

Research Article

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Investigation of Stress Corrosion Cracking Behaviour of Mg-Al-Zn Alloys in Different pH Environments by SSRT Method

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Abstract: In this study, stress corrosion behaviors of AZ31, AZ61 and AZ91 Mg alloys which contain different amounts of Al were investigated under acidic, basic and neutral environments having chloride ions using Slow Strain Rate Test (SSRT) method. Stress corrosion indexes (I_{SCC}), ultimate tensile strength (UTS) and elongation of AZ31, AZ61, and AZ91 Mg alloys were determined and compared. Slow strain rate test showed that three Mg alloys in basic environments were the least stress corrosion susceptible, while the most stress corrosion susceptible occurred in acidic environments. Also, it has been shown that the stress corrosion indexes of AZ91 Mg alloys are less than AZ31 and AZ61 Mg alloys in all environments. UTS and elongation of AZ61 Mg alloys were higher than those of AZ31 and AZ91 in all media. The fracture of surface images also examined in the scanning electron microscope (SEM) and both intergranular stress corrosion cracking (IGSCC) and transgranular stress corrosion cracking (TGSCC) were observed in all three alloys.

Keywords: Magnesium alloys; stress corrosion; SSRT; pH.

1 Introduction

Mg alloys have many unique properties such as low density, damping capacity, castability, good thermal conductivity, low heat capacity, outstanding recyclability, excellent biocompatibility and non-toxicity to human body and environment. In addition to all these properties

their use in structural applications such as the automotive, aerospace and medical sectors has been increasing due to their high specific strength [1]. With this increase, stress corrosion cracking (SCC) susceptibility is a great risk for Mg alloys and limits its use in these areas [2]. SCC is one of the most dangerous types of corrosion. With the use of the above mentioned structural elements, SCC causes slow and subcritical crack growth even in cases of mechanical loading which is considered safe. When it reaches the critical crack size, it causes sudden and catastrophic fast fracture with the applied load [3]. In addition to this, service conditions become more severe especially when Mg components (engine blocks, transmission bodies, structural body elements etc.) are used in bulk in the automotive industry [4]. For these reason many researchers have investigated the SCC of Mg alloys. The effects of alloy composition, environment, coating and welding processes on the SCC of Mg alloys were investigated and also the mechanism of fracture and its causes were discussed [5-6]. Mg-Al alloys have low stress corrosion resistance in air, distilled water, and chloride-containing solutions [5-7] and AZxx Mg alloys containing Al and Zn are also susceptible to SCC in similar environments [8]. The researchers stated that the intergranular stress corrosion crack (IGSCC) and transgranular stress corrosion crack (TGSCC) fracture mechanisms were observed in Mg alloys and they were discussed the reasons [3,8-10]. Sozanska et al. investigated SCC of WE43 magnesium alloy by quantitative fractography methods [11]. Winzer et al. [7] investigated AZ91 Mg alloy behavior of SCC in aqueous media. Ebtejah et al. [12] examined the influence of chloride and chromate ion concentration on SCC susceptibility of Mg-9Al. The stress corrosion behavior of AZ91D Mg alloy in modified simulated body fluid was investigated. [13]. Harandi et al. [14] investigated the effect of the addition of bovine serum albumin (BSA) to Hanks' solution in corrosion and SCC susceptibility of AZ91D magnesium alloy. Corrosion and SCC behavior of ZK60 magnesium alloy was investigated under different conditions, i.e., thin electrolyte layer (TEL)

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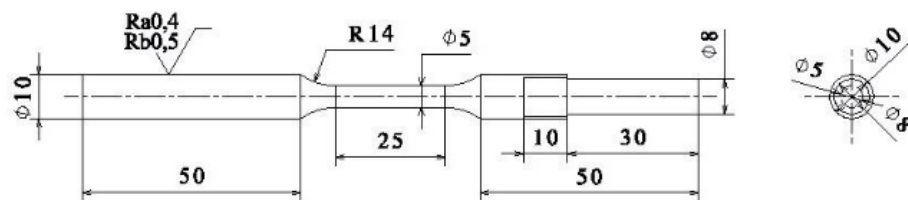


Figure 1: Used tensile samples for SSRT (Unit:mm).

and solution, by SSRT and electrochemical techniques [15]. The effects of coating [16-18] and welding [19-21] processes on the SCC of Mg alloys were investigated. There are a few reports on the influence of pH on SCC in the literature. The effect of pH and chloride ion concentration on corrosion of ZE41 Mg alloy was investigated and observed corrosion of the alloy in NaCl chloride solutions depend on the pH and chloride ion concentration [22]. He et al. studied the corrosion and SCC behavior of AZ31B Mg alloy in 3.5 wt.% Na_2SO_4 solution with different pH values and said that corrosion and stress corrosion increased with increasing pH [23]. However, in literature review, on the effects of pH on SCC of commonly used of AZ31, AZ61 and AZ91 Mg alloys which different amounts of Al were not found. The aim of this study is to investigate and compare the stress corrosion behavior of these alloys under acidic, basic and neutral environments which contain chloride ions using Slow Strain Rate Test (SSRT) method. As a result of experimental studies, stress corrosion indexes for all three alloys were highest in acidic media while the lowest values were obtained in basic media. Furthermore, stress corrosion resistance of AZ91 Mg alloy is higher than AZ31 and AZ61 Mg alloys.

2 Experiments

2.1 Test Materials

The chemical compositions of AZ31, AZ61 and AZ91 Mg alloys used in the experiment were presented in Table 1. Untreated 16 mm diameter rod specimens were produced on CNC machines in accordance with ASTM E8 Test Method standards (Standard Test Methods for Tension Testing of Metallic Materials). The drawing of the produced samples is given in Figure 1.

The specimen preparation steps are as follows:

1. The surfaces of all samples were polished using 1000, 2000 grit silicon carbide papers,
2. All samples were cleaned with distilled water and acetone,
3. Dried in cool air.

Table 1: Chemical compositions of the magnesium alloys used (% weight) (On the scale, no elements with a compounding ratio below 0.001% are given).

Alloy	Al	Zn	Mn	Fe	Cu	Si
AZ31	3.069	1.133	0.486	0.019	0.001	0.131
AZ61	6.242	1.246	0.369	0.029	0.001	0.137
AZ91	9.231	1.348	0.286	0.012	0.004	0.148

2.2 Slow Strain Rate Test (SSRT)

In this study, slow strain rate test (SSRT) was applied and the stress corrosion behavior of AZ31, AZ61 and AZ91 Mg alloys was investigated. The experiments were carried out in accordance with ASTM G-129 00 standard. Slow deformation tests were performed by Shimadzu brand universal tensile testing device. The corrosive environment was provided by acidic (pH 2), basic (pH 11) and neutral (3.5 wt.% NaCl) using artificial corrosion cell mounted on the tensile testing device. Acidic solution was prepared by adding HCl to 3.5 wt.% NaCl solution to decrease the pH value to 2.0, and NaOH was added to 3.5 wt.% NaCl solution until pH 11.0 to obtain a basic solution. The stress corrosion crack index was calculated by cracking the test specimens in the abovementioned media and at a speed of pulling jaws of 0.005 mm/min. Stress corrosion crack index can be calculated as follows by the failure time [24].

$$I_{scc} = 1 - t_{solution} / t_{air} \quad (1)$$

where $t_{solution}$ and t_{air} are failure time in environment and air, respectively. When the value of I_{scc} approaches to 1, it is assumed that the alloy is highly susceptible to SCC.

After the SSRT experiments, fracture surface images of the samples were taken by SEM (FEI Nova Nano SEM 450) and the fracture types were evaluated. Also, Nade NMM-800 TRF light microscope was used for microstructure images for examine the grain size of the samples and to investigate the phases.

Ethical approval: The conducted research is not related to either human or animal use.

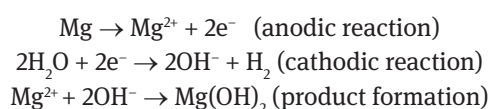
Table 2: SSRT results of magnesium alloys.

Specimen	Corrosion Environment	Fracture Time (Hour)	I_{sc}	UTS (MPa)	Elongation (%)
AZ31	Air	34.62	-	146.960	41.544
	Acidic (pH=2)	5.89	0.830	63.605	7.068
	3.5 wt.% NaCl	7.72	0.778	60.406	9.264
	Basic (pH=11)	9.58	0.724	84.979	11.496
AZ61	Air	56.40	-	171.151	67.680
	Acidic (pH=2)	13.46	0.761	99.711	16.152
	3.5 wt.% NaCl	15.71	0.721	115.929	18.852
	Basic (pH=11)	18.34	0.675	126.771	22.008
AZ91	Air	13.03	-	140.702	15.636
	Acidic (pH=2)	6.27	0.519	76.484	7.524
	3.5 wt.% NaCl	6.80	0.479	87.628	8.160
	Basic (pH=11)	7.46	0.428	108.961	8.952

3 Results and Discussion

Stress corrosion tests of AZ31, AZ61 and AZ91 Mg alloys were carried out in acidic, basic and 3.5 wt.% NaCl solutions by the SSRT method.

The fracture times of AZ31, AZ61 and AZ91 Mg alloys in air, acidic, natural and basic media as a result of SSRT test are shown in Figure 2. Also, Table II shows the stress corrosion cracking indexes (I_{sc}), ultimate tensile strength (UTS) and elongation values obtained from the SSRT results. According to these results, stress corrosion indexes for all three alloys were the highest in acidic environment while the lowest values were obtained in basic environment. In other words, all three alloys have the lowest stress corrosion resistance in acidic environment while have the highest resistance in the basic environment. This clearly demonstrates that the stress corrosion cracking is strongly dependent on pH value. Depending on the pH value of solution, stressed corrosion behavior appears to have a similar effect to corrosion behavior of Mg alloys. This effect of solution pH on corrosion behavior can be explained by the EH-pH (potential/pH) diagram of magnesium (Pourbaix diagram) [25]. The dissolution of magnesium in aqueous solutions occurs primarily by reducing water to produce magnesium hydroxide $Mg(OH)_2$ and hydrogen gas (H_2) [26].



In magnesium-aluminum alloys, $Mg(OH)_2$ formation is facilitated when the pH value is higher than about 9, depending on the magnesium concentration. The required pH value for formation of $Mg(OH)_2$ is about 11. Since $Mg(OH)_2$ formed on the surface is protective against corrosion, corrosion rate increase at high pH values. SCC in Mg alloys is generally said to consist of two mechanism groups. The first is the continuous crack spread with anodic dissolution at the crack end, and the second is the discontinuous crack spread with mechanical cracks [3]. It can be said that anodic dissolution is effective for IGSCC and that the second mechanism is effective for TGSCC. Considering that the parameter which is effective in anodic dissolution is corrosion, it can be said that $Mg(OH)_2$ compound, which is protective against corrosion, actually improves SCC.

As shown in Figure 3, stress corrosion indexes were calculated for AZ31 Mg alloy to be 0.830, 0.778, and 0.724, while 0.761, 0.721, and 0.675 for AZ61 Mg alloy and 0.519, 0.479 and 0.478 for AZ91 Mg alloy in acidic, natural and basic media, respectively. From these values, it is obvious that stress corrosion indexes of AZ91 Mg alloy are less than AZ31 and AZ61 Mg alloys in all three environments. In other words, stress corrosion resistance of AZ91 Mg alloy is higher than AZ31 and AZ61 Mg alloys ($AZ31 < AZ61 < AZ91$ for stress corrosion resistance in all three environments). Ghali [27] and Song et al. [28] found that the amount of aluminum in the alloy affects corrosion and stress corrosion. Mg-Al alloys and Mg-Zn alloys have high stress

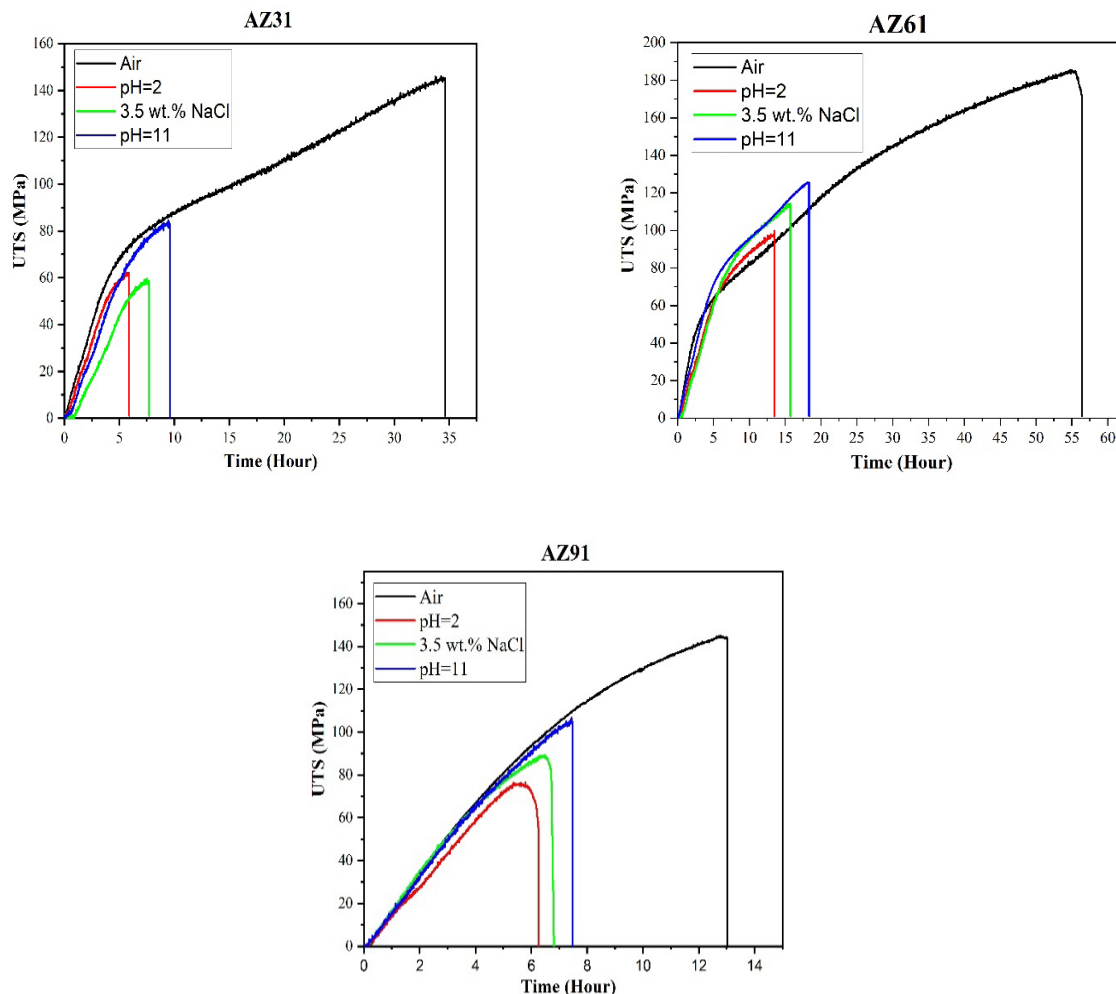


Figure 2: The fracture times of AZ31, AZ61 and AZ91 Mg alloys in air, acidic, natural (3.5 wt.% NaCl) and basic media as a result of SSRT test.

corrosion resistance whereas Al and Zn free-Mg alloys have low stress corrosion resistance. It has been reported that an aluminum content above a threshold level of 0.15–2.5% should be required to induce SCC behavior in Al-containing magnesium alloys [27–28]. In addition, since the addition of Zn also leads to SCC susceptibility in magnesium alloys, it will not be surprising that the stress corrosion resistance of AZ series Mg alloys is low. Increasing concentrations amount of Al from 2 to 8% in Mg-Al alloys reduces the corrosion rate. Approximately, addition of Al about 2–4wt% results in α -Mg dendrites surrounded by the eutectic double-phase $\alpha + \beta$ at grain boundaries. On the other hand, distinct β particles tend to precipitate along grain boundaries due to the higher solidification rate in high additions of 6 to 9 wt.%. If the amount of Al is up to 10%, the Al-rich α phase will cause micro segmentation during solidification and this

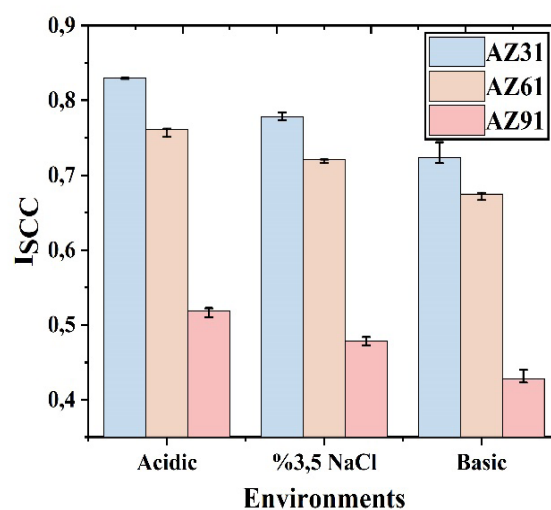


Figure 3: Stress corrosion indexes of AZ31, AZ61 and AZ91 Mg alloys in different media.

value will be the appropriate local concentration [3-29]. β phase is very stable and effective in solution. β phase can play dual functionality as an anodic barrier and galvanic cathode depending on β particles content. If the β phase is present in small quantities in the precipitate between the particles in the α matrix, this acts mainly as a galvanic cathode and accelerates the corrosion of the α matrix. If the amount of β phase is large, then β phase can act as an anodic barrier to prevent corrosion of the alloy. The β phase is beginning to appear when Al content is above 2% by weight. As a result, an increase in Al content up to 10% can increase corrosion and stress corrosion resistance [27]. Winzer et. al. [7] also characterized SCC of AZ91 and AZ31 Mg alloys in distilled water using constant elongation rate test (CERT) and the linearly increasing stress test (LIST). They said that AZ91 consists of an α -matrix with a significant amount of β -phase, whereas AZ31 consisted essentially only of an α -matrix with an Al-concentration similar to that in the α -phase of AZ91. In this study, it was observed that as the amount of Al increases, the stress corrosion resistance of Mg alloys increases which is well-matched with the literature.

UTS and elongation values were calculated from the SSRT test results and are shown in Figure 4 and 5. UTS values are the ultimate tensile strength values that the samples reach before breaking, and elongation values are obtained from fracture times. In addition, UTS, elongation and ISCC results of AZ31, AZ61 and AZ91 Mg alloys are summarized in Table 2 for acidic, basic and neutral environments, respectively. It is evident that the UTS and elongation values for three kinds of Mg alloys were significantly decreased in solutions media with respect air.

When all these results were analyzed, the ultimate tensile strength values were found out to be AZ91 < AZ31 < AZ61 in all three environments. On the other hand, the same behavior was not observed in all three environments for elongation. In air, 3.5 wt.% NaCl and basic environments elongation relation were AZ91 < AZ31 < AZ61 while AZ31 < AZ91 < AZ61 relation is valid for acidic environment (Table 2, Figure 4 and Figure 5). As can be seen, the AZ61 Mg alloy has the highest elongation and ultimate tensile strength values in all environments. For this reason, Mg alloys are considered to have the optimum strength and ductility value of 6% aluminum additive [28].

After the SSRT test, a boiling solution (%15 CrO_3 + %1 Ag_2CrO_4 + distilled water = 100 ml) was used to remove the corrosion products of the samples. Cleaned products samples were dried distilled water and acetone. The fracture surface images of these samples were then examined by scanning electron microscopy. Figure 6

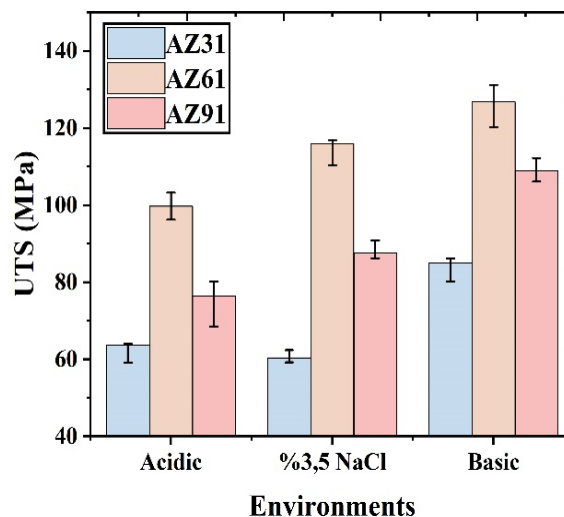


Figure 4: Ultimate tensile strength of AZ31, AZ61 and AZ91 Mg alloys in different media.

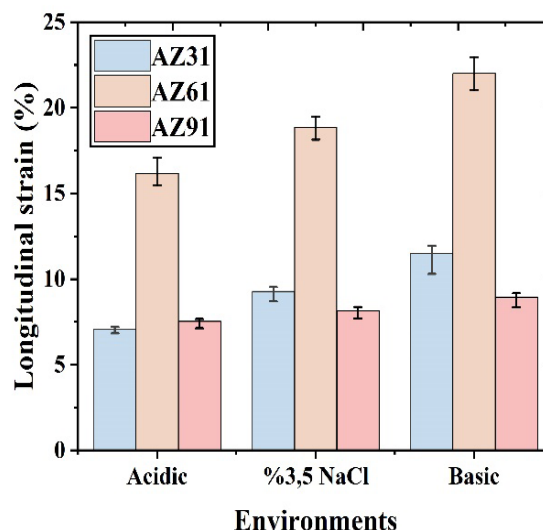


Figure 5: Elongation (%) of AZ31, AZ61 and AZ91 Mg alloys in different media.

shows the fracture surface images of AZ31, AZ61 and AZ91 Mg alloys in all solutions. From the images, intergranular stress corrosion cracking (IGSCC) and transgranular stress corrosion cracking (TGSCC) are observed for all samples and media.

There are different mechanisms for IGSCC and TGSCC. TGSCC is discontinuous and involves alternating fracture and dissolution whereas IGSCC is continuous and completely electrochemical [30]. The formation mechanisms of IGSCC and TGSCC in the literature are explained by microstructure and environment. Hydrogen embrittlement mechanisms may produce both IGSCC and TGSCC, especially depending on the grain size. While

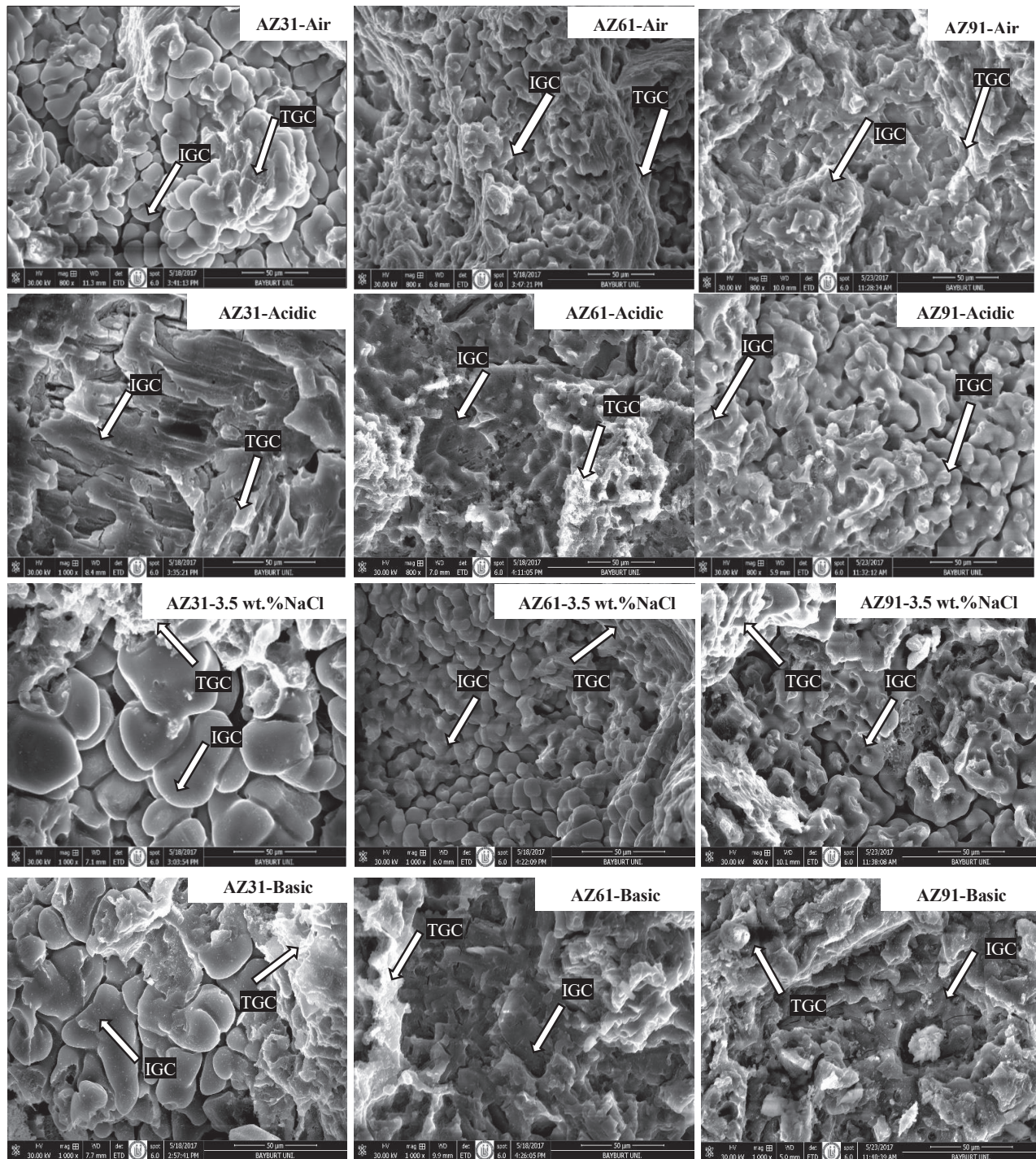


Figure 6: SEM images of the fracture surfaces in different media for AZ31, AZ61 and AZ91 Mg alloys.

TGSCC is observed in fine-grained commercial-purity Mg alloy (0.025 mm), mixed TGSCC and IGSCC are found in large-grained high-purity Mg alloy (0.075 mm) in a dilute 10^{-3} M Na_2SO_4 . It has been said that the IGSCC is due to a higher dislocation pile-ups at large grain boundaries [4]. Crack morphology is influenced by the environment. TGSCC has occurred in different solutions such as

saturated MgCO_3 solution, 0.5% KF solution, 0.5% KHF solution, and 0.5% HF solution of Mg-5Al, but IGSCC has taken place in 0.05% potassium chromate solution of Mg-5Al.

Microstructure studies have been carried out to examine the grain size of the samples and to investigate the phases. For metallographic characterization, samples

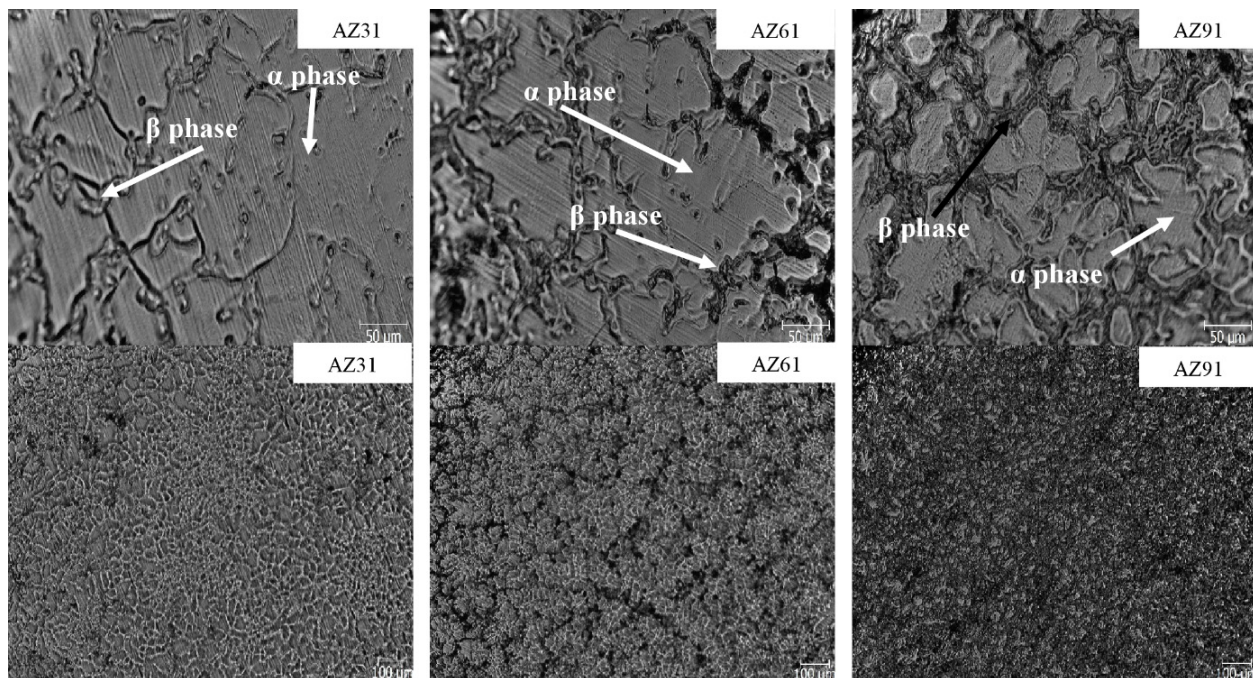


Figure 7: Microstructure images of AZ31, AZ61 and AZ91 Mg alloy samples.

were sanded to a 2500 grit sandpaper, followed by polishing with alumina suspension and subjected to microstructure studies by etching with a solution of 5 mL acetic acid, 6 g picric acid, 10 mL distilled water, 100 mL ethanol, 5 mL HCl, and 7 mL nitric acid for 1-2 s [32]. Micrographic observations from metallographically prepared surfaces were performed using the Kameram image analysis program on images taken with a Nade NMM-800 TRF light microscope and a Kameram digital camera connected to it. The resulting images of microstructure studies are given in Figure 7. The images were taken at 10x and 50x magnification. Average grain sizes were measured as 0.042 mm, 0.037 mm, and 0.031 mm for AZ31, AZ61 and AZ91 Mg alloys, respectively. According to literature findings, these values of grain size are suitable for IGSCC and TGSCC fracture patterns to be observed. Furthermore, as seen in the microstructure images, intermetallic β phase ($Mg_{17}Al_{12}$) is present and this phase can be said to be a factor in the coexistence of TGSCC and IGSCC. The popular model for IGSCC cracking indicates that, in single-phase alloys, TGSCC is due to the absence of $Mg_{17}Al_{12}$ [30].

4 Conclusions

The effect of pH value on SCC behavior of AZ31, AZ61 and AZ91 Mg alloys can be summarized as follows.

I. Stress corrosion indexes for all three alloys were highest in acidic media while the lowest values were

obtained in basic media. It is evident that as the pH value of the solution decreases in all three alloys, the stress corrosion index increases. In other words, stress corrosion resistance in basic media is the highest whereas in acidic environment is the lowest.

- II. Stress corrosion indexes of AZ91 Mg alloy are less than AZ31 and AZ61 Mg alloys in all three environments ($AZ31 < AZ61 < AZ91$ for I_{sc} in all three environments). It means that stress corrosion resistance of AZ91 Mg alloy is higher than AZ31 and AZ61 Mg alloys.
- III. The ultimate tensile strength values have relation of $AZ91 < AZ31 < AZ61$ in all three environments. On the other hand, the same behavior was not observed in all three environments for elongation. In air, 3.5 wt.% NaCl and basic environments elongation relation is $AZ91 < AZ31 < AZ61$ while is $AZ31 < AZ91 < AZ61$ in acidic environment. As can be seen, the AZ61 Mg alloy has the highest elongation and ultimate tensile strength values in all environments.
- IV. Intergranular stress corrosion cracking (IGSCC) and transgranular stress corrosion cracking (TGSCC) are observed in all media.
- V. Studies to improve the stress corrosion resistance of magnesium alloys in industrial applications, automotive, defense industry, aerospace, and especially in implant applications in recent years are inadequate, and extensive studies are needed to be done in this regard.

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Conflict of interest: Authors declare no conflict of interest.

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