

## Behavior of Electrodeposited Layer on Mg (ZE41A) Alloy Under Tribo-Corrosion Condition

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In this paper the Tribological-Corrosion resistance of Tagnite coating referred to as chromate free electrodeposited coating on magnesium alloy (ZE41A). The electrode layer was evaluated in 3% NaCl was assessed. An environmental compatible Tagnite treatment coating was utilized on a ZE41A magnesium alloy. Two application were used for the electrodeposition of coating by immersion method and brush method. The results showed an improvement in current density and Tribo-corrosion resistant surface for the Brush Tagnite electrodeposition coating method. The Tagnite anodic electrodeposition of coating appeared to consist of a thin and cracked coating layer with 'dry-mud' morphology. The Tribo-Potential wear tests (TP) indicated a reduction in coefficient of friction as load increases with poor film adhesion compared to Tribo-wear tests (T); this effect has resulted from an oxidation wear mechanism that has dominated the surface of the magnesium alloy. The domination of abrasive wear mechanism in the tribo tests indicated by a high volume wear rate values, which concluded that it was better to polarize magnesium alloy through anodic oxidation in artificial seawater environment. The improved tribological-corrosion behavior of the electrodeposited magnesium alloy was attributed to the formation of thick magnesium oxide and magnesium hydroxide films occurring on the contacted surfaces.

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**Keywords:** Corrosion, Tribology, Tagnite coating, Mechanical wear, Potentiodynamic polarization, Magnesium (ZE41A), Seawater.

## 1. INTRODUCTION

Magnesium offers a set of highly attractive properties for the manufacturing industry like lightness, durability, high specific strength, good castability and long life. Since magnesium (ZE41A) is a leading alloying element with Zn, Zr and Re as principal alloying elements. It has been very widely used for heavy load mechanical engineering applications. Magnesium and its alloys are used in manufacturing helicopter components, cars, aircraft engines, airframes, light trucks, automotive parts, and computers [1]. One of the important challenges that makes its application very limited is the relatively poor corrosion resistance of the alloy but coating is commonly used to overcome this problem. In order to protect magnesium, both chemical and electrochemical methods are used for surface modification of magnesium. Chemical methods are known to be environmentally hazardous. While the common electrochemical methods result in a more environmentally compatible material. This provide an effective base but offer little corrosion protection [2]. Murray. et al.,[3] used a polarization curve method to evaluate coating. A lot of research utilized cerium and synthesized fluoride conversion coating on magnesium alloys to improve corrosion resistance and pitting potential of the alloy[4-7]. Huang et al.,[8] found that borate without active element is not effective at reducing the friction and wear of magnesium alloy. Ma Youpinga, et al.,[9] found that diffusion alloying layer acts as effective corrosion barrier to decrease the corrosion rate for (ZM5) magnesium alloy when exposed to 3% NaCl solutions and greatly contribute to the enhancement of wear resistance. Apparently, anodic film was mainly composed of MgO when modified with Al<sub>2</sub>O<sub>3</sub> has shown to exhibit the superior corrosion resistance for AZ91D Mg alloy in salt solution[10], while the wear mechanism of the alloy during sliding involves the formation of thin, narrow shards along the edges of wear grooves that break off to produce loose particles[11].

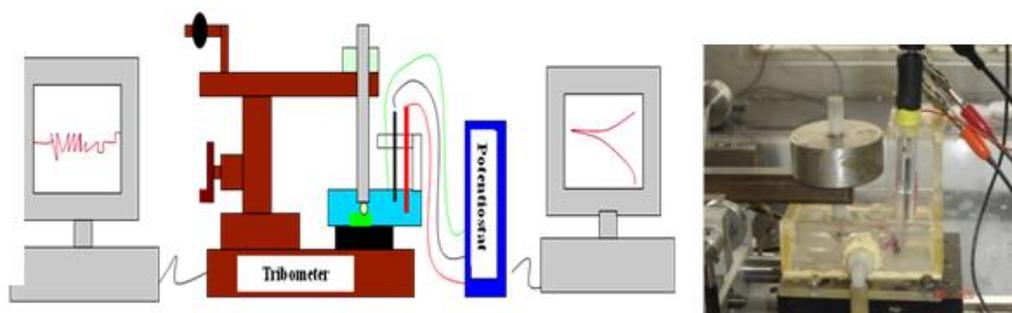
The main objective of the present work is to gain a better understanding of the mechanism of Tagnite coated magnesium alloy (ZE41A) under tribo-corrosion condition (when mechanical wear and chemical/electrochemical processes interact with each other). Comparative studies of two application methods of Tagnite anodic electro-deposition with surface treatment coating were performed. A low voltage formula brush Tagnite (ZC275) with thickness about 217 microns and conventional immersion Tagnite (ZC276) of 0.35 millimeter in thickness. The Tagnite Coating is a chromate-free anodic surface treatment with more corrosion and abrasion resistance than any chromate based coating [12]. The coating occurs using alkaline solution free from chromium (VI) or other heavy metals and operates below room temperature (40° to 60 °F) this solution consists of hydroxide, fluoride and silicate species which are also incorporated into the layer [13, 14]. Evaluation was conducted on magnesium (ZE41A) alloy, which is used on both military and commercial helicopter gearboxes. However, Tribological-corrosion behavior of (ZE41A) magnesium alloys in aqueous seawater condition has seldom been reported, therefore, it is of a great theoretical and practical importance to carry out investigations of this alloy. The study also explored the relationship between stand alone tribology oxide film and corrosion oxide film growth in seawater solution.

## 2. EXPERIMENTAL DETAILS

**Table. 1,** Processing parameters for Tagnite., [14]

<b>Preliminary treatment</b>	Alkaline
<b>Activation (stage one)</b>	0.5–1.2 M $\text{NH}_4\text{F}$ , 70–80°C, 30–40 min
<b>Electrolyte (stage two)</b>	5–7 g/l potassium hydroxide KOH 15–20 g/l potassium silicate $\text{K}_2\text{SiO}_3$ 8–10 g/l potassium fluoride KF pH 12.5–13
<b>Parameters</b>	$i = 1\text{--}5 \text{ A/dm}^2$ , within 30 s increase voltage to 150 V → then continue to anodise at constant current of $3 \text{ A/dm}^2$ End-point voltage (depending on thickness) = 200–400 V $T_{\text{EL}} 10\text{--}20^\circ\text{C}$
<b>Sealing</b>	12% $\text{KH}_2\text{PO}_4$ , 5 min, 60°C

Tagnite coated (ZE41) magnesium alloy was studied, contains 0.7%Zr, 4.2%Zn, 1.2%Re. Wet friction sliding test combined with accelerated corrosion test were performed using a CSM Tribometer and Gamry computer controlled potentiostat with a new erosion- corrosion cell design as shown in Figure 1. Magnesium samples were cut into rectangular shape with area of the working surface of  $1 \text{ cm}^2$ . A single core electrical wire was attached to the samples by conductive electrical resin then the samples were cold resin molded. The EIS experiments were performed at open circuit potential over the frequency range 100 kHz to 0.2 Hz. The potential perturbation was 5 mV in amplitude. All electrochemical measurements were performed with a Potentiostat/Galvanostat/Zra analyzer (Gamry PCI300/4). A personal computer with DC 105, EIS300 and Echem Analyst 5.21 was used for data fitting and calculation.



**Figure 1.** Cell design and Tribo-Corrosion test rig

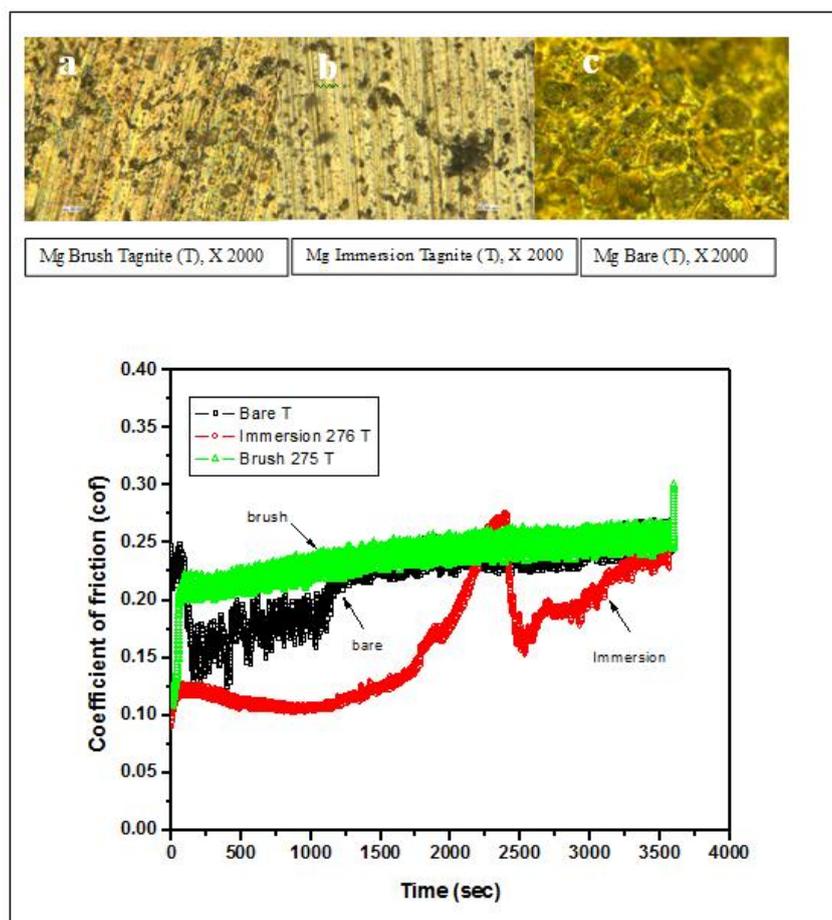
The surface preparation procedure of the magnesium bare sample was consisted of grinding surfaces by 320 and 1200 grit SiC papers, respectively then polished with 9, 3 and 1 microns diamonds paste oil based, followed by a polish with silica paste and colloidal silica. The polished surfaces were cleaned with acetone solution.

Tribology wear test (T) was carried out in a sliding velocity range of 3.00 cm/s for a constant sliding distance of 68.8 m and a load range of 3, 5 and 9 N for 45 to 55 minutes. Tribo-Potential oxidation wear test (TP) combined both tribology wear test and potentiodynamic corrosion test. The scan rate was 0.22 mV/sec, scan range -1.8 to -1.4 V for 1 hour. An average volumetric wear rate from both techniques was determined as a function of time at each load condition. EIS under prolonged condition were conducted on the specimen under test at a frequency ranging from 100 KHz to 0.01 Hz.

### 3. RESULTS AND DISCUSSION

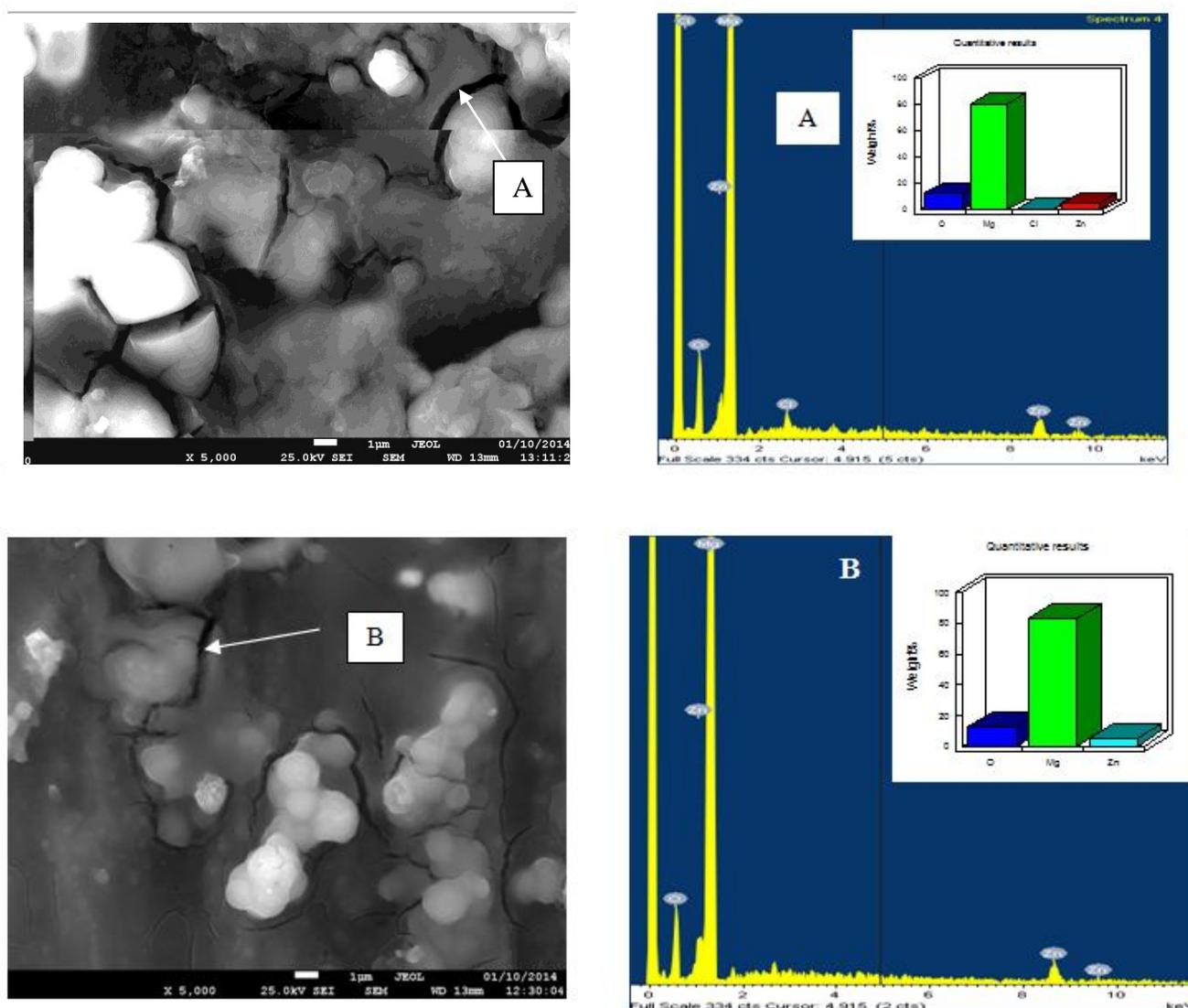
#### 3.1 Tribo-wear test (T)

Both mean friction coefficients (C.O.F) behavior of Brush Tagnite (275) coated magnesium and bare samples was identical after 55 minutes duration of exposure. This revealed that the (275) brush sample with its film thickness has been removed during the first 1.2 minutes of the (T) test. After that then a stable increase in oxide film thickness were observed under this process combination of abrasive effect of the alloy surface oxidation and coating corrosion products, with C.O.F reaching a value of 0.25 were observed, as shown in Figure 2.



**Figure 2.** Tribological (T) Coefficient of friction for ZE41A magnesium alloy coated with Tagnite by immersion and brush method in seawater solution.

Meanwhile, immersion Tagnite (276) coated sample showed a stable mean C.O.F, at the first 17 minutes of the wet reciprocating wear test, this was due to the residual thick Tagnite coating and the less entrapment of corrosion products or fine oxide particles between the sliding (ZE41A) surface and the static alumina ball. A gradual increased in mean C.O.F up to 40 minutes was caused by thickening of the oxide film and larger oxidation ionic exchange between alloy surface and the seawater electrolyte.



**Figure 3.** The morphology and EDS Spectrum (5000X) show the Erosion (T) inside the groove of Magnesium oxide film formed on the surface of magnesium ZE41A immersed in Tagnite (A) and Brush Tagnite (B) in 3.5% NaCl solution.

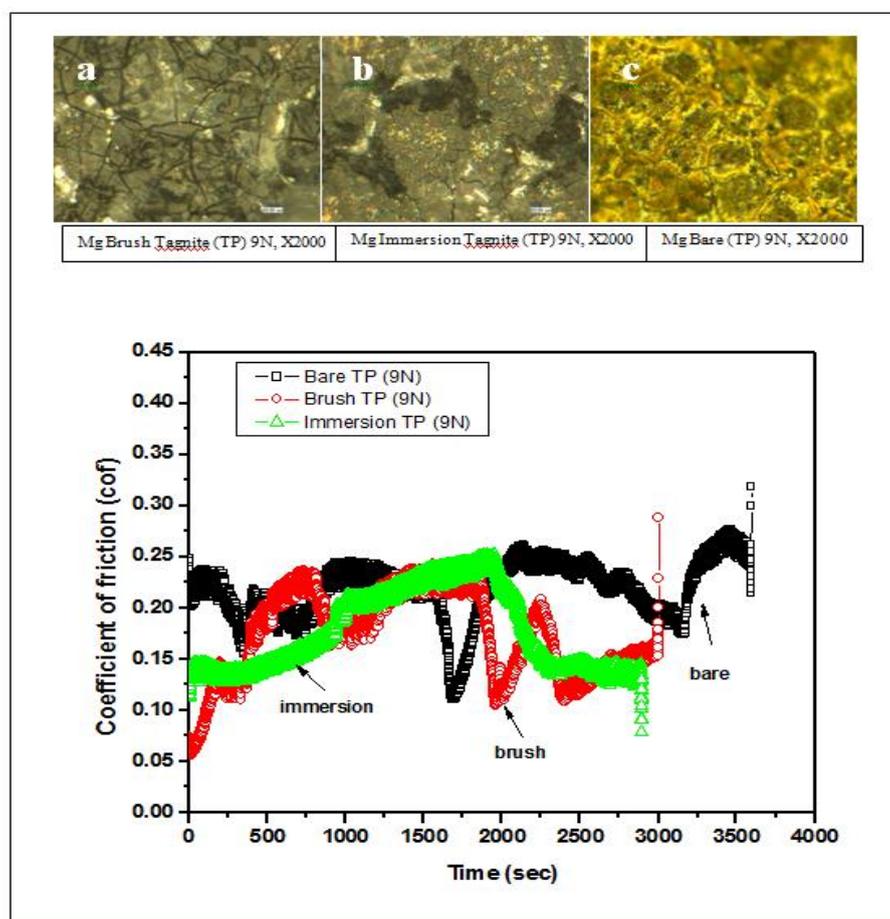
Beyond 40 minutes time duration, a sharp reduction in coefficient of frictions occurred due to the, thickening of the oxide film on the surface of the immersion coated magnesium, which makes it less adherence to the alloy surface. This process has subsequently causes oxide film detachment. Also the reduction was related to the hardness of the sliding magnesium surface compared to the static ball and the oxide films speed of initiation and adhesion to the surface. According to Hoche et al., [15]

magnesium are susceptible to the formation of surface oxidation films even after short exposure to atmosphere.

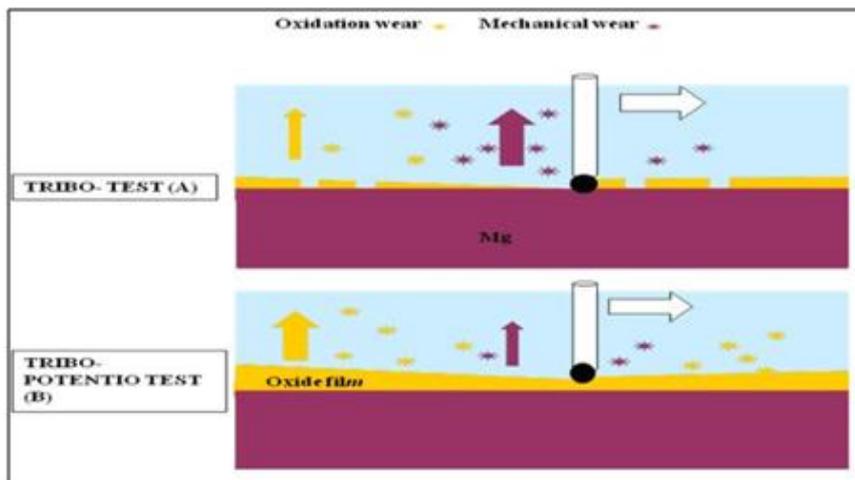
According to research oxidation film on the magnesium is known to be fragile and unstable [16, 17]. In this study, oxide film started to redevelop and thickening at 42 minutes it continued until it reach an equilibrium state of initiation of adherent film in relation to loads and the electrolyte condition involved in the test. Small oscillations of both the friction coefficient and the corrosion potential are observed during the tribocorrosion test, which might be attributed to the ejection of wear debris from the contact area similar to that observed by Barril et al., [18].

Metallographic examination showed similar wear scar lines on the bare and the coated ZE41A alloy samples surface as indicated in figure. 2a, 2b, 2c for immersion Tagnite (276), evidence of mechanical abrasion mechanism has occurred with electrochemical corrosion reactions occurred at the magnesium grain boundary and pits sites; variation of surface discoloration was evidence on the bare alloy than the coated ones. In SEM characterization showed in figure 3, a thin and cracked coating layer with ‘dry-mud’ morphology and similar oxidation percentage.

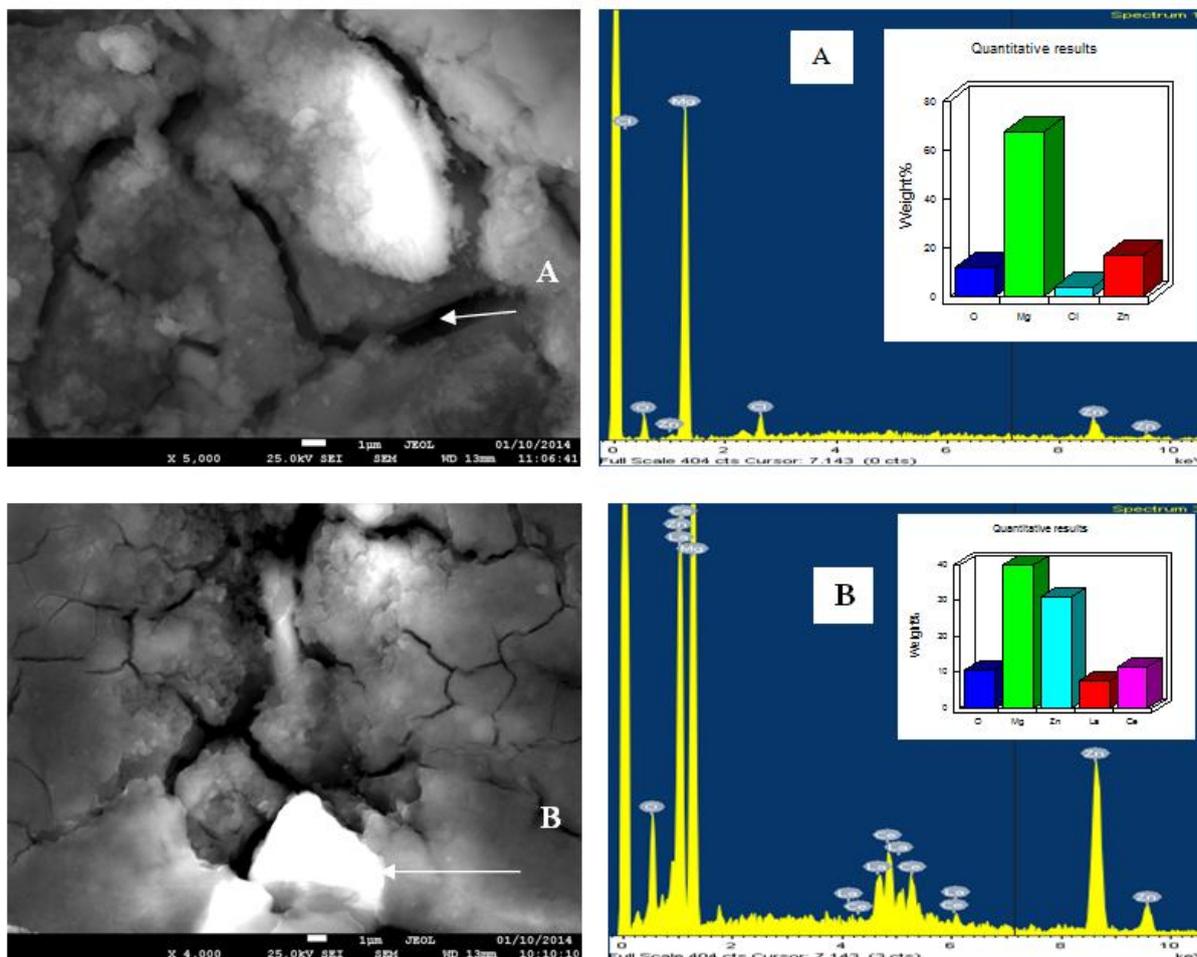
### 3.2 Tribo-Potential wear test (TP)



**Figure 4.** Tribo-Potential (TP) of friction for ZE41A magnesium alloy coated with Tagnite by immersion and brush method in seawater solution.



**Figure 5.** Tribological wear (T) and Tribo-Potential (TP) mechanisms for magnesium (ZE41A) in seawater solution.



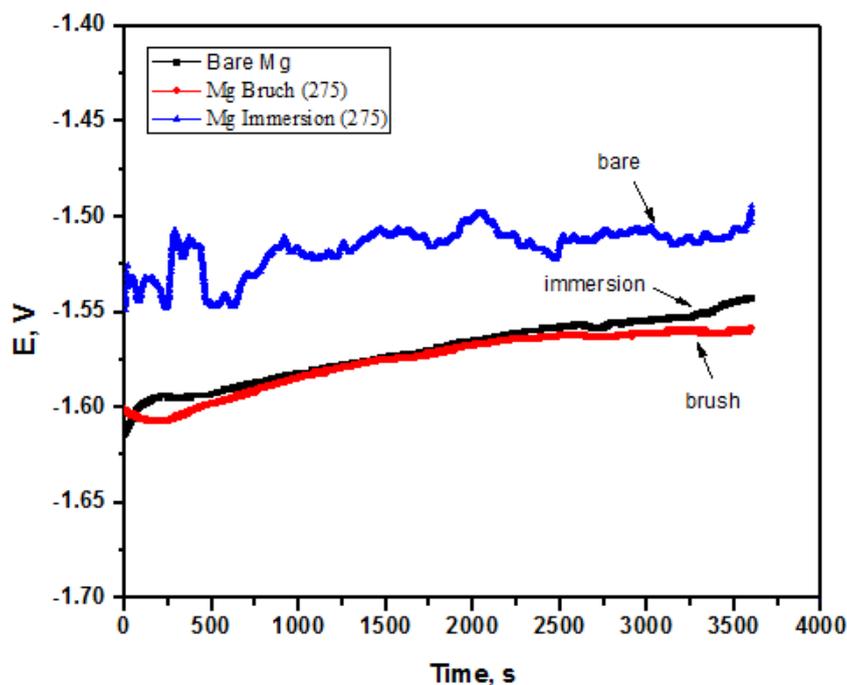
**Figure 6.** The morphology and EDS Spectrum (5000X) show the Tribocorrosion (TP) inside the groove of Magnesium oxide film formed on the surface of magnesium ZE41A immersed Tagnite (A) and Brushed Tagnite (B) in 3.5% NaCl solution.

During Tribology wear test with potentiodynamic scan as shown in Fig. 4, the initial C.O.F for Immersion Tagnite indicated a gradual increase up to 0.24, during the first 32 minutes sliding time duration, coating showed an oxide film thickening, its surface adhesion was reduced and oxide film rupture commences due to the wear load stresses at 9 N. This followed by a sharp decrease in coefficient of friction to a value of 0.14. On the Brush Tagnite coating after 30 minutes a sharp drop occurred C.O.F then a fast regrowth of the oxide film commences, due to accelerated dissolution of the Mg alloy elements at high anodic potential then film breakdown occurred again, until a constant stable value of 0.17 was achieved at longer time duration. This revealed the mechanism of intact oxide film initiation and re-growth then film breakage.

Microscopic examination of ZE41A coated immersion Tagnite sample under (TP) test, indicates a complete surface dissolution as shown in figure. 4 infested with deep corrosion pits which prohibited the existence of wear scars lines. Traces of white Tagnite coating pigments and a thick cracked oxide film was exhibited on the surface.

In general the (TP) test coated for magnesium showed a C.O.F ranging between 0.14-0.17, while the (T) test resulted in higher values of C.O.F, 0.2-0.25. Apparently, resulted in earlier rupture of oxide film at 32 minutes for the (TP) test and 40 minutes for the (T) test. This concluded that at the (TP) test produces a thicker oxide film that is poor in surface adhesion as illustrated in the mechanism in Figure 5. In SEM analysis indication of lower oxidation level in brush Tagnite than immersion Tagnite coatings as shown in figure 6, combined with dry mud feature morphology.

### 3.3 Open Circuit Potential Measurements



**Figure 7.** Open circuit potential measurements of Bare Magnesium (ZE41A) alone and coated with Tagnite by Immersion (276) and brush (275) methods in static seawater solution.

Figure 7 show the corrosion potential as a function of time for the bare magnesium and the two coated Magnesium samples (immersion and brush) in 3.5% NaCl solution. It is well known that Mg is very active in salt water and undergoes anodic dissolution as follow [19].



After that Mg forms an oxide layer according to the following equation:



From figure 1, it is obvious that in case of the bare magnesium sample the corrosion potential value recorded to be -1.62 V at zero time then the potential value rises sharply with time to reach about -1.59 V after about 3 minutes due to the aggressive attack of the chloride ions after that, the potential increases very slowly with time which attributed to the stable growth of oxide film on the surface this is agreement with the work done by [19] and [20].

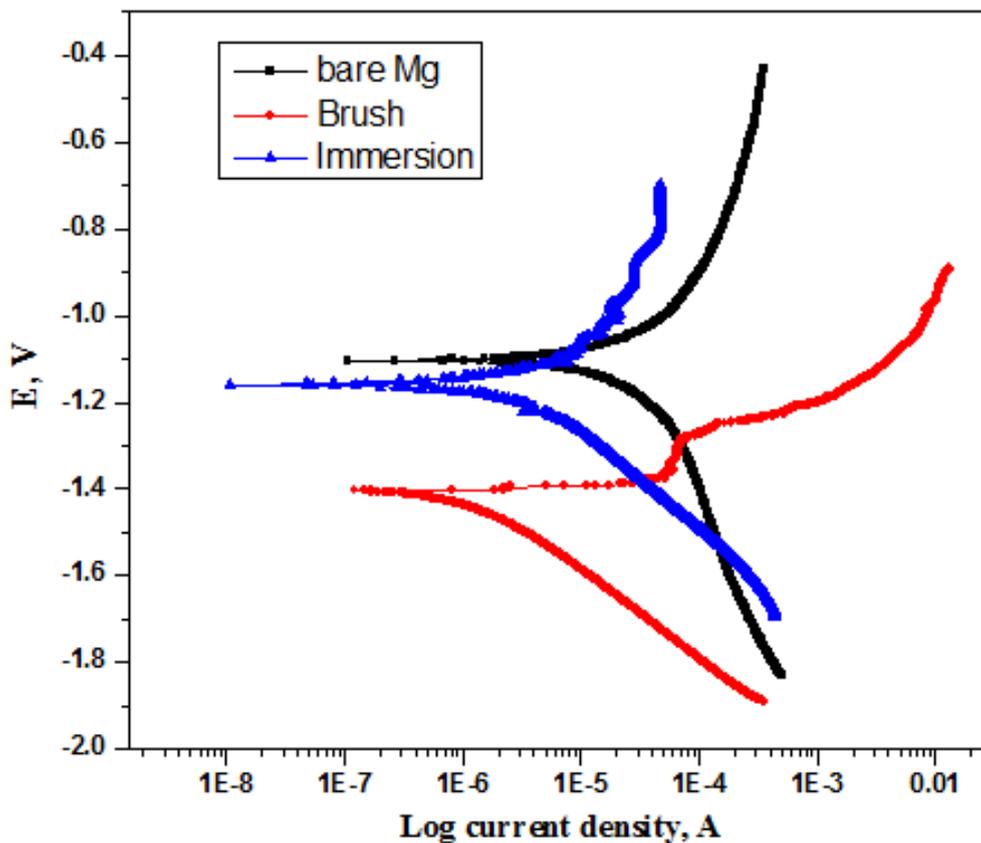
For immersion Tagnite (276) coated sample, the potential increases firstly from -1.55 V at zero time to -1.53 V through the first minute followed by shift in potential in the active direction consistent with removal of the oxide film.

Subsequently, the potential increase again to more positive potential due to buildup of oxide film and then continuous decrease in potential due to detachment of the thicker anodic layer presented on Mg surface occurred with immersion time. In other words, the decrease in potential may be due to a reduction in the corrosion properties of the substrate/coating system due to the penetration of the electrolyte through the pores and defects in the coatings as observed by [21]. In the brush Tagnite (275) coated sample, the potential drop from -1.59 V at zero time to reach -1.61 V after about 3 minutes due to the destruction of the coated layer as a result of permeation of chloride ions from salt water into the coating. After the sliding was stopped the corrosion potential increased in the noble direction. Therefore, it can be said that the repassivation due to oxide film formation has been occurred. The steady state potential was reached when equilibrium occurs between the rate of formation and the rate of removal of the oxide film. In conclusion, it is clear that brush coated sample has better intact to the Mg surface than that of immersion one.

### 3.4 Tafel Polarization measurements

Tafel polarization curves for bare magnesium alloy type ZE41A sample alone and coated with Tagnite (immersion and brush) were recorded in 3.5% NaCl solution as shown in Figure 8.

In the bare alloy the polarization curve showed that in the anodic part the current increases due to aggressive attack of chloride ions and dissolution of the alloy has happened until it reached a passive behavior that present due to the formation of oxide layer on the alloy surface with low anodic current densities in a wide range of potentials (from -1.4 to -1.1 V<sub>SCE</sub>). The dissolution of the bare magnesium alloy in neutral medium is due to the transformation of Mg(OH)<sub>2</sub> to easily soluble MgCl<sub>2</sub> [22, 23].



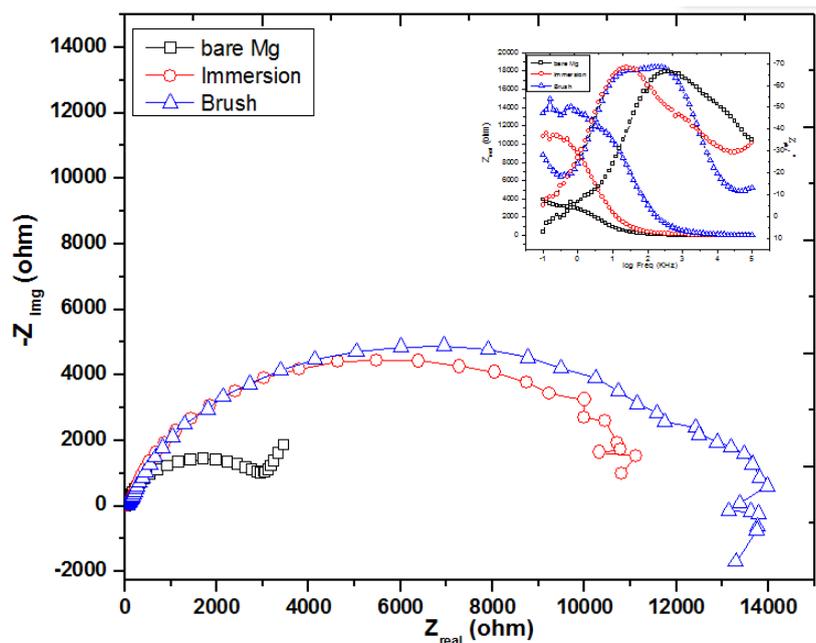
**Figure 8.** Tafel plots of Bare Magnesium (ZE41A) alone and coated with Tagnite by Immersion (276) and brush (275) methods in static seawater solution.

**Table 2.** Polarization data obtained for Bare Magnesium (ZE41A) alone and coated with Tagnite by Immersion (276) and brush (275) methods in static seawater solution.

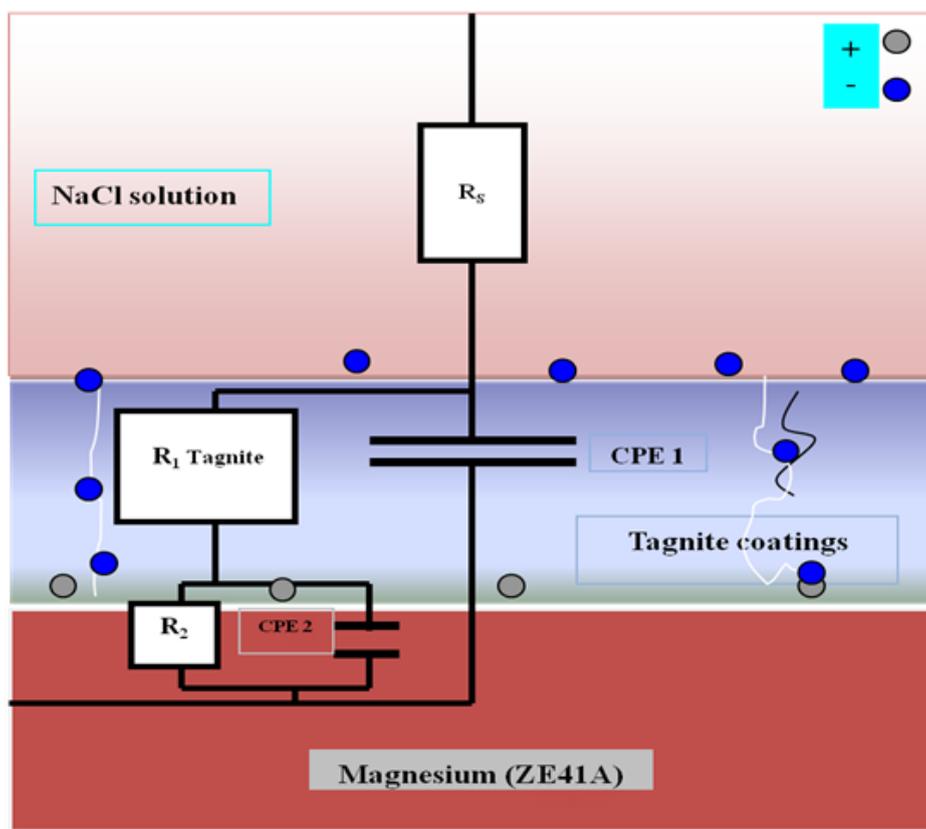
Media	$E_{corr}$ , mV	$i_{corr}$ , $\mu A\ cm^{-2}$	$\beta_a$ , mV dec <sup>-1</sup>	$-\beta_c$ , mV dec <sup>-1</sup>	CR mm y <sup>-1</sup>
Mg in 3.5% NaCl	-1.1	92.1	94.3	127	3.79
Brush coat	-1.4	1.11	67.4	197	0.45
Immersion coat	-1.16	2.62	78.5	204	1.08

In presence of both Tagnite coatings it was found that presence of coatings exhibited dramatically lowers corrosion currents ( $i_{corr}$ ) and also lower corrosion rates (CR) than the bare alloy and hence higher corrosion resistance as shown in Table 2. The lowest corrosion current density obtained in the case of brush Tagnite coating due to the presence of thin and compact layer of the coat resist the aggressive attack of chloride ions. In the immersion Tagnite coat a thicker layer of the coat are present but its resistance to chloride ions are lower than brush coat due to the steady permeation of chloride ions through the coat as result to the capillary effect.

3.5 Electrochemical impedance spectroscopy (EIS)



**Figure 9.** Nyquist plots for Bare Magnesium (ZE41A) alone and coated with Tagnite by Immersion (276) and brush (275) methods in static seawater solution.



**Figure 10.** Equivalent circuit model used to fit the impedance spectra (models two relaxation time constants).

The impedance spectra for bare magnesium alloy type ZE41A sample alone and coated with Tagnite (immersion and brush) in 3.5% NaCl solution are presented as Nyquist plots as shown in Figure 9. From AC impedance nyquist plot, It is clear that the diameter of the semicircle obtained for coated Mg alloy greatly increased than that obtained for bare Mg. An equivalent circuit was proposed to represent the corrosion of Mg alloy in chloride solution shown in Figure 10, where  $R_s$  is the solution resistance between Mg and the platinum electrode,  $R_1$  is the resistance of a film on the surface of Mg, and  $R_2$  is the polarization resistance at the Mg surface and CPE 1 and CPE 2 are the constant phase elements. CPE is substituted for the capacitive element to give a more accurate fit, where the capacitive loop obtained is a depressed semicircle rather than a regular one.

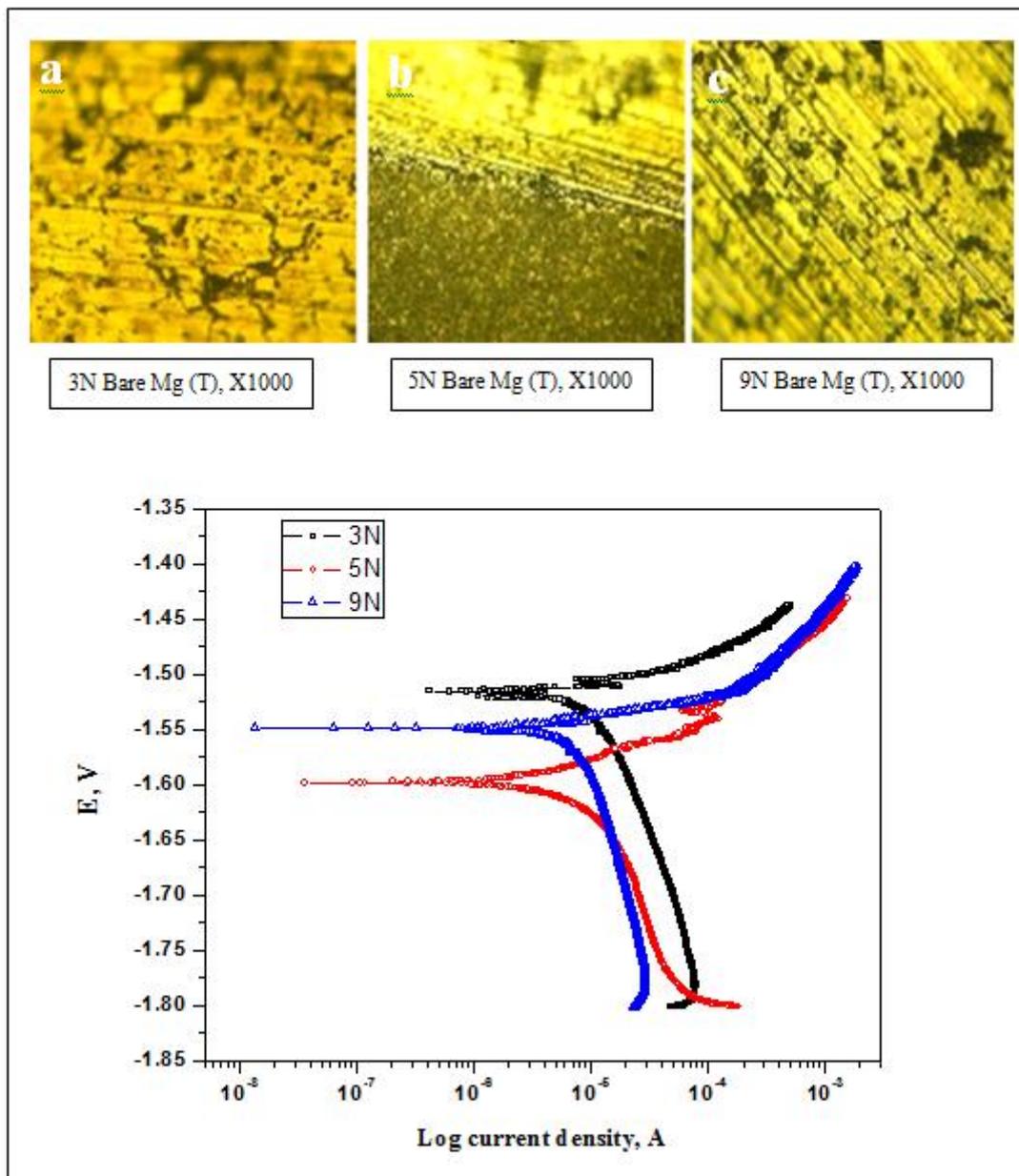
Deviation from the ideal semicircle is usually attributed to frequency dispersion, roughness and inhomogeneity of the surface, and to resistance to mass transport [24]. The obtained data showed that the CPE decreases significantly in presence of the brush Tagnite from 29.1 to 0.86  $\mu\Omega^{-1}s\text{ cm}^{-2}$  and in presence of immersion Tagnite from 29.1 to 8.3  $\mu\Omega^{-1}s\text{ cm}^{-2}$ . The reduction in CPE may be due to the decrease in local dielectric constant and/or a reduction in the thickness of the double layer suggesting an increase in the protection of coats. Moreover, the polarization resistance ( $R_2$ ) increases in the presence of brush Tagnite from 3.6 to 13.42  $k\Omega\text{ cm}^2$  and from 3.6 to 11.96  $k\Omega\text{ cm}^2$  in the presence of immersion Tagnite. The presence of coats successfully improves the corrosion resistance of magnesium alloy compared to the uncoated one. Moreover, the Brush Tagnite coat is more beneficial in improving the corrosion resistance of magnesium alloy than the immersion Tagnite coat. The EIS results are in agreement with the data obtained from potentiodynamic polarization measurements.

### 3.6 Friction wear mechanism

Mechanical abrasive wear mechanism dominated the Tribology wear (T) test as shown in Fig, 5. When the sliding magnesium surface passes the static ball, the oxide film was removed and the surface was scared, the film try to re-grow again, but the electrochemical reaction is not fast enough to maintain a considerable film thickening with effective film/surface adhesion to overcome the ball friction forces. Eventually, the oxide film was ruptured and broke away under the assigned load stresses. In figure 4 illustrate the high oxidation and metal dissolution mechanism on the alloy surface during the Tribo-Potential (TP) test. The oxide film was thickened due to high oxidation level as a result of high anodic polarization potentials and seawater immersion. The static ball does not get in contact with the alloy surface, which related to the high film thickness and the fast oxide films re-growth.

Figure 11 shows a potentiodynamic scans, which illustrates the effect of friction wear load and potentials on polarization curves during tribology wear test. The Brush Tagnite (275) sample at anodic potential of -1.5 V, produced the lowest value current density of  $2.19E^{-4}$  and a corrosion rate of 5 mm  $y^{-1}$  at  $i_{corr}$  of 5.6  $\mu A$ . As a result of a thicker coating with fewer coating capillaries and on the other hand, the mechanism of abrasion oxidation of the oxide film which limited the ionic exchange at surface-solution interface. This was confirmed by Song et. al [25], that passive films on magnesium alloy cannot exist in acid solutions but there are partial films on magnesium alloy in neutral and basic

solutions, which provide moderate protection to the matrix alloy, resulted in the increase of corrosion potential and decrease in film surface.

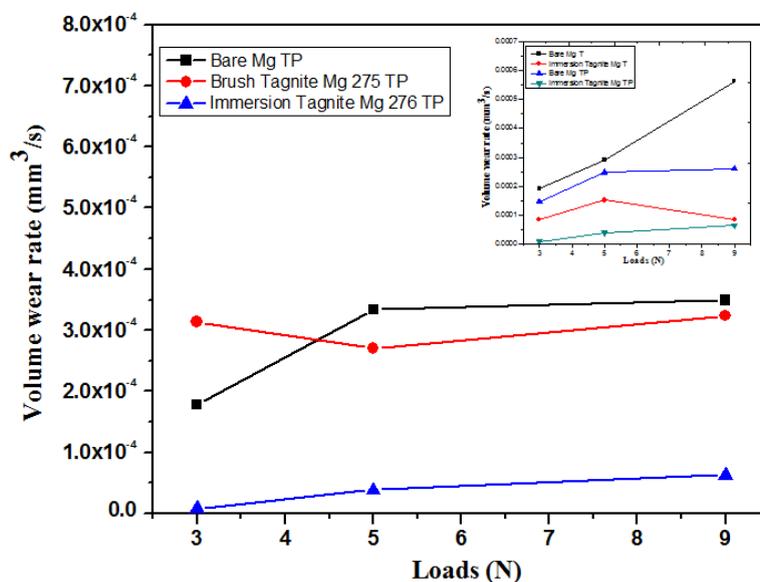


**Figure 11.** Tribo potentiodynamic scans of Mg coated with brush Tagnite (275) under 3, 5 and 9 N loads in seawater solution.

### 3.7 Volume wear rate calculation

The volumetric wear rate of the ZE41A alloy in Fig. 12 during the (TP) test, validated the reduction in C.O.F. The (TP) tests showed a lower volume wear rates than the (T) tests, while the (T) tests volume wear rates increased linearly as applied load increases and value (T) test for the coating is lower than the bare sample (TP) test, which was due to the existence of the coatings anodic barrier on

the surface of the sample. The findings concluded that in both tests coefficient of frictions for (ZE41A) magnesium alloy will increase as wear load increased in seawater solution after prolong period of time, this were caused by the different friction wear mechanisms, abrasive wear in Tribology wear (T) test and oxidation in the Tribo-Potential wear (TP) test, until an equilibrium state of oxidation reaction and load stress is reached, C.O.F tends to stabilized.



**Figure 12.** Volume wear rate for bare Magnesium (ZE41A) alloy alone and coated with immersion and brush Tagnite methods.

As reported by Rai. et al.,[26] the wear rate is significantly dependent on the applied load. In (TP) test there was an apparent relationship between the low current densities at -1.5 V and the reduction in coefficient of friction values. This was due to less metal dissolution of the protective coating by brush than the immersion Tagnite one.

The increase in volume wear rates in Tribology wear (T) tests in Figure 12, were mainly due to the domination of abrasion mechanical wear mechanism over the oxidation one, while a reduction in volume wear rate occurred in the Tribo-Potential (TP) tests were attributed to the oxidation domination dissolution mechanism, hence, thick oxide films were protecting the metal surface.

**4. CONCLUSIONS**

- In absence of wear (Tribocorrosion analysis) the studied coats act as an excellent barrier for the transportation of electrons and provide good protection to the Mg alloy against corrosion.
- The synergistic effect of corrosion and wear at the same time can accelerate the coating failure and mass loss to the studied alloy.
- Magnesium ZE41A alloy shows better wear resistance under Tribo-Potential wear test (TP) than Tribology wear test (T).

- Tagnite coating by brush method have a better corrosion resistance than the immersion (276) method.
- Tribo-Potential test (TP) magnesium samples produced poor adhesion oxide film compared to the Tribo-wear test (T).
- Domination of oxidation wear mechanism in Tribo-Potential wear (TP) tests, while the domination of mechanical abrasive wear mechanism in Tribology wear (TW) tests.
- Sever pitting corrosion and magnesium dissolution in (TP) tests which was attributed to the dissolution of second phase elements and corrosion in the anodic regions on the Mg alloy surface.

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