Study of an Mg₉₀Y₇Zn₃ alloy (WZ73) in sodium chloride solution — An analysis by correlated polarization and climate chamber testing

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Abstract
In the present work, the corrosion behavior of a twin-roll cast WZ73 magnesium alloy is investigated by potentiodynamic polarization and climate chamber tests in different sodium chloride solutions. The corrosion parameters $E_{corr}$ and $j_{corr}$ are extracted from the polarization curves, and the critical concentration for the initiation of pitting corrosion is thus identified. The critical concentration for the initiation of pitting is confirmed by means of climate chamber tests. As a consequence, the WZ73 magnesium alloy is found to be highly susceptible to pitting corrosion in sodium chloride solutions of concentrations higher than $10^{-4}$ mol/L. Moreover, when the NaCl concentration increases, the corrosion mechanism changes from pitting to filiform corrosion, which is attributed to micro-galvanic effects. At low NaCl concentrations, the corrosion attack remains localized. When the NaCl concentration increases, pitting occurs and, in addition to the pits that form, micro-galvanic corrosion leads to filiform corrosion attack. This is caused by the electrochemical potential of the long period stacking ordered phase, which is higher than that of the $\alpha$-Mg matrix. Consequently, the corrosion attack is focused on the phase boundary between the $\alpha$-matrix and LPSO phases.

KEYWORDS
climate chamber test, Mg-Zn-Y alloy, micro-galvanic corrosion, pitting corrosion, potentiodynamic polarization, uniform corrosion

1 | INTRODUCTION

In recent years, the importance of lightweight structures and assemblies in industrial applications has increased enormously and, as a result, research into and the development of light materials are of great interest across broad fields of application. Due to their excellent properties, magnesium and its various alloy systems have considerable potential for replacing heavier components made of steel. The very low density of magnesium (1.74 g/cm³) compared to other construction materials – for example, aluminum at 2.78 g/cm³ – can facilitate further significant weight reductions.¹,² In addition, the high specific strength and good damping capacity are further factors that support the use of magnesium in
structural applications. Magnesium and its alloys also incorporate the significant advantages of metallic materials, which include, for instance, favorable mechanical properties, thermal and electrical conductivity, good processability, and excellent recycling potential. In addition to the large number of positive aspects, however, magnesium is highly susceptible to corrosion when appropriate protection measures are not considered. Accordingly, the use of magnesium alloys is limited due to their insufficient corrosion resistance, which is a key point for commercial applications. To solve this problem, a range of new alloy systems are being developed that focus on enhanced corrosion resistance. In this context, magnesium alloys that contain rare earth elements, such as yttrium, are promising developments. The WZ (Mg-Y-Zn) magnesium alloy series provides the opportunity to improve corrosion resistance through their unique LPSO (long period stacking ordered) structure. Studies of these systems indicate that WZ magnesium alloys exhibit higher corrosion resistance than the conventionally used AZ magnesium alloy series.

In this study, the electrochemical corrosion behavior of a recently developed WZ73 magnesium alloy containing 7 wt% Yttrium and 3 wt% Zinc was examined in sodium chloride solutions by means of potentiodynamic polarization to evaluate the critical sodium chloride concentration for the initiation of pitting corrosion. Additionally, the magnesium alloy was investigated using a climate chamber test in two different sodium chloride solution concentrations, with \( c_1 = 10^{-4} \text{ mol/L} \) and \( c_2 = 10^{-3} \text{ mol/L} \). The results of the short-term polarization and climate chamber tests were correlated to evaluate the corrosion behavior of the WZ73 magnesium alloy.

## EXPERIMENTAL

### Material and preparation

In this study, a twin-roll cast (TRC) WZ73 magnesium alloy with the chemical composition given in Table 1 was used. The TRC process was developed at the Institute of Metal Forming at TU Bergakademie Freiberg to manufacture magnesium sheets to standard industrial levels. The advantage of the TRC technology is that the solidification and the forming process of the alloys takes place in one step, making TRC an energy- and cost-efficient technology. In addition, further benefits of this method include reductions in segregations, the finer distribution of precipitates, and improvements in microstructural homogeneity. A detailed description of the manufacturing process can be found in the literature. The WZ73 magnesium alloy sheet used in this study had a thickness of 5.4 mm and was analyzed in the as-received state. The focus of this study was on the evaluation of the corrosion sensitivity of the WZ73 magnesium alloy directly after the manufacturing process described above.

For microstructural observation, the samples were ground with 4000-grit SiC grinding paper, polished with an OP-S suspension, washed with water and ethanol, and dried.

### Electrochemical polarization and climate chamber test

The WZ73 magnesium alloy samples for the polarization tests (sample size: 2 cm \( \times \) 2 cm) were potentiodymatically polarized in the as-received state. Therefore, no special surface treatment (e.g., grinding, polishing) was applied. Five different sodium chloride solutions with varying concentrations were used, namely \( c_1 = 10^{-4} \text{ mol/L} \), \( c_2 = 10^{-3} \text{ mol/L} \), \( c_3 = 10^{-2} \text{ mol/L} \), \( c_4 = 10^{-1} \text{ mol/L} \), and \( c_5 = 0.885 \text{ mol/L} \). \( c_1 \) and \( c_2 \) were chosen to facilitate identification of the critical NaCl concentration of a harmful pitting corrosion attack. \( c_3, c_4, \) and \( c_5 \) were used to analyze the influence of high NaCl concentration, whereby \( c_5 \) corresponds to the concentration commonly used for the salt spray test that simulates a highly aggressive corrosion attack. To this end, a VSP potentiostat (BioLogic Science Instruments), EC-Lab® software and a conventional three-electrode-arrangement were used for the test, with the magnesium alloy acting as the working electrode, a saturated Ag/AgCl electrode as the reference, and a platinum mesh as the counter electrode. Before examination, the samples were washed with distilled water and then ethanol, and dried. The sample/electrolyte interface had an area of 1 cm\(^2\) and the polarization was carried out with a scan rate of 6 mV/min. Prior to polarization, the open circuit potential was measured for 15 min to ensure the existence of steady state conditions. The corrosion current density \( j_{\text{corr}} \) was

| Mg  | Y   | Zn  | Zr  | Si  | Fe  | Ni  | Cu  |
|-----|-----|-----|-----|-----|-----|-----|-----|
| Bal.| 6.8 | 2.5 | 0.4 | 0.01| 0.005| 0.001| 0.0001|
extracted from the polarization curves by means of the Tafel extrapolation method. Each test was repeated three times to ensure reproducibility.

For the climate chamber test, as-received WZ73 magnesium alloy samples (sample size: 13.5 cm × 13.5 cm) were exposed in the climate chamber (CCT 400-S CWC, VLM GmbH) to sodium chloride solutions of \( c_1 = 10^{-4} \) mol/L and \( c_2 = 10^{-3} \) mol/L. These concentrations were identified from the polarization tests as critical for the initiation of pitting corrosion. The tests were carried out at a temperature of 35°C ± 2°C and had a total test duration of 672 h (4 weeks). For analysis of the damage evolution as a function of time, the sample surface was investigated at intervals of 168 h (1 week). Before examination, the samples were washed with distilled water, then ethanol, and dried. The tests were carried out according to DIN EN ISO 9227.\(^{10}\)

The chemical composition and damage evolution behavior were investigated by means of scanning electron microscopy (SEM – TESCAN, MIRA 3 XMU) with an integrated energy dispersive X-ray (EDX) detector. EDX spectroscopy was performed for evaluation of the element composition in the \( \alpha \)-matrix and the LPSO phases. For the EDX analysis, a primary beam energy of 20 kV and a probe current of 1 nA were used.

Due to excessive corrosion-product formation after the polarization and climate chamber tests and for analysis of the damage evolution, the samples were immersed in a 20% chromic acid solution\(^{11-13}\) before washing with distilled water and ethanol, and drying. For analysis of the pit evolution as a function of time, the pit depth was evaluated by laser interferometry (BMT, Breitmeier Messtechnik). In addition, the weight loss was determined by using a conventional analytical balance and by calculation of the mass difference before and after the corrosion test.

### 3 | RESULTS AND DISCUSSION

#### 3.1 | Microstructure of TRC WZ73

Figure 1(A) shows a micrograph of the WZ73 alloy used with the typical binary microstructure of the Mg-Y-Zn alloy system visible, as described in the literature.\(^{14,15}\) The \( \alpha \)-Mg phase (primary matrix) was dendritic and the secondary phase was attached in the interdendritic zone at the grain boundaries. With respect to the results reported in the literature,\(^{14-18}\) the secondary phase could be allocated to the Mg\(_{12}\)YZn phase (X-phase), with a LPSO structure. The SEM micrograph (Figure 1(B)) indicates the dark gray magnesium matrix and the surrounding network of LPSO structures that were analyzed by EDX (see Table 2). The magnesium matrix (A in Figure 1(B)) did not contain significant amounts of dissolved alloying elements. The alloying elements were found primarily in the LPSO phase (B in Figure 1(B) and Table 2). Kittner et al\(^{18}\) report that in particular, the white, shining locations inside the LPSO structure are enriched with Yttrium. Krbetschek et al\(^{19}\) determine that the X-phase of the TRC state analyzed in this study has an 18R-type structure.

![Figure 1](image1.png)

**Figure 1** TRC WZ73 (A) optical micrograph, (B) scanning electron micrograph. A and B indicate the locations for EDX analysis

| Zone | Mg   | Y    | Zn   |
|------|------|------|------|
| A    | 97.70| 1.01 | 1.29 |
| B    | 72.44| 14.01| 13.55|

**Table 2** Chemical composition of microstructural constituents (A – magnesium matrix, B – LPSO phase) in wt% determined by means of energy dispersive X-ray spectroscopy (EDX) analysis.
3.2  |  Potentiodynamic polarization test

Figure 2 presents the potentiodynamic polarization curves, which indicate the expected increase in $E_{\text{corr}}$ and decrease in $j_{\text{corr}}$ when the sodium chloride concentration decreased. These observations are fully in agreement with the behavior found for the AZ31 magnesium alloy by Lange et al.\textsuperscript{12} The extracted parameters $E_{\text{corr}}$ and $j_{\text{corr}}$ are summarized in Table 3.

As indicated by the polarization curves and the parameters $E_{\text{corr}}$ and, in particular, $j_{\text{corr}}$, a significant increase in corrosion sensitivity was noted when the concentration exceeded $c_2 = 10^{-3}$ mol/L. The current density increased by two orders of magnitude when the NaCl concentration increased from $c_2$ to $c_3$, indicating severe corrosion attack. The electron micrographs observed after the polarization tests (see Figure 3) revealed an extensive loss of passivity and changing of the corrosion mechanism when the NaCl concentration increased from $c_2$ to $c_3$. The corrosion attack, which was predominantly local depassivation (see Figure 3(B),(C)) for $c_1$ and $c_2$, extended to the whole surface and transformed to uniform corrosion for $c_3$ (see Figure 3(D)). The attack intensified further when the concentration increased up to $c_4$ and $c_5$ (see Figure 3(E),(F)). It was concluded that when the sodium chloride concentration reached a critical limit, the number of local weak points generated within the passive oxide increased significantly, and the anodic metal dissolution process accelerated enormously. As a consequence of the lateral expansion, these local depassivated sites coalesced and the damage evolution appeared macroscopically as a uniform corrosion attack.

Figure 3(B) reveals in greater detail that after polarization in concentration $c_1$, significant corrosion attack did not occur and the surface appeared to be similar to the initial state (see Figure 3(A)). The cluster of cavities and crevices observed were related to the TRC process.\textsuperscript{8} Consequently, a chloride-containing aqueous solution with the concentration $c_1$ may be considered harmless. When the sodium chloride concentration increased to $c_2 = 10^{-3}$ mol/L, the local corrosion attack observed was focused on weak points such as pores and local cavities, and initiated pitting corrosion (Figure 3(C)). Furthermore, lateral expansion of these local corrosion attacks was observable, which is classified by other authors as a kind of filiform corrosion.\textsuperscript{14}

The electron micrographs observed after polarization in $c_3$ (see Figure 3(D) and, for more detail, Figure 4(A)) revealed that corrosion was initiated at the interface between the $\alpha$-Mg matrix and the X-phase. It was predominantly the $\alpha$-Mg matrix (dark) that was attacked, while the X-phase (bright) remained intact and protruded from the surface as a basic structure. Chiu and Liu\textsuperscript{20} report that corrosion attack is highly favored at the $\alpha$-Mg matrix/X-phase interface, and suggests a micro-galvanic effect.\textsuperscript{14,21-23} The X-phase acts as the micro-cathode and the $\alpha$-Mg matrix as the micro-anode,\textsuperscript{6} which is a result of the higher electrochemical potential of the X-phase in comparison to the $\alpha$-Mg matrix due to the alloying element enrichment.\textsuperscript{15} As a result, the X-phase is more corrosion resistant. After polarization in $c_5$, the surface appeared approximately similar to the state after polarization in $c_4$ (see Figure 4(B)), though it was more highly fissured, the corrosion attack was much more intense and the pit size increased drastically (see Figure 4(C)). In general, for NaCl concentrations higher than $c_3$, the corrosion attack remained uniform but the corrosion velocity was clearly enhanced.

In sum, the corrosion attack increased severely when the sodium chloride concentration exceeded $c_2 = 10^{-3}$ mol/L, which was indicated by larger pit sizes and a change from localized corrosion to uniform corrosion.
Scanning electron micrographs of TRC WZ73 after potentiodynamic polarization in various sodium chloride solutions

**FIGURE 3**

(A) Initial state  
(B) $c_1 = 10^{-4} \text{ mol/l}$  
(C) $c_2 = 10^{-3} \text{ mol/l}$  
(D) $c_3 = 10^{-2} \text{ mol/l}$  
(E) $c_4 = 10^{-1} \text{ mol/l}$  
(F) $c_5 = 0.885 \text{ mol/l}$

**FIGURE 4**

Scanning electron micrographs of TRC WZ73 after polarization in  
(A) $c_3 = 10^{-2} \text{ mol/L}$  
(B) $c_4 = 10^{-1} \text{ mol/L}$  
(C) $c_4 = 0.885 \text{ mol/L}$ sodium chloride solution. The arrows mark the location of the microgalvanic corrosion attack.

The initiation of pitting corrosion observed for $c_2$ was of great interest because it marked the critical NaCl concentration for the beginning of the local depassivation process on the passive oxide, the initiation of pitting corrosion, and the subsequent severe degradation of the magnesium alloy. Therefore, the solutions with the concentrations $c_1$ and $c_2$ were chosen for the subsequent climate chamber test.

### 3.3 **Climate chamber tests**

During the climate chamber test, a whitish-gray layer of voluminous Mg(OH)$_2$ assimilated rapidly on the sample surface.$^{20,24,25}$ Figure 5 compares the corrosion product evolution in the chosen test solutions of concentration $c_1$ and
c_2 as observed after 24 h of exposure. It was obvious that the effect was more pronounced at higher sodium chloride concentrations, indicating a higher level of corrosion attack at the surface.

The electron micrographs observed after several time intervals of the climate chamber test indicated that the corrosion behavior was strictly a function of the sodium chloride concentration (see Figure 6). For the concentration c_1, Figure 6(A)-(D) reveals that pitting did not occur for this sodium chloride concentration. Furthermore, a significant increase in the corrosion attack could not be detected up to the end of the climate chamber test. It was assumed that the

**Figure 5** Photographs of the TRC WZ73 magnesium alloy for (A) the initial state, and after 24 h of climate chamber testing in (B) c_1 = 10^{-4} \text{ mol/L} and (C) c_2 = 10^{-3} \text{ mol/L} NaCl solution.

**Figure 6** Scanning electron micrographs of the TRC WZ73 magnesium alloy after climate chamber testing in c_1 = 10^{-4} \text{ mol/L} (A-D) and c_2 = 10^{-3} \text{ mol/L} (E-H).
layer of corrosion products formed rapidly on the surface in the $c_1$ NaCl concentration (see Figure 5(B)), which reduced the corrosion susceptibility of the alloy to a negligible level.

Unlike with $c_1$, the concentration $c_2$ had visible effects on the surface. After removing the corrosion products, pitting corrosion and micro-galvanic corrosion attack could be observed after 168 h of exposure (see Figure 6(E)). The pits were the initiation points for the subsequent micro-galvanic corrosion attack, which was clearly observable by contrasting the X-phase with the remaining LPSO phase, which was not attacked as strongly. In comparison, the corrosion attack in $c_1$ was less intense and revealed a homogeneous damage evolution pattern (see Figure 6(A)) after the same exposure time. For this reason, it was proven that a sodium chloride concentration of $10^{-3}$ mol/L ($c_2$) is critical for the WZ73 magnesium alloy in the TRC condition. After 336 h, lateral expansion of the local corrosion attacks was observed (see Figure 6(F)). With respect to the potentiodynamic polarization, this expansion could be classified as a kind of filiform corrosion. When the test duration increased further, the corrosion attack was more extensive, which was made obvious by the enhancement of pit depth and size (see Figure 6(G),(H)). In comparison, for the test solution of concentration $c_1$, the corrosion attack was less intense and did not result in a significant increase in damage evolution (see Figure 6(A)-(D)). Therefore, it was concluded that along with NaCl concentration, the corrosion mechanism was also a function of the test duration. As a result, the degradation of the WZ73 alloy in $c_2$ was, in principal, defined by localized corrosion attack followed by a filiform corrosion with the pits as the origin and, finally, a widespread corrosion attack on the entire surface when the passive oxide had been completely dissolved.

Figure 7(A) presents the pit-depth evolution on the WZ73 magnesium alloy as a function of the exposure time during the climate chamber test in the $c_2$ sodium chloride solution concentration. The curve progression clearly indicated a drastic increase in pit depth up to a test duration of 504 h. Afterwards, the pit depth evolution remained constant, indicating that the corrosion attack had perhaps reached a final state. Similar to the observations made for $c_1$, it was concluded that a certain quantity of corrosion products had formed on the alloy surface, thus reducing the ongoing corrosion attack significantly. Consequently, the pitting attack stagnated and may have reached a metastable steady state. This assumption was consistent with the sample weight loss measured (see Figure 7(B)).

In sum, the results presented above prove that slight changes in sodium chloride concentration can have enormous effects on the corrosion behavior of the WZ73 magnesium alloy. Although stagnation of the corrosion attack due to intense corrosion-product formation seems possible, a highly effective anti-corrosion coating should be recommended for the WZ73 magnesium alloy even when exposed to low concentrations of NaCl solution.

4 | CONCLUSION

In this study, the electrochemical corrosion behavior of a twin-roll cast WZ73 magnesium alloy was analyzed by potentiodynamic polarization and climate chamber tests in a range of sodium chloride concentrations.

From the polarization tests, the expected increase in corrosion sensitivity was observed when the NaCl concentration increased, and the critical sodium chloride concentration for pit initiation was identified in the range of $10^{-4}$–$10^{-3}$ mol/L. Based on the results of the polarization tests, 4-week climate chamber tests were carried out in $10^{-4}$ and $10^{-3}$ mol/L NaCl solution. The damage evolution observed as a function of time indicated similar corrosion behavior to that observed in the course of the polarization tests.

For NaCl concentrations higher than $10^{-4}$ mol/L, the corrosion attack on the WZ73 magnesium alloy was characterized by pit initiation, a subsequent filiform corrosion attack with its origin at the pits, and a final uniform corrosion attack over the entire surface.
The damage evolution observed after the polarization and climate chamber tests indicated that micro-galvanic corrosion had occurred between the highly electrochemically active $\alpha$-matrix and the electrochemically more noble LPSO phase. The increased corrosion durability of the LPSO phase resulted from the enrichment of the alloying elements.

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Robert Hornig: Conceptualization; investigation; methodology; validation; visualization; writing-original draft; writing-review and editing. Marcel Mandel: Conceptualization; methodology; writing-original draft; writing-review and editing. Krüger Lutz: Supervision; writing-review and editing. Sven Bräunling: Writing-review and editing.

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