# Study The Corrosion Behavior for AS41 MMCS

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**Abstract:** The corrosion behavior of carbon fiber reinforced Mg alloy (AS41)-based metal matrix composites has been studied and compared with that of AS41.The corrosion behavior was studied using gravimetric corrosion measurement and electrochemical techniques. The corrosion experiments have been carried out in neutral, acid and alkaline solutions. The presence of carbon fiber deteriorated the corrosion resistance of magnesium alloy. Carbon, as an electrically conductive element, is electrochemically unstable despite its almost chemical stability in aqueous chloride environments. Therefore, the polarization diagram of MMCs were demonstrated in terms of the electrochemical activity of carbon fibers.

**Keywords:** Corrosion, magnesium alloys, AS41, (AS41+C)

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## "(1)". Introduction

Magnesium alloys reinforced with carbon or graphite are one of the promising candidate materials for applications in which as high strength/weight ratio is required. Magnesium serves as an excellent matrix for Metal-Matrix composites as it has an excellent bonding affinity to the reinforcing materials. Currently, the three main systems of magnesium alloys used commercially from die casting processes are magnesium-aluminum-zinc (AZ) such as AZ91, magnesium-aluminum-manganese (AM) such as AM60 and magnesium-aluminum-silicon (AS) such as AS41[1]. Magnesium composites can be manufactured using continuous casting, squeeze casting, diffusion bonding and powder metallurgy. The main principle of Metal-Matrix composites is the incorporation of a high performance second phase, such as carbon, metallic or ceramic addition, into a conventional engineering material, such as aluminum, magnesium or titanium, to produce a combination with features that are not obtained from any of the individual constituents by itself [2]. Mg-MMCs show nearly the same corrosion behavior as standard magnesium alloys. However in the case of carbon fibers, more care has to be taken. Magnesium-Matrix composites are being developed in order to reach the same properties as those reached by Aluminum-Matrix composites. In practice, the two main criteria among which lies the choice of using either aluminum or magnesium as a matrix are the weight versus the corrosion resistance. The corrosion of magnesium is highly dependent on the presence of cathodic constituent phases and thereby galvanic corrosion get up between the Mg-matrix alloy and the noble reinforcement constituent [3,4].The corrosion behavior of magnesium differs according to the surrounding environment. Chloride contaminants on the surface destroy that surface film as magnesium chloride is formed which causes the accumulation of anodic sites leading to micro galvanic couples and severe local attack. Salt and in particular chlorides increase the corrosion rate. Moderate attack is suffered in industrial atmospheres. The corrosion resistance of magnesium and magnesium alloys depend on the same factors that are critical for other metals [4] The corrosion products formed on the surface of magnesium alloys differ depending on the location of exposure of these alloys. Analysis of corrosion products taken from the surface of magnesium alloys that were exposed to rural atmosphere for 18 months showed the presence of various hydrated carbonates of magnesium. For magnesium alloys subjected to industrial atmosphere, hydrated as well as carbonated together with magnesium sulphates and magnesium sulphites where found. Similar analysis to the previous done on samples exposed for shorter periods showed that the main corrosion reaction for magnesium alloys was the formation of magnesium hydroxide followed by a secondary reaction with carbonic acid converting the hydroxide to hydrated carbonate. In sulfur contaminated atmospheres, sulfates may be present in the corrosion compounds. The aim of this study is to study the corrosion behavior of carbon fibers reinforced metal - matrix composite AS41 magnesium alloys compared to corrosion behavior of AS41 magnesium alloy by using gravimetric corrosion measurements and electrochemical techniques.

| **Alloy** | **Elements (wt %)** | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|
| AS41 | **Al** | **Si** | **Zn** | **Mn** | **Ni** | **Cu** | **Fe** | **Pb** | **Sn** | **Mg** |
| | **4.37** | **0.93** | **0.09** | **0.35** | **<0.0005** | **<0.0002** | **0.0028** | **0.0027** | **0.0049** | **Balance** |

## "(2)". Experimental Procedures

### 2.1: Chemical Composition Analysis

The chemical composition of the samples was detected using ICP-AES (Inductive Coupled Plasma by Atomic Emission Spectroscopy) for detection of very low alloying concentrations. The chemical composition is shown in the table (1).

**Table 1:** The chemical composition of the samples:

The carbon reinforced magnesium-matrix composite AS41 alloy differs only in composition from the AS41 alloy in the presence of about 25% of short carbon fibers. The composite was produced by squeeze casting in which the molten magnesium alloy was forced into the carbon short fiber performs.

### 2.1.1 : Surface Preparation

All test samples were grinded and then polished to have a uniform surface which is clear of scratches and clean of any impurities. The grinding process is done on the grinding/polishing machine. The grinding papers used were 320, 500, 800, 1000 and finally 1200.Polishing is then done on the same machine by using a set of finer grinding papers. The lubricant used was a diamond paste. The dimensions of test samples are indicated in Table (2 ).

**Table 2 :** The dimensions of test samples:

| The materials | Autoclave Test (Area mm$^2$) | Industrial Atmosphere (Area mm$^2$) | Marine Atmosphere (Area mm$^2$) | Electrochemical Corrosion test (Area mm$^2$) |
|---|---|---|---|---|
| **AS41** | 258.38 | 1033 | 448.23 | 120 |
| **AS41+C** | 488.42 | 778.65 | 550.22 | 120 |

### 2.2 : Microstructure Investigation

The samples have been itched by using etching solution (25 mL distilled water, 75 mL ethylene glycol and 1 mL HNO3). Optical microscopy was used to investigate the itched samples under magnification of 50X. The pictures have been transferred to the computer through a digital camera.

## 2.3 : Gravimetric Corrosion Measurement

### 2.3.1: Autoclave Test (Highly Accelerated Stress Test)

The autoclave test is used to determine the weight change of the samples under hydrothermal conditions. Before the test, the samples were first cleaned by distilled water and then weighted. The three samples were then suspended using a Teflon wire inside the autoclave. The autoclave contained distilled water at a temperature of 150°C and a pressure of 5 bars. The samples were removed every 12 hours for 60 hours in order to investigate the weight change. Each samples was weighted and the new weight was subtracted from the initial weight ($\Delta W = W0 - W1$) where W0 is the initial weight and W1 is the new weight. The resulting weight change was divided by the area ($\Delta W/A$) and a curve was drawn where the weight change per area in g/mm$^2$ is plotted against time in hours.

### 2.3.2 Salt Spray Test

The salt spray apparatus is designed to allow the samples to be subjected to similar atmospheres in industrial and marine conditions. The samples were placed 5 cm from the specific solution where a magnetic stirrer is placed to apply the spraying effect. At a temperature of 40°C and a revolution rate of 8- 9 rpm, the samples were subjected to corrosive conditions that lead to either reduction or increase in the samples weight. Accordingly, the initial weight of each sample was taken before being subjected to any of the different atmospheres.

### 2.3.2.1 : Industrial Atmosphere (ASTM G87)

The test solution for this experiment consists of 0.22 mL of sulphuric acid and 0.12 g of sodium sulphate dissolved in 1 liter of distilled water. The samples are then removed each 2 hours for the following 12 hours to be weighted. The weight change was then compared to the initial weight of each sample and the resultant was divided by the sample area. A curve was finally drawn where $\Delta W/A$ was plotted against time.

### 2.3.2.2 : Marine Atmosphere (ASTM B-287-62)

The test solution for this experiment consists of 5.8 mL of acetic acid and 30 g of sodium chloride dissolved in 1 liter of distilled water. The samples are then removed each 2 hours for the following 54 hours to be weighted. The weight change was then compared to the initial weight of each sample and the resultant was divided by the sample area. A curve was finally drawn where $\Delta W/A$ was plotted against time.

## 2.4 : Electrochemical Corrosion Measurement

The samples were connected to an aluminum wire using TIG welding for the electrochemical tests. The samples were mounted in Serifix resin that is a cold mounting polyester resin used for routine embedding of metallographic samples. The mixture forming the mount consists of 50 mL of resin and 1 mL hardener. A stock of 0.5 M of sodium chloride solution which is the solution used in all the electrochemical test, was prepared by accurate weighting of 29.22 g of sodium chloride and dissolution in 1 Liter of distilled water. Prior to each experiment, the test solution was nitrogen saturated by means of bubbling for 30 minutes. The open circuit potential was measured by potentiostatic/galvanostatic, PGZ 100 (Voltalab, Radiometer analytical, France). The variation in OCP was followed as a function of time until a steady-state value was reached. Steady-state potentials were considered to be attained when the change in potential was less than 5 mV in 20 minutes. In this experiment, a relation between the potential and log of the current density is determined. The tafel sequence is set to operate in a region that is 150 mV above and below the saturation potential measured from the OCP experiment. The analysis is carried out at a scanning rate of 2 mV/sec. The output of each run consists of a polarization curve through which the corrosion parameters can be determined using a software package supplied by the manufacture using Tafel extrapolation technique. Linear Polarization is another method used to determine the corrosion current and the corrosion rates of the sample. This experiment provides a relation between the potential and the current density of the material. The linear polarization sequence operates in a region that is 50 mV above and below the saturation potential measured from the OCP experiment. The analysis is carried out at a scanning rate of 0.5 mV/sec.

## "(3)". Results and Discussion

### 3.1. Microstructure Analysis

The microstructure of test samples are shown in Fig.1a and 1b. Figure 1a indicates the presence of silicon in the form two types of $Mg_2Si$ precipitates. The first type is the Chinese script like products and the second type of $Mg_2Si$ precipitation is formed from the massive silicon in the preform [6]**.** Secondly, it can be notice the presence of aluminum the form of dark circular spots of $Mg_{17}Al_{12}$. Microstructure in Fig.1b dark carbon fibers within the alloy substrate are clearly notice.

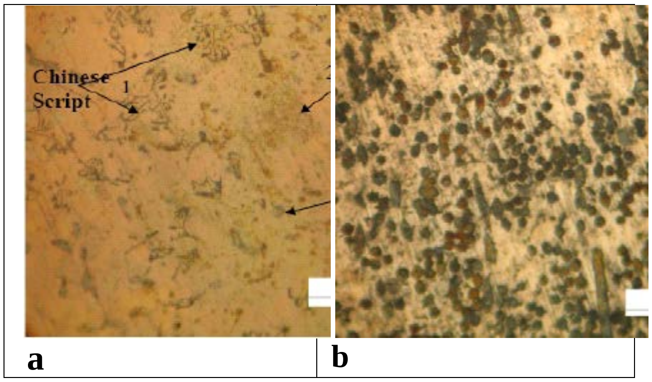

**Fig.1:** Microstructure at 50X, (a) AS41, (b) (AS41+C).

### 3.2.Gravimetric Corrosion Measurement

#### 3.2.1Autoclave Test

In autoclave test the alloys subject to a very high humidity atmosphere accompanied by both high temperature as well as high pressure for 60 hours. The weight change (g/mm$^2$) for test samples is summarized in Fig.2a. As shown in Fig.2a, the weight of AS41 and AS41+C was gradually increasing for the first 24 hours; this increase may be justified by the formation of a very thin layer of gray amorphous Mg (OH)2 film . After 36 hour it can be noticed that a huge weigh drop occurs for AS41+C sample meaning that the corrosion products were able to penetrate the formed protective layer and localized as well as galvanic corrosion started to occur severely into the sample. On the 48th hour measurement, the AS41+C sample failed and could not complete the test showing the huge effect of such an aggressive environment on this alloy. The severe extent of the corrosion effect can be clearly noticed in Fig.2b and the weight loss can be easily visualized through simple visual inspection. On the other hand the weight of AS41 alloy increased up to the end of the experiment. The further weight increase can be attributed to the development of heavier tarnish films of principal corrosion product of crystalline Mg (OH)$_2$ [7 ].

**Fig.2a:** Test Sample behavior in autoclave test

**Fig.2b:** Test Sample after autoclave test

### 3.2.2.Salt Spray Test

The salt spray test was used to evaluate the corrosion behavior of the samples in both industrial and marine environments. The results obtained after experimental procedures in each of these stimulated environments are summarized in Fig.(3,4) .

#### 3.2.2.1.Industrial Atmosphere

As indicated in Fig.3a, test samples gained weight along the path of this experiment which lasted for 12 hours. It can be noted that AS41 alloy did not show much weight gain during the first 4 hours of the experiment. However, after 4 hours a continuous linear increase in the sample weight can be detected. Also it can be observed that a gradual increase in the weight gain of AS41+C alloy in the first 6 hours. After that a sudden increase in the weight gain of AS41+C alloy can be noted. This weight increase can be justified by the formation of corrosion products, which hugely increased in the case of AS41+C alloys. As shown in Fig.3b, the AS41 alloy, showed the accumulation of precipitates on the surface as well as darkening of the sample surface which might be due to the formation of the protective layer as well as the accumulation of corrosion product that can damage this protective film and accelerate the corrosion process. While the AS41+C alloy showed the formation of small dark spots on the sample surface as well as dark regions on the sample edges. These dark regions are a sign of galvanic corrosion occurring in the sample in the regions were the carbon fibers are exposed. The dark spots on the sample surface show localized pitting corrosion. The extent of pitting and galvanic corrosion can be noticed in the above graphical representation. The final weight measurement show a drop in the sample weight due to the weight loss resulting from pitting and galvanic corrosion.

**Fig.3a:** Test Sample behavior in salt spray industrial test

**Fig.3b:** Test Sample after salt spray industrial test

### 3.2.2.2.Marine Atmosphere

The weight change of AS41 and AS41+C alloys is illustrated in Fig.4a. As shown in Fig.4a, AS41 alloy showed an increase in their weight that was almost kept constant throughout the experiment. AS41+C alloy also showed a weight increase however with a much bigger rate than AS41alloy. However, starting from the 48th hour, the sample started dropping weight increasingly and huge weight loss can be seen. As mentioned earlier, the samples were subjected in this salt spray test to a corrosive media containing acetic acid and sodium chloride, both of which are very dangerous chemicals that have strong corrosive effect on the samples. Acetic acid is one of the most known acidic materials. As shown in previous experimental work, solutions of low pH levels or those of acidic nature tend to increase the corrosion rate of magnesium alloys.

This corrosion behavior is a result of the corrosion products formed on the surface of the alloy which are not thick enough to act as a passive layer protecting the alloy and preventing further corrosion. However, these layers of corrosion products are easily broken and participate in increasing the extent of corrosion. Another contributing factor to this sever corrosion is the presence of sodium chloride, the presence of salts and chlorides in particular help in breaking down the protective layer formed on the alloy surface and thus increase the corrosion rate from the beginning of the experiment, even prior to the accumulation of corrosion products as shown in Fig .4b. These severe environmental factors, accompanied by the presence of impurities in AS41 alloy, as well as the carbon present in AS41+C alloy. Yao et al. studied the corrosion of melt-spun Mg in 3% NaCl and concluded that the oxide film formed on Mg was saturated with Mg (OH)2The presence of two distinct oxygen species was identified, with one assigned to $O^{-2}$ in MgO, and the other to $OH^-$ in $Mg(OH)_2$. The two species revealed different depth distribution in the film from which it was concluded that $Mg(OH)_2$ was present predominantly as the top layer. It decreased gradually with the depth while MgO exhibited the reverse trend. The presence of chloride ions thickens the corrosion product formed at the surface, due to their easy penetration in the hydroxide film [8,9].

## 3.3.Electrochemical Corrosion

Open circuit potential (OCP) measurements and potentiodynamic polarization scans have been used to evaluate the corrosion behavior of magnesium alloys. The OCP results recorded in Table (3). As shown in Table (3) AS41 was ( -1.605 V), and AS41+C was ( -1.585 V). This means that AS41 had more negative open circuit potential than AS41+C. The more negative the open circuit potential, the more sensitive to corrosion the sample. Accordingly, these results do not give an accurate representation of the actual corrosion behavior of the alloys. From the gravimetric experiments, AS41+C alloy suffered the most sever corrosion effects. Accordingly, one should have expected it to have the most negative open circuit potential. Justifying this behavior lies in the presence of large carbon content within this sample. Carbon is a very noble material and it should have participated in raising the AS41+C alloy potential. While in real life application, the presence of carbon has led to severe galvanic corrosion proving that it is more harmful then beneficiary to the sample [3].

**Fig.4a:** Test Sample behavior in salt spray marine test

**After 18 hours** **After 54 hours**

**Fig.4b:** Test Sample after salt spray marine test

**Table 3:**Corrosion paramters determination by Tafel extrapolation

| Parameters | AS41 | AS41+C |
|---|---|---|
| Open circuit potential(OCP),V | -1.605 | -1.595 |
| icorr (A/cm2) | 0.178 x $10^{-}4$ | 1 x $10^{-4}$ |
| Corrosion Rate ,mm/Year | 0.72 | 2.29 |

Figure 5. shows the potentiodynamic polarization curves of the test samples in 0.5 M NaCl . It can be noticed that AS41 and AS41+C alloys showed anodic dissolution. The corrosion rate of the samples were determined from the potentiodynamic polarization curves using Tafel extrapolation method and summarized in Table (4). It can be confirmed that AS41+C had the higher corrosion rate, than AS41 alloy. Due to the carbon content as well as the impurities found in the AS41+C alloy, the highest corrosion rate should be expected. In contrast to aluminum, magnesium forms non-corrosion resistant oxide film when exposed to air. The magnesium oxide (MgO) film, formed naturally as a result of exposure to air, has no protective properties, especially in humid atmospheres , rather in neutral, acidic or saline solutions, a heavy corrosion takes place [10,11]. Trzaskoma [12] exhibited severe degradation of the graphite fibers/magnesium composites in aqueous chloride environments. He confirmed the important role of galvanic interaction in the aqueous corrosion rather than the rapid reaction of magnesium with the solution. Hihara and Kondepudi [13,14] investigated the galvanic corrosion behavior of Mg MMCs based on two matrices, pure Mg and ZE41A-Mg alloy.

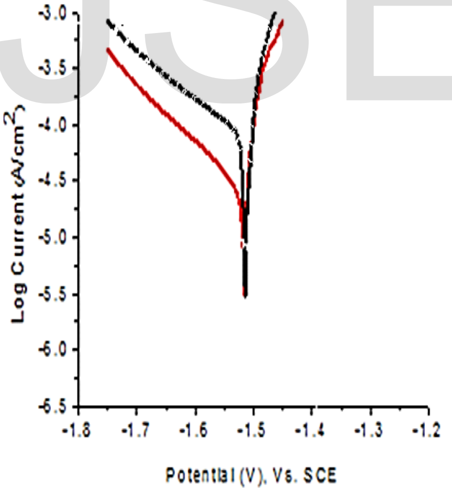

**Fig.5:** Polarization curves of Test Samples 0.5M NaCl solution.

**Table 4:** showing the weight and the weight loss of the samples during the salt spray marine test:

| **Time (H)** | **AS41+C Weight (g)** | **(Weight Diff)/ Area** | **AS41 Weight (g)** | **(Weight Diff)/ Area** | **AZ31 Weight (g)** | **(Weight Diff)/ Area** |
|---|---|---|---|---|---|---|
| 0 | 1.5698 | 0 | 1.1298 | 0 | 0.9794 | 0 |
| 2 | 1.5729 | 5.63411E-06 | 1.1305 | 1.5617E-06 | 0.9802 | 2.08171E-06 |
| 4 | 1.5843 | 2.63531E-05 | 1.1322 | 5.35439E-06 | 0.9827 | 8.58704E-06 |
| 6 | 1.5908 | 3.81666E-05 | 1.1344 | 1.02626E-05 | 0.9871 | 2.00364E-05 |
| 8 | 1.6095 | 7.2153E-05 | 1.137 | 1.60632E-05 | 0.9855 | 1.5873E-05 |
| 10 | 1.623 | 9.66886E-05 | 1.1388 | 2.0079E-05 | 0.9858 | 1.66537E-05 |
| 12 | 1.6313 | 0.000111683 | 1.1396 | 2.18638E-05 | 0.9863 | 1.79547E-05 |
| 14 | 1.6641 | 0.000171386 | 1.1466 | 3.74808E-05 | 0.9895 | 2.62816E-05 |
| 16 | 1.6825 | 0.000204827 | 1.1497 | 4.43968E-05 | 0.9894 | 2.60213E-05 |
| 18 | 1.7039 | 0.000243721 | 1.1559 | 5.8229E-05 | 0.9909 | 2.99245E-05 |
| 20 | 1.7303 | 0.000291702 | 1.152 | 4.95281E-05 | 0.9918 | 3.22665E-05 |
| 22 | 1.753 | 0.000332958 | 1.1547 | 5.55518E-05 | 0.993 | 3.5389E-05 |
| 24 | 1.7571 | 0.000340409 | 1.1556 | 5.74482E-05 | 0.9934 | 3.62998E-05 |
| 26 | 1.7945 | 0.000408382 | 1.1636 | 7.54077E-05 | 0.9945 | 3.92922E-05 |
| 28 | 1.8162 | 0.000447821 | 1.156 | 5.84521E-05 | 0.9903 | 2.83633E-05 |
| 30 | 1.8363 | 0.000484352 | 1.1536 | 5.30977E-05 | 0.9868 | 1.92558E-05 |
| 32 | 1.8877 | 0.000577769 | 1.1506 | 4.64047E-05 | 0.9847 | 1.37913E-05 |
| 34 | 1.9113 | 0.000620661 | 1.1487 | 4.21659E-05 | 0.983 | 9.36768E-06 |
| 36 | 1.9540 | 0.000698175 | 1.1475 | 3.93771E-05 | 0.9812 | 4.68384E-06 |
| 38 | 1.9556 | 0.000701174 | 1.1621 | 7.20612E-05 | 0.9834 | 1.04085E-05 |
| 40 | 1.9778 | 0.000741522 | 1.1644 | 7.71925E-05 | 0.9853 | 1.53526E-05 |
| 42 | 1.9949 | 0.0007726 | 1.1665 | 8.18776E-05 | 0.9863 | 1.79547E-05 |
| 44 | 1.9941 | 0.000771146 | 1.1625 | 7.29536E-05 | 0.9874 | 2.08171E-05 |
| 46 | 1.993 | 0.000769147 | 1.162 | 7.18381E-05 | 0.9899 | 2.73224E-05 |
| 48 | 1.9081 | 0.000614845 | 1.1640 | 7.61886E-05 | 0.9911 | 3.0445E-05 |
| 50 | 1.8519 | 0.000512704 | 1.178 | 0.000107534 | 0.9914 | 3.12256E-05 |
| 52 | 1.8378 | 0.000487078 | 1.1861 | 0.000125605 | 0.9892 | 2.55009E-05 |
| 54 | 1.8329 | 0.000478172 | 1.1837 | 0.000120251 | 0.9902 | 2.8103E-05 |

Two types of reinforcements were used separately; SiC monofilament (MF) made of a carbon core coated by a carbon-rich SiC layer and pure SiC particles. The results exhibited indication of higher galvanic corrosion rates in the presence of SiC (MF), whose electrochemical behavior was similar to that of graphite, compared with pure SiC particles. Hall [15] detected the evidence of galvanic corrosion for carbon fibre/magnesium MMCs in a laboratory atmosphere with 60% relative humidity at 20 ◦C. The determined rate of penetration was about 100µm/year. The magnesium matrix contained 1 wt.% Al and Al carbides were formed at the fibre/matrix interface during squeeze casting but they had no significant effect on the atmospheric corrosion behavior. Galvanic corrosion, in general, of MMCs based on matrices such as Al, Ti and Cu reinforced with carbon or graphite was cited as a major problem. This is due to the high electrical conductivity of carbon [3].

## "(5)". Conclusions

The galvanic coupling with C-fibres leads to severe corrosion of Mg-matrix composites. On the 48th hour measurement in autoclave test, the AS41+C sample failed and could not complete the test showing the huge effect of such an aggressive environment on this alloy. In marine atmosphere test, AS41+C alloy showed a weight increase however with a much bigger rate than AS41alloy. However, starting from the 48th hour, the sample started dropping weight increasingly and huge weight loss can be seen.

The open circuit potential did not vary greatly or do not give an accurate representation of the actual corrosion behavior of the alloys. However, an increase in the degree of corrosion was attributed to the presence of carbon fibres. Electrochemical test results and gravimetric test data showed that the AS41+C MMC possessed a corrosion behavior comparable to AS41 matrix alloy.

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