

# On the capability of in-situ exposure in an environmental scanning electron microscope for investigating the atmospheric corrosion of magnesium



M. Esmaily<sup>a,c,\*</sup>, N. Mortazavi<sup>b,c</sup>, M. Shahabi-Navid<sup>a,c</sup>, J.E. Svensson<sup>a,c</sup>, L.G. Johansson<sup>a,c</sup>, M. Halvarsson<sup>b,c</sup>

<sup>a</sup> Department of Chemical and Biological Engineering, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden

<sup>b</sup> Department of Applied Physics, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden

<sup>c</sup> Department of Materials & Manufacturing Technology, Chalmers University of Technology, 2A, SE-412 96 Gothenburg, Sweden

## ARTICLE INFO

### Article history:

Received 18 November 2014

Received in revised form

19 February 2015

Accepted 21 February 2015

Available online 23 February 2015

### Keywords:

In-situ microscopy

ESEM

Chamber pressure

Atmospheric corrosion

## ABSTRACT

The feasibility of environmental scanning electron microscope (ESEM) in studying the atmospheric corrosion behavior of 99.97% Mg was investigated. For reference, ex-situ exposure was performed. A model system was designed by spraying few salt particles on the metal surface and further promoting the corrosion process using platinum (Pt) deposition in the form of  $1 \times 1 \times 1 \mu\text{m}^3$  dots around the salt particles to create strong artificial cathodic sites. The results showed that the electron beam play a significant role in the corrosion process of scanned regions. This was attributed to the irradiation damage occurring on the metal surface during the ESEM in-situ experiment. After achieving to a reliable process route, in a successful attempt, the morphology and composition of the corrosion products formed in-situ in the ESEM were in agreement with those of the sample exposed ex-situ.

© 2015 The Authors. Published by Elsevier B.V. This is an open access article under the CC BY license (<http://creativecommons.org/licenses/by/4.0/>).

## 1. Introduction

Environmental scanning electron microscopy (ESEM) has established itself as a promising method for studying the interaction of different materials with humid air or water in different disciplines such as geology, medicine, biology, life and food science [1–3]. However, the use of ESEM in materials science research has been delayed for much time. This is due to the essential needs and considerations in preparation techniques and the problems associated with the pressure inside the microscope chamber. A practical example of potential uses of ESEM is the atmospheric corrosion of metals, where there is an interaction of metal surface with the micro-droplets, the transition of micro-droplets to an electrolyte layer, and then the formation of corrosion products on the metal surface [4–9]. In this regard, the ESEM method is expected to provide useful information on the early stages of corrosion, which will have an impact on the later stages of a corrosion process. Although the initial stages of the corrosion process can be investigated ex-situ using well-controlled corrosion chambers and field exposures [6–11], the problem with this approach is that the

dynamic phenomena in the corrosion process, including the formation of the electrolyte layer and corrosion products cannot be monitored as it occurs. In-situ exposure within an ESEM can be considered as a powerful tool to investigate the very initial stages of corrosion “live”. The localized and electrochemical nature and relatively fast kinetics of NaCl-induced atmospheric corrosion of some metallic materials such as magnesium (Mg) and Mg alloys imply that it would be possible to study the atmospheric corrosion by in-situ microscopy.

In the ESEM, the state of hydration of the specimen is determined by its temperature and the water vapor pressure in the microscope chamber [2,3]. Thus, ESEM can provide a combination of the required aggressive environment and continuous imaging throughout the corrosion process. This combination gives a unique opportunity to investigate the initiation and growth processes of the corrosion products on different metal grains. There are few in-situ ESEM corrosion studies on Mg and Mg alloys available in the literature [12–16]. Chen et al. [14] investigated the corrosion behavior of Mg alloys by changing the RH of the ESEM chamber. They examined the corrosion of the alloy AZ91 at a fixed value of RH in wet and dry cycles to study the effect of process parameters on corrosion product formation. Rossi et al. [16] studied initial stages of the NaCl-induced corrosion process on the surface of pure zinc (Zn) and Mg by means of ESEM using a humidostatic chamber. They promoted the corrosion process by increasing the RH of the

\* Corresponding author at: Department of Chemical and Biological Engineering, Chalmers University of Technology, SE-412 96 Gothenburg, Sweden.

E-mail address: [mohsen.esmaily@chalmers.se](mailto:mohsen.esmaily@chalmers.se) (M. Esmaily).

ESEM chamber and claimed that the ESEM could simulate an aggressive environment.

The ESEM method has also been successfully employed for oxidation experiments on steels and ceramics at high temperatures [17–20]. Jonsson et al. [20], who studied the initial oxidation of low-alloyed steel in the presence of small amounts of KCl (s) through ESEM in-situ exposure, stated that the in-situ results were in good agreement with corresponding tube furnace exposures in terms of the composition, thickness and morphology of the oxide scale. Despite of these attempts, still, there is a need to understand the effect the lower exposure pressure ( $\sim 2\text{--}4$  Torr) and the presence of an electron beam (e-beam) on the corrosion mechanisms occurring in the ESEM. The atmospheric corrosion behavior of Mg and Mg alloys under outdoor conditions and in the laboratory has been extensively investigated [21–26]. In this study, the influence of the two mentioned inherent parameters of the ESEM in-situ exposure on the NaCl-induced atmospheric corrosion process of pure Mg was examined. The corrosion mechanism inside the ESEM chamber was described and the results were compared to those of the ex-situ exposures.

## 2. Experimental

Pure Mg (99.97% Mg) with a chemical composition of 0.005% Zn, 0.003% Al, 0.003% Si, 0.0023% Mn and 0.001% Fe and balance Mg (by weight) was used as the test material. The in-situ exposures were performed by means of a FEI Quanta 200 FEG ESEM (FEI Company, Hillsboro, OR, USA) with a Schottky Field Emission Gun (FEG). We used an oil free pump to prevent carbonaceous depositions over the scanned areas. Imaging was performed throughout the degradation process using an acceleration voltage of 20–30 kV. The instrument was equipped with Oxford Inca energy dispersive X-ray detector (EDX).

Post-exposure analyses were performed by SEM/EDX for local chemical composition analysis of the corrosion products as well as elemental mapping of corroded metal surfaces. Samples were machined from ingot to obtain small-sized cylinders with dimensions of 3 mm diameter and 4 mm height according to the required dimension for placing the samples inside the Peltier heating/cooling stage for the ESEM in-situ experiments, see Fig. 1. The Peltier stage enables operating exposures in temperature between  $-20$  and  $100$  °C [2]. In this study a temperature of  $4$  °C was used for the in-situ exposures, see below. Prior to corrosion

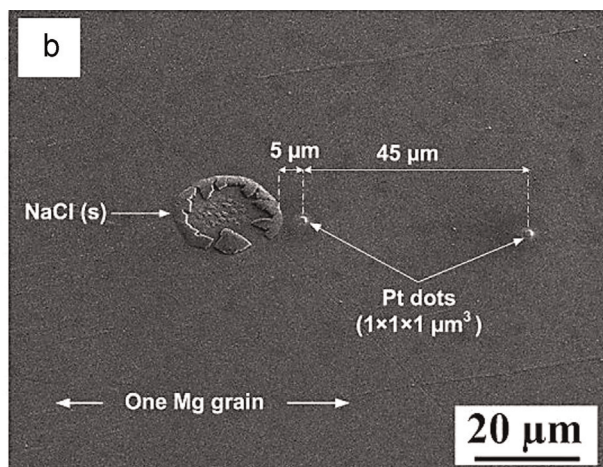
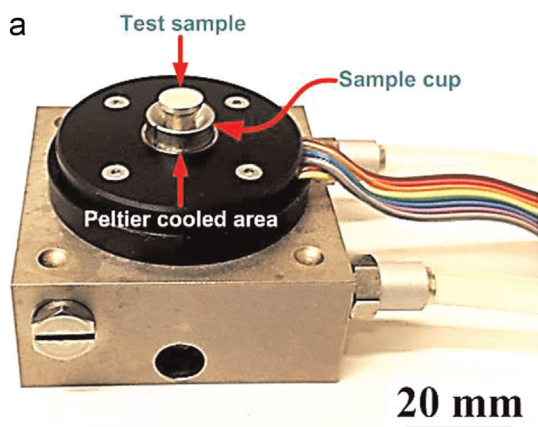


Fig. 1. (a) Photo of the Peltier cooling stage of the FEI Quanta 200 FEG ESEM used in this study and (b) SE SEM image showing the position of the two Pt spots with respect to the salt droplet.

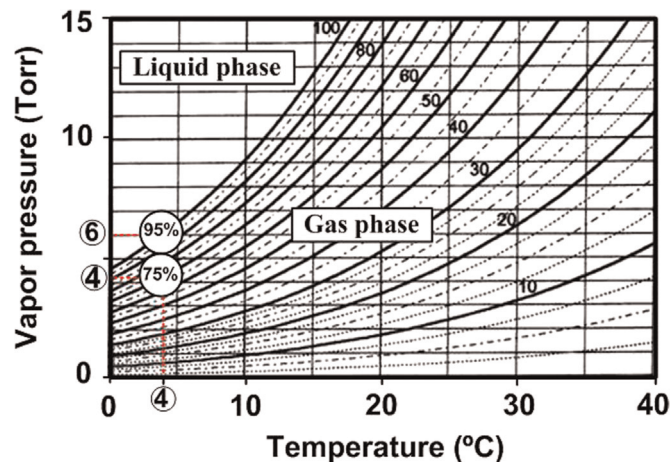


Fig. 2. Relative humidity (RH) isobar [10] chart used for the in-situ corrosion experiment. Showing the temperature and pressure used for the in-situ experiments to produce relative humidity (RH) of 75% and 95%.

experiments, a solution of 20 ml distilled water, 80 ml ethanol and 1 g NaCl was sprayed on the samples, which in total were contaminated with  $5 \mu\text{g}/\text{cm}^2$  NaCl (equal to  $40 \mu\text{g Cl}^- \text{C m}^{-2} \text{y}^{-1}$  salt).

In ESEM, water can be condensed on a sample surface by decreasing the Peltier cooling stage temperature to *few degrees* and increasing the chamber pressure. The liquid/gas phase border is 6.1 Torr at  $4$  °C according to the relative humidity (RH) isobar shown in Fig. 2. Hence, a higher pressure than 6.1 Torr will result in liquid condensation (100% RH) on the metal surface, while using a lower pressure controls the RH in the gas phase ( $< 100\%$  RH). As local analyses were performing in individual grains, it was essential to identify the crystallographic orientation of the grains studied before the exposures. This was done using a LEO Ultra 55 FEG-SEM equipped with an HKL Channel 5 Electron Backscatter Diffraction (EBSD) system. The sample preparation procedure for the EBSD measurement is described elsewhere [27].

The exposures performed in this study are explained in Table 1. Three ESEM in-situ exposures were performed at two different RH (75% and 95%). Different levels of RH were used to observe the formation of the electrolyte layer in different humid environments. The exposures time and temperature were 320 min (5 h) and  $4$  °C, respectively. Different RH values were obtained by employing different chamber pressures, see Fig. 2.

Download English Version:

<https://daneshyari.com/en/article/8038124>

Download Persian Version:

<https://daneshyari.com/article/8038124>

[Daneshyari.com](https://daneshyari.com)