Aluminum loss in KF-NaF-AlF₃ based electrolyte

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Abstract
Aluminum loss experiments in KF-NaF-AlF₃ based electrolyte were tested with graphite crucible, corundum crucible and carborundum crucible respectively. The results showed that aluminum loss was of 16 % or more in graphite crucible under static immersion conditions, much higher than that of in corundum crucible (< 0.3 %) and carborundum crucible (average value 1.53 %). The electrolyte crust of high alumina content and high molecular ratio on the surface of liquid aluminum was formed both in graphite crucible and in carborundum crucible because of the reaction of Al₄C₃ formation. The mechanism for higher aluminum loss in graphite crucible and in carborundum crucible under static immersion conditions was: Aluminum and carbon formed a Galvanic cell, that is, Aluminum reacted with oxygen ion in the electrolyte to form Al₂O₃; carbon contained in crucible reacted with aluminum ion to form Al₄C₃; and then the Al₄C₃ was dissolved in electrolyte or was decomposed by oxidation, which induced continuous loss of aluminum.

Keywords: Aluminum loss; KF-NaF-AlF₃ based electrolyte; low-temperature electrolyte; aluminum carbide (Al₄C₃).

1. Introduction
The NaF-AlF₃-Al₂O₃ based electrolyte is used in conventional aluminum reduction cells. The loss of aluminum dissolution in molten electrolyte is one of important factors which decreases current efficiency of electrolysis [1]. The solubility of aluminum in molten electrolyte (NaF-AlF₃-Al₂O₃) is not very high, only of 0.1 % - 0.2 % at 1000 °C [1, 2]. However, the dissolution loss of aluminum could be continuous in the actual electrolytic process. In industrial aluminum reduction cell, the loss of aluminum is divided into four continuous steps:
(1) Aluminum is dissolved in the electrolyte of the interface;
(2) Dissolved aluminum diffuses outward from the interface;
(3) Dissolved aluminum goes into the molten bath and diffuses toward anodic gas layer;
(4) Dissolved aluminum is oxidized by the anodic gas.

In general, the most accepted point of view is that the mass transfer process of dissolved aluminum is control step in the static molten electrolyte without electrolysis, and the dissolution of aluminum is control step in the molten bath with strong mixing(such as in industrial cells), in which the diffusion is very intense[1, 4]. So the solubility of aluminum determines the loss rate of aluminum in industrial cells.

The solubility of aluminum is related to the factors of melt temperature, cryolite ratio, additives (CaF₂, MgF₂), alumina concentration, viscosity and surface tension and so on [1, 4, 5].Generally, the higher of reduction cell temperature the larger of aluminum solubility. The solubility of Al decreases with the decrease of cryolite ratio and alumina concentration, but the range of cryolite ratio should be between 2.2 and 2.8. After MgF₂ or CaF₂ is added or the viscosity of melt is increased, the solubility of Al would decrease as well.

Besides, crucible materials used in the tests have large effect on the aluminum dissolution loss. In 1997, Qiu Zhuxian et al used graphite crucible as containers, and determined the solubility of aluminum in low cryolite ratio bath by mass method [6]. The loss of aluminum was only
0.2 % in a graphite crucible in which the lining had been coated with boron nitride; while the loss was about 1.2 % without the boron nitride coated lining. The reason of the increased loss of aluminum could be that aluminum reacted with graphite sidewall to form Al$_4$C$_3$ since electrons flow passed through aluminum and graphite sidewall in the molten bath. In the industrial cell, however, the graphite sidewalls were covered with a layer of frozed bath, which helped reducing the Al$_4$C$_3$ formation.

Sterten et al [7] carried out system research, such as aluminum loss, cathodic current density, current efficiency in different composition of electrolytes, which contained potassium cryolite, sodium cryolite, lithium cryolite, aluminum fluoride, calcium fluoride and alumina. It was found that the loss of aluminum dissolution in graphite crucible was larger than that in quartz crucible, and the loss in low temperature electrolyte was much smaller than that in traditional electrolyte. But the current efficiency decreased with the addition of K$_3$AlF$_6$, probably because a large amount of Al$_4$C$_3$ was generated.

Low temperature electrolysis (below 900 °C), which is considered as the most potential energy-saving technology and the main route to reduce inert anode corrosion, has become one of the most concerned and most active research topics in the international aluminum-smelting field [4, 8 - 10]. While the NaF-KF-AlF$_3$-based electrolyte with low cryolite ratio (1 < CR < 2) is considered as a promising electrolyte system for low temperature aluminum electrolysis [11 - 13].

The composition of KF-NaF-AlF$_3$ based electrolyte has more KF than that of the traditional NaF-AlF$_3$-Al$_2$O$_3$ electrolyte system, so the dissolved loss of Al in the electrolyte would be also different. According to references [7] and [14], the solubility of Al increased because of the presence of KF. But the dissolution loss of Al in the KF-NaF-AlF$_3$ based electrolyte (1 < CR < 2), and the effect of crucible materials on current efficiency and aluminum loss in the actual process of electrolysis is not yet very clear.

In the present work, the tests for dissolution loss of Al in KF-NaF-AlF$_3$ based electrolyte (1 < CR < 2) were carried out, and the effect of crucible materials on the current efficiency was explored with 20 A electrolysis.

2. Experimental

The KF-NaF-AlF$_3$ based electrolyte was used, KF content was around 20 wt%, cryolite ratio was between 1.3 - 1.6 ([NaF]+[KF]) / [AlF$_3$]), alumina content was between 4.2 wt% and 5.2 wt%, temperature was about 820 °C. The electrolyte, consisted of industrial grade potassium fluoride, industrial grade sodium cryolite, industrial grade aluminum fluoride, was treated through pre-melting process. The ingredients of the materials are shown in Tables 1 – 3.

<table>
<thead>
<tr>
<th>Material name</th>
<th>Chemical Constituents, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Potassium fluoride (KF)</td>
<td>KF</td>
</tr>
<tr>
<td>≥ 98.5</td>
<td>≤ 0.5</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Material name</th>
<th>Chemical Constituents</th>
</tr>
</thead>
<tbody>
<tr>
<td>Sodium cryolite (Na$_3$AlF$_6$)</td>
<td>F</td>
</tr>
<tr>
<td>51.76</td>
<td>15.11</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Material name</th>
<th>Chemical Constituents, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Aluminum fluoride (AlF$_3$)</td>
<td>AlF$_3$</td>
</tr>
<tr>
<td>89.2</td>
<td>6.82</td>
</tr>
</tbody>
</table>
A self-made cell was heated externally, whose lining was made of graphite material, temperature was controlled automatically, and the cell cover was made of high alumina refractory materials. As shown in Figure 1 below.

Figure 1. Molten bath in a self-made cell. Figure 2. Graphite crucible with aluminum.

The tests of aluminum loss were carried out in the cell above under static immersion condition at 820 °C. A weighed aluminum block was put into the graphite crucible, and then they were placed into an oven, dried for 4 h or more at 150 °C. After that, the graphite crucible with the aluminum block was dropped into the bath.

After a certain time, the crucible was removed from the bath. After the crucible was cooled, the aluminum mass in the crucible was weighed to check the aluminum loss.

The 20 A electrolysis tests were carried out in the KF-NaF-AlF₃ based electrolyte, cryolite ratio 1.4, 810 °C, saturated alumina in a cell shown in Figures 3 and 4. The anode was made of an alloy, and cathode was made of TiB₂–C composite.

Figure 3. Photo of the 20 A cell. Figure 4. Structure of the 20 A cell.

where, 1 refractory brick ; 2 stainless steel crucible ; 3 graphite crucible ; 4 electric furnace ; 5 thermocouple of temperature control ; 6 thermocouple of temperature measurement ; 7 crucible lid ; 8 furnace sealing cover (with cooling water) ; 9 bath ; 10 cathode ; 11 anode ; 12 nitrogen gas pipe.

3. Results and Discussion

3.1. Aluminum loss tests

3.1.1. Graphite crucible

It was noticed when the graphite crucible touched the molten electrolyte, there were a lot of bubbles emitted immediately from the crucible wall. When the graphite crucible completely immersed into the electrolyte melt, more bubbles erupted, and the bubbles almost covered the graphite crucible and the aluminum block. At this point, the aluminum block was found to be
wrapped with frozen electrolyte. When the frozen electrolyte was re-melted, black fog occurred around the aluminum block, and at the same time the aluminum block was becoming liquid.

![Figure 5. Crust formation after aluminum melts.](image1)

![Figure 6. A large amount of black fog and yellow flame appeared after the crust broken.](image2)

The liquid aluminum gathered into a ball shape after melted, and a layer of crust was formed on the aluminum surface (as Figure 5). After the crust was formed, the block fog of aluminum disappeared and electrolyte resumed clear. The number of bubbles released on the wall of graphite crucible was gradually reduced. When the crust was broken (as Figure 6), there seemed to have an intensive reaction on the aluminum surface, a large amount of black fog was formed and spread upward, electrolyte became cloudy and yellow flame could be seen on the electrolyte surface.

When the black fog disappeared, the crust on the aluminum surface was found reformed, and the bath resumed clear but slightly yellow.

The tests conditions and results were shown in Table 4.

<table>
<thead>
<tr>
<th>Test serial number</th>
<th>Process and conditions</th>
<th>Electrolyte</th>
<th>Pre-test weight (g)</th>
<th>Post-test weight (g)</th>
<th>Loss rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>New graphite crucible, crust was broken once, total immersion time, 24 h.</td>
<td>4.42</td>
<td>200</td>
<td>141.8</td>
<td>29.1</td>
</tr>
<tr>
<td>2</td>
<td>New graphite crucible, crust was not broken, total immersion time, 17 h.</td>
<td>4.27</td>
<td>255</td>
<td>213</td>
<td>16.5</td>
</tr>
<tr>
<td>3</td>
<td>New graphite crucible, crust was not broken, total immersion time, 36 h.</td>
<td>4.69</td>
<td>271</td>
<td>225</td>
<td>16.9</td>
</tr>
<tr>
<td>4</td>
<td>Graphite crucible was pre-immersed for 6h, and then the aluminum block was put in, total immersion time, 20 h.</td>
<td>4.75</td>
<td>104</td>
<td>93.7</td>
<td>9.9</td>
</tr>
<tr>
<td>5</td>
<td>Graphite crucible was pre-immersed for 24 h, and then the aluminum block was put in, crust was not broken, total immersion time, 24 h.</td>
<td>4.37</td>
<td>494</td>
<td>460.8</td>
<td>6.72</td>
</tr>
<tr>
<td>6</td>
<td>Cylindrical graphite crucible in an air-tight furnace with nitrogen flow, total immersion time, 24 h.</td>
<td>4.84</td>
<td>120.1</td>
<td>114.4</td>
<td>4.7</td>
</tr>
</tbody>
</table>
In the first test, the crust was broken once for checking the phenomenon, and the aluminum loss was of 29.1%. The main purpose of the second and third tests was to observe the crust’s protection to reduce the aluminum loss. It was found out that if the crust were not broken, the aluminum loss rate in 17 h immersion and 36 h immersion were basically the same.

There were a lot of bubbles emitted from the graphite crucible when touched bath in all the precede tests. The bubbles came from air in pores of graphite contained oxygen which could cause aluminum loss directly. And at another hand, air and Al would form the Al-Air galvanic cell as follows:

Anode reaction: \[4\text{Al} - 12\text{e} \rightarrow 4\text{Al}^{3+}\] (1)

Cathode reaction: \[3\text{O}_2 + 12\text{e} \rightarrow 6\text{O}^{2-}\] (2)

Total reaction: \[4\text{Al} + 3\text{O}_2 \rightarrow 2\text{Al}_2\text{O}_3\] (3)

In the fourth and fifth tests, the graphite crucibles were pre-immerged into the electrolyte melt so that the most bubbles were discharged in advance, in order to observe whether the aluminum loss could be reduced. The results showed that the graphite crucible was pre-immerged for 6 h, the rate of aluminum loss was reduced to 9.9%; the graphite crucible was pre-immerged for 24 h, the rate of aluminum loss was reduced to 6.72%. It indicates the possibility of the reaction described by Equations (1), (2), (3) or only Equation (3).

In the third test, the aluminum surface crust was sampled and analyzed as shown in Table 5.

<table>
<thead>
<tr>
<th>Name</th>
<th>NaF</th>
<th>KF</th>
<th>AlF3</th>
<th>Al2O3</th>
<th>CR</th>
<th>Al(metal)</th>
<th>B</th>
<th>Ti</th>
<th>Si</th>
<th>Remains</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
<td>(%)</td>
</tr>
<tr>
<td>Crust</td>
<td>25.62</td>
<td>21.38</td>
<td>39.33</td>
<td>7.65</td>
<td>2.09</td>
<td>5.72</td>
<td>0.034</td>
<td>0.030</td>
<td>0.004</td>
<td>0.233</td>
</tr>
</tbody>
</table>

In all the tests in Table 4, the color of electrolyte was becoming slightly yellow, and some yellow materials were also found at the interface between bottom of the aluminum block and the graphite crucible. It was speculated that the Al4C3 was formed.

Aluminum carbide can be dissolved in the electrolyte (according to literature [1], the solubility of Al4C3 in sodium cryolite electrolyte is of 2.5%), and the dissolved Al4C3 could be brought to the surface of electrolyte and oxidized by oxygen in air. The overall reaction formula can be expressed as follows:

Al4C3 formation: \[3\text{C} + 4\text{Al} \rightarrow \text{Al}_4\text{C}_3\] (4)

Al4C3 oxidation: \[\text{Al}_4\text{C}_3 + 6\text{O}_2 \rightarrow 2\text{Al}_2\text{O}_3 + 3\text{CO}_2\] (5)

The sixth test was conducted in an air-tight furnace, the aluminum block and electrolyte were placed together in a graphite crucible, and then they were put into the furnace protected by nitrogen gas, the rate of aluminum loss decreased to 4.7%. The main purpose of the test was to drain the oxygen above bath surface.

According to the reaction Equation (4) and (5), when the oxidation of Al4C3 reduced, the dissolution velocity of Al4C3 in electrolyte slows down, and the generation of Al4C3 would be reduced as well. So the aluminum loss rate in the sixth test was significantly reduced.

3.1.2. Carborundum crucible

The phenomenon in carborundum crucible was similar to that in graphite crucible, when carborundum crucible touched the electrolyte melt bath, there were also bubbles being emitted, but the amount of bubbles was significantly less. And a layer of crust was also formed on surface of liquid aluminum. Most likely porosity in carborundum crucible was
much smaller.

Three different carborundum crucibles were used (different manufactures, slightly different material composition), and the results of aluminum loss were different, but less than that of in graphite crucible, as shown in Table 6.

**Table 6. The aluminum loss in carborundum crucibles.**

<table>
<thead>
<tr>
<th>Test serial number</th>
<th>Process and conditions</th>
<th>Electrolyte</th>
<th>Pre-test weight (g)</th>
<th>Post-test weight (g)</th>
<th>Loss rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Carborundum A, crust was not broken, total immersion time, 18 h.</td>
<td>5.2</td>
<td>1.436</td>
<td>214.4</td>
<td>0.46</td>
</tr>
<tr>
<td>2</td>
<td>Carborundum B, crust was not broken, total immersion time, 24 h.</td>
<td>4.8</td>
<td>1.47</td>
<td>583.2</td>
<td>1.3</td>
</tr>
<tr>
<td>3</td>
<td>Carborundum C, crust was not broken, total immersion time, 24 h.</td>
<td>4.84</td>
<td>1.43</td>
<td>123.2</td>
<td>1.95</td>
</tr>
<tr>
<td>4</td>
<td>Carborundum C, crust was not broken, total immersion time, 24 h.</td>
<td>4.84</td>
<td>1.43</td>
<td>194.1</td>
<td>2.37</td>
</tr>
<tr>
<td>5</td>
<td>Carborundum C, crust was not broken, total immersion time, 24 h.</td>
<td>4.271</td>
<td>1.456</td>
<td>189.2</td>
<td>1.59</td>
</tr>
<tr>
<td></td>
<td>Average</td>
<td></td>
<td></td>
<td></td>
<td>1.534</td>
</tr>
</tbody>
</table>

**3.1.3. Corundum crucible**

Phenomenon in corundum crucible was different from that in graphite crucible and carborundum crucible. There were basically no bubbles emitting from the crucible wall, only a few bubbles were steamed out during the aluminum block melting since corundum crucible is much denser than graphite and carborundum crucibles, as shown in Table 7.

**Table 7. Density and porosity of graphite, carborundum and corundum.**

<table>
<thead>
<tr>
<th>Items</th>
<th>Graphite</th>
<th>Carborundum</th>
<th>Corundum</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bulk density (g/cm³)</td>
<td>1.72</td>
<td>2.76</td>
<td>3.7</td>
</tr>
<tr>
<td>Theoretical density (g/cm³)</td>
<td>2.25</td>
<td>3.2</td>
<td>3.9</td>
</tr>
<tr>
<td>Apparent porosity (%)</td>
<td>15.49</td>
<td>7.5</td>
<td>(close to 0)</td>
</tr>
</tbody>
</table>

When aluminum block was immerged in bath, the first phenomenon was that the aluminum block was wrapped by frozen electrolyte. After the frozen electrolyte was re-melted, the aluminum block became liquid gradually. When stable, the aluminum surface was covered by a layer of black fog, looked like black crust, but gently touch could disperse the black fog.

The black fog could be the so-called aluminum fog. The aluminum loss rates were very small in those tests, some of which the post-test weight of aluminum block were increased because of the bath conglutination. The results were shown in Table 8.
Table 8. Aluminum loss in corundum crucibles.

<table>
<thead>
<tr>
<th>Test serial number</th>
<th>Process and conditions</th>
<th>Electrolyte</th>
<th>Pre test weight (g)</th>
<th>Post test weight (g)</th>
<th>Loss rate (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Corundum crucible, total immersion time, 18h.</td>
<td>4.696</td>
<td>1.481</td>
<td>104.6</td>
<td>104.3</td>
</tr>
<tr>
<td>2</td>
<td>Corundum crucible, total immersion time, 24h.</td>
<td>4.802</td>
<td>1.37</td>
<td>331</td>
<td>330.9</td>
</tr>
<tr>
<td>3</td>
<td>Corundum crucible, total immersion time, 24h.</td>
<td>4.802</td>
<td>1.37</td>
<td>82.5</td>
<td>83</td>
</tr>
<tr>
<td>4</td>
<td>Corundum crucible, total immersion time, 24h.</td>
<td>4.802</td>
<td>1.37</td>
<td>89.6</td>
<td>90.1</td>
</tr>
</tbody>
</table>

4. Electrolysis tests

From the precede tests, it could be seen that aluminum loss in graphite crucible was quite large, even in nitrogen atmosphere, the loss was still 4.7 %. During electrolysis, if existence of graphite, electrolyte and oxygen occurs especially with inert anodes, it provides the conditions for formation, dissolution and oxidation of Al₄C₃. Therefore the current efficiency of electrolysis would be jeopardized.

The 20A tests with graphite crucible were carried out three times (Test1-Test3), and the test with corundum crucible was carried out once (Test0). Test conditions: KF-NaF-AlF₃-based remelted bath was used; Initial cryolite ratio was 1.40; alumina concentration was saturated; Electrolytic temperature was about 810 °C; Nitrogen flow rate was 0.4 L/min; Alloy inert anodes and TiB₂ ceramic cathodes were used.

Aluminum was pre-added in the graphite crucibles of Test1 and Test2 tests, which were different from Test 3 and Test 0; there were no pre-added aluminum in the graphite crucible of Test3 and in the corundum crucible of Test0. The voltage curves of those tests were shown in Figure 7.

![Figure 7. Curves of the 20A tests.](image)

As compared to the voltage curve of Test0, the voltage curves wave up and down during the first periods in Test1, Test2 and Test h. So the Test3 was stopped at 12 h to check what happened.

After the tests, the crucibles were taken out and broken; the current efficiency could be calculated by weight of aluminum block. The current efficiency of Test0 was 95 %; the current efficiency of Test1 was 64 %; the current efficiency of Test2 was 45.8 %; there was no aluminum block in the graphite crucible of Test3, but a mass of black materials, so there was basically no current efficiency.
Aluminum fog would be generated during the process of aluminum melting in tests of aluminum loss under static immersion, no matter in graphite crucible, or in corundum crucible, or in carborundum crucible, and color of the fog was always black, while according to the literature [1] and [15], the color of aluminum fog was purple and blue or brown. The different color of the aluminum fog could be related to the electrolyte composition, temperature and light intensity. Aluminum fog might result from either electrochemical dissolution or chemical dissolution of aluminum [15, 16] as follows:

Electrochemical dissolution : \[ \text{Al} - e \rightarrow \text{Al}^+ \]  
Chemical dissolution : \[ 2\text{Al} + \text{AlF}_3 \rightarrow 3\text{AlF} \]

Under static immersion, aluminum loss in corundum crucible was little, which indicates that the solubility of Al in KF-NaF-AlF$_3$ electrolyte system (CR = 1 - 2) was quite low.

Under static immersion, aluminum loss was increased greatly, when the graphite crucible and carborundum crucible were used. There were two main reasons:

1. Aluminum and oxygen which was brought into the electrolyte from pores of graphite formed galvanic battery (Al-Air battery). So the aluminum was oxidized. The process of reaction could be described by Equations (1) - (3).

2. Aluminum reacted with carbon contained in the crucible material to produce Al$_4$C$_3$ which had a higher solubility in electrolyte. The Al$_4$C$_3$ could be oxidized by oxygen on bath surface where the Al$_4$C$_3$ was moved onto by electrolyte flow or diffusion. So aluminum loss would be continuous. The process of reaction could be described by Equations (4) - (5). And this was the reason for lower current efficiency of Test1, Test2 and Test3.

Under electrolysis condition, the electrochemical dissolution (Equation 6) and chemical dissolution (Equation 7) of aluminum co-existed, but it could not cause substantial aluminum loss and a substantial decrease in current efficiency. Because the 20 A electrolysis test (Test0) was done in corundum crucible, the current efficiency could reach 95 %. Even in the industrial aluminum reduction cell, aluminum loss was relatively low, and the loss rate was about $0.53 \times 10^{-3}$ g/cm$^2$-min $\times$ [4]. Of course, liquid aluminum as cathode inhibited electrochemical dissolution of aluminum.

In the process of Al$_4$C$_3$ formation, not only at the Al-C interface of contact, the whole surface of aluminum was involved in the produce reaction of Al$_2$O$_3$. The local concentration of Al$_2$O$_3$ was increased, result in the segregation of electrolyte, and then the high melting point electrolyte crust was formed. The reaction process could be described as follows:

Reaction on the liquid aluminum surface :

\[ 4\text{Al} + 6\text{O}^2- \text{ (complex) } - 12e^- = 2\text{Al}_2\text{O}_3 \]  

Reaction on the carbon surface :

\[ 3\text{C} + 4\text{Al}^{3+} \text{ (complex) } + 12e^- = \text{Al}_4\text{C}_3 \]  

Overall reaction (Al$_4$C$_3$formation) :

\[ 3\text{C} + 4\text{Al} \rightarrow \text{Al}_4\text{C}_3 \]  

This also happens to explain why the crust would be formed on the surface of liquid aluminum, only in graphite crucible and carborundum crucible, and not in a corundum crucible.

5. Conclusions

(1) Aluminum loss in KF-NaF-AlF$_3$ electrolyte (CR = 1 - 2) was tested, and had a strong relationship with the crucible materials. The aluminum loss was the highest in graphite crucible and lowest in corundum crucible.

(2) The solubility of aluminum in KF-NaF-AlF$_3$ electrolyte (CR = 1 - 2) was relatively low,
but the aluminum fog would be generated during aluminum melting.

(3) Under static immersion, the two main reasons causing high aluminum loss in graphite crucible and carborundum crucible were: aluminum reacted with oxygen carried by crucible to form Al₂O₃; Aluminum reacted with carbon contained in crucible to form Al₄C₃, and then the Al₄C₃ was dissolved or oxidized. However, the latter was the key factor to the continuous loss of aluminum, which could seriously affect the current efficiency.

(4) The crust on aluminum surface was composed of high cryolite ratio and high alumina concentration in graphite crucible and carborundum crucible due to the reaction of Al₄C₃ formation.

6. References

