Electrochemical Performance of SiC Composite Anode in Aluminum-Air Battery

Mingcheng BING, Fan MO,* and Zhengfei HU*

Shanghai Key Lab for R&D and Application of Metallic Functional Materials, School of Materials Science and Engineering, Tongji University, Shanghai 201804, People’s Republic of China

* Corresponding authors: mofan61@163.com, huzhengf@tongji.edu.cn

ABSTRACT

The commercial pure aluminum (1A95) and its micro-alloyed with Mg and further particle composited with SiC were prepared using the vortex casting method, their electrochemical performances as the anodes in Aluminum-air batteries were investigated using open-circuit potential (OCP), potentiodynamic polarization, electrochemical impedance spectroscopy (EIS) and galvanostatic discharge. The results indicate that Al-Mg/SiC composite anode shows much better corrosion resistance and discharge performance than others. Impedance spectra and surface observation show that SiC addition obviously changed the anode dissolution morphology, which means that the SiC particles serve as attachment points for hydroxide products and impurities and reduce the parasitic corrosion reactions. As SiC particles fall off the substrate, SiC shedding disrupts the Al(OH)3 film, reducing the accumulation of hydrogen molecules on the pore surface, the more fresh surface is exposed, which promoted the corrosive effect of the anode. So, the Al-Mg/SiC composite anode has higher electrochemical activity and the open-circuit potential shifts more negatively. Even though the shedding of SiC particles might cause a relatively higher rate of dissolution during the spontaneous oxide growth/oxide dissolution process, the formation of MgAl2O4 cladding SiC particles reduces the Al/SiC interface energy, lowers corrosion rate and improves the composite anode efficiency and capacity density.

1. Introduction

Aluminum has immense potential as an anode of metal-air batteries for its high energy density (8100 Wh kg⁻¹), and negative standard potential (−2.37 V vs. SHE).1–3 However, due to its high activity, aluminum is rapidly oxidized to form a dense passivation film (Al₂O₃), which hinders the anodization of aluminum and increases polarization. At the same time, self-corrosion of Al anodes in alkaline electrolytes will release hydrogen and reduce the utilization ratio, this will increase the cost and the difficulty of designing the battery. Many efforts have been made, such as using high-grade aluminum doped with Ga, Sn, Mg, In, Zn, and Mn, to improve the electrochemical parameters of Al-air batteries, including improving OCP and reducing parasitic corrosion reactions at the anode. Researchers have developed a variety of solutions.4–11 The route of controlling the behavior of the aluminum anode by the addition of alloying agents is known as “activation”.12 The mechanism of the aluminum activation is related to (a) moderating the thickness of the oxide film; (b) reducing the rate of the direct reduction of water by aluminum and (c) controlling the dissolution morphology.

SiCp/Al metal matrix composites (MMCs) represent attractive materials for its significant corrosion resistance.13 Some researchers suggested possible interfacial reactions between the matrix components and the SiC particles during the fabrication process and the heat treatment has an important influence on the corrosion behavior of SiCp/Al MMCs.14–16

Mg is highly effective in wettability improvement of ceramic particles by molten aluminum compared to alloy compositions without Mg.17 Adding alloying element Mg in aluminum anode material can improve the uniformity of anode material corrosion. When the content of impurity Si reaches a certain range in aluminum alloy, Mg can be combined with Si to form Mg₂Si phase, which decreases the galvanic corrosion. The potential value (Ecorr = −1.079 V) of the Mg₂Si phase is very close to that of the α-Al matrix (Ecorr = −1.046 V), which reduces the adverse effects of impurity elements.18 Mg can also improve the corrosion resistance of the alloy and can transfer the potential of the alloy.

So far, we know that SiCp/Al metal matrix composites and Mg can improve the corrosion performance of Al material. However, it is still uncertain whether the two materials used as anode materials will also help to improve the electrochemical performance of aluminum. This article discusses the electrochemical performance of SiCp/Al materials and compares common methods to improve anode performance by adding elements to investigate whether SiCp/Al materials can be used as anode materials.

2. Experimental

2.1 Material preparation

The commercial as-cast pure aluminum (1A95) ingot, magnesium ingot (99.99%), SiC particles with an average size of 10 µm were used as raw materials for melt and cast anodes. The working composites of the anodes were pure Al, Al-1Mg (wt%) (the sample
The solution was 4 mol L\(^{-1}\) KOH. The open-circuit potential (EOCP) was measured by heating up to 750 ± 5°C. The molten alloys were poured into a preheated cast iron die. The as-cast Al as anode was mostly linear with time. It is distinctly that the as-cast Al as anode after discharge was recorded for anode utilization ratio calculation. The morphology of the sample before and after discharge was characterized by scanning electron microscope (SEM) of DEXM and energy dispersive spectrometer (EDS).

### 3. Results and Discussion

#### 3.1 H\(_2\) evolution rate

During the discharge process, the dissolution of aluminum in the electrolyte occurs on the anode side while the oxygen is electrochemically reduced at the cathode side under the action of the catalyst. However, active material also suffers from self-discharge induced by corrosion proceed in open circuit conditions. The self-corrosion of the aluminum alloy anode is the main reason for the low anode utilization efficiency in the aluminum-air battery.

The hydrogen evolution rates and the rates of the samples are shown in Fig. 3 and Table 1, respectively. The hydrogen evolution is mostly linear with time. It is distinctly that the as-cast Al as anode has the highest self-corrosion rate compared with Al-Mg alloy and Al-Mg/3SiC composite, resulting in less utilization of the metal. Mg with a more negative electrode potential than aluminum matrix can form a galvanic couple and aluminum is protected as a cathode.

### Supplemental Information

**2.4 Battery performance test**

In this study, the anode materials were prepared pure Al, Al-Mg alloy, and Al-Mg/3SiC composite. The cathode was a traditional three-layer structure, which was formed by pressing the gas diffusion layer, foam nickel mesh, and catalytic active layer. The working area of the cathode was 25 cm\(^2\) and the electrolyte was 4 mol L\(^{-1}\) KOH solution. The schematic diagram of the aluminum-air battery prepared for the test was shown in Fig. 2. Constant current discharge test was used to judge the discharge performance of the different anodes. The current density controlled at 10 mA cm\(^{-2}\) for a duration of 5 h. The mass loss of the aluminum anode after discharge was recorded for anode utilization ratio calculation. The morphologies of the samples before and after discharge were characterized by scanning electron microscope (SEM) of DEXM and energy dispersive spectrometer (EDS).

The anode utilization rate was calculated by the following formula:

\[
\eta = \frac{100I/2F(\Delta m)}{t}\tag{2}
\]

Where \(\eta\) is the anode utilization, %; \(I\) is the current, mA; \(t\) is the time, s; \(\Delta m\) is the actual weight loss, g; \(F\) is the Faraday constant.

Each experiment was repeated at least three times to ensure reproducibility.

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**2.3 Electrochemical measurement**

The electrochemical tests were carried out with a three electrodes system by the Gamry Reference 600+ electrochemical workstation. The solution was 4 mol L\(^{-1}\) KOH. The open-circuit potential (EOCP) of working electrodes was measured with an exposed area of 1 cm\(^2\). The samples were immersed in 4 M KOH solution for 1 h to reach a stable potential before the electrochemical impedance spectroscopy (EIS) test. The frequency of the EIS measurements ranged from 100 kHz to 0.01 Hz and the amplitude was 5 mV. The potentiodynamic polarization test was measured at 1 mV s\(^{-1}\) scanning rate.

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**Figure 1.** Schematic of the experimental setups for hydrogen gas collection.

**Figure 2.** Schematic diagram of the house-made Al-air battery.
The corrosion rate of Al-Mg alloy in 4 M KOH solution is $0.43 \pm 0.013$ ml min$^{-1}$ cm$^{-2}$, and Al-Mg/SiC composite is $0.38 \pm 0.003$ ml min$^{-1}$ cm$^{-2}$, they are obviously lower than that for pure Al ($0.51 \pm 0.004$ ml min$^{-1}$ cm$^{-2}$). Under the pre-oxidation condition, it is believed that the MgAl$_2$O$_4$ produced at the Al/SiC interface contributes to improving the corrosion resistance. It is to be expected that the existence of SiC will decrease the self-corrosion of aluminum-air batteries.

3.2 Battery performance

Figure 4 shows the open-circuit potential and galvanostatic discharge curves of the various anodes and Table 2 summarizes the stabilization voltage and efficiency of the various materials at 10 mA cm$^{-2}$ current density in 4 M KOH solution. The discharge voltage shows a rapid drop at the initial stage, which may be caused by the internal resistance of the battery. Then the potential tends to be stable over a period as the electrochemical equilibrium is reached. A comparison of the discharge curves of the investigated materials in Fig. 4(b) shows that the discharge voltage and the anode efficiency of Al-Mg alloy were higher than pure Al. This indicates that the addition of Mg element can improve the discharge voltage in 4 M KOH solution. The discharge voltage of the Al-Mg/SiC composite shows the highest value, about 1.38 V. In contrast, the as-cast Al shows a lower discharge voltage after 5 h under the discharge experiments. The addition of SiC increases the discharge voltage.

The discharge data shown in Table 2 indicate that the Al-Mg alloy shows much lower weight loss and higher anodic utilization. Combined with the previous hydrogen evolution test, the self-corrosion of the anode is effectively suppressed. The commercial pure aluminum possesses a lower anodic efficiency, it is still unsuitable as the anode of Al-air battery due to its high self-corrosion rate and low anodic utilization. The Al-air batteries based on Al-Mg alloy and Al-Mg/SiC composite anodes show better discharge performance. Though SiC detached from the aluminum matrix, causing part of quality loss, the addition of SiC into aluminum matrix can activate the aluminum anode. SiC takes off the Al(OH)$_3$ layer on the surface of the aluminum matrix while falling off and increases the electrolyte flow rate. Increasing the electrolyte flow rate may lead to a fast supply of OH$^-$ to the interface of anode/electrolyte and rapid removal of Al(OH)$_4$ away from the aluminum anode surface, allowing more Al(OH)$_3$ layers to be dissolved into the electrolyte. Compared the results of the hydrogen evolution test with discharge performance, Al-Mg/SiC composite showed the highest performance.

3.3 Corrosion morphology

Figure 5 shows the corrosion morphologies of the 3 samples after 5 h discharge in 4 M KOH solution. It can be seen that the surface morphologies of the examined samples are different which should be associated with their discharge performance. Much larger and deeper corrosion pores and pits were formed in pure aluminum (Fig. 5(a1)) while Al-Mg alloy showed a uniformly corroded surface (Fig. 5(b1)). The deep corrosion holes observed in the pure aluminum samples should be related to the existence of impurities or inclusions identified as Fe and Si latter. The aluminum oxide film will be broken in the alkaline solution and impurities and inclusion act as hydrogen evolution centers which will not detach with the dissolution of aluminum matrix. The concentration of OH$^-$ ions to the aluminum surface and the amount of Al(OH)$_3$ in the electrolyte will influence the aluminum dissolution rate. However, along with
the dissolution of aluminum, the formation of Al(OH)$_3$ films prevents the transfer of OH$^-$ ions, H$_2$ accumulates in the corrosion sites and causes the change of the anode current density, exacerbating the galvanic coupling reaction with impurity elements, resulting in the enlargement of the pore size. Mg can be combined with Si to form the Mg$_2$Si phase, which decreases the galvanic corrosion. The Al(OH)$_3$ films form soluble aluminate ions in the electrolyte and regenerate a bare aluminum surface site. As SiC falls off the Al substrate, reducing the accumulation of hydrogen molecules on the pore surface, the more exposed surface appears. The reaction (3) and (4) will occur simultaneously. This caused the appearance of Fig. 5(c1), which displays a rough surface with many bare surface sites.

The formation of aluminum hydroxide layer:

$$\text{Al} + 3\text{OH}^- \rightarrow \text{Al(OH)}_3 + 3\text{e}^- \quad (3)$$

$$\text{Al(OH)}_3 + \text{OH}^- \rightarrow \text{Al(OH)}_4^- \quad (4)$$

The SEM micrographs and EDS line analysis of Al-Mg/SiC composite are presented in Fig. 6. Figure 6a shows that the distribution of SiC particles in the matrix is macroscopically uniform, even though agglomeration can be seen in some areas and few pores are formed close to SiC particles. This is a common phenomenon caused by the stirring casting method. And the reaction layer can be seen around the SiC particles (Fig. 6b). According to the relevant literature, during the preparation of composite materials, Mg will remove oxygen on the surface of the dispersed phase and increase its surface energy, which helps to improve wettability. As a surfactant, Mg will reduce the solid-liquid interface energy and help interface reactions to generate new phases. Figure 6c is the corresponding EDS line analysis at the Al-Mg/SiC composite interface. The Mg and O are present at the interface, which shows oxide is formed. Past research presents that when Mg is added in the SiC/Al composite, Al$_2$O$_3$, MgAl$_2$O$_4$ or MgO will be generated at the Al/SiC interface, and the main phase is MgAl$_2$O$_4$ when the content is in the range of 0.098%~4%. The Mg content is about 1% in the examined sample, which is in the range, so the oxide mostly is MgAl$_2$O$_4$ at the SiC/Al interface. Some bright-colored phase (θ) also appeared around the SiC particles, which is confirmed as Fe-rich phase (Fig. 6d). The Fe-rich phase is a normal inclusion in the matrix material. The distribution of the Fe element has an influence on the overall performance of the aluminum alloy. According to thermodynamics and research works, silica can react with the aluminum alloy (in the presence of magnesium) to form spinel and magnesium oxide. The XRD pattern shows in Fig. 7 Al, SiC, MgAl$_2$O$_4$, and Mg$_2$Si phases might present in the sample. As expected, the SiO$_2$ helped to protect the SiC particles and avoid the formation of the unwanted Al$_4$C$_3$.

$$2\text{SiO}_2 + \text{Mg} + 2\text{Al} \rightarrow \text{MgAl}_2\text{O}_4 + 2\text{Si} \quad (5)$$

The reaction (5) at the interface consumes magnesium in the matrix while the Si element released. However, only the content of the Si element reaches a certain amount there will be more solid
Mg₂Si formation which is the key phase to alloy strengthening, such as Al-Si casting alloys (A356 aluminum alloy) and 6XXX series aluminum alloys. With the formation of MgAl₂O₄, the volume of this dispersive phase would contract within the metallic Al matrix about 27%, and this contraction might lead to some gaps appeared. These gaps are used as the “diffusion channels” of metallic Al, Mg, and the generated Si of reaction (5), so discontinuous MgAl₂O₄ phases will appear near SiC particles. When the material solidifies and crystallizes, the remaining Mg in the matrix and the generated Si will form Mg₂Si phases, as shown in Fig. 6b. Mg₂Si is also observed at the interface near SiC particles. However, the content of Mg₂Si depends on the effective content of Mg in the matrix, so tiny amount of Mg₂Si is formed and the main interface phase is MgAl₂O₄.

The presence of MgAl₂O₄ is beneficial to the combination of SiC and Al matrix, meanwhile, both MgAl₂O₄ and Al are face-centered cubic structures, and the lattice mismatch with Al is very small, which reduced the interface energy of MgAl₂O₄/Al, this helps to lower the corrosion rate of Al-Mg/SiC composite.

Figure 6. Microstructures of a composite containing Mg, low (a) and high (b) magnification SEM images; (c) EDS line analysis of Al-Mg/SiC composite; (d) EDS spectrum obtained on the θ phases.

Figure 7. XRD patterns obtained in the Al-Mg/SiC composite samples.
3.4 Electrochemical analysis

Figure 8 illustrates the EIS plots of the different materials in the 4 M KOH solution. The Nyquist plot of pure Al was fitted using the equivalent circuit in Fig. 9(a), respectively, while both Nyquist plots of Al-Mg alloy and Al-Mg/SiC composite anodes were fitted using the equivalent circuit in Fig. 9(b). For the elements in the equivalent circuits, R1 represents the solution resistance, and R2 represents the charge transfer resistance. The Nyquist diagram of the pure Al consisted of a high-frequency capacitive loop might be related to the dissolution of Al and a low-frequency capacitive loops related to the film growth. The EIS plots of Al-Mg alloy and Al-Mg/SiC composite include a high-frequency loop and a low-frequency loop as well as two inductive loops at high and middle frequencies. The high-frequencies inductive loop (L1) appears in the imaginary region, which is related to the potential distributions on the electrode surface. The middle-frequency inductive loop (L2) may be related to the hydrogen evolution reaction. The high-frequency capacitive loop is attributed to the charge transfer resistance Rt (R2) in parallel with the double-layer capacitive (CPE1), and a larger R2 represents a lower corrosion rate. The second one is the low-frequency loop that might be related to the hydroxide dissolution on the anode surface. In this case, CPE2 and R4 depict corresponding parameters for the dissolution.

Figure 9 shows the equivalent circuits of the investigated samples, the fitting lines of the equivalent circuit are shown in Fig. 8, which exhibit good coincidence with the Nyquist plots. Table 3 shows the fitted results. The X^2 is the precision of the simulated data and the X^2 in Table 3 are all small which means the fitting data are coincidence with the experiment results. In general, the higher R1 (R2) reflects lower corrosion since the exchange current is directly associated with the electrochemical process of corrosion. The R2 values listed in Table 3 increased in the following order: pure Al < Al-Mg/SiC composite < Al-Mg alloy; the corrosion rates of 3 anodes are in the opposite order: Al-Mg alloy < Al-Mg/SiC composite < pure Al. One could say that the surface of Al-Mg alloy is more inhibited than the others, leads to high anodic utilization during galvanostatic discharge. However, although the addition of a small amount of SiC reduces the anode utilization rate due to SiC shedding, Al-Mg/SiC composite has a higher discharge voltage compared with Al-Mg alloy and pure Al.

3.5 Potentiodynamic polarization

Figure 10 and Table 4 present the potentiodynamic polarization curves and the corresponding corrosion parameters of the samples. As the reproducibility of the tests being particularly good, only one curve was drawn under each condition. The values of corrosion potentials of Al-Mg alloy and Al-Mg/SiC composite were negative than pure Al. Combined with the OCP test of pure Al, it means that the addition of Mg, SiC can promote the electrochemical activity of anodes in 4 M KOH solution. The Al-Mg/SiC composite has the most negative corrosion potential means its activation performance
Table 4. Corrosion parameters of the different anode materials in 4 M KOH solution.

| Materials          | $E_{corr}$ (V) | $I_{corr}$ (mA cm$^{-2}$) | $R_p$ (Ω cm$^2$) |
|-------------------|----------------|---------------------------|-----------------|
| Pure Al           | -1.347         | 31.1                      | 1.45            |
| Al-Mg alloy       | -1.401         | 26.1                      | 1.82            |
| Al-Mg/SiC composite| -1.426         | 26.8                      | 1.69            |

is better than the other anode materials. The corrosion current density ($I_{corr}$) of Al-Mg alloy and Al-Mg/SiC composite anodes were lower than pure Al. The resistivity of the reaction interface will influence the reaction rate. The decrease of anodic polarization current density means the anodic dissolution of the aluminum anode in the electrolyte is suppressed. Those results are consistent with H$_2$ evolution rate tests and discharge experiments before.

The relatively negative corrosion potential in 4 M KOH is mixed with the cathodic process of water reduction based on the reaction (6), which acts as a subsidiary cathode and reduces the efficiency of the anode, the reduction of water and the growth of aluminum hydroxide layer according to the reaction (3) and (4).

$2\text{H}_2\text{O} + 2\text{e}^- \rightarrow \text{H}_2 + 2\text{OH}^-$ (6)

$\text{Al(OH)}_3 \leftrightarrow \text{Al(OH)}_2^+ + \text{OH}^-$ (7)

The presence of surface films and the accumulation of H$_2$ in the pores decreases the dissolution of aluminum. The formation and transfer of Al(OH)$_2^+$ from anode surface to the electrolyte is a slow step limited by the reaction (7). It has been confirmed that with increasing OH$^-$ ions concentration to the aluminum surface, the Al(OH)$_3$ films become thinner. In our paper, the addition of SiC destroys the initial film, and increases the bare surface, SiC takes off the Al(OH)$_3$ layer on the surface of the aluminum substrate while falling off and increasing the electrolyte flow rate. Increasing the electrolyte flow rate may lead to a fast supply of OH$^-$ to the interface of anode/electrolyte.

4. Conclusions

Three aluminum anodes of commercial pure aluminum (1A95), its micro-alloyed with Mg, and further particle composited with SiC have been investigated comparably to evaluate the effects of the addition of Mg and SiC on the aluminum-air battery performance. The results of impedance spectra and surface observation show that SiC provided attachment points for hydride products and changed the dissolution morphology. The battery performance of Al-Mg/SiC composite anode is higher and better than pure-Al and Al-Mg alloy anodes for its higher OCP voltage, high-anode efficiency, and high capacity density. Al-Mg/SiC composite anode also shows better corrosion resistance and lower self-corrosion. A different "activation" way was used to change the performance of the aluminum-air battery by adding SiC. It is inferred that the micro-alloyed and particle composited the commercial pure aluminum can improve their performance in Aluminum-air battery.

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