Comparing Modeled and Experimental Accelerated Corrosion Tests on Steel

N. Van den Steen,a, z H. Simillion,a D. Thierry,b, H. Terryn,a and J. Deconinck a

aResearch Group Electrochemical and Surface Engineering (SURF), Vrije Universiteit Brussels (VUB), 1050 Brussels, Belgium
bInstitut de la corrosion, Technopôle de Brest Iroise 220, F-29200 Brest, France

The high economical impact of atmospheric corrosion1 can be reduced by enhanced corrosion management and prevention based on accurate predictions. Large advancements could be accomplished by providing new insights in different mechanisms and a viable alternative for expensive and time consuming experiments on the long run. The thickness and composition of the electrolyte influence the whole process, affecting the reactions, accessibility of oxygen, concentrations of species, etc. In spite of the large impact of the electrolyte thickness and composition, most models focus on the pure electro-chemical aspect of the problem, largely ignoring the dynamic film formation.2–5 A general review of the advances in atmospheric corrosion modeling can be found in Ref. 6. This work is an effort to fill the void in this domain.

The first results of the Dynamic Electrolyte Film Corrosion (DEFC) model were presented in a previous paper,7 focussing on the physics and applicability of the proposed model. While this paper, based on the work of Baklouti et al.,8 still concentrates on the liquid film formation, simulated general corrosion depths are compared with experimental data for different accelerated corrosion tests obtained with the Electrical Resistance (ER) sensors.9 From the abundance of accelerated corrosion standards used in industry, four different tests are considered: VDA233-102, GMW 14872, Volvo Act 1 (STD 423-0014) and Volvo Act 2 (VCS 1027,1449 equivalent to Ford CETP 00.00-L-467). All four tests are still relevant and differ from each other in complexity and application of the salt spray.

The outline of this paper is the following. The first objective is to demonstrate the importance of dynamic film evolution when modeling atmospheric corrosion. The second objective is to evaluate the conditions for which a similar trend between the simulated and experimental data is observed. By comparing different tests, coincidental similarities are avoided and points of attention/objectives for future work can be evaluated.

First a description of the different accelerated corrosion tests is provided. Understanding the differences between the different tests is essential to evaluate the (dis)similarities between the model and experiments. Afterwards a short introduction to the ER monitoring system, used to obtain the experimental data, is provided. Next, an introduction to the DEFC model and the (elementary) corrosion model is given. A more in-depth overview is given on the sections of the model which have been improved compared to previous work.7 The goal of this work, the comparison between the simulated and experimental results, is done in the next section. Finally, conclusions are formulated with suggestions for future work.

Accelerated Corrosion Tests

Accelerated corrosion testing has been used since the 1900 s to evaluate the performance of coatings and materials when exposure to the actual conditions is neither practical nor available.10,11 An accelerated corrosion test is commonly a cabinet test method to test material samples exposed to a climatic stress for a given period of time. For most tests, the main objective is to establish a ranking of materials, depending on their corrosion resistance. Two main types are distinguished: static atmosphere testing and cyclic corrosion testing.11–13 During static atmosphere testing, a constant single environment is applied throughout the complete duration of the test. A well known example is the salt fog test (ASTM B 117).12 The search for reliable test methods, mainly for the automotive industry, led to a wide variety of cyclic corrosion tests. Nowadays there are almost as many accelerated corrosion tests as there are car manufacturers. Also multiple efforts have been made to evaluate the correlation between accelerated corrosion tests and real-world environment tests, highlighting the importance of selecting the appropriate test corresponding with the application.13–14

The cyclic tests addressed in this work are the VDA 233-102, GMW (14872), Volvo Act 1 (STD 423-0014) and Volvo Act 2 (VCS 1027,1449 equivalent to Ford CETP 00.00-L-467). Short descriptions of the tests are given below. Detailed information on the application of the addressed cycles can be found in the standards.15–18

VDA 233-102.—The VDA 233-102 is the most complex test used in this work. The temperature and relative humidity evolutions for the different sub-cycles are given in Figure 1. The salt spray with a 1% sodium solution and freezing periods are indicated by a darker section in sub-cycles A and C respectively.

GMW 14872.—The temperature and relative humidity evolution of the sub-cycles are given in Figure 2. One notices immediately that this cycle is much less complex than the VDA 233-102. The salt spray is applied twice during cycle A as indicated by the darker red zones. The spray is performed with a 1% salt solution, consisting of 0.9% sodium chloride, 0.1% calcium chloride and 0.075% sodium bicarbonate. Because sodium bicarbonate has no significant hygroscopic effect, it is ignored in the model. In contrast with the VDA 233-102 cycle, a rather broad range of relative humidities is allowed during the sub-cycle A, indicated in the figure by the purple area. The exact humidity applied within this range can have a non-negligible effect.

Volvo Act 1.—The Volvo Act 1 cycle is composed of two sub-cycles. Both cycles, each with a duration of 12 hours, are illustrated in Figure 3. Sub-cycle 1 consists only of relative humidity and temperature control, while the first part of sub-cycle 2 is meant to keep

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the samples visibly wet by using a salt spray (1% sodium chloride solution). This salt spray is of a totally different type than the salt spray in the VDA 233-102 and GMW. Instead of a fog as used in these tests, a shower like spray is applied. A standard week consists of a repetition of sub-cycle 1 (SC1) where on Monday and Friday, the second sub-cycle is replaced by sub-cycle 2 (SC2).

**Volvo Act 2.**—This cycle consists of two longer sub-cycles: one 24 h cycle for weekdays (SC1) and a 48 h cycle for the weekends (SC2). The temperature and relative humidity evolutions are shown in Figure 4. SC1 consists of a wet phase with four salt sprays with a 0.5% sodium solution: a first 10 min long spray and later three short (3 min) sprays to assure a wet surface. On Figure 4 the salt sprays are
indicated with arrows. SC2 is a simple cycle with constant humidity and temperature control.

Experiments

The electrical resistance (ER) monitoring system AirCorr™ has been used in aggressive environments, such as on operating vehicles and ships, weathering sites and accelerated tests. For these applications, the corrosion depth measured by the sensor appeared in good correlation with parallel exposed metallic coupons.

The ER method relies on the evolution of the electrical resistance R of a conductor as a function of its resistivity (\(\rho\)) and geometry; length (l), cross section (S), width (w) and thickness (t) according to:

\[
R = \rho \frac{l}{S} = \rho \frac{l}{w \cdot t}.
\]  

[1]

For a given resistivity \(\rho\) and when \(w >> t\), R is inversely proportional to t. Because the resistivity is dependent on the material and the temperature, the ER method uses a differential measurement based on a sensitive track exposed to the environment, i.e. subjected temperature variations and corrosion, and a second protected track as reference, i.e. only subjected to temperature variation. A schematic view of the geometry (55 × 3 mm) of the ER probe is presented in Figure 5. More details about the ER probe dedicated to aggressive environments are specified elsewhere.

Using the resistance of the sensitive and reference track, initially before exposure (\(R_{\text{ref, init}}\) and \(R_{\text{sens, init}}\)) and during the exposure (\(R_{\text{ref}}\) and \(R_{\text{sens}}\)), the mean corrosion depth (CD) can be calculated according to:

\[
CD = t_{\text{init}} \left(\frac{R_{\text{ref, init}}}{R_{\text{sens, init}}} - \frac{R_{\text{ref}}}{R_{\text{sens}}}\right).
\]  

[2]

In this work mild steel sensors (AISI 1010) were used. The thickness of the sensitive track was 250 \(\mu\)m. The measurements were recorded automatically with a data logger that was exposed together with the sensors in the climatic chambers during the cyclic corrosion tests described previously. Hence in-situ measurements were performed in real time during various cyclic corrosion test procedures. All measurements were only performed once.

It should be noted that the ER technique used in this work is also subjected to error. If for example, the sensor is covered with a highly conductive electrolyte, the device does not only measure the resistance of the metal track, but the combined electrolyte-track system. Still, these independent measurements are valuable for comparing the trend of the corrosion depth evolution during different tests.

Physics and Model

**Basic film condensation/evaporation model.**—Heat and mass transfer are the two main phenomena involved in the dynamic evolution of the electrolyte film. Coupled by the latent heat, it is impossible to solve the two problems separately. The driving force of the mass transfer (evaporation/condensation) is the temperature difference (\(\Delta T\)) between the surface temperature (\(T_s\)) and the surrounding air (\(T_{\infty}\)). This temperature difference is a function of the material properties (specific heat capacity, density), geometry (thickness), air temperature, heat transfer from/to the air and the latent heat flux. The value of the relative humidity (\(RH_{\infty}\)) defines whether the temperature difference causes evaporation or condensation of the liquid film. A schematic representation is shown in Figure (6).

Some pollutants induce hygroscopic effects and will attract moisture from the air when the relative humidity exceeds the deliquescent
relative humidity (DRH). The presence of these hygroscopic salts will add an additional driving force, which depends on the relative humidity, the type of salts and their concentrations.

To obtain the temperature evolution at the surface of a specimen, the partial differential equation

\[
c_p \rho \frac{dT}{dt} = \nabla (-k \nabla T),
\]

which depends upon the heat conduction coefficient \(k\), the specific heat capacity \(c_p\) and the material density \(\rho\), is solved. The boundary condition, the heat transfer between the material and the air \(q_{\text{core}}\), can be written as

\[
q_{\text{core}} = h(T_s - T_s). \tag{4}
\]

This convection flux depends upon the heat transfer coefficient \(h\). With the temperature evolution, the mass transfer \(\dot{m}\) can be found using

\[
\dot{m} = k_m(w_{\text{inf}} - w(T, \text{salts})), \tag{5}
\]

with \(k_m\) the mass transfer coefficient. The Lewis number \((Le)\) links the mass transfer coefficient \(k_m\) with the heat transfer coefficient \(h\):

\[
h = c_p Le^2, \tag{6}
\]

with \(c_p\) the specific heat capacity. The absolute air humidity \((w_{\text{inf}})\) can be calculated from the relative humidity of the air, given by

\[
w_{\text{inf}} = \frac{0.622 p_{\text{inf}} RH_{\text{inf}}}{p - p_{\text{inf}} RH_{\text{inf}}}, \tag{7}
\]

with \(p\) the pressure, \(p_{\text{inf}}\) the vapor pressure and RH the relative humidity of the air. The local absolute air humidity \((w)\) is given by

\[
w = \frac{0.622 p'_{\text{inf}}}{p - p'_{\text{inf}}}, \tag{8}
\]

with \(p'\) the vapor pressure. The vapor pressure can be adapted to take into account the experimentally observed vapor pressure above salt solutions. The latent heat \((Q_{\text{latent}})\), given by

\[
Q_{\text{latent}} = \dot{m} L_v, \tag{9}
\]

links the heat and mass transfer problems through the specific latent heat \((L_v)\). A thorough overview of the governing equations can be found in Ref. 7.

When a salt spray/fog is modeled, the following assumption is made. Run-off (with droplets) is a complex phenomenon and would require a complete study on its own. Therefore a constant uniform electrolyte is assumed during this phase. The salt distribution on the surface is assumed to be equal to the quantity of salt present in this film. One can argue that this constant film is to be seen as the averaged value.

Compared to our previous model, the treatment of mixed hygroscopic salts has been improved. Now, the combined effect of the salts in the mixture is taken into account. In case of the presence of hygroscopic salts, the vapor pressure above the surface is different from the saturated vapor pressure and equation 8 becomes:

\[
w = \frac{0.622 p'_{\text{inf}}}{p - a_{\text{inf}} p'_{\text{inf}}}. \tag{10}
\]

In Figure 7 the calculated water activity \((a_{\text{inf}})\) above a surface with an area density of 0.9 g m \(^{-2}\) of sodium chloride and 0.1 g m \(^{-2}\) of calcium chloride is shown as a function of the film thickness. The calculated values for the water activity take into account the solubility of each salt and the combined vapor pressure lowering.23

DRH and efflorescent relative humidity (ERH) values for single salts are taken from the literature.24–26 The effect of the DRH is included in the vapor pressure measurements as a function of the salt concentration. Size effects of the particles are not taken into account with this approach.25 For determining the ERH values, dedicated experiments in a RH controlled environment are needed.27,28

All the accelerated corrosion tests considered in this work, have a salt spray phase. The modeling of this phase is not included into the model, as runoff is a complex phenomenon. During this phase, a constant film thickness of 100 \(\mu\)m is assumed. From the assumed thickness, the area densities of the salts are calculated based on the concentrations of the spray. This choice will affect the values of the simulated film thicknesses, but as we are primarily interested in the trend of the corrosion depth evolution, the impact will be minor.

**General corrosion model**—This work focuses on the modeling of the dynamic film formation through evaporation and condensation. However, a corrosion model is needed to evaluate the impact of the film thicknesses on the metal removal rate. A second reason for using a general corrosion model is to facilitate the comparison with experimental results, as experimentally the corrosion depth is the measured quantity.

Corrosion of iron in wet conditions is largely dictated by oxygen reduction at limiting current density except at very thin film thicknesses.29–32 This results in a corrosion current density \((j)\) inversely proportional with the film thickness \((h)\), given by

\[
j = \frac{nF D_{O_2} C_{O_2}}{h}. \tag{11}
\]

In this equation, \(n\) is the number of electrons involved in the reaction, \(F\) is the Faraday constant, \(D_{O_2}\) and \(C_{O_2}\) are the diffusion coefficient and bulk concentration of oxygen respectively. In the literature, a maximum corrosion current density around 1 \(\times\) 3 \(^{-3}\) A cm \(^{-2}\) is observed during drying. After further drying, a decrease to zero is reported. Therefore, the corrosion rate is limited at 1 \(\times\) 10 \(^{-3}\) A cm \(^{-2}\). Below 10 \(\mu\)m, the corrosion is no longer controlled by oxygen diffusion and a linear decrease to 0 A cm \(^{-2}\) is assumed. The linear decrease is assumed to avoid the implementation of a complex corrosion model, as the focus of this work lies on the dynamic behavior of the film thickness. It is expected that this assumption will only cause a minor deviation, as a similar behavior is found experimentally.31,33 A more extensive overview of equations and applied limits as given previously.1 This model is applicable only when dealing with a clean steel substrate, excluding all discussions on the role of oxide layers, passivation, corrosion products, etc. All elements could be added, but would deviate from the focus on the role of varying environment in presence of salts.

The bulk concentration of oxygen, which can be calculated by

\[
c_{O_2} = c_{O_2} \exp \left( \frac{3.64 \times 10^{-4} \text{RH} - 5844}{h} \right). \tag{12}
\]

is already a function of the salt concentration in the electrolyte.34 In this equation, \(c_{O_2}^{\text{bulk}}\) is the bulk concentration without salts. The dependency
of the diffusion coefficient of oxygen on the salt concentration was added by

\[ D_{O_2} = D_{O_2,0} \exp(-a \cdot c_{salt}). \]  

Where \( D_{O_2,0} \) is the reference diffusion coefficient without salts, \( a \) is a coefficient depending on the salt (\( 1 \times 10^{-4} \text{ m}^2\text{s}^{-1} \) in the case of \( \text{NaCl} \)) and \( c_{salt} \) is the salt concentration. The maximum concentrations of the salts are set to the corresponding saturation concentrations, preventing the concentrations used in the calculations to reach infinity when the film thickness reaches zero. The effects of the limited salt concentration are clearly visible in Figure 8 at a thickness of 5.5 \( \mu \text{m} \) when 2 g m\(^{-2} \) of salt is present. Below a film thickness of ±5.5 \( \mu \text{m} \) the solution is saturated, resulting in a constant bulk concentration and diffusion coefficient of oxygen in this range. A further decrease of the film thickness results in an increased accessibility to oxygen, as the length of the diffusion path is decreased, thus causing a higher corrosion current density until the linear imposed maximum is reached at a thickness of 3.2 \( \mu \text{m} \).

The corrosion rate can be derived from the current density by assuming a loss of \( 1.32 \times 10^{-1} \text{ mh}^{-1} \) of steel at a corrosion current density of \( 1 \times 10^{-4} \text{ A cm}^{-2} \).

A schematic overview of the workflow of the corrosion model is provided in Figure 9 to summarize this section.

**Impact of the model improvements.**—The improvements made compared to the previous model focus on the deliquescence and efflorescence of salt mixtures. Of the four tests considered in this work, the GMW 14872 makes use of a sodium chloride and calcium chloride mixture. In Figure 10 the simulated thickness during a 24 h interval with the previous and improved model are illustrated. The significant differences are between 3 h and 8 h. During this period, the film thickness predicted by the improved model is almost eight times higher (0.23 \( \mu \text{m} \) and 1.8 \( \mu \text{m} \)). Combined with the corrosion current density illustrated in Figure 8, it is clear that this will have a big impact.

**Comparison of simulations and experimental data**

Note that the simulations presented here were performed in a one-dimensional space, assuming ideal uniform material properties, film thicknesses and other parameters.

The initial output of all the simulations is the film thickness evolution with time. In post-processing, this simulated film thickness is combined with the corrosion current model (Figure 8) and results by integration with the corrosion depth evolution in time. The time dependent film thicknesses are only provided for the VDA 233-102 cycles as an example of the workflow.

**VDA 233-102.**—Experimental data for two different sequences were available for the VDA 233-102: \( \text{ABA}_1\text{CABB} \) and \( \text{BACABB}_1 \). The same samples were subjected twice to the cycle with sequence \( \text{ABA}_1\text{CABB} \). The difference between the measured corrosion depth of the
two curves is significant as shown in Figure 12: the increase in corrosion depth after the first week is almost 31 \( \mu m \) or 61% of the corrosion depth after the second week. A major contribution to this difference is already present after the first sub-cycle (12 \( \mu m \)). As the corrosion test progresses, the corrosion depth increases at a slower rate during the first cycle than during the second cycle. This suggests that there is a period needed to initiate the corrosion process or destroy a pre-existing oxide layer, causing a different behavior between the experimental results of the first and the second week. This observation immediately points out that a fully quantitative prediction of the corrosion depth is only possible with this model in ideal circumstances. But here the aim is to demonstrate the importance of the electrolyte film thickness.

To facilitate the comparison of the trend, both experimental (week 2) and simulated corrosion depths were scaled to unity in Figure 12. Remarkable similarity of the trend is observed, but a delay between the experiment and simulation is visible, especially in sub-cycles A and C.

The agreement between simulated and experimental corrosion depth is much less obvious for the alternative VDA 233-102 cycle (Figure 13). Notice that only one week of experimental results are available. Scaling the complete range as shown in Figure 13 suggests that the agreement improves after the first three/four sub-cycles, supporting the idea of an initiating period. In the first two sub-cycles (B and A), there are almost no similarities in the trend. In sub-cycle C, a shift is present: in the experimental results, the corrosion stops immediately as soon as this sub-cycle starts, while the corrosion depth of the simulation continuous to increase until the temperature drops below the freezing point. For the following sub-cycles (ABBA), the trends are similar, but not identical.

**GMW.**—Figure 14 shows the simulated and experimentally measured corrosion depth for 288 h in the GMW test. A gradual increase
in corrosion rate is observed in the experimental results. To aid in the comparison, the experimental results are divided into three parts as shown in Figures 15. In each time interval the corrosion depth is aligned. Note that there were no particular reasons to subdivide the cycle in these specific parts. Similarities between the simulation and experiment are clearly visible. However, as the cycle progresses, sudden increases in the corrosion depth at the beginning of each sub-cycle C (green) are measured. When transitioning from sub-cycle B to C, the relative humidity is decreased while the temperature is increased. This causes the film thickness to quickly reduce. Because the film thickness is reduced so quickly, the corrosion depth increase caused by the higher corrosion current rates in thin films, is almost not visible in the simulated results. The increasing nature of this effect over time strengthens the hypothesis that the buildup of iron corrosion products lies at the base of this phenomenon. The iron corrosion products probably cause additional hygroscopic and capillarity effects, increasing the drying time. Consequently a thin electrolyte film, which causes the highest corrosion current rates, will be present for a longer period of time.

Volvo Act 1.—The experimental and simulated corrosion depth during the Volvo Act 1 corrosion test are shown in Figure 16. The effects of corrosion processes are clearly visible in the experimental results. Starting from every salt spray phase, an increase in the corrosion rate (slope) is observed in the experiments. This observation is used to distinguish four regions, separated by vertical dotted lines in Figure 16. In the further evaluation, the focus will lie on the sections indicated by a and b.

In Figure 17 the sections a and b are compared individually by aligning the corrosion depth at the beginning. In both figures, the corresponding trends between simulation and experiment are clearly visible, especially in section a. Only during the salt spray phase there is a little similarity. In the other phases, the behavior of the corrosion depth of the experiments is smoother. A possible explanation could be the corrosion products limiting the evaporation of the electrolyte as described above.

Volvo Act 2.—Figure 18 compares the corrosion depth of the experiments and the simulation for the Volvo Act 2 test. Experimental data for two week cycles are provided. The downward surges in the experimental data, which are observed for both experimental curves, could be caused by a sudden decrease in solution resistance because of the heavy salt solution sprays. This decrease could (partially) shortcut the sensor, resulting in a lower measured resistivity. Because these sprays take place at the beginning of the red phase, a small time shift of the data is suspected. It is also clear that the increased corrosion rate visible in the simulated results when going from the blue to the green zone is not found in the experiments. On the other hand, the experiments show an increased corrosion depth rate when going from...
tests we can suspect that the combination of the dynamic film thickness model with the corrosion model works best in strongly varying accelerated corrosion tests where a fog is applied as salt spray. The corrosion depth predictions of the model for accelerated corrosion tests with longer periods of constant environmental conditions correspond less with the experimental data. The environmentally constant conditions increases the relative impact of the corrosion products on the total observed corrosion depths, which are not yet included into the model.

Despite the simplifications in the film and the elementary corrosion models, the importance of a dynamic electrolyte film model has been proven. We conclude that for a correct modeling of accelerated corrosion tests, or atmospheric corrosion, the electrolyte film model is a crucial component.

Part of the differences between the simulations and results can be attributed to the effect of iron corrosion products. As iron corrosion products build up, hygroscopic and capillary effects influence the drying and wetting of the sample. A second phenomenon is the possible initial passivation of the surface. By stepwise adding an elaborate corrosion model, simulating e.g. also pH and corrosion products distributions, also these effects could be simulated.

Even without the quantitatively correct corrosion predictions, this model may prove useful. We show the important role of the hygroscopicity of salts, which is responsible for the presence of a film. A thin electrolyte film is the precursor of corrosion. During the design of a component, the geometry could be optimized to reduce the number of corrosion sites or key area’s can be identified to receive additional protection or maintenance.

For the prediction of long-term (outdoor) corrosion, both the corrosion and film model should be improved. First of all, the film model should be combined with an elaborate corrosion model, including the formation and role of corrosion products and complex corrosion mechanisms. For the dynamic film model, the biggest difference will be the hygroscopic effect of the corrosion products and possibly the impact of a porous layer.

Conclusions

Four different accelerated corrosion tests were modeled. Simulated results were compared with experimental data and three tests showed good agreement in the trend of the corrosion depth. Hygroscopic salts have a big impact on the film thickness and corrosion depth. An electrolyte film thickness model is a necessary component of an atmospheric corrosion model.

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