Secondary spreading of acidified aerosols on the surface of Zn

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\textbf{A B S T R A C T}

Secondary spreading of acidified sodium chloride (NaCl) aerosol droplets on the surface of zinc have been studied. The in-situ observation of the secondary spreading, corrosion morphology, corrosion products under different position and volume loss are used to understand the secondary spreading of the acidified aerosol. The results reveal that the secondary spreading effect of NaCl aerosol droplet is more pronounced compared to the acidified aerosol droplets which are mainly embodied in larger spreading region and faster spreading rate. The difference of the secondary spreading effect is associated with acidification which inhibits the increase in the pH at the periphery. Furthermore, lower corrosion rate was observed for the acidified aerosol droplets due to smaller secondary spreading area (cathodic region). The main corrosion product in the center of NaCl aerosol droplet and the aerosol droplet acidified by HCl is Simonkolleite, while for the aerosol droplet acidified by H\textsubscript{2}SO\textsubscript{4} which shows several different corrosion morphologies in the center, the main corrosion products are Gordaite, Simonkolleite and Zn\textsubscript{3}Cl\textsubscript{2}(OH)\textsubscript{4}SO\textsubscript{4} · SH\textsubscript{2}O. The formation of protective corrosion products Gordaite that can inhibit the anodic reaction leads to the smallest spreading area and the lowest corrosion rate for the aerosol droplet acidified by H\textsubscript{2}SO\textsubscript{4}.

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1. Introduction

Zinc and its alloys are widely used in many fields because of the relatively lower prices and good corrosion resistance. Atmospheric corrosion as the common corrosion type of zinc has been studied in both the laboratory simulated environment and field exposures [1–10]. The sodium chloride deposited on the surface of metal can accelerate corrosion significantly and induce the failure of structures [2,9,11,12]. Hence, the aerosol droplets as a main source of salts play a significant role in the marine atmospheric corrosion of zinc and amount of work in this field has been done [5,13–19]. In marine environment, aerosols are formed by turbulent ocean states and surf action and transported inland predominantly by coastal wind patterns [20]. The marine aerosols can travel long distances, even hundreds of kilometers from their source. During the transportation, aerosols will be acidified by absorbing different kinds of gas, such as HCl, SO\textsubscript{2}, NO\textsubscript{x} and NH\textsubscript{3}, and the typical pH value of aerosols is 1–4 [6].

Secondary spreading, as a critical phenomenon of aerosol-induced corrosion, was first observed on the surface of zinc by...
Neufeld et al. [13]. And the phenomenon is contributed to the change of surface tension caused by the high pH at the periphery of aerosol droplets [13,14,21]. Then, the aerosol droplets become unstable after corrosion for a certain time because of the change of surface tension and a thin film will spread out from the periphery [13,14,22,23].

In-situ observation is an effective method to study the process of secondary spreading. Neufeld et al. found that the distance from the periphery of the original droplet to the edge of the spreading region and the square root of time was linear regression relation [13]. Tsuru et al. revealed the relationship between the spreading rate and relative humidity and found higher spreading rate in higher relative humidity [22]. In addition, Azmat et al. observed the changing process of pH under the droplets. The pH of the periphery increased soon before the secondary spreading of acidified droplets, indicating that the edge of the droplet acted as cathode during the corrosion and hydroxyl ions were formed due to the oxygen reduction [23]. Chen et al. found that in the environment with high concentration of CO₂ and SO₂, NaCl droplet showed lower spreading rate and small spreading region, and sometimes no secondary spreading was even observed [13,14,21,24].

The acidification of aerosol droplet has a significant effect on the corrosion rate. On one hand, the acidification can case the dissolution of oxide and change the composition of the corrosion products. On the other hand, the secondary spreading effect is pH dependent and secondary spreading area acting as cathode in the corrosion process can increase the corrosion rate under the aerosol droplet. Azmat et al. studied the corrosion under acidified fine aerosol droplets (<5 μm) and large droplets (≈2 mm) [17,23]. The result revealed the synergistic effect between the concentration of chloride and the type of acid. And the corrosion of Zn under sulfur-containing aerosol droplets exhibited a lower corrosion rate, which was attributed to the formation of sulfur-containing complexes. However, fewer studies focused on the effect of the area of secondary spreading region on the corrosion rate.

Although amount of work has been done on the aerosol droplet corrosion, the study of work of acidification on the process of secondary spreading is still lacking, especially the kinetic data. The objective of this article is to study the corrosion process of Zn under acidified aerosol droplets including the spreading process, corrosion products and corrosion rate. The secondary spreading process was observed in-situ with the use of stereomicroscopy. The species and distribution of corrosion products were analyzed using SEM/EDS and Raman spectroscopy. Furthermore, the corrosion rate was calculated based on the volume loss measured by laser scanning confocal microscope. The result obtained from this work is helpful to understand the mechanism of secondary spreading.

with 2.5-μm diamond paste. Finally, the samples were rinsed with deionized water and ethanol and dried with cold flowing air. The samples were stored in a desiccator (<10% RH) for at least two days before the experiment.

To investigate the secondary spreading of acidified aerosol droplets, NaCl solutions were acidified by HCl and H₂SO₄ to pH = 1 respectively. For the all acidified solutions, the concentration of Cl⁻ was 0.6 mol/L, and 3.5% NaCl solution that was not acidified served as a comparison. The aerosol droplets were deposited on the surface of samples by a sprayer, and the diameters ranged from 50 μm to 250 μm. After deposition of aerosol droplets, the samples were immediately put in an environment chamber with 90% ± 3% RH. The experiments with different solutions were repeated for three times at least. After corrosion for 6 h, the samples were taken out from the environment chamber and stored in the desiccator (<10% RH) before the following analysis.

2.2. In-situ observation of secondary spreading

The processes of secondary spreading were monitored by stereomicroscope. A schematic diagram of the in-situ observation experimental setup was shown in the previous articles [25]. The relative humidity controlled by the glycerol-water solution at the bottom of the humidity chamber was set as 90% ± 3%. And the temperature of the experiment was 25° ± 3°. The RH and temperature were monitored using a temperature/humidity sensor. The optical images of aerosol droplets at different time were taken by stereomicroscopy equipped with a digital camera through the hole in the upper face covered by a thin (t ≈ 0.15 mm) glass.

2.3. Analysis of corrosion products

The corrosion morphologies under different positions of the aerosol droplets after corrosion for 6 h were studied using an XL30-PHILIPS SEM at 25 kV. Energy dispersive spectroscopy was performed by energy dispersive spectrometer (Oxford Instruments).

Furthermore, the corrosion products of different positions were identified through Raman spectroscopy (Horiba, HR Evolution) equipped with multiple Olympus and a Feltier-cold charge-coupled device (CCD) detector. The excitation source is a 532 nm laser with an incident laser power of 1mw. The accumulation time was 60 seconds.

2.4. Measurement of volume loss under the original droplets

The corrosion rate of Zn under the original aerosol droplets was quantified by the measured of volume loss. The corrosion products formed on the surface of zinc were removed by immersing the samples in the specific solution (prepared by putting 100 g NH₄Cl in distilled water to make 1000 mL solution) at 70°C for 5 min according to ISO 8407. Then, the samples were rinsed with deionized water and alcohol, dried with cold flowing air, and stored in desiccator. The volume loss (ΔV) under the original droplets was measured using laser scanning confocal microscope (Olympus OLS4000).

3. Results

3.1. Results of field observation of secondary spreading

The development process of spreading was observed in-situ to better understand the effect of acidification on the secondary spreading. For the three aerosol droplets, the entire processes of secondary spreading were recorded every 5 min by stereomicroscopy with a camera system. Fig. 1 shows the in-situ optical images of aerosol droplet acidified by HCl. After the aerosol droplets impinged on the surface of Zn for 20 min, micro droplets appeared at the outer edge of the original droplet. And then thin film spread outward from edge of the droplets in the following corrosion process. It should be noted that the thin film spread faster and farther in the direction of the scratch. Finally, the thin film would stop spreading within 120 min. The spreading processes of the other two aerosol droplets not shown here were similar with the NaCl aerosol droplet acidified by HCl. After the secondary spreading stopped, the diameter of the secondary spreading region attained a value 3–4 times that of the original droplets for the NaCl aerosol droplets, while it was 2–3 times for the NaCl aerosol droplets acidified by HCl and 1–2 times for the NaCl aerosol droplets acidified by H2SO4.

Furthermore, the area of secondary spreading region of the three aerosol droplets at different times were measured using image analysis software of the video camera system. Diameters of the spreading regions were calculated by assuming that the secondary spreading regions were circular. As illustrated in Fig. 2, the secondary spreading of the three aerosol droplets showed the similar development trend. The spreading rate was very fast in the initial stage of secondary spreading, and then slow down until the stop with corrosion time. However, the initial stable period was shorter for NaCl aerosol...
droplets (5 min) compared with the acidified aerosol droplets (20 min). In addition, secondary spreading of NaCl aerosol droplets showed a faster spreading rate compared with the acidified aerosol droplets that had the similar spreading rate. The spreading time was also different for the three different aerosol droplets. The spreading time of NaCl aerosol droplets and NaCl aerosol droplets acidified by HCl was about 120 min, while it was about 60 min for NaCl aerosol droplets acidified by H₂SO₄.

3.2. Distribution of corrosion products under the aerosol droplets

As shown in the previous work, the central and periphery of the aerosol droplets acted as anode and cathode, respectively [13]. Hence, the compositions of corrosion products were also different in these regions. Raman spectroscopy as a powerful microanalysis method was used to study the spatial distribution of corrosion products [23,26,27].

Fig. 3 showed Raman spectra of the corrosion products in different regions of the NaCl aerosol droplets. The Raman
The spectrum of the central aerosol droplet (Fig. 3a) showed the corrosion product was Simonkolleite (Zn₃(OH)₂Cl₂·H₂O). In more detail, the peaks at 212, 256 and 267 (shoulder) cm⁻¹ were attributed to Zn-Cl bond and the peak at 394 cm⁻¹ was related to the Zn-O vibration characteristic of Simonkolleite structure. Furthermore, two peaks at 3455 and 3488 cm⁻¹ assigned to O–H fundamental stretching bands were also observed [23,28,29]. The main corrosion products in the periphery of the droplet were ZnO and Hydrozincite (Zn₅(CO₃)₂(OH)₆). The band at 1086 cm⁻¹ was the ν1 symmetric stretching mode of the carbonate unit of Hydrozincite and the bands in the range of 2845–2935 cm⁻¹ were belonged to the O–H stretching Hydrozincite [23,27,30]. The peak at 567 cm⁻¹ was assigned to ZnO [28,31]. The Raman spectra of NaCl aerosol droplet acidified by HCl was similar with NaCl aerosol droplet and not shown here.

Fig. 4 depicted the Raman spectra of corrosion products under the NaCl aerosol droplet acidified by H₂SO₄. The result showed the presence of Simonkolleite, Gordait (NaZn₄Cl(OH)₆SO₄·6H₂O), Zn₄Cl₂(OH)₆SO₄·5H₂O, ZnO and Hydrozincite in the center of the aerosol droplet (Fig. 4a). The Raman bands at 256, 267, 393, 3450, 3486 and 3588 cm⁻¹ were assigned to Simonkolleite [28,29,31]. The peaks at 970 cm⁻¹ assigned to SO₂⁻ and 3422 cm⁻¹ assigned to the O–H stretching identified the corrosion product NaZn₄Cl(OH)₆SO₄·6H₂O [31]. The Raman characteristic peaks of Zn₄Cl₂(OH)₆SO₄·5H₂O were also identified at 206, 288 and 1003 cm⁻¹ [31,32]. In addition, the peak at 327 cm⁻¹ was assigned to ZnO and peaks at 347 and 2945 cm⁻¹ were attributed to Hydrozincite [28,33]. The Raman spectrum of corrosion products at the periphery of the aerosol droplet was shown in Fig. 4b and the corrosion products contained ZnO, ZnCO₃ and Zn₅(CO₃)₂(OH)₆. The peaks at 328, 412, 434 and 1072 cm⁻¹ were attributed to ZnO [28,33]. Peaks at 1086 and 1407 cm⁻¹ were assigned to ZnCO₃, while the peaks at 342, 1086 and 2945 were assigned to Hydrozincite [23,33].
3.3. **Morphologies and element composition of corrosion products**

The morphologies and element composition of corrosion products under different aerosol droplets were analyzed by SEM-EDS. Fig. 5 showed the morphologies and element composition of corrosion products in the central and the periphery of NaCl aerosol droplets. Corrosion products in the central of NaCl aerosol droplet was flake-like structure, and the flake-like corrosion products crossed together (Fig. 5a). In addition, the corrosion product was composed of Zn, O and Cl, and was most likely Simonkolleite (Figs. 5b and 3a). The morphology at the periphery of the droplet was layer stack structure (Fig. 5c), and the corrosion product composed of Zn and O was likely ZnO and Zn(OH)₂ (Figs. 5d and 3b). The corrosion morphology and element composition of the NaCl aerosol droplets acidified by HCl were similar with that of NaCl aerosol droplets.

Fig. 6 showed the corrosion morphology and the corresponding element composition of corrosion products in the center of the NaCl aerosol droplet acidified by H₂SO₄. There were two types of morphologies in the center of the droplet (Fig. 6a and 6b) and the compositions were both Zn, O, Cl, S (Fig. 6c and 6d), while there was a great difference on the atomic ratio of different elements. Furthermore, the corrosion products containing more S distributed in the center dispersedly and uniformly (Fig. 6a and 6b). The result of corrosion morphology and elementary composition indicated that there were several different types of corrosion products in the center of the aerosol droplet which was in consistent with the result of Raman spectra and the main corrosion products were Gordaite, Simonkolleite, Zn₄Cl₂(OH)₄SO₄ · 5H₂O. The corrosion morphologies of ZnO and Hydrozincite which were identified by Raman spectra were not observed in the center of the aerosol droplet, meaning that the amount of the two corrosion products was small. In addition, the elements distribution of cross section of corrosion products in the center of aerosol droplet acidified by H₂SO₄ was also studied and shown in Fig. 7. And from the bottom to the top of corrosion products, the content of element S decreased first and then increased. The variation of the content of element S was owed to the different forming sequence of corrosion products (Section 4.2 for detail).

3.4. **Volume loss of Zn under the original aerosol droplets**

Volume loss under the original aerosol droplets was measured through laser scanning confocal microscope after the corrosion products were removed from the surface. The volume loss under aerosol droplets with different diameter was shown in Fig. 8. The result showed that the volume loss increased with diameter of the original aerosol droplets. The volume loss under the NaCl aerosol droplet was larger compared with...
the acidified aerosol droplets. And NaCl aerosol droplets acidified by H2SO4 induced the minimum volume loss. Moreover, the differentiation of volume loss between the three aerosol droplets was relatively small when the size of droplets was small, but then became larger with the increasing of diameter.

4. Discussion

4.1. Effect of acidification on the secondary spreading

Secondary spreading of NaCl droplets was observed on zinc and copper surfaces in recent years [13,21]. However, few works had been done on the process of secondary spreading, especially for the acidified aerosol droplets. In present study, the acidified aerosol droplets showed smaller secondary spreading region compared with NaCl aerosol droplets and Chen et al. obtained the similar result in the environment with different concentration of CO2 and SO2 [14]. In addition, the NaCl aerosol droplets showed a faster spreading rate compared with the acidified aerosol droplets. The change of the secondary spreading effect for the acidified aerosol droplets was supposed to be associated with the pH of the periphery. The secondary spreading effect was attributed to galvanic couple between the central of the aerosol droplets (anode) and the periphery of the aerosol droplets (cathode). For NaCl aerosol
droplets, the distribution of anode and cathode is decided by the differentiation of oxygen concentration, and the periphery of droplets act as cathode because of higher oxygen concentration. And the anodic and cathodic reactions were as follows:

\[
\text{Zn} - 2e \rightarrow \text{Zn}^{2+} \quad (1)
\]

\[
\frac{1}{2} \text{O}_2 + \text{H}_2\text{O} + 2e \rightarrow 2\text{OH}^- \quad (2)
\]

The oxygen reduction reaction taking place in the cathode would generate more \(\text{OH}^-\). The high pH at the periphery results in a negative surface charge which decreases the surface tension at the oxide/electrolyte interface, and further leads to the flow of electrolyte [14,21].

While for the acidified aerosol droplets, anodes and cathodes distributed randomly and homogeneously which avoided the segregation of different ions in the initial stage. After corrosion for a certain time, cathodes would develop at the periphery of the aerosol droplets resulting in alkaline condition. In addition, the high concentration of \(H^+\) in the center would also inhibit the increase of pH at the periphery, thereby inhibiting the decrease of surface tension at the oxide/electrolyte interface. Therefore, the secondary spreading of acidified aerosol droplets started later than NaCl aerosol droplets as shown in Fig. 2. The surface tension at the oxide/electrolyte interface is the highest at the isoelectric point (IEP) which is around pH 8.8 for ZnO and will decrease at higher pH values [14,34]. And the higher pH at the periphery will induce larger secondary spreading effect. However, for the acidified aerosol droplets, more \(H^+\) in the center would migrate to the periphery of the droplet which sustained a lower pH at the cathodic areas compared with NaCl aerosol droplet. The less alkaline environment would lead to smaller changes on the surface tension at the oxide/electrolyte interface which reduced the secondary spreading effect. Therefore, NaCl aerosol showed a faster spreading rate and a larger spreading region compared with the acidified aerosol droplets. As the corrosion time increasing, \(CO_2\) would be absorbed at the periphery and secondary spreading area, which decreased the pH and limited the cathodic reaction due to the formation of zinc hydroxycarbonates. Hence, the spreading rate decreased in the later stage of spreading process (Fig. 2). In addition, the corrosion product Gordaite formed under the aerosol droplet acidified by \(H_2SO_4\) may inhibit the anodic reactions, which resulted in the secondary spreading to stop earlier than the other two aerosol droplets.

### 4.2. Effect of acidification on the corrosion products

The separation of anodes and cathodes under the aerosol droplets would lead to the migration of ions between anodes and cathodes. Hence, \(Cl^-\) and \(SO_4^{2-}\) would migrate to the center of the aerosol droplet and no corrosion products containing element Cl and S were observed at the periphery (Figs. 3 and 4). The alkaline condition at the periphery favored the absorption of \(CO_2\) and the formation of carbonate ions which would react with \(Zn^{2+}\). Therefore, the main corrosion product in the central was Simonkolleite, a typical corrosion product formed in the Cl-containing environments, and the main corrosion products at the periphery were ZnO and Hydrozincite (Figs. 3 and 5) for NaCl aerosol droplets. This result was in accordance with the previous study [13,23]. For the aerosol droplet acidified by \(H_2SO_4\), the main corrosion products in the center were Gordaite, Simonkolleite and Zn$_4$Cl$_2$(OH)$_6$SO$_4$·5H$_2$O according to the result of the SEM and Raman spectra (Figs. 4 and 6). Gordaite is the common corrosion product in the environment containing element S [35-37]. In the condition of relatively lower pH values and existence of \(SO_4^{2-}\), Gordaite is expected to be formed earlier than Simonkolleite as the formation of Gordaite need only small concentration of \(Zn^{2+}\). And Gordaite can exist in the slightly acidic to highly acidic environment, while Simonkolleite tend to form in the slightly acidic to neutral conditions [1,15,23,38]. Besides, Zn$_4$Cl$_2$(OH)$_6$SO$_4$·5H$_2$O is formed by the transformation of Simonkolleite in the solution containing sulfate ion [31], and therefore two types of morphologies were observed in the center. Hence, the content of element S was comparatively higher at the bottom and top of the corrosion products compared with the middle part of the corrosion products layer (Fig. 7). The corrosion products formed as the follow reactions:

\[
\text{Zn}^{2+} + \text{2OH}^- \rightarrow \text{Zn(OH)}_2 \rightarrow \text{ZnO} + \text{H}_2\text{O} \quad (3)
\]

\[
3\text{ZnO} + \text{Zn}^{2+} + \text{SO}_4^{2-} + \text{Na}^+ + \text{Cl}^- + 9\text{H}_2\text{O} \rightarrow \text{NaZn}_4\text{Cl(OH)}_6 \cdot 6\text{H}_2\text{O} \quad (4)
\]

\[
4\text{ZnO} + \text{Zn}^{2+} + 2\text{Cl}^- + 5\text{H}_2\text{O} \rightarrow \text{Zn}_5\text{(OH)}_6\text{Cl}_2 \cdot \text{H}_2\text{O} \quad (5)
\]

\[
\text{Zn}_5\text{(OH)}_6\text{Cl}_2 \cdot \text{H}_2\text{O} + 2\text{SO}_4^{2-} + 3\text{Zn}^{2+} + 2\text{Cl}^- + 4\text{H}_2\text{O} \rightarrow 2\text{Zn}_4\text{Cl}_2(\text{OH})_4\text{SO}_4 \cdot \text{H}_2\text{O} \quad (6)
\]

### 4.3. Effect of acidification on the corrosion rate

In the present study, NaCl aerosol droplet showed a higher corrosion rate than the NaCl aerosol droplet acidified by HCl, which was not in accordance with the previous studies [17,23]. In addition, the difference of corrosion rate between the aerosol droplets became larger with the increase of diame-
The difference of the size of secondary spreading region accounted for the difference of corrosion rate. The size of the secondary spreading region is proportional to the diameter of the original aerosol droplets [13]. The NaCl aerosol droplet showed larger spreading region compared with the aerosol droplet acidified by HCl (Fig. 2) and larger secondary spreading region means larger effective cathode area which would increase the corrosion rate under the original droplet. When the size of the original aerosol droplet was small, the difference of the spreading region was relatively small, and the increase of corrosion rate caused by the difference of spreading region was comparable to that caused by acidification. With the increase of diameter of the original aerosol droplet, the increase of corrosion rate caused by the difference of spreading region was much larger than that caused by pH. Hence, the difference of corrosion rate became larger with the increase of diameter.

The aerosol droplets acidified by H₂SO₄ showed a lower corrosion rate compared with the other two aerosol droplets, which was thought to be associated with the corrosion products in the center of the aerosol droplet. On one hand, Gordaite is proposed to be an effective barrier which could reduce the corrosion rate [38,39]. On the other hand, the aerosol droplets acidified by H₂SO₄ showed a smaller secondary spreading area compared to the aerosol droplets acidified by HCl because of the formation of Gordaite. Hence, the aerosol droplets acidified by H₂SO₄ showed the lowest corrosion rate.

5. Conclusion

The spreading process, corrosion products and volume loss of acidified and unacidified NaCl aerosol droplets on the surface of Zinc were studied. The results indicated the acidification had a significant effect on the secondary spreading and the following main conclusions can be drawn.

The secondary spreading of the acidified aerosol initiated later compared with NaCl aerosol droplet. Furthermore, acidified aerosol droplets showed a lower spreading rate and a smaller spreading area. The aerosol droplet acidified by H₂SO₄ showed shorter spreading time which was due to the Gordaite formed in the center inhibiting the anodic reactions.

The main corrosion product was Simonkolleite for NaCl aerosol droplet and droplet acidified by HCl, while for droplet acidified by H₂SO₄, Gordaite, Simonkolleite and Zn₂Cl₂(OH)₄SO₄ · 5H₂O were the main corrosion products. Two types of corrosion morphologies were observed in the center of the droplet acidified by H₂SO₄ because of the different formation sequence of corrosion products.

The acidified aerosol droplets showed a lower corrosion rate because of the smaller secondary spreading area compared with NaCl aerosol droplet. And the difference of the secondary spreading area became lager with the increase of diameter, thus leading to larger difference of the corrosion rate.

Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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