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Chapter V

5. Reinforcement of anodes by metal oxides

5.1. Introduction

The major drawback of aluminium alloy sacrificial anode is the non-coulombic corrosion loss and low galvanic efficiency. Incorporation of metal oxides may refine the grains and their boundaries suppressing the grain boundary corrosion of the anodes that results in improvement of the anode performance [1-3]. Metals of Mn, La and V are widely used as micro composites in sacrificial anodes for improving the galvanic performance [4-12]. Similarly oxides of Al and Mn are also used for production of high wear resistant sacrificial anodes [1,2]. A 5% of Al₂O₃ in aluminium anode can result in metallurgical improvement of the alloy [1]. Mn and V have been used for inhibiting crevice corrosion of the alloy [9,13]. In the development of aluminium alloy composite anodes, the infiltration of the reinforcement network by molten aluminium and the homogeneity of the matrix are partially governed by wetting nature of the reinforcement by aluminium and the chemical interaction between the reinforcement and the aluminium matrix [14-18]. One of the composites selected and used for development of aluminium alloy anode in the present work is Al₂O₃. Even though Al₂O₃ is the widely applied composite material in aluminium alloy anodes, many of the reports available in the literature indicates that Al₂O₃ is not wetted by molten aluminium below 1000 °C and it is considered to be inert in molten aluminium [14,19]. In contrast, the wettability of Al₂O₃ in molten aluminium and the physical interaction between Al₂O₃ and molten aluminium have also been rarely reported [20].
The chemical interaction between lanthanum oxide with aluminium in molten stage will lead to the formation of lanthanum aluminates [21], which make the anode passive. La$_2$O$_3$ has also been selected for the present work with the prediction that the existence of reinforcement La$_2$O$_3$ after partial conversion with aluminium layer may lead to substantial reduction in self-corrosion value. In addition to these two inert metal oxides namely Al$_2$O$_3$ and La$_2$O$_3$, two other catalytic oxides namely MnO$_2$ and V$_2$O$_5$ have also been selected and studied during the present work. These oxidation catalysts have been selected for the present work with the criteria of their wide use in electrochemical reactions and rare citation of its usage in sacrificial anodes. Presence of these oxides in the reinforced state will prevent passivation of aluminium and induced aluminium activation. MnO$_2$ incorporated anodes showed poor galvanic performance with a galvanic efficiency of around 70%. The V$_2$O$_5$ incorporated anodes performed well and showed high galvanic efficiency up to 83%. All these results are discussed in the following sections in detail.

5.2 **Reinforcement by inert metal oxides**

5.2.1 **Physical characteristics**

Different batches of anodes containing varying concentrations of inert metal oxides like Al$_2$O$_3$ and La$_2$O$_3$ were prepared and preliminary tested by SEM to ensure uniform morphology and composition of the reinforced anodes. Scanning electron micrograph of the Al$_2$O$_3$ and La$_2$O$_3$ incorporated Al + 5% Zn alloy anodes are presented in figure 5.1.a and 5.1.b. The SEM photographs reveal the morphology of the cross-sectional area of the respective electrodes after significant polishing by a series of emery
paper. The morphology of pure Al + 5% Zn alloy anode has been already shown in figure 4.2. Inclusion of Al₂O₃ caused minor precipitation or segregation of the Al₂O₃ in the grain boundaries. It has also caused substantial metallurgical defects in the Al + Zn alloy matrix. It needs tailoring of the casting parameters to get highly refined matrix without any voids and excess growth. Since this is not the role parameter that governs the galvanic performance, the study was continued ahead. Inclusion of Al₂O₃ up to a concentration of 5% in pure aluminium substrate has been studied by different investigators also[ 8 ]. It has also been established that Al₂O₃ segregation does not occur up to the concentration of 5%. However, no literature is available on such type of incorporation of Al₂O₃ in Al-Zn alloy substrate for metallurgical improvement of the alloy. The concentration of Al₂O₃ in the present study was limited up to the level of 0.5%, since higher amount of the inert metal oxides in the sacrificial anode would decrease the energy density of the anode mass. The SEM photographs of 0.5% La₂O₃ incorporated anodes show much improved grains and their uniform distribution (figure 5.1.b). A trace amount of precipitate of La₂O₃ is seen with higher magnification (2000 times) of SEM photographs. No other significant change is found with magnification of 4000 times. Thus La₂O₃ in contrast to Al₂O₃, substantially improves the metallurgical properties of the reinforced alloy. Since achievement of high galvanic performance is the primary objective of the present study, no in-depth understanding of the metallurgical characteristics of the anode system was attended.
Figure: 5.1.a. SEM photographs of the $\text{Al}_2\text{O}_3$ incorporated Al + 5% Zn alloy anode at three different magnifications (i, x 500, ii, x 2000, iii, x 4000).
Figure: 5.1.b. SEM photographs of the $\text{La}_2\text{O}_3$ incorporated Al + 5% Zn alloy anode at three different magnifications (i, x 500, ii, x 2000, iii, x 4000).
The Vickers hardness numbers of the Al + 5\% Zn alloy anode reinforced with 0.5\% Al_2O_3 and LaO_2 were measured to be 39.9 and 38.8 respectively. The hardness of the bare substrate was 39.92. As inclusion of inert metal oxides up to a percentage of 0.5 does not significantly influence the hardness of the alloy, the metallurgical parameters like Young’s modulus, rigidity modulus, compressibility test etc were also not attempted.

5.2.2. Performance evaluation

Six different batches of electrodes containing different concentration of either La_2O_3 or Al_2O_3 were prepared and their galvanic performance were evaluated by different technique like OCP decay measurement, CCP variation measurement, determination of self corrosion and galvanic and galvanostatic polarization. The OCP decay curves of all these anodes are given in figure.5.2. Additions of La_2O_3 at a concentration of 0.1\% substantially decrease the electrode potential more than 50mV. Incorporation of higher concentration of La_2O_3 above 0.1\% causes higher polarization, the least performance with 0.5\% La_2O_3 which is almost equivalent to that of pure substrates. Hence high initial OCP as a result of addition of La_2O_3 cannot be considered favourable. The decrease in the performance with increase in La_2O_3 can be attributed to the formation of trace amount of precipitate of La_2O_3 in the interior mass of the anode as already evidenced with SEM photographs. Such precipitation of La_2O_3 may be negligible or even may not happen if the La_2O_3 concentration is very low. This precipitate may be of the form of lanthanum aluminates, the trace amount of which is very difficult to characterize with conventional analytical tools. Similar crystal structure of lanthanum aluminates has also been investigated by several investigators with lanthanum
incorporated pure aluminium matrix. Lanthanum aluminate can be predicted to cause passivation and low self corrosion of Al-Zn alloy with low activation of aluminiun.

In contrast to La$_2$O$_3$ incorporated aluminium alloy anode, the Al$_2$O$_3$ incorporated anodes show more nobler initial OCP and lesser polarization, all together seemed to be inferior to that of La$_2$O$_3$ incorporated anodes. All the OCP values of Al$_2$O$_3$ incorporated anodes lie in the range of -0.950 to -0.960 V initially and -0.930 to -0.940 V after three months. No significant observation is made from the figure as well as the OCP values for a period of three months, which lies in the range of just few tense of mV higher than that of the bare anode. In this context monitoring of CCP variations when the anodes are galvanically coupled with steel cathodes drew much attention in terms of interpretation of galvanic performance of the anodes.

Figure 5.3 shows CCP variation curves of Al + 5% Zn alloy anodes incorporated with La$_2$O$_3$ or Al$_2$O$_3$. While La$_2$O$_3$ incorporated anodes show more cathodic initial CCP around -0.950V, the anode incorporated with 0.5% Al$_2$O$_3$ show the least polarization and attain a CCP of -0.870 V after a period of three months. The difference between CCP’s of any two anodes is seemed to be less than 60 mV after any period of immersion. However, a little different trend is noticed from the galvanostatic polarization curves of these anodes (figure 5.4). While the bare anode shows highest nobler potential than any other electrode within the entire range of polarization, other polarization curves are seemed to be in two different groups based on either incorporated with Al$_2$O$_3$ or La$_2$O$_3$. Though La$_2$O$_3$ incorporated anode show higher cathodic initial CCP, they steeply-
Figure: 5.2. OCP decay curves of Al + 5 % Zn alloy sacrificial anodes reinforced with different concentrations of La$_2$O$_3$ and Al$_2$O$_3$

( X - Al + 5% Zn \\
Δ - Al + 5% Zn + 0.25% La$_2$O$_3$, \\
● - Al + 5% Zn + 0.1% Al$_2$O$_3$, \\
■ - Al + 5% Zn + 0.5% Al$_2$O$_3$ )

( Electrolyte : 3% NaCl solution, Temperature : 30 °C, stagnant condition).
Figure: 5.3. CCP decay curves of Al + 5 % Zn alloy sacrificial anodes reinforced with different concentrations of La$_2$O$_3$ and Al$_2$O$_3$

( x-Al + 5% Zn  
Δ-Al + 5% Zn + 0.25% La$_2$O$_3$,  
●- Al + 5% Zn + 0.1% Al$_2$O$_3$,  
■- Al + 5% Zn + 0.5% Al$_2$O$_3$ )

( Electrolyte : 3% NaCl solution, Cathode : mild steel, Temperature : 30 °C, stagnant condition).
Figure: 5.4. Anodic polarization curves of Al + 5% Zn alloy sacrificial anodes reinforced with different concentrations of La$_2$O$_3$ and Al$_2$O$_3$

( X - Al + 5% Zn
Δ- Al + 5% Zn + 0.25% La$_2$O$_3$,
○- Al + 5% Zn + 0.1% Al$_2$O$_3$,
■- Al + 5% Zn + 0.5% Al$_2$O$_3$)

(Electrolyte: 3% NaCl solution, Cathode: Platinum foil, Temperature: 30 °C, stagnant condition).
polarizes when the current density exceeds 10mA/cm² in contrast to Al₂O₃ incorporated anodes which show steady polarization even beyond the current density exceeds 10mA/cm². Based on these observations, it is clear that La₂O₃ incorporation improves the morphology and thereby the galvanic performance of the anodes to a little extent when compared with that of Al₂O₃ incorporation. However, at higher current density passivation characteristics of La₂O₃ and lanthanum aluminate dominates and causes higher polarization. These also cause high polarization with prolonged time as seen from figure 5.3, which shows steady polarization of CCP of the La₂O₃ incorporated anodes with time. Even though the La₂O₃ incorporated electrodes show around 40mV higher CCP compared with other anodes, these anodes cannot be considered for practical applications due to their passivation characteristics.

The overall galvanic performance of both La₂O₃ and Al₂O₃ incorporated aluminium alloy anodes are presented in table 5.1. Though La₂O₃ incorporated anodes show better galvanic performance like high OCP, CCP and efficiency with low self-corrosion, the anodes cannot be considered for practical applications based on their short-term tests as evident from the table. As illustrated in figure 5.3, these anodes steeply polarize at high current density or with prolonged time. High galvanic efficiency of La₂O₃ incorporated anodes can be attributed to low self-corrosion caused by La₂O₃ and lanthanum aluminate, which inhibit aluminium corrosion. There is no significant observations with Al₂O₃ incorporated Al-Zn alloy anodes as there galvanic performance are almost equivalent to that of the bare anode. In this context reinforcement by catalytic metal oxides receives much attention.
Table: 5.1. Galvanic performance of La\textsubscript{2}O\textsubscript{3} and Al\textsubscript{2}O\textsubscript{3} reinforced Al + 5\% Zn alloy sacrificial anode (Electrolyte: 3\% NaCl solution, Cathode: mild steel, Temperature: 30 °C, stagnant condition).

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Conc. of La\textsubscript{2}O\textsubscript{3}</th>
<th>Conc. of Al\textsubscript{2}O\textsubscript{3}</th>
<th>OCP V/SCE</th>
<th>CCP V/SCE at (i=)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>%</td>
<td>%</td>
<td>mA/cm\textsuperscript{2}</td>
<td>0.01</td>
</tr>
<tr>
<td>1</td>
<td>---</td>
<td>---</td>
<td>-0.930</td>
<td>-0.925</td>
</tr>
<tr>
<td>2</td>
<td>0.1</td>
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<td>-0.985</td>
<td>-0.981</td>
</tr>
<tr>
<td>3</td>
<td>0.25</td>
<td>---</td>
<td>-0.983</td>
<td>-0.980</td>
</tr>
<tr>
<td>4</td>
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<td>---</td>
<td>-0.967</td>
<td>-0.965</td>
</tr>
<tr>
<td>5</td>
<td>---</td>
<td>0.1</td>
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<td>-0.945</td>
</tr>
<tr>
<td>6</td>
<td>---</td>
<td>0.25</td>
<td>-0.955</td>
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<tr>
<td>7</td>
<td>---</td>
<td>0.5</td>
<td>-0.960</td>
<td>-0.955</td>
</tr>
</tbody>
</table>

5.3. Reinforcement by catalytic metal oxides

5.3.1 Physical characteristics

The bare Al + 5\% Zn alloy anodes containing varying concentrations of catalytic oxides namely MnO\textsubscript{2} and V\textsubscript{2}O\textsubscript{5} were first tested for improvement in surface morphology. The SEM photographs of cross sectional view of 0.5\% MnO\textsubscript{2} and 0.5\% V\textsubscript{2}O\textsubscript{5} incorporated anodes are shown in figure 5.5.a and 5.5.b respectively at three different magnifications viz 500, 2000 and 4000. In case of MnO\textsubscript{2} incorporated anodes the precipitation of MnO\textsubscript{2} is seen with in the grains and grain boundaries. Such precipitation normally reduces the galvanic efficiency of the anode. Barring the voids visible in the micrograph, two distinct phases are clearly visible are with MnO\textsubscript{2} precipitation and other being clear phase. During addition of MnO\textsubscript{2} in the alloy melt-
Figure 5.5.a. SEM photographs of the MnO$_2$ incorporated Al + 5% Zn alloy anode at three different magnifications (i, x 500, ii, x 2000, iii, x 4000).
Figure: 5.5.b. SEM photographs of the V$_2$O$_5$ incorporated Al + 5% Zn alloy anode at three different magnifications (i, x 500, ii, x 2000, iii, x 4000).
itself, the precipitation of MnO₂ was visible which was negligible when concentration of MnO₂ was very low. In the case of V₂O₅ incorporated anodes no such type of precipitation is observed in the grains and almost a negligible phase is observed with minor random precipitation of V₂O₅. The grains were found to be almost uniform throughout the alloy matrix. Thus incorporation with these catalytic metal oxides does not possess a uniform matrix in both the cases.

The Vicker hardness numbers of 0.5% MnO₂ and 0.5% V₂O₅ incorporated anodes were found to be 37.92 and 38.90 respectively. As the hardness of the bare substrate was also measured to be 39.92, no significant deviation in terms of hardness number due to catalytic oxide incorporation is noticed, the anodes were not characterized by other physical tests.

5.3.2 Performance evaluation

Galvanic performance of the catalytic metal oxide incorporated anodes were also tested by different techniques viz. OCP decay followed by CCP variations which was followed by galvanostatic polarization along with determination of self-corrosion and galvanic efficiency. The OCP decay curves of Al-Zn alloy anodes incorporated with varying concentrations of MnO₂ and V₂O₅ are shown in figure 5.6. These oxides show higher initial OCP and low polarization when compared with that of their anode. Both V₂O₅ and MnO₂ exhibit catalytic effect of which V₂O₅ is seemed to be still better than that of MnO₂, the best being with the anode incorporated with 0.5% V₂O₅. However, the maximum potential difference between any two electrodes is seemed to be less than -
Figure: 5.6. OCP decay curves of Al + 5 % Zn alloy sacrificial anodes reinforced with different concentrations of MnO₂ and V₂O₅

- Al + 5 % Zn
- Al + 5 % Zn + 0.25 % MnO₂,
- Al + 5 % Zn + 0.1 % V₂O₅,
- Al + 5 % Zn + 0.5 % V₂O₅

(Electrolyte: 3% NaCl solution, Temperature: 30 °C, stagnant condition).
50 mV at any time during the test period of three months. Around 10 mV of polarization of the 0.5% V₂O₅ incorporated anode in a span of three months test period is an interesting observation. The anode also showed less than 30 mV of polarization during monitoring of CCP variation after galvanically coupled with steel cathodes of fixed surface area (figure 5.7). Almost the same performance is observed with 0.5% MnO₂ incorporated anode also. Both MnO₂ and V₂O₅ incorporated anodes also show better performance when compared with that of pure Al + 5% Zn alloy anode. Almost similar order of galvanic performance of these anodes is noticed from the galvanostatic polarization curves (figure 5.8). Thus the catalytic effects of the metal oxides are found to be increasing with increase in the respective concentrations. Even then, observing more metallurgical defect and precipitation of these oxides at high concentration of 0.5%, and predicting low galvanic efficiency of unit mass of the anode, incorporation with higher concentration of these oxides were not attempted. The overall galvanic performance of these anodes along with that of pure Al + 5% Zn alloy anode are summarized in table 5.2. More than 40% reduction of the self-corrosion value and around 40% enhancement of galvanic efficiency due to incorporation of 0.5% V₂O₅ is predominantly noticed.

Table 5.3 compares the galvanic efficiency of the anodes incorporated with all the metal oxides selected in the present work. The galvanic efficiency was determined in this accelerated test by impressing an anodic current density of 2.5 mA/cm² across the respective anodes against mild steel cathodes of fixed surface area, for a period of 48 hours within study also. The galvanic efficiency of V₂O₅ incorporated anode is seemed
Figure: 5.7. CCP decay curves of Al + 5% Zn alloy sacrificial anodes reinforced with different concentrations of MnO₂ and V₂O₅

(x) Al + 5% Zn
(Δ) Al + 5% Zn + 0.25% MnO₂,
(●) Al + 5% Zn + 0.1% V₂O₅,
(■) Al + 5% Zn + 0.5% V₂O₅

( Electrolyte: 3% NaCl solution, Cathode: mild steel, Temperature: 30 °C, stagnant condition).
Figure: 5.7. Anodic polarization curves of Al + 5 % Zn alloy sacrificial anodes reinforced with different concentrations of MnO₂ and V₂O₅

( x - Al + 5% Zn
Δ- Al + 5% Zn + 0.25% MnO₂,
●- Al + 5% Zn + 0.1% V₂O₅,
■- Al + 5% Zn + 0.5% V₂O₅ )

(Electrolyte : 3% NaCl solution, Cathode : Platinum foil, Temperature : 30 °C, stagnant condition).
better than that of MnO₂ incorporated anodes. No significant observations other than discussed earlier is noticed with inert metal oxides incorporated anodes.

**Table: 5.2.** Galvanic performance of MnO₂ and V₂O₅ reinforced Al + 5% Zn alloy sacrificial anode (Electrolyte: 3% NaCl solution, Cathode: mild steel, Temperature: 30 °C, stagnant condition).

<table>
<thead>
<tr>
<th>Sl. No</th>
<th>Conc. of MnO₂ %</th>
<th>Conc. of V₂O₅ %</th>
<th>OCP V/SCE</th>
<th>CCP V/SCE at i= 0.01 mA/cm²</th>
<th>CCP V/SCE at i= 0.1 mA/cm²</th>
<th>CCP V/SCE at i= 1.0 mA/cm²</th>
<th>Self corrosion X 10⁻⁶ g/cm²/h</th>
<th>Efficiency %</th>
<th>Energy-Density Ah/kg</th>
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<tr>
<td>1</td>
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<td>-0.993</td>
<td>-0.985</td>
<td>5.720</td>
<td>83.1</td>
<td>2169</td>
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**Table: 5.3.** Galvanic performance of Al + 5% Zn alloy sacrificial anodes reinforced with different metal oxide at an impressed current density of 2.5 mA/cm² (Electrolyte: 3% NaCl solution, Cathode: mild steel, Temperature: 30 °C, Condition: stagnant).

<table>
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<tr>
<th>Composition</th>
<th>Galvanic efficiency %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al + 5% Zn</td>
<td>35.0</td>
</tr>
<tr>
<td>Al + 5% Zn + 0.5% LaO₂</td>
<td>43.0</td>
</tr>
<tr>
<td>Al + 5% Zn + 0.5% Al₂O₃</td>
<td>35.5</td>
</tr>
<tr>
<td>Al + 5% Zn + 0.5% MnO₂</td>
<td>62.1</td>
</tr>
<tr>
<td>Al + 5% Zn + 0.5% V₂O₅</td>
<td>68.1</td>
</tr>
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5.4. **Summary**

Two each inert and catalytic metal oxides were selected and studied for evaluation of their role when they are incorporated in Al-Zn alloy anodes. Among these oxides, catalytic metal oxides showed good results leaving scope for further significant improvement along with other type of activators also. The important conclusions arrived from the present study are listed below.

- Even though incorporation of Al-Zn alloy anodes with inert metal oxides namely $\text{La}_2\text{O}_3$ and $\text{Al}_2\text{O}_3$ does not significantly change the morphology of the anodes, it results in little better galvanic performance of the anodes yielding no scope of practical applicability.

- Incorporation of $\text{La}_2\text{O}_3$ exhibits passivation characteristics of anode with higher current density or longer duration.

- Incorporation of $\text{MnO}_2$ and $\text{V}_2\text{O}_5$ shows good catalytic effect in aluminium activation and the catalytic effect increases with increase in concentration of their catalytic oxides. Higher concentration of these catalytic oxides causes precipitation in the alloy matrix.

- Among the different anodes studied the anode incorporated with 0.5% $\text{V}_2\text{O}_5$ showed excellent galvanic performance.
Scope for the further work

Even though good results have been achieved by means of incorporation with catalytic metal oxides, activation of aluminium in this process is substantially less when compared with doping of Sn or In. Hence double catalytic activation along with matrix reinforcement will result in significant improvement of the anode. Since surface activation, a new type of activation process first explored and evaluated in the present work, is also experimented in the course of the study, such process can also be experimented in this context. The next chapter deals with the surface activation of sacrificial anodes.

5.5. References


