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THE CORROSION INHIBITION EFFECT ON THE MAGNESIUM ALLOY AZ91C'S FATIGUE BEHAVIOR IN THE SALINE ENVIRONMENT

Mohammed S.R. SALEH[1], Magdi E.M. EL-GAROSHI[1], Ali F. Ali FADIEL[1,*], Hafiez M.B. KHALID[1]

[1]Mechanical Engineering Department, Higher Institute for Sciences and Technology, Tobruk, Libya

Abstract

Investigations were made into the fatigue in the magnesium alloy AZ 91C cast in corrosive fluids and air at varied NaCl solution concentrations. Fatigue specimens will be subjected to surface stress testing in corrosive conditions with a stress amplitude of 75 MPa both after and before coating. To comprehend the behavior of the magnesium material, its fatigue life, corrosion product (X-ray spectra), and fracture properties will be examined. The corroding medium (NaCl) has been found to significantly shorten the fatigue life of the magnesium alloy AZ91C. Chemical conversion treatment was applied to the AZ91C alloy to improve corrosion resistance and corrosion fatigue resistance. MgSnO3 was found in the XRD patterns of the stannite-treated AZ91C alloy, and this treatment slightly improved the alloy's corrosion resistance in an alkaline stannite solution with a pH of 7. H2O and SEM images demonstrate that chloride ions can penetrate the substrate through porous structures.

Keywords*:* *magnesium alloy, corrosion resistance, fatigue life, Chemical conversion treatment*

Introduction

In all of the trials conducted for this investigation, the under-test medication, AZ91C, whose chemical make-up is shown in Table 1, was used. The two phases that make up the early microstructure are the phase and the -phase. The intermetallic phases, Mg17Al12, which contain more than 40% aluminum, are the -phase, a solid mixture of magnesium, aluminum, and zinc that shares the same crystal structure. The free corrosion potential of this intermetallic complex, which is divided at the grain boundaries, is almost -1.0 V as opposed to -1.73 V for the -phase [1].

Metals General Co. in the area supplied the as-cast AZ91C plate, It was machined to a length of 120 mm and a diameter of 50 mm. 30 x 30 x 90 mm and was used to fatigue test samples with a thickness of 3 mm [2]. A variety of circumstances, including corrosion fatigue and the alloys studied were tested for weight loss during immersion. In order to ascertain the fatigue life in various settings, corrosion fatigue experiments were performed on flat tensile specimens with a 3 mm thickness and the dimensions as seen in Fig. 1.

Table 1. AZ91C Mg alloy chemical composition (wt. %) [3]

Alloy	Al	Zn	Mn	Si	Cu	NI	Fe	Mg
AZ91C	8.80	0.71	0.19	0.029	0.002	<0.001	0.0010	Bal.

*Corresponding author: dr_ali.f@hotmail.com

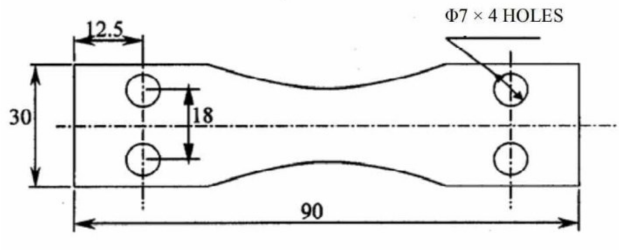


Fig. 1. Test sample dimensions in a 3 mm thickness

All test specimens were washed, degreased, and dried in the air after being polished with consecutive grits of 280, 400, 800, and finally 1200. The corrosion resistance of a material is evaluated using immersion testing under various circumstances. We calculated weight loss using a Rectangular sample with dimensions 30 x 30 x 4.80 mm. Following cleaning and degreasing, Before and after testing, the samples were weighed on an analytical scale with a measurement accuracy of 0.0001 g. The tests were placed over the course of two days.

After removing the corrosion products from the sample surfaces using conventional methods, the test samples were weighed.

Additional specimens of the same sizes were used in the accelerated corrosion studies. Table 2 illustrates the alloy's mechanical properties.

Table 2. AZ91C Mg Alloy Mechanical Properties [3]

Yield Strength, MPa	Tensile Strength MPa	Elongation % in 50 mm g.l.	Hardness HB	Elastic modulus GPa
145	275	6	68	45

The samples were sanded on fine paper with 1200 grit followed by a polishing process using pads and aluminum oxide for continuous immersion testing. Then Each sample was first cleaned in accordance with ASTM G 172-03[4]. The polished and reweighed samples were exposed to 1%, 5%, and NaCl solutions at different time intervals. In the end, wash the samples well by immersing them in 100 ml of a 15% CrO_3 solution in boiling water. This is followed by an acetone wash. Each experiment ends with annual weight loss measurements and corrosion rate determination in millimeters.

Methods and Methods

The corrosion fatigue studies were conducted bending fatigue testing on a plane apparatus Fig. 2. The apparatus is set up to allow for the evaluation of specimen fatigue life in diverse contexts. Multiple fatigue stress cycles with varying maximum and minimum stresses were part of the conditions for each trial. They are both in tension, the mean stress (m) is 75 MPa, the maximum stress (m) is 100 MPa, the proportion of the lowest to maximum stress (R) is 0.5, and the frequency is a constant 13.3 Hz (800 rpm) [5].

Environment

The inhibitor of choice for this study was the theorem. Complete immersion test in 1%, 5%, and, NaCl. The exposure time is two days. Complete immersion tests were performed in an aqueous (5%) sodium chloride solution at pH = 7 in The presence of inhibitors. The overall amount of inhibitory tincture Present in 5% NaCl aqueous solution at different concentrations (0.01, 0.05) g/l. The test specimen's weight loss is used to calculate the corrosion rate. For this purpose, corrosion products are removed [6].

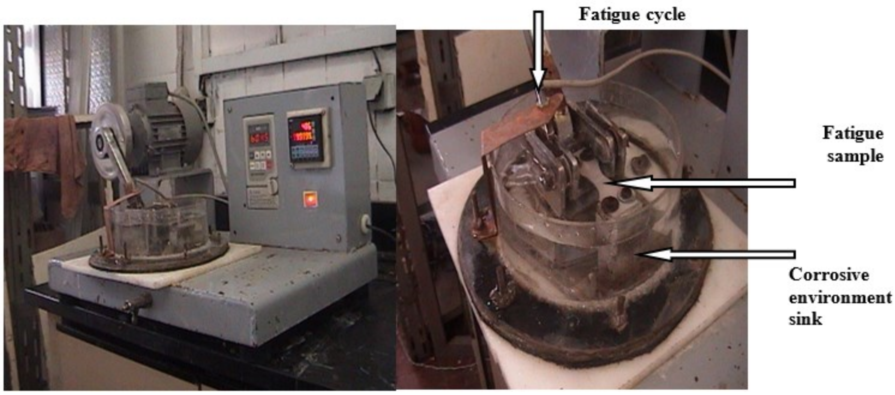


Fig. 2. Machine for assessing plane-bending fatigue

Different Procedures of Experimental Work

A flowchart of the experiments employed in this study's tests is shown in Fig. 3.

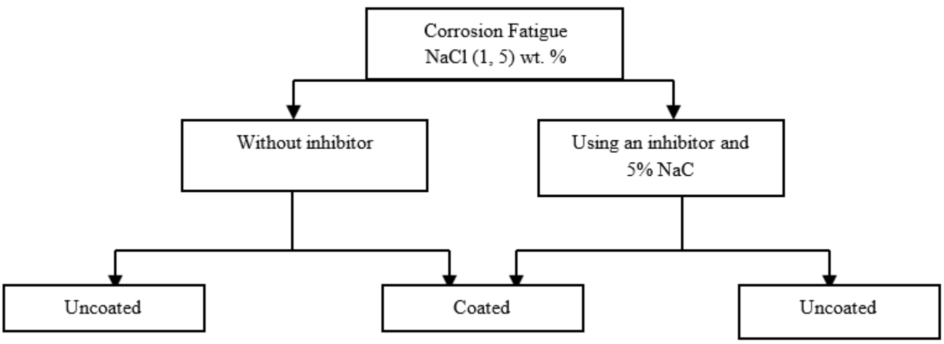


Fig 3. Different procedures of experimental

Technique

Test specimens or components are put through enough stress cycles to start showing cracks in cycles-to-failure testing, which propagate until complete breaking occurs. Examining smooth or notched specimens is usually how this information is found. However, it can be difficult to distinguish between CFC propagation life and CFC initiation using this type of testing.

Rates of existing cracks advancing using fracture mechanics techniques, crack propagation testing evaluates crack propagation under cyclic stresses. A material's ability to initiate cracks is reduced or completely eliminated by pre-existing cracks or acute flaws. Testing of both kinds is crucial. Though it seems that fracture growth predominates thick-section component durability, crack initiation appears to be more significant in the failure process of comparatively thin sections [7]. Corrosion fatigue experiments were performed at continuous frequencies of 13.3 Hz, a stress ratio of 0.5, and a mean stress of 75 MP (complete fracture). For each specimen, the estimated number of cycles to failure (Nf) was calculated. It was looked into how Nf and the type and amount of the chemical environment related to one another.

In order to examine the surface morphology of the specimens and their fracture mechanism, corrosion fatigue testing specimen surfaces were characterized using XRD thin films (Panalytical Xpert Pro), optical microscopy, and scanning electron microscopy (Jeol-5410).

Results and Discussion

Extensive metallographic examinations on the fatigue specimens were done in order to drive a life-prediction concept based on microstructural damage mechanisms. Similar observations of fracture initiation for die-casting AZ91C were reported, and it appears that crack initiation during fatigue occurs at contraction cavities at or below the surface. The essential discontinuity's location and nature, as well as any obvious patterns in the corrosion fractures, forms, and degree of damage, were all revealed by fractography. The correlation between the effect of NaCl concentration and fatigue life (Nf) is depicted in Fig. 4. It is before the coating that the material's structure is significantly damaged by the NaCl content. and, as a result, lowers the material's corrosion resistance and fatigue strength. Additionally, it is discovered that for the magnesium alloy AZ91C in the corrosive media was attained at 5108 cycles, which is a significant difference in the number of cycles to failure (Nf).

and that obtained in the air Nf=3103 cycles. The material exhibits a comparatively high fatigue life at 1% NaCl (Nf=140000 cycles). The fatigue life was lowered to Nf=109000 and 90000 cycles with ratios of -22 and -35.7%, respectively when the NaCl concentrations were increased to 3 and 5%. The fatigue life Nf is drastically reduced to 55300 cycles, or -60.5%, by raising the NaCl concentration to 7%. It is evident that the NaCl gradually damages the structure of the Mg alloy after coating. Additionally, it has been discovered that the coating layer enhances and protects fatigue life. The fatigue life for 1% NaCl rises from 140000 cycles before coating to 230220 cycles after coating (an increase in Nf of approximately 64.4%). The fatigue life significantly worsens at 3% NaCl concentration (45% reduction in Nf). The fatigue life is somewhat reduced by increasing the NaCl concentrations to 7%, going from 11000 to 103000 (3.6% less Nf).

Figures 5 and 6 show typical surface fractures of the rope surface after exposure time. Localized corrosion affects the entire surface and persists, leading to general corrosion after the exposure period. The degree of erosion indicates the corrosiveness of the corrosive medium. Cracks begin at the surface, usually at points of high-stress concentration (also called stress intensity factors), and propagate farther and farther with each increase in load until the remaining stress area eventually becomes too small to statically support the load [8] are: Are as follows: The crack spreads because the crack tip zone is too small.

During its expansion, there was a crack deflection and crack branching. The crack tip is reduced when the fracture is diverted off its typical growth plane, increasing the fatigue crack growth resistance [9, 10].

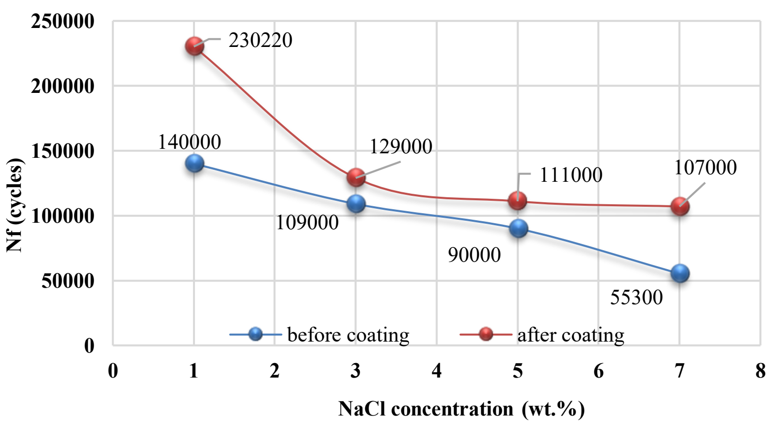


Fig. 4. The concentration of NaCl affects the fatigue life of bare and coated samples

As the crack length rises, crack closure may also contribute to slower crack propagation. Fig 5. AZ91C following corrosion in 1 % NaCl, in a SEM micrograph before coating. It seems clear, that the NaCl moderately attacks the surface of AZ91C giving crack initiation. However, Total fatigue surface aspects ensure a lower severity of 1% NaCl (see image b in fig. 5)

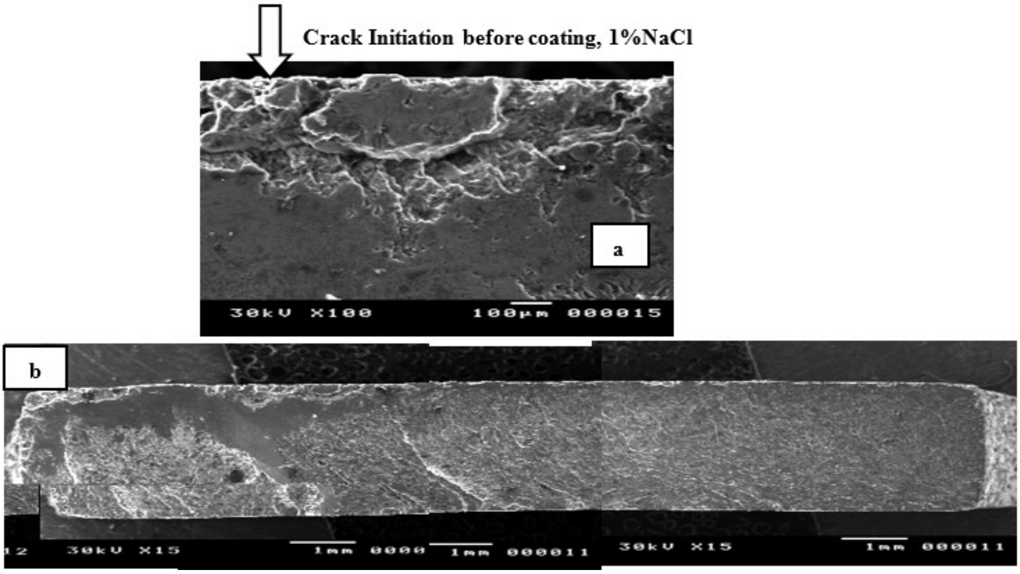


Fig. 5. SEM micrograph demonstrating the impact of 1% NaCl concentration on the fracture surface

Fig. 6 shows a SEM micrograph of AZ91C before coating following corrosion in 5% NaCl. It shows catastrophic crack initiation. It seems clear that the NaCl severely attacks the surface of AZ91C. However, Total fatigue surface aspects exhibit high severity of surface roughness due to 5% NaCl concentration (see image b in fig. 6).

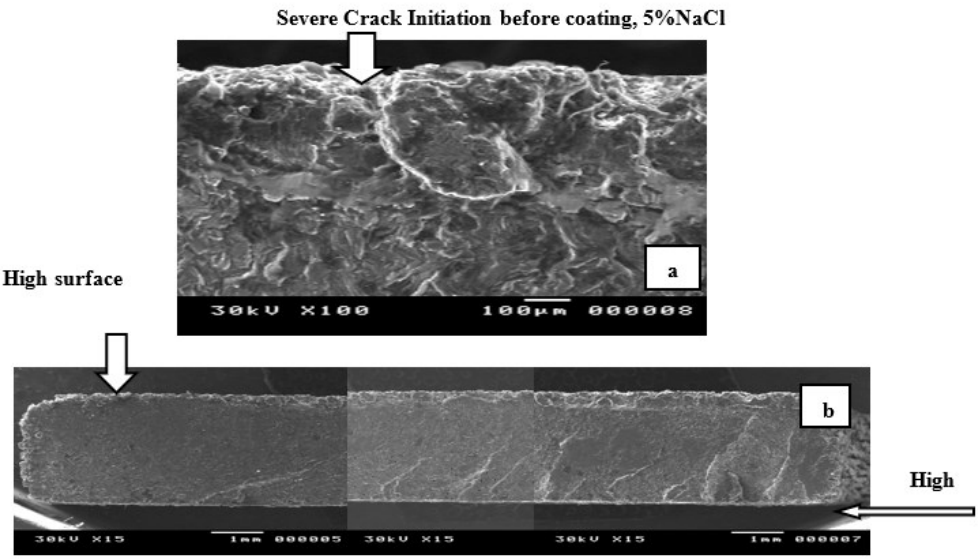


Fig. 6. SEM micrograph illustrating the impact of 5% NaCl concentration on fracture surface

After coating, the material in 1% NaCl exhibits uniform distribution of the fatigue crack initiation as shown in Fig. 7. and the crack initiation resistance is enhanced than uncoated samples. Therefore, the fatigue life relatively increases.

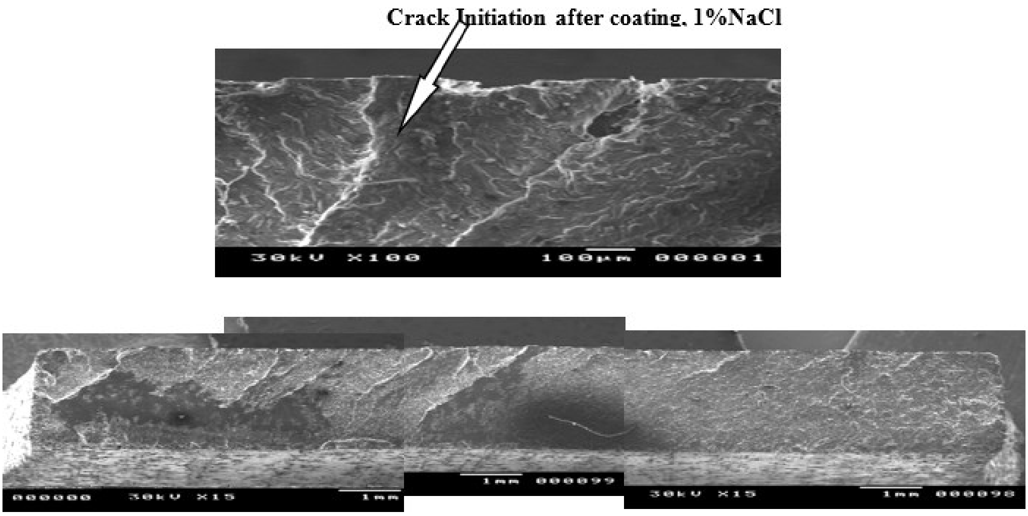


Fig. 7. SEM micrograph of showing the Effect of 1%NaCl concentration on fracture surface after coating

For 5% NaCl, the material exhibits severe attack of corrosion pits leading to a high concentration of surface roughness (a lot of the fatigue crack initiation sites) as Shown in Fig. 8. While the crack initiation resistance decreases by 1% NaCl of coated samples. Therefore, the fatigue life relatively deteriorates.

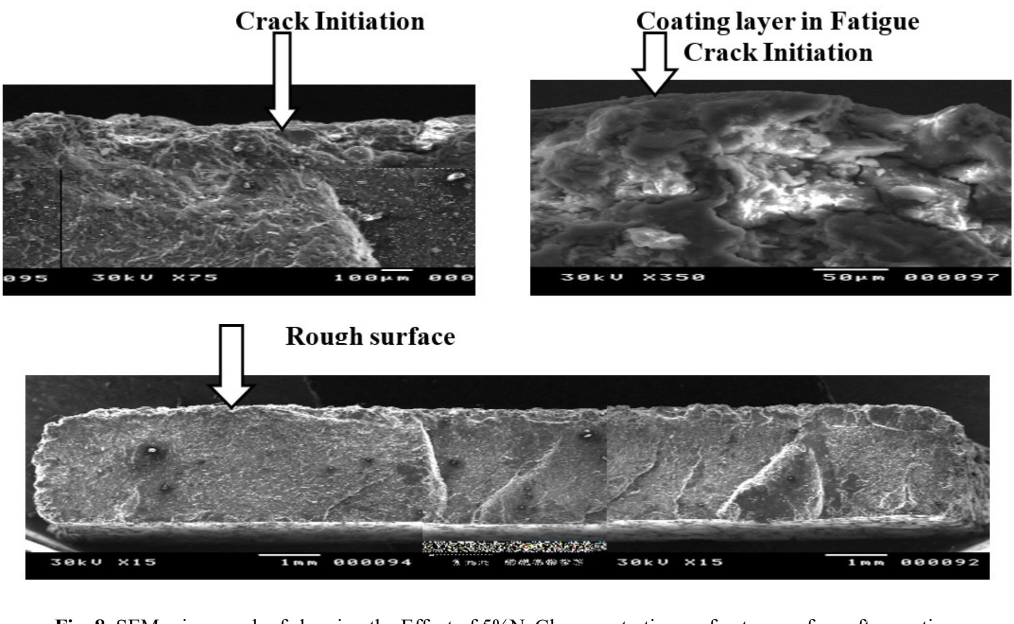


Fig. 8. SEM micrograph of showing the Effect of 5%NaCl concentration on fracture surface after coating

Effect of a theorem on fatigue life

Fatigue tests are conducted in an aggressive corrosive medium (NaCl) with inhibitors (thiourea in various concentrations) both before and after coating in order to increase the fatigue life of AZ91C.

In corrosive corrosion media (5 percent NaCl) concentration, the effect of different thiourea concentrations on the fatigue life of Mg-alloy AZ91C (bare and coated samples) is depicted in Fig. 9. As can be observed in the same figure, the fatigue life of Mg-alloy AZ91C generally increases whether it is coated with the thiourea inhibitor before or after.

As thiourea concentrations rise, the fatigue life of AZ91C before and after coating increases until it reaches a maximum value at 0.03g/l thiourea, at which point it starts to decline. At 0.03 g/l thiourea, the largest number of cycles—118000 on the bare sample and 145964 on the coated sample—are obtained; at 0.01 g/l thiourea, the lowest number of cycles—70200 on the bare sample and 9130 on the coated sample—are obtained. The coated AZ91C alloy has a considerably longer fatigue life than the uncoated sample.

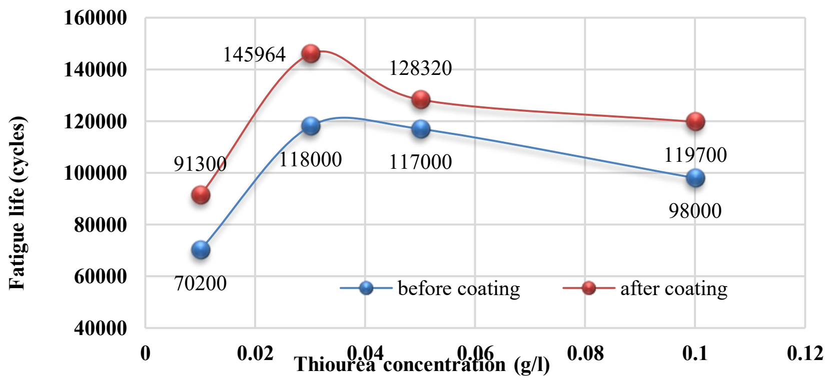


Fig. 9. The influence of different concentrations of theorem of fatigue life of Mg-alloy AZ91C, on uncoated and coated samples in aggressive corrosion medium (5%NaCl)

Figs. 10 and 11 show SEM micrographs of AZ91C after corrosion in 5%NaCl before coating. They show the crack initiation of uncoated AZ91C samples (subjected to 5%NaCl in the existence of 0.01 or 0.05 Thoreau as an inhibitor). It is clear that the fatigue crack initiation is highly affected in the case of 0.01thiorea than 0.05 g/l theorem. As opposed to that, coated AZ91C samples see Figs. 12 and 13 show relatively high fatigue life in relation to uncoated samples. However, the fatigue crack initiation shows a higher delay of 0.05 g/l than of 0.01 g/l.

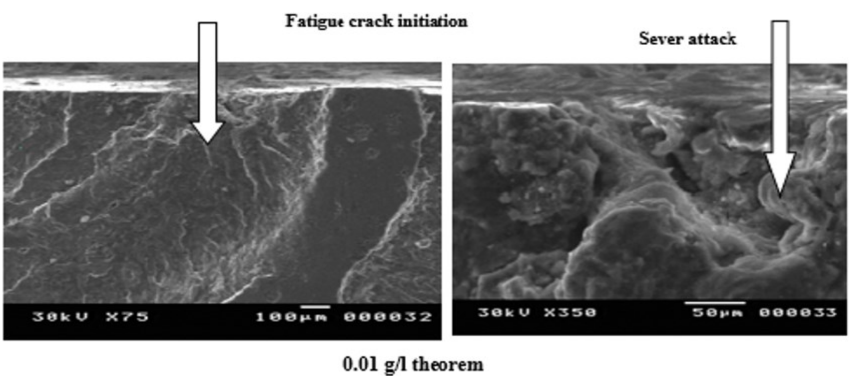
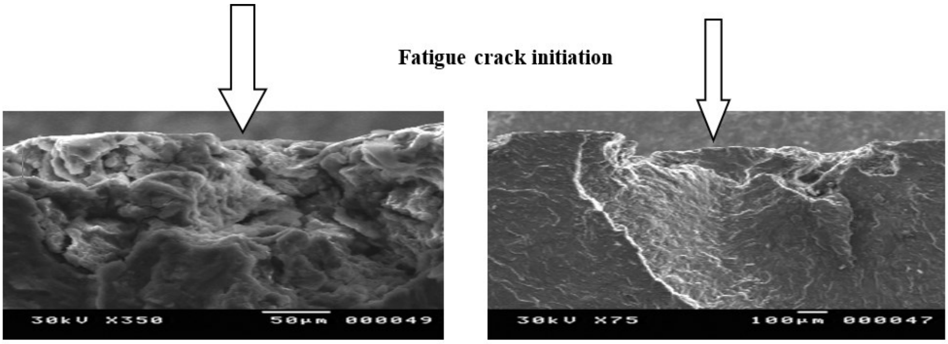


Fig. 10. SEM micrographs showing effect of theorem concentration on crack initiation before coating (5%NaCl)



0.05 g/l theorem

Fig. 11. SEM micrographs showing effect of theorem concentration on crack initiation before coating (5%NaCl)

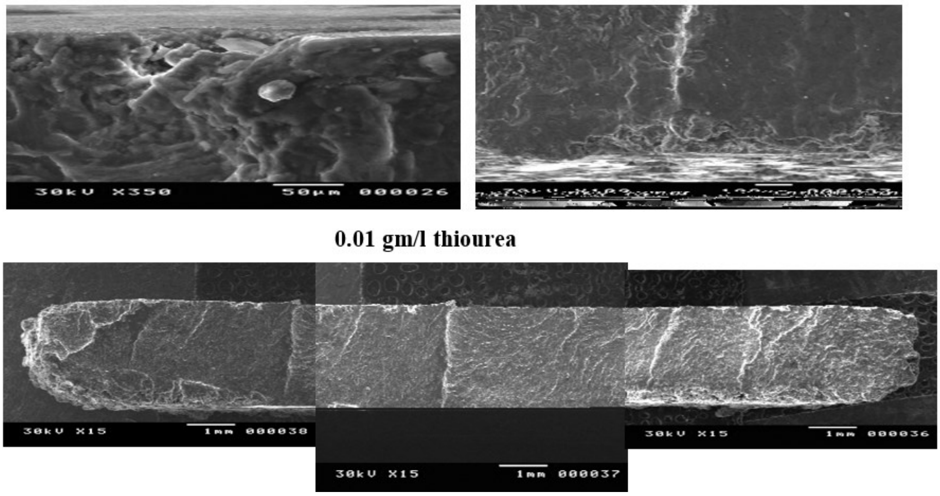


Fig. 12. SEM micrographs showing theorem concentration (0.01g/l) effect on crack initiation for coated samples

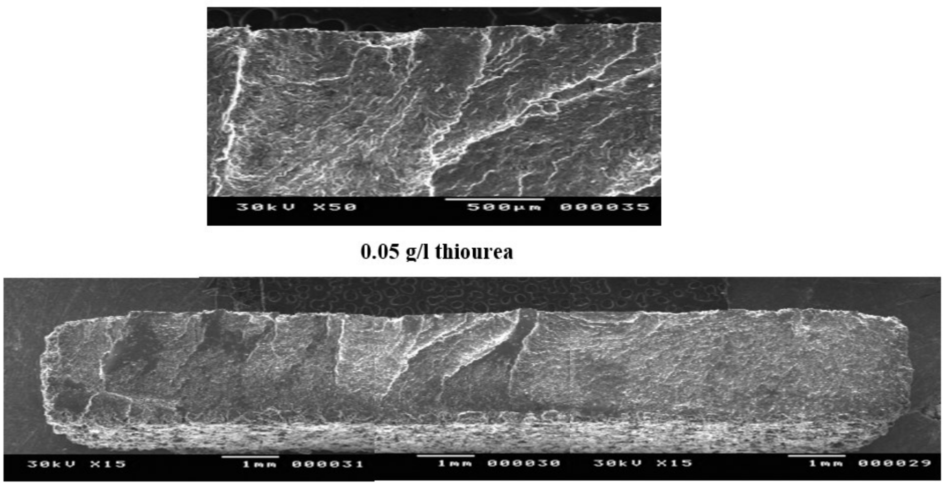


Fig. 13. SEM micrographs showing effect theorem concentration 0.05 g/l on crack initiation for coated samples

Inhibitor and Coating Efficiency

The overall effectiveness (IE) of the corrosion fatigue test is calculated using the following equation (1).

$$BE(\%) = \frac{Nfwith - Nfwithout}{Nfwithout} \times 100 \quad (1)$$

Where: Nf without Fatigue life without inhibitor and without a coat; Nf with Fatigue life with inhibitor and with a coat

Table 3. Percentage of the coating layer's boosting effectiveness at varied NaCl concentrations (pH = 7)

Solution	Coating Efficiency (CE), % Corrosion fatigue test
1% NaCl	64.4
5% NaCl	23.3

Table 4. The percentage inhibition efficiency of Thoreau in 5% NaCl (pH=7)

Solution	Inhibiting Efficiency (IE), % Corrosion fatigue test
5% NaCl + 0.01 g/l Thoreau	-22
5% NaCl + 0.05 g/l Thoreau	30

Table 5. At 5% NaCl (pH=7), the percentage-enhancing effectiveness of both coating and inhibitor

Solution	Efficiency Combined (BE), % Corrosion fatigue test
5% NaCl + 0.01 g/l Thoreau	1.5
5% NaCl + 0.05 g/l Thoreau	42.6

Conclusions

The conversion coating has many non-penetrating pores and is about 2 m thick.

Before the coating, it was clear that increasing the NaCl level seriously damaged the material's structure and decreased its fatigue strength and corrosion resistance.

For samples that aren't coated, the fatigue-emanating crack starts at the surface, whereas it starts in corrosion pits.

The paint layer reduces the rate of corrosion by approximately 46.5% when sodium chloride is used.

When the NaCl concentrations were raised to 5%, the fatigue life was somewhat reduced to Nf =109000 and 90000 cycles with ratios of -22 and -35.7%, respectively. Raising the NaCl content to 5% substantially reduces the fatigue life Nf to 55300 cycles, or -60.5%. It is obvious that after coating, the NaCl gradually weakens the structure of the Mg alloy.

The fatigue life is improved and protected by the coating layer. With 1% NaCl, the fatigue life increases from 140000 cycles prior to coating to 230220 cycles following coating (an increase in Nf of almost 64.4%).

Future Directions and Limitations

Corrosion of magnesium alloys is a complex process, and how corrosion acts is heavily influenced by the environment. Experimentation is the main research method used to study the particular corrosion mechanisms of magnesium alloys. The process underlying wear behavior as well as the properties of materials in this field require in-depth study.

To stop magnesium alloys from corroding, many people are interested in alloying. The application scope of the material can be further broadened by incorporating rare earth elements, which display greater effects and improve the corrosion performance of magnesium alloy. Further study is required to comprehend how the rare earth elements added to magnesium alloy affect corrosion resistance because there is no single model for the corrosion resistance mechanism.

Coating techniques can successfully boost corrosion resistance by limiting contact between the surface and the corrosive media by producing a film with a distinct structure on the surface of the magnesium alloy. The advancement of this technology will determine how well magnesium alloys are used. The coating is one of these elements that help with local corrosion treatment. It is necessary to create models that precisely explain the processing stages and govern the microstructures and compositions on the surface in order to increase the treated magnesium alloy's corrosion resistance after the coating has been damaged.

The coatings on modern magnesium alloys must not only prevent corrosion but also be corrosion-resistant in a variety of environments due to the vast range of uses for these materials. This necessitates complex surface preparation techniques. It is necessary to promote research into the surface engineering of magnesium alloys and to build relevant databases for corrosion and protection.

References

[1] M. S. Saleh, A.F.A. Fadiel, H.M. Khalida, *Corrosion Rate Study of AZ91C Magnesium Alloy in Sodium Chloride Solution of Different Concentrations Using Immersion Method for Coated and Uncoated Samples*, **International Journal of Advances in Engineering Research, 26 (II)**, 2023, https://doi.org/10.6084/m9.figshare.24077319.v1

[2] A. Powell, *A Magnesium Clean Energy Ecosystem Vision*. In: Maier, P., Barela, S., Miller, V.M., Neelameggham, N.R. (eds) Magnesium Technology 2022. The Minerals, Metals & Materials Series. **Springer, Cham**, 2022, https://doi.org/10.1007/978-3-030-92533-8_20

[3] L.D.K. Catherine, D.A. Hamid, *Mechanical properties and machinability of magnesium alloy AZ31 and AZ91–a comparative review*, **IOP Conference Series: Materials Science and Engineering**, 1062(1), 2021, p. 012054

[4] D. Singh, D.A. Basha, L. Wadsö, D. Orlov, Y. Matsushita, A. Singh, S.S. Hosmani, *Evolution of gradient structured layer on AZ91D magnesium alloy and its corrosion behavior,* **Journal of Alloys and Compounds**, **882**, 2021, p. 160659.

[5] K. Eldwaib, M. Aldarwish, M. Ballem, S. Garrab, S. Adrwish, *The Corrosion Inhibition Effect on Fatigue Behavior for Aluminum Alloy 5052 in the Saline Environment,* **Journal of Pure & Applied Sciences, 21(2)**, 2022, pp. 18-22.

[6] Z. Shi, M. Liu, A. Atrens, *Measurement of the corrosion rate of magnesium alloys using Tafel extrapolation*, **Corrosion Science**, **52(2)**, 2010, pp. 579-588

[7] W. Zhou, N.N. Aung Y. Sun, *Effect of antimony bismuth and calcium addition on corrosion and electrochemical behavior of az91 magnesium alloy,* **Corrosion Science**, **51(2)**, 2009, pp. 403–408. https://doi.org/10.1016/j.corsci.2008.11.006

[8] G. Subhash, S. Ridgeway, *Mechanics of Materials Laboratory Course,* **Springer Nature**, 2022

[9] H.A. Richard, M. Sander, *Fatigue crack growth, Vol. 18,* **Springer, Cham**, 2016, https://doi.org/10.1007/978-3-319-32534-7

[10] C. Berlanga-Labari, M.V. Biezma-Moraleda, P.J. Rivero, *Corrosion of Cast Aluminum Alloys: A Review,* **Metals**, 10, 20201384. https://doi.org/10.3390/met10101384

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