in the wear properties mentioned above can be further revealed by observing the worn surface morphologies of the investigated alloys under the load of 12 N, sliding speed of 1.67 m s⁻¹ and emery paper of 600 grit size for 30 min as shown in figure 14.

It is obvious that the grooves and scratches spread over the worn surfaces of all samples. Continuous grooves and scratches parallel to the sliding direction are obtained. The scratches and grooves are resulted from the ploughing action [37]. However, the observation of the bare alloy shows that the alloy offers large width and deep
The depth of grooves compared with the other alloys. The predominant wear mechanism is abrasion. Addition of Sn particles into the matrix offers narrow grooves on the worn surfaces. The width and depth of the grooves decline with the increase of Sn content. The compositions of the debris and the matrix after wear tests of Al-Zn-Mg-Cu are shown in figure 15. The alloy without Sn consists of Al, Zn, Mg and O. Increasing Sn content leads to fine particles of debris on the surfaces of Al-Zn-Mg-Cu-Sn alloys. Fine debris is a characteristic of low wear conditions. The compositions of the debris and matrix are shown also in the figure 15. This proves that oxidation layer containing Sn on the surface of alloys is a good lubricant. According to Lu et al [38] addition of Mg in presence of Sn in Al-Sn system remarkably reduced the size of loose debris attached to worn surface, facilitating the formation of a stable oxide tribolayer and effectively increased the wear resistance. To further analyze the damage mechanism, the generated debris during dry sliding wear tests were separately collected after the tests and analysed using scanning microscope. The magnifications were selected so that a maximum number of unagglomerated particles of debris were in view. Figure 16 shows that the debris of the investigated alloys have different shapes and sizes. The main types of debris are flakes, machining chips and fine particles. It can be also noted that the incorporation of Sn into the matrix leads to a decrease in the size of debris. This behaviour shows that Sn particles act as a solid internal lubricant and therefore diminishes the contact between the abrasive materials and the surfaces.

Figure 17 shows the change of friction coefficient ($\mu$) for Al-Zn-Mg-Cu containing different contents of Sn versus elapsed time under a load of 12 N, speed of 1.67 m s$^{-1}$ and 600 grit size. The behaviour of $\mu$ reveals the stick-slip performance for all alloys under the dry sliding wear test conditions. Once both surfaces of abrasive emery and the pin contact each other, the adhesion occurs and causes increasing in abrasive sliding force. Then, the friction coefficient is increased. After that, the friction coefficients is appeared as random pattern oscillations. At some dots, the tangential friction forces may be beat the adhesive bonds along the interface lines. As the wear test continues, the repeating slip processes causes interfacial adhesion bonds to break. The increase of effective strain values drives to the reduction in the $\mu$ average of friction force. This behavior might be a response to form an oxide film on the worn surface [27]. Further, figure 17 illustrates that $\mu$ decreases from 0.69 to 0.6, 0.51, 0.34,
0.29 and 0.22 with increment of Sn content from 0.0 to 0.2, 0.4, 0.6, 0.8 and 1.0 wt%, respectively. The behaviour is attributed to the homogeneous distribution of Sn particles in the Al matrix benefits the formation of uniform tin oxide layer on worn surface. This trend affirms that Sn particles act as a solid internal lubricant and hence reduces the contact between the abrasive materials and the surfaces. Further, a smooth tribolayer is formed and the tendency for abrasive wear is decreased.

Figure 16. (a)–(d). Debris morphologies of the as-cast alloys containing Sn (a) 0, (b) 0.2, (c) 0.6, and (d) 1.0 wt%.

Figure 17. Effect of Sn content on friction coefficient of the investigated alloys.
4. Conclusions

The effect of Sn content on microstructure, corrosion and wear properties of Al-Zn-Mg alloy was investigated. It was concluded that increment of Sn content in Al-Zn-Mg alloys, declined the grain size and produced a more uniform grain size distribution. This led to improvements in hardness, corrosion and wear resistance. Further, the study illustrated that increasing Sn, decreased the corrosion rate and shifted the corrosion potentials to more negative values. On the other hand the uniform corrosion on the surface of alloys has been stimulated. The Sn particles acted as a solid internal lubricant and hence reduced the contact between the abrasive materials and the surfaces, causing an improvement in wear properties.

Data availability statement

The data generated and/or analysed during the current study are not publicly available for legal/ethical reasons but are available from the corresponding author on reasonable request.

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