Carbothermic Reduction of Alumina A Study & A Progress Report

Valerie Gervais MIME572: Computational Thermodynamics McGill University, Winter 2013

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1. Hall-Héroult Process

Electrolytic reduction of alumina

 \rightarrow Currently the only industrial process for the production of Aluminum

•The Aluminum Industry is the primary source of industrial energy consumption, consuming about 1% of the total electric energy produced globally.

•The Hall-Héroult process is the most energy consuming stage in aluminum production, while also being highly expensive and only economically feasible in large scale production.

•The Hall-Héroult process is also a CO₂ intensive process and is known to be responsible for producing 2.5% of the world's anthropogenetic Greenhouse gas emissions.

**Based on 2011 data [1]



Alumina (Al_2O_3) is dissolved in molten salt (cryolite) by direct current electrolysis during which CO_2 is released at the carbon anode and Al is recovered at the bottom of the cell.

1.1 Moving towards Sustainable Alternatives

Carbothermic Reduction of Alumina
 Has been suggested and studied by many researchers in the last 50 years.

Based on 2011 documentation, the implementation of such a process would reduce energy consumption by 21% and would reduce Greenhouse gas emissions by up to 52%. [1]

2. Basics of Carbothermic Reduction of Alumina

2.1 DATABASE SELECTION

Development of thermodynamic data

KEY: Understanding the thermodynamics of the Al2O3-Al4C3 slag system and the Al-C liquid metal [2]

- Gibbs free energies
- Activities

NEW DATABASE in FactSage: FACT FTOxCN

 Developed for performing equilibrium calculations in the AI-(Si-Ca-Mg-Fe)-C-O-N System at very high temperatures

Project Database selection: FTOxCN & FactPS for gaseous products

* The addition of a very small amount of argon (10E-4 mol) is sometimes required for convergence of PhaseDiagram and Equilb module calculations. [3]

2.2 Carbothermic Reduction of Alumina

General equation:

 $AI_2O_3 + 3C = 2AI + 3CO$

As shown on the right, utilizing the reaction module, this reaction is thermodynamically favorable at high temperatures. However, it occurs simultaneously with other reactions which complicate the direct reduction process.



The direct reduction of alumina using carbon suffers from critical issues such as

- Aluminum carbide and oxycarbide formation, and
- Aluminum vaporization

These reduce the aluminum yield considerably \rightarrow Visual demonstration using FactSage on the next slide

2.3 Thermodynamic analysis

For a system initially containing 1 mole of alumina and 3 moles of carbon, the following displays the molar variations in the species containing Aluminum at equilibrium for a range of temperatures.



Important Factors:

For 1940°C <T< 2050°C, aluminum is mainly in the slag phase (red & blue).

For 2020°C <T< 2160°C, Al₄C₃ carbides are stable outside of the slag phase (pink).

For 2080°C< T< 2330°C, Aluminum liquid alloy is stable (green).

At T = 2160°C, maximum aluminum yield is reached.

Near max. yield T, a significant amount of AI is lost to $AI_{(g)}$ (gray) or $AI_2O_{(g)}$ (teal).

Figure 2. Distribution of Aluminum content at equilibrium for an input of $AI_2O_3 + 3C$

**Note that the molar variations of CO(g), CO2(g) and C(s) along with all other species present at negligible content have been omitted from this graph.

Primary R&D Goal: Increase Aluminum yield and reduction process efficiency by reducing losses to carbides or aluminum vaporization.

3. Two Stage Reactor →Patent issued in 1981 By C.N. Cochran et al.

Overall process [4]

Stage 1: Slag Production

Upper reaction zone $3AI_2O_3 + 9C \rightarrow (AI_4C_3.AI_2O_3)_{slag} + 6CO$

Stage 2: Alloy Formation

Lower reaction zone $(AI_4C_3.AI_2O_3)_{slag} \rightarrow 6 AI_{alloy} + 3CO$ (At higher temperature than stage 1)

A grate or screen (13) separates the upper and lower reaction zones in the reactor, allowing only liquid phases to pass through to stage 2. Most of the carbon dioxide gas is produced in the first stage and ejected through the top preventing interference with alloy formation in the lower stage.



reaction zone (stage 1) and lower reaction zone (stage 2). [4]

Since the issue of this patent, most of the research done by Alcan and Alcoa has revolved around Cochran's idea. [1]

Latest development in relation to this process has been done through the collaboration of Alcoa and Elkem who developed the Advanced Reactor Process ARP, a flow chart of which is shown in Figure 4. This process resolves previous issues such as

- \rightarrow heat supply
- \rightarrow slag transfer

→ vapour losses through the addition of a Vapour Recovery Reactor [1]

3.1 Slag production stage

(Upper reaction zone; stage 1)

$3Al_2O_3 + 9C \rightarrow (Al_4C_3.Al_2O_3)_{slag} + 6CO$

<u>Key factor:</u> Al_2O_3 and Al_4C_3 can either be produced as separate phases or as a slag phase. As shown previously in Figure 2, this is dependent upon the **temperature** at which the reactor is operated.

Next>> Determining the optimal temperature for slag production



Slag Production Stage: Upper Reaction Zone $3AI_2O_3 + 9C \rightarrow (AI_4C_3.AI_2O_3)_{slag} + 6CO$ **Temperature range for slag production**



Figure 5. Stable phases created by $3AI_2O_3 + 9C$ as a function of equilibrium temperature.

Phase Species Gas: CO, AI, AI_2O Slag#1: AI_2O_3 , AI_4C_3 AIC#1: $AI_4C_3(s)$ Liq#1: AI-C alloy

Stable Slag Temperature range 1907–2049°C but 1907-1948°C has ≈1E-5moles Best estimate: 1948-2049°C

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Slag Production Stage: Upper Reaction Zone $3AI_2O_3 + 9C \rightarrow (AI_4C_3.AI_2O_3)_{slag} + 6CO$ Temperature range for slag production



Figure 6. Molar amount of each species in the slag phase as a function of temperature

Concluding Note: An operating temperature of 2000°C is chosen as optimum temperature. This value will be used for further calculations based on the slag production stage (upper reaction zone)

Zooming in on the stable slag temperature range:

In Figure 6. the components of the slag phase are displayed based on their mole content in relation to the total mole content of the slag phase (red).

Stage 1 slag production is concerned with creating a slag phase without the production of AI_4C_3 carbides out of phase and with minimum aluminum vaporization.

From previous analysis, Figure 2. showed that AI_4C_3 carbides become stable at T=2020°C therefore this temperature must be avoided. Furthermore, aluminum vapours increase with temperature for the slag temperature range.

Finally, a relevant amount of AI_4C_3 in the slag phase is required for an efficient carbothermic reduction. (this will be shown later)

The operating temperature of the Slag Production Stage has been determined as 2000°C. This is lower than the temperature proposed in Cochran's patent in 1981 (2050°C).

Next we consider the lower reactor stage which receives the slag produced in the upper reactor region. The lower reactors is known to operate at a higher temperature than that above it and so it heats up the slag to produce the aluminum alloy.



on the lower reactor stage [5]

Alloy Formation Stage: Lower Reaction Zone (Al₄C₃.Al₂O₃)_{slag}→ 6 Al_{alloy} + 3CO @2100°C (patent temperature) Optimization of AI-C Yield from Al₄C₃/Al₂O₃ Ratio

On right, Figure 8. shows the stable phases present at equilibrium for $T = 2100^{\circ}C$, based on the mole fraction of Al_4C_3 initially present in the slag phase. This temperature was chosen for optimization because it is the temperature chosen by Cochran in 1981. (It will be shown later that this approximation gives the correct result)

Phase Species

Gas: CO, AI, AI_2O Slag#1: AI_2O_3 , AI_4C_3 AIC#1: $AI_4C_3(s)$ Liq#1: AI-C alloy



Figure 8. Aluminum yield as a function of mole Fraction of Al_4C_3 in the slag

Alloy Formation Stage: Lower Reaction Zone $(AI_4C_3.AI_2O_3)_{slag} \rightarrow 6 AI_{alloy} + 3CO @2100^{\circ}C (patent temperature)$ Optimization of AI-C Yield from AI_4C_3/AI_2O_3 Ratio

Al-C alloy yield is maximum when the slag in the lower reaction region has a nearly equimolar concentration of Al_4C_3 and Al_2O_3

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Point # 53 : x = 0.5 y = 2.537					
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Point # 59: x = 0.55 y = 2.735					
Point # 60 : x = 0.56 y = 2.6742					

The formation of carbides $AI_4C_3(s)$ separate from liquid phases occurs when the mole fraction of AI_4C_3 is, $X_{AI4C3} \le 0.53$.

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Point #	58: x = 0.54 y = 0.022219					
Point #	59: x = 0.55 y = 0.043476					
Point #	60: x = 0.56 y = 0.064732					
Point #	61 : x = 0.57 y = 0.085988					
Point #	62 : x = 0.58 y = 0.107244					

Concluding Note: A slag ratio of 1:1 is chosen. For further calculations involving the slag phase, a mole ratio of $0.5Al_2O_3$ - $0.5Al_4C_3$ will be used.

Not that the slag produced in the upper reactor and through to the lower stage does not have this equimolar ratio. Therefore, additional carbon must be added to the lower reactor to meet the required stoichiometry

Next>> Determining the operating temperature in the lower reactor

Alloy Production Stage: Lower Reaction Zone (Al₄C₃.Al₂O₃)_{slag}→ 6 Al_{alloy} + 3CO Temperature Range for Highest Aluminum Yield from Slag

Using the optimized slag composition, the temperature at which the lower reaction stage must be operated is determined.

Figure 9 shows the variation in the number of moles of Aluminum (liqu#1) produced as a function of the reactor temperature.

Graphed Species & Phases

• Displayed are both the entire Liquid phase (Liqu#1) and the Aluminum portion of that Al(liqu#1).

• The Gas phase (GAS) is displayed as well as the aluminum vapour portions of it (Al₂O, Al)



Figure 9. Aluminum yield as a function of the operating temperature of the lower reactor

Note: The graph agrees with the 1981 patent where the second stage was performed at 2100°C since this is within the approximate optimum range: 2055- 2125°C.

Alloy Production Stage: Lower Reaction Zone (Al₄C₃.Al₂O₃)_{slag}→ 6 Al_{alloy} + 3CO Temperature Range for Highest Aluminum Yield from Slag

The aluminum yield is measured based on the input number of moles vs the output number of moles of Al in the liquid metal. An optimum temperature of 2070°C was chosen for the lower reactor region.

At this temperature, the Al yield is 75%.

Concluding Note: For further calculations based on the alloy formation stage (lower reaction zone), an operating temperature of 2070°C will be used



Figure 9. Aluminum yield as a function of the operating temperature of the lower reactor

3.3 Phase Diagram: Al₄C₃-Al₂O₃

Validating that all assumptions and conclusions collide together and form a viable process.



Figure 10. Al₄C₃-Al₂O₃ phase diagram with focus on phases present for 0.5 mol fraction

Summary

→ A thermodynamic evaluation of the basic carbothermic reduction of alumina showed aluminum content losses to the formation of carbides or that of vapours. → The development of a two stage process by Cochran et al. was presented in which aluminum vaporization is avoided in the first stage and carbide formation is unstable in the second

→ The evaluation of Stage 1: slag production resulted in an optimal temperature of 2000°C

→ A study on the optimum molar ratio of slag demonstrated that a $0.5AI_2O_3$ -0.5 AI_4C_3 mole ratio gives the maximum Aluminum metal output, explaining the charge mole ratio of 1 moles of AI_2O_3 to 3 moles of C in the theoretical carbothermic equation

 \rightarrow Finally,the evaluation of Stage 2: aluminum alloy formation set the operating temperature at 2070°C

Flux Addition

In industry, Flux is often added to help in the smelting process. Here, will we study the effect of the addition of CaO, MgO and SiO_2 in alumina carbothermic reduction.

Objective: Determine the optimum flux composition to obtain the lowest possible melting temperature forming the AI_2O_3 - AI_4C_3 Slag and increasing the Aluminum yield.



Figure 11. Schematic of the addition of Flux in the two stage

4. Flux Composition

CaO-MgO-SiO₂

Determination of the best flux composition to lower operating temperatures and increase aluminum yield.

<a>CaO, MgO, <c> SiO₂

The total flux charge in the upper reactor zone is approximated at 20% of the total charge. Therefore, a + b + c = 0.20 moles INPUT: $Al_2O_3 = 0.4$ moles $Al_4C_3 = 0.4$ moles

& Argon gas is added at 0.0001mol for convergence of the phase diagram and equilb modules

- Determine the effect of each slag component individually
- Observe the combined effect of two components at a time and determined the most adequate molar ratio between the two
- Analyze the data to determine what ratios increase the aluminum yield and decrease the operating temperatures.

4.1. Effect of Each Component on the process 4.1.1 An example: MgO- Al₄C₃-Al₂O₃ system

Step by step demonstration of flux component effect on the temperature at which slag is formed, Tslag, the temperature at which liquid AI is formed, Tliquid and the Aluminum yield.



4.1. Effect of Each Component on the process 4.1.1 An example: MgO- Al₄C₃-Al₂O₃ system Variable Selection







4.1.1 Effect of Each Component on the process; MgO example Using the Equilb Module to determine Al yield

The slag seems to form at relatively low temperatures but as shown in the last slide, Slag formation with AI_4C_3 only occurs around 1740°C.

Furthermore, for an equimolar input of AI_4C_3 and AI_2O_3 , this analysis is more relevant to the lower reactor stage.

Figure 13. shows that the liquid phase is initially produced around 1990°C. The Aluminum yield at 2070°C is 1.5337 moles for an input of 2.4 moles, therefore **63.9%.**



Figure 13. Stable phases present + AI (liqu) as a function of Temperature with 20% MgO addition

Note: The addition of MgO gives an AI yield that is lower than the initial yield of 75%



Figure 14. Al₄C₃-Al₂O₃ Phase Diagram including 20% CaO

4.1.2 Effect of Each Component on the process; CaO addition Using the Equilb Module to determine T_{slag} , T_{liquid} and Al_{yield}

The slag containing Al_4C_3 forms at a relatively low temperature of 1655.3°C. This temperature is much lower than the original slag production temperature of 2000°C





Figure 15. Stable phases present + Al (liqu) as a function of Temperature with 20% CaO addition

4.1.2 Effect of Each Component on the process; CaO addition Using the Equilb Module to determine T_{slag} , T_{liquid} and AI_{yield}

Figure 15. also shows that the liquid phase is initially produced around 2085°C. This temperature is higher than the initial liquid alloy temperature of 2070.

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Furthermore, the Aluminum yield at 2070°C is 0.000118 moles for an input of 2.4 moles, therefore **0%.**

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Note: the same procedure was used to determine the effect of SiO_2 addition. The following table displays the effect of each on the key variables.

4.1 Effect of Each Component on the process 4.1.3 Conclusions

Variables:

 T_{slag} = Temperature at which slag is formed in the upper reactor

 T_{liquid} = Temperature at which AI liquid alloy is formed in the lower reactor

Al_{yield @ 2070} = Mole percentage of Aluminum converted to the Aluminum liquid alloy at a lower reactor temperature of 2070°C

Max. Al_{vield @ T} = Maximum aluminum yield at a given temperature in the lower reactor

Added Component (20%)	T _{slag} (°C)	T _{liquid} (°C)	Al _{yield} @ 2070°C	Max. Al _{yield} @ T
None	2000	2070	75%	
MgO	1747.1	1990	63.9%	66.37% @ 2042.1
CaO	1655.3	2085	0%	69.29% @ 2103.5
SiO ₂	1931.3	2027.3	61.08%	61.75% @ 2055

Table 1. Effect of individual flux addition

Note: All Aluminum yield whether they be at 2070 or the maximum aluminum yield for a given temperature, are lower than the Aluminum yield without flux addition.

4.2. Effect of Adding Component Combinations on the process The same procedure was followed but an additional component was added. The aluminum yield for a two-flux component system was determined. As before, Initial amount of Al in slag $0.4AI_4C_3-0.4AI_2O_3=0.4(2) + 0.4(4) = 2.4$ moles

Table 2. Effect of CaO –SiO₂ addition

CaO %	SiO2 %	Tslag (C)	Tliquid (C)	Max. Al (moles) @ T	AI (moles) @2070C**	% Al Yield @2070
10	10	1657.5	2045	1.5839 @ 2045	1.5094	62.89%
20	0	1655.3	2085	1.663 @ 2103.5	1.834E-5	0%
0	20	1931.3	2027.5	1.4819 @ 2055	1.466	61.08%
15	5	1657.2	2055	1.6258 @ 2095	0.533	22.21%
5	15	1663.2	2031.7	1.5381 @ 2070	1.5381	64.09%

Table 3. Effect of MgO –SiO₂ addition

MgO %	SiO2 %	T slag (C)	Tliquid (C)	Max. Al (moles) @ T	Al (moles) @ 2070C**	% Al Yield @2070
10	10	1742.9	1990	1.5338 @ 2050	1.4863	61.93%
20	0	1747.1	1990	1.5928@ 2042.1	1.5337	63.90%
0	20	1931.3	2027.3	1.4819@ 2055	1.466	61.08%
15	5	1742.9	1990	1.5638@ 2041.5	1.4906	62.11%
5	15	1740.9	2015	1.5079 @ 2050	1.4781	61.59%

Summary of the Flux Analysis

→ The presence of CaO significantly decreases the temperature at which slag (including AI_4C_3) is formed. This means that composition of CaO in the flux reduces the operating temperature of the Upper Reactor region.

→ The presence of SiO₂ as well as that of MgO reduces the temperature at which the liquid alloy is formed in the second stage of the two stage reactor. → Despite this affect of reducing considerably the operating temperatures of the reactor stages, it was shown that the presence of these flux components either individually or in pairs, decreases the original Aluminum yield of 75%.

5. Effect of Oxygen content on Aluminum Yield

In industry, the addition of flux is usually known to be beneficial to the smelting process. For the case of the carbothermic reduction of alumina, it was found that this is not the case.

A possible explanation for this result could be the effect of the presence of oxygen in contact with the Aluminum liquid that is forming. Cochran's two stage reactor had a two stage process partially in order to avoid the interference of carbon monoxide gas with the liquid aluminum. Therefore,

interaction with oxygen atoms could be the downfall of process efficiency.

Next>> The effect of oxygen atoms on aluminum yield

5.1 A Previous study on mole ratios of oxygen to aluminum 2011 Study by School of Mining and Metallurgical Engineering, Greece [1]

A study recently performed in Greece showed that the mole to mole ratio of oxygen and Aluminum in the initial charge (usually AI_2O_3) affects the final aluminum yield.[1]

Result: As you decrease the number of moles of oxygen to the number of moles of Aluminum, you increase your Aluminum yield and reduce losses to Al(g) and $Al_2O(g)$

Perhaps, the inverse can be applied also. Addition of CaO, MgO and SiO₂ increases the amount of oxygen present which could potentially account for reduction in yield.

Next>> Variation in amount of oxygen added as a function of Aluminum yield.



igure 4: Calculated aluminium molar speciation and alumina to metallic aluminium reduction yield at 2200 °C in system with varying initial aluminium to oxygen atomic ratio and excess carbon. For ratio 3 oxygen atoms / 2 Al atoms the initial system is equivalent to the composition of Al₂O₃+10C, for 2 oxygen atoms / 2 Al atoms to ½·(Al₄O₄C+19C), for 1 oxygen atoms / 2 Al atoms to Al₂OC+9C, and for 0 oxygen atoms / 2 Al atoms to ½·(Al₄C₃+17C)

5.2 Variation in the moles of oxygen reacting with the slag A possible explanation for the reduction in Aluminum yield by addition of Flux

Input:

1 mol of (Al2O3)0.5(Al4C3)0.5
<A>mol of O
Vary <A> from 0 to 2.5
by 0.01
T= 2070°C

Figure 16 shows that the number of moles of Al produced decreases as the input number of moles of oxygen added to the slag increases.

Note: This confirms the initial assumption that the presence of oxygen atoms in the flux reduce the aluminum yield.



Figure 16. Aluminum yield as a function of the number of moles of oxygen present.

Thank you.

References

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