
Summary

Previously the assumption have been that emission of PFC gases only occurred during conventional anode effects, where the concentration of dissolved alumina in the electrolyte are between 0.5 - 2 wt%. Until recently these assumptions have been proven wrong. Today there are several reports which can document generation of PFC under apparently normal operating conditions. A key to understand and to prevent the evolution of PFC gases is to know which parameters in the cell that contribute to the emissions. The goal of this study is to measure PFC by use of a Quantum cascade laser (QCL) directly on the exhaust duct of a single electrolysis cell. Campaigns from a single cell can be of help to develop reliable models for estimation of fluorocarbon species from the industry. This simple study have shown that the formation of PFCs are highly sensitive to imbalances in anode currents, but also to small concentrations of alumina in the cell.

Preface

This project has been done in collaboration with Alcoa Mosjøen, whom suggested the current topic. The project is an preamble to mount and test equipment to collect data to the master's thesis. I would like to thank everyone at Alcoa for the opportunity to do experiments and tests at their plant during the summer and autumn. In addition, I would also like to thank Prof. Morten Hovd, my supervisor at NTNU, for giving me the chance to carry out the project in collaboration with Alcoa.

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Abbreviations

ACD	=	Anode-Cathode Distance
AE	=	Anode effect
CCD	=	Critical current density
GHG	=	Greenhouse gases
LVP-AE	=	Low voltage propagating anode effect
LV-AE	=	Low voltage anode effect
NP-AE	=	non-propagating anode effect
PFC	=	Perfluorocarbon
QLC	=	Quantum cascade laser

Introduction

1.1 Background and motivation

From an environmental point of view, it is expected that a company or even an entire industry operates as environmentally friendly as possible. The aluminium industry is a large contributor to emissions globally but it has made great progress in reducing greenhouse gases from the smelting process over the last decades. This is achieved by technological improvements, but also by continuous search for new knowledge and better understanding of the smelting process. The rest of this chapter requires some general knowledge about the electrolysis process and for a reader which is not familiar with this, I would encourage to read chapter 2 before proceeding from here.

Perfluorocarbons (PFCs) are highly potent greenhouse gases with long lifetimes in the atmosphere that are emitted from the aluminium smelters. Previously there was a broad unanimity in professional circles that PFC generation only occurred during conventional anode effects (AEs) where the concentration of alumina in the electrolyte became too low, followed by a rapid increase in cell voltage. However, recent studies have shown that this is not always the case and PFC gases are also produced during apparently normal cell conditions. An example of these conditions could be during anode change or in the end of an underfeeding period (Batista et al., 2016). This indicates that the reported amount of greenhouse gases actually is higher than what is being reported, since estimation of PFCs from smelters today is based on the anode effect. According to reports from Chinese smelters the emissions of PFC from non-conventional anode effects contribute significantly to the total amount of produced PFC in the aluminium industry.

A key to understand and to prevent the evolution of PFC gases is to know which parameters in the cell contribute to the emissions. Cell design- and technology are much related to the probability of gas formation. It has been shown that the co-evolution of fluorocarbon species is highly sensitive to imbalances in anode current distribution, which (Rye et al., 1998) and others have connected to gradients in the alumina concentration. Measuring campaigns directly to one single industrial cell have previously not been feasible, because the measurement campaigns have been done by use of Fourier-transform

infrared spectroscopy (FTIR) which is a reliable, but demanding process. FTIR are also generally considered to be connected with high cost and require maintenance during measurement campaigns (Espinoza-Nava et al., 2016). However, the emerging quantum cascade lasers (QCL) have the property to monitor pollutants in the mid-infrared range and the possibility to measure continuous, in-situ, generation of PFC from the duct on one single cell. Since QCL offer low maintenance and the possibility to mount the equipment directly to the duct, this gives the opportunity to gain valuable insight to which cell conditions contributing to the generation of PFC. Campaigns from a single cell can be of help to develop reliable models for estimation of fluorocarbon species from the industry. In addition, when the decisive conditions are known, the smelting process can be further optimized to prevent the states where the emissions are high.

1.2 Objectives

The objective for this thesis is to perform in-situ PFC measurements by use of QCL on an aluminium electrolysis cell during normal operation. Specifically the objectives can be described by:

1. Literature review on previous work. Several studies have been performed and to be able to contribute to the discussion it is important to know what have been done in the past.
2. Get familiar and know how to operate the equipment to be used. This includes the QCL, monitoring of anode currents and the overall process monitoring of the electrolysis cell.
3. Develop and execute an experimental plan to see which cell parameters that contribute to the generation of PFC gases.
4. Evaluate and analyze the results.

Even though the goals are stated like this, the overall thesis will not cover them explicitly. The main part of the project work is to assemble, run and carry out the experimental plan and to analyze the results.

1.3 Outline

Chap 1 - Introduction
Chap 2 - Aluminium
Chap 3 - Experimental
Chap 4 - Results and discussion
Chap 5 - Conclusion and further work

Chapter 2

Aluminium

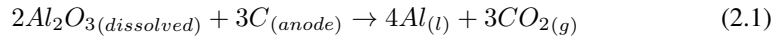
This chapter is intended to introduce the reader to general concepts in aluminium electrolysis. If not otherwise stated, section 2.1 and 2.2 are mainly based on the work done by (Grjotheim and Kvande, 1993) and (Hestetun, 2009) and gives an introduction to development and key concepts of the Hall-Héroult process. The goal of section 2.3 is to enlighten the reader to the complexity of all the intuitively simple variables contributing to the process. In the last two sections abnormalities and environmental aspects of the smelting process is addressed.

2.1 Aluminium production

If you take a look around you would find this metal everywhere. In anything from packing material to jet planes. aluminium is light and strong, and new applications are continuously discovered. With about 8 % of the earth's crust, aluminium is the most abundant metallic element. Because of its strong binding with oxygen it is not found in its purest form in nature, but only as combinations with other materials. The main compound in aluminium production is aluminium oxide (Al_2O_3), often referred to as alumina. The most available source of alumina is bauxite, and it is recovered through a chemical transformation called the Bayer process. All of the industrial smelters in the world produce aluminium by the Hall-Héroult process, invented and patented in 1886 by Charles Hall in the USA and Paul Héroult in France, independently of each other. No other process have until today been able to measure up with this method, and it still is the only industrially viable method for production of aluminium.

2.2 The Hall-Héroult process

In the Hall-Héroult process alumina (Al_2O_3) is dissolved in an electrolyte mainly containing molten cryolite (Na_3AlF_6). The electrolyte is often referred to as bath. In figure 2.1 a simple sketch of an electrolysis cell is shown. Aluminium has a higher density than bath and is found in a liquid phase in the bottom of the cell. The carbon anodes are located on the top of the cell and they are dipped into the bath. When the alumina dissolves in the bath the oxygen is discharged onto the carbon anodes, thus forming gaseous carbon dioxide (CO_2). Based on this the main chemical reaction during production can be written as



As we can observe from equation 2.1 the two main components in the process are alumina and carbon, and the products are aluminium and carbon dioxide. In addition to these substances the bath contains other additives like cryolite (Na_3AlF_6), aluminium fluoride (AlF_3) and calcium fluoride (CaF_2).

2.2.1 Cell and potline design

Mainly there are two different technologies regarding cell design. The older Söderberg cells and the one that is considered the preferred one today, the prebake cells. The main difference between the two designs is how the carbon anode(s) are made. The Söderberg cells are considered somewhat outdated, and only the prebake cell design are taken into account for this thesis.

The anodes for the electrolysis cell are produced and prebaked in a separate anode baking furnace, before the anodes can be used in the aluminium electrolysis cell, hence the name, prebake cell. This is done to ensure a higher quality of the anodes and the product produced. Due to the reactions in the bath, the anodes are worn over time and they have to be replaced at a regular interval. Typically the anodes are 1/3 to 1/4 of their original size when they are replaced. In figure 2.1 a sketch of a prebake electrolysis cell is illustrated and in figure 2.2 an end-by-end potline is shown. The electrolytic cells are connected in series and with modern design they can carry up to $600kA$ (IAI., 2018). Although the cells can vary in size and configuration from plant to plant, the fundamental process is still the same. The line current enters the cell through the bus bars, distributes between the anodes and then leaves the cell through collector bars joined into the cathode carbon lining in the bottom of the cell.

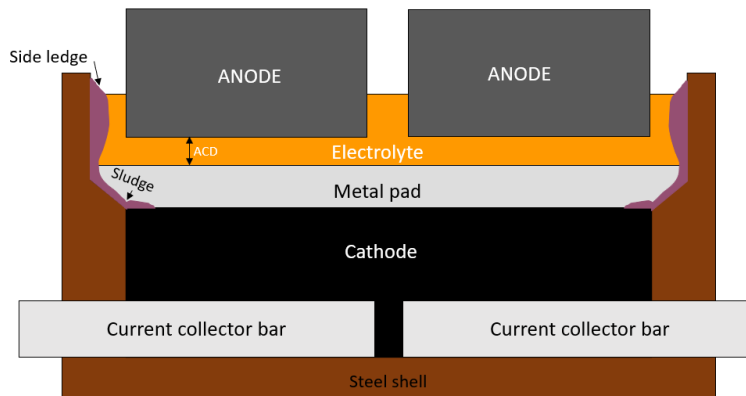


Figure 2.1: Schematic drawing of an aluminium electrolysis cell.



Figure 2.2: End-to-end potline at Alcoa Mosjøen.

2.3 Cell operation

The day to day operation of the electrolysis cell varies. The overall goal is to keep the environment in the cell as close to given parameters as possible. Some operations are done at a regular interval. The main operations are measurements of the electrolyte, changing anodes and metal tapping. Most of the operations are done manually with help of specialized equipment. Cranes or specialized vehicles are mainly used to ensure safe operation when worn anodes are replaced or metal is siphoned from the cell. All operations and additions affect the cell in some way and they will be thoroughly described in section 2.3.2.

2.3.1 Cell performance

Environmental

The environmental aspects of the aluminium electrolysis process can be linked to the cell performance. The formation of undesired gases happens under conditions which lowers the general performance of the cell. In general the pollutants from the aluminium industry is of interest to decrease, not only from an environmental point of view, but also from an economic view. The prebake cells are hooded to increase capturing, recovering and cleaning of the gases from the cells. In addition, the government have environmental standards which sets maximum limits of emissions per kg aluminium produced. If these limits are exceeded there may be fines or other penalties. There also is taxes to be paid to some of the emissions.

Perfluorocarbons are highly potent greenhouse gases (GHG) with long lifetimes in the atmosphere that are emitted from the aluminium smelters. Two of the most common PFC gases are tetrafluoromethane (CF_4) and hexafluoroethane (C_2F_6). Common for all the PFCs are their high global warming potentials and long lifetimes (Åsheim, 2017). Their global warming potentials are about 6000 times higher than CO_2 and the increasing attention these gases have gained the last couple of years are justified. These gases can have a significant impact on the GHG emissions from one plant, and to be able to estimate the total amount of PFC emissions is of great importance.

2.3.2 Production variables

In an ideal process in the cell, as much aluminium would be made with the minimum amount of required energy and raw materials. This is however hard to achieve due to all the different variables contributing to the process. If one of the variables deviate from its optimum value or composition, this would again affect other variables and the overall performance of the cell is lowered.

Line amperage

From a day to day perspective the line amperage is not something that is taken into consideration. Most of the time it is kept at the highest possible value for the given cell design to ensure a high production rate. Although, there are scenarios when the line amperage possibly have to be reduced. This could be because of problems with the alumina feeding

or in worst case a power outage. Also since the cell resistance vary independently, each cell operate at different cell voltages, this again can affect the line amperage at a small time interval if several cells have deviating behaviour, like an anode effect. This is because of the transformer being insufficient to deliver enough power to keep the amperage steady when the cells are outside their normal operating state.

Anode-cathode distance (ACD)

The average distance from the bottom of the anode to the metal pad, the anode to cathode distance, is a variable with significant contribution to the overall cell resistance. Since all the anodes are connected to the anode beam, the ACD can be adjusted as desired to achieve control of the cell resistance. By increasing the ACD, the cell resistance increases and vice versa. Based on this, the ACD should be minimized to keep the resistance at a minimum, but in practise there is a minimum distance that must be kept to avoid short-circuiting between the anodes and the metal surface. In addition the CE increases with increasing ACD, hence there is an optimal ACD that minimizes energy consumption.

Addition of alumina

To keep the production going, alumina have to be added regularly to the bath. For modern aluminium plants, so-called point-feeders are mounted into the cell. This feeder technology keep the rate of alumina steady by automatically adding small amounts of alumina from two to six volumetric feeders into the bath at a specified interval. Since the bath and anodes are covered with a mixture of frozen bath and fine alumina powder, a pneumatic hammer creates a hole in the crust, allowing the alumina to drop easily into the bath. Small additions are preferred due to the increased ability for the alumina to dissolve, mix, and disperse rapidly in the electrolyte (Kvande and Drabløs, 2014). If the dissolution of alumina are insufficient, the alumina sink to the bottom of the cell forming what is called sludge. This sludge leads to increased electrical resistance, and thus increased cell voltage.

The optimal concentration of alumina in the bath is between 1.5-3.5 wt% for modern cells (Whitfield et al., 2004). Actually, the resistance in the bath is a non-linear function of alumina concentration, and it varies with different ACD. If the cell is operated in the higher end, the problem with sludge formation would appear. On the other hand, if the concentration becomes to low, the phenomenon known as anode effect (AE) would occur. The anode effect is further described in section 2.4.

Heat balance

Since the electrolyte (bath) is a highly corrosive liquid it can't be in direct contact with the side walls of the cell. To prevent this, a solid side ledge is made during startup. To maintain this protective layer, control of the superheat is crucial. Superheat is the difference between the bath temperature and the liquidus temperature. The liquidus temperature is the temperature where frozen bath starts melting. In figure 2.3 an illustration of the side ledge is shown. With increasing superheat, the heat loss to the surroundings also increases, causing melting of the side ledge. On the other hand, if the superheat decreases, bath will freeze and the side ledge would increase, reducing heat loss to the surroundings.



Figure 2.3: Different side ledge thickness

Measurements of the bath- temperature and acidity are done manually at a frequent interval, usually once or every other day. For deviating cells even more. Acidity is the concentration of excess aluminium fluoride (AlF_3) to the cryolite composition. These two measurements give useful information about the state of the cell and are closely related to the superheat. High bath temperature could be an indication of a too high energy input to the cell for a given bath composition, while the acidity affects the liquidus temperature of the bath. Except control of the ACD, addition of (AlF_3) is the primary input to control the heat balance. Since the alumina contains Na_2O , some of the aluminium fluoride will react with this, thus reducing the acidity of the bath. Also there are losses due to evaporation and release of carbon fluoride gases. To keep the concentration stable, about 20-30 *kg/day* have to be added to the cell. For modern cells the aluminium fluoride is added similarly as the alumina through point-feeders.

In addition the bath and metal level are of importance to the heat balance. Energy is required to keep the bath in liquidus form and it is of importance to have the desired volume of bath and metal to ensure control of heat leaving the cell. Since the side ledge is constantly changing and evaporation of the electrolyte is present, the bath volume can change over time. If the volume is outside the boundaries, addition or removal of bath might be necessary to keep the bath temperature under control. Keeping control of the metal depth is also necessary to ensure that the amount of metal removed from the cell corresponds to the amount produced for the given period of time.

2.4 Disturbances

Generally all conditions that can bring a cell away from the optimal state can be named a disturbance. The ideal for a cell would be that none of the variables stated in the previous section varies with time, or that replacing anodes and tapping metal would be necessary. Based on this, also the routine work can be seen as a disturbance since it brings the cell outside its optimal operating state. However, there are many conditions that can be named a disturbance and in particular, the anode effect is thoroughly explained due to the fairly new terminology introduced regarding this undesirable state.

Anode effect (AE)

Anode effects have been a topic of extensive study due to the quite harmful fluorocarbon gases which are being produced in this state (Wond et al., 2014). To prevent the anode effect from forming the alumina concentration have to be maintained at a desired level. If too much alumina is added the problem of sludge formation emerges as mentioned previously, but an insufficient amount of dissolved alumina can cause the AE to develop. The wetting of the anode deteriorates with decreasing alumina content, leading to increased gas coverage under the anode (Thonstad et al., 2000). The gas coverage under the anode acts as an insulating layer, causing the resistance underneath the anode to increase which again leads to a massive increase in the cell voltage. The cell voltage could possibly rise abruptly to 20-50 V without any corresponding increase in metal production (Hestetun, 2009). The AE could also lead to over-heating of the cell and to melting of the side-ledge if the duration is long. This type of anode effect is considered to be a *conventional* AE and was the only type known for a long time. However, since the discovery of PFC formation outside the conventional AE at what appears to be normal cell voltage (3.7 - 4.5 V) there was a need for new terminology. (Wond et al., 2014) uses the terms *low voltage propagating* anode effect (LVP-AE) and *non-propagating* anode effect (NP-AE). The LVP-AE is a result from localized AEs which rapidly propagate to a limited number of anodes with the cell still remaining below the conventional AE voltage. The last one, NP-AE is typically the one connected with the continuous formation of PFCs. This type is also named low-voltage anode effect (LV-AE). This continuous formation of PFCs during what appears to be low-voltage is the one of interest for this thesis.

In figure 2.4 the distribution of individual anode currents are shown. Here one of the feeders was closed, thus starving the cell of alumina and leading to the conventional anode effect. Here it can be observed that minutes before the AE occurred, there is a significant drop in the current through at least one of the anodes. This drop leads to higher current through the other anodes, bringing them closer to the critical current density (CCD), and eventually, causing the anode effect to develop on the cell. The parts in figure 2.4 marked with a line is where one of the anodes start to decrease due to a possibly local depletion of alumina. This local depletion of alumina doesn't necessarily lead to an AE, but the formation of PFCs can still be present and will be further investigated.

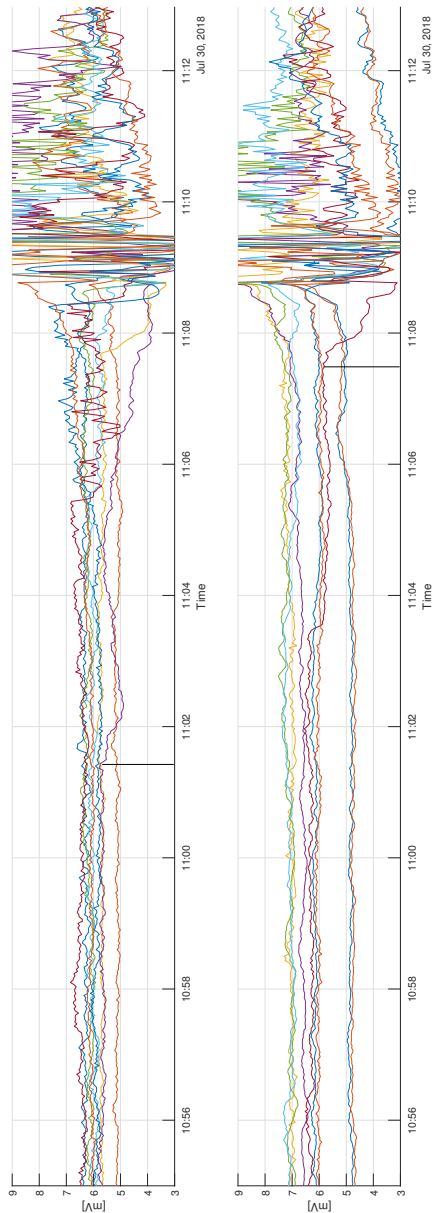


Figure 2.4: Individual anode currents showing initiation of an conventional anode effect.

Experimental

Section 3.1 will cover the different equipment which have been used, while section 3.2 describes the different experiments which has been completed.

3.1 Experiment set up

3.1.1 Anode current measurements

Each of the anode rods on the prebake cells at Alcoa Mosjøen is connected to the bus bar by an individual flexible. By measuring the ohmic voltage drop across the flexible the current passing through each individual anode can be easily monitored. The cells are also equipped with displays with columns of LED bars to represent the current through each individual anode on the cell (Rye et al., 1998). This display gives the operators valuable information regarding the state of the cell and deviating anodes. These anode current readings were not automatically stored, so to record the anode current measurements, a multi-channel logger had to be connected to the measuring device recording the voltage signals to the LED's on the cell. This logger gives the possibility to sample data down to 1 sec.

3.1.2 Quantum cascade laser

The QCL is a LaserGas™ Q CF4 delivered by Neo Monitors. The QCL was installed on the exhaust duct of a single aluminium electrolysis cell so that continuous concentration measurements of CF_4 from the cell could be logged. In figure 3.1 and 3.2 the QCL mounted to the duct is shown. The installation of the transmitter and detector was done by securing the equipment onto mounted flanges on the exhaust duct. The tuning of the QCL to obtain the required transmission was done directly by adjusting screws on the flanges. In the QCL configuration software the light path was set equal to $0.4m$ and the temperature was set to $125\text{ }^{\circ}C$. The temperature measurement of the gas was done by use of a thermocouple. Since these two parameters were fixed, this is a possible source of

concentration offset for the QCL. The ideal would have been to feed the instrument with temperature and pressure values directly from transducers so the software could calculate the concentration based on these parameters directly.



Figure 3.1: LaserGas™ Q CF4 laser head.



Figure 3.2: LaserGas™ Q CF4 detector.

3.2 Field test at Alcoa potroom

Since the PFC emissions from non-propagating or low-voltage anode effect appear to be connected to typical activities in the potrooms, such as changing of anodes, metal tapping, troubled pots and other occasions that may could induce a localized increase in anode current density or a local depletion of alumina in one part of the pot, these are the type of events that should be investigated. For this part the following events have been studied:

Smaller ACD for a single anode

The impact of lowering one of the anodes by 2.0cm . This should increase the current density through the lowered anode.

Closed feeder

To see how an individual point-feeder of alumina contributes to the current distribution. The feed-rate to the cell was manipulated and the current through each anode was monitored. The manipulation of feeding consisted of closing one individual feeder, and hence under-feeding the cell. The feeding was taken back to normal operation when an conventional anode effect occurred.

Anode change

When new anodes are placed in the cell the current distribution change. This change in current distribution through the anodes are because of the freeze that forms under the new anodes. The newly set anodes need hours to warm up before they are starting to draw current. This leads to a non-uniform current distribution for the rest of the anodes in the cell.

Results and discussion

4.1 Smaller ACD for a single anode

In figure 4.1 anode 6 was lowered about 2.0cm at 19:13 bringing it from about 5.5mV to 7.2mV and there is an immediately response in the concentration of CF_4 as seen in figure 4.2. The increase in CF_4 concentration is from about 70ppb before it approximately settles at 300ppb after 4 hours. The anode was kept at this level until 07:55 the next day as seen in figure 4.3. When the anode was taken back to its original reference, the CF_4 concentration decreased rapidly the next minutes before settling at around 10ppb .

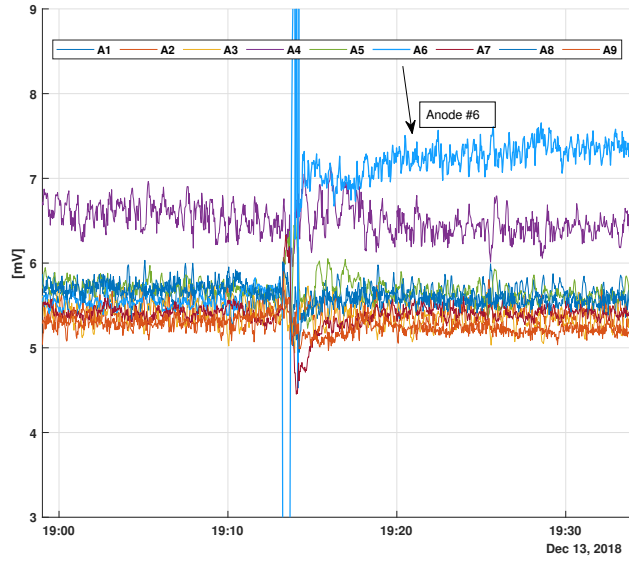


Figure 4.1: Current distribution when lowering single anode (anode #6).

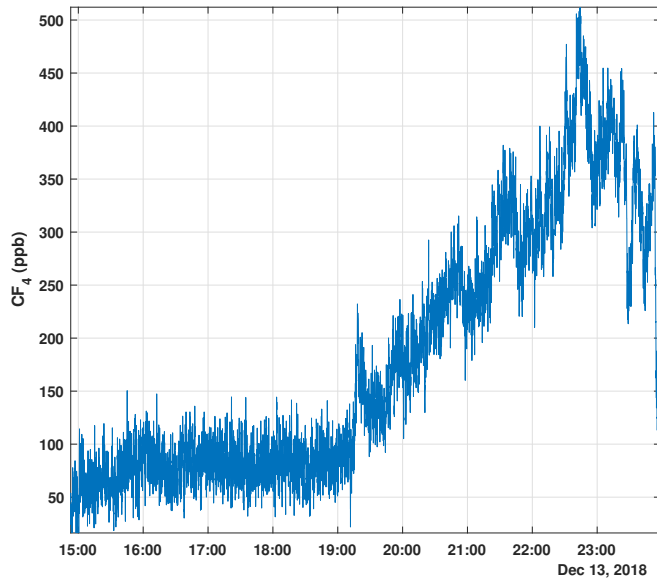


Figure 4.2: Response of CF_4 concentration when lowering single anode (anode #6).

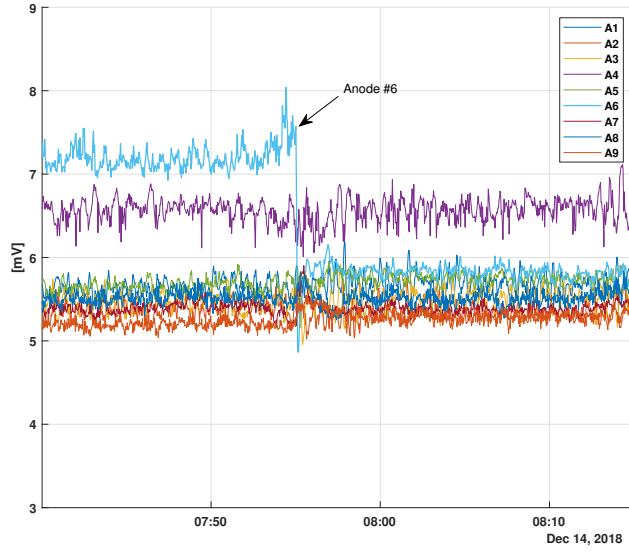


Figure 4.3: Current distribution when anode is placed at correct reference (anode #6).

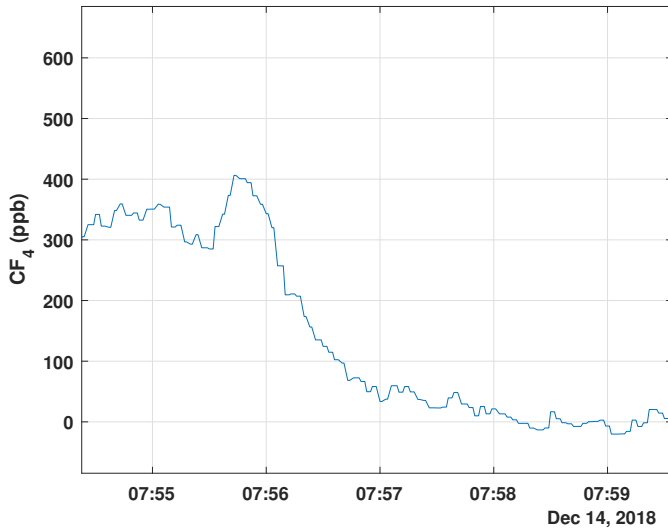


Figure 4.4: Response of CF_4 concentration when anode is placed at correct reference (anode #6).

4.2 Closed feeder

To provoke an anode effect the feeder on the front side of the cell was closed. The pot would eventually get starved of alumina and the anode effect would appear. Since the feeder on the front of the cell was closed, where the anodes ranging from 1 to 9 are present, it was expected that one of these anodes should have a decrease in the current distribution prior to the anode effect. In figure 4.5 it can be observed that anode 2 have a drop at 22:23 which is an indication of the onset of a anode effect. In figure 4.6 at the same time the concentration of CF_4 is increasing.

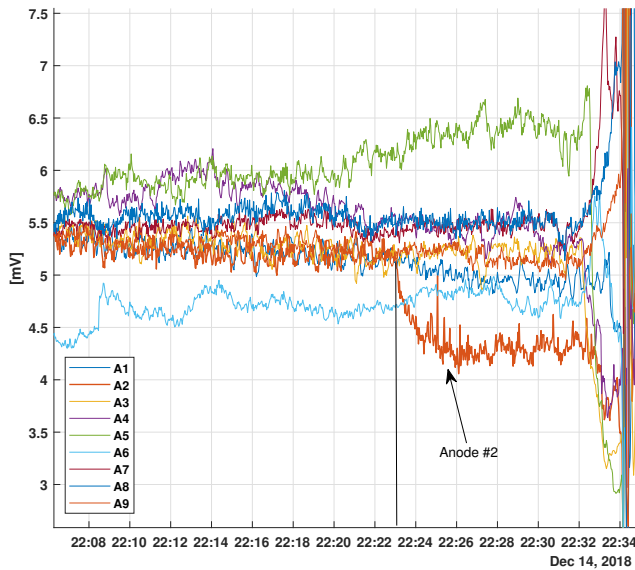


Figure 4.5: Current distribution when one feeder is closed.

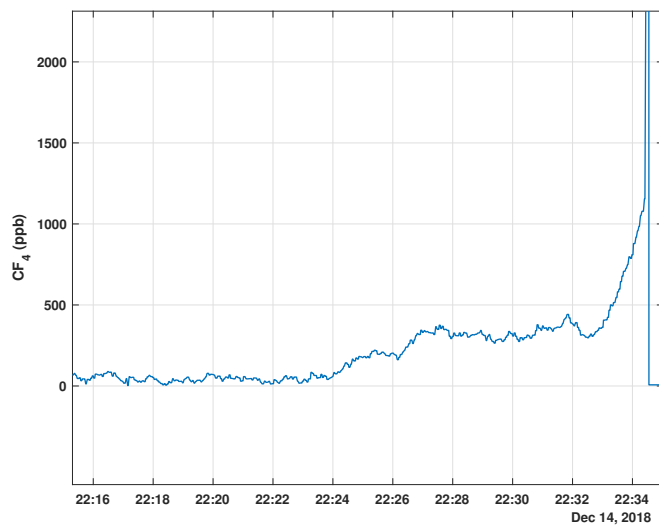


Figure 4.6: Response of CF_4 concentration when one feeder is closed.

4.3 Anode change

In figure 4.7 the current distribution when anode 10 and anode 11 are replaced can be observed. After the anode change the potential off all the other anodes are increased, and in figure 4.8 and figure 4.9 the CF_4 concentration can be observed. The concentration increases from about 20ppb to 200ppb after the anode have been changed and the cell have settled at the higher potential.

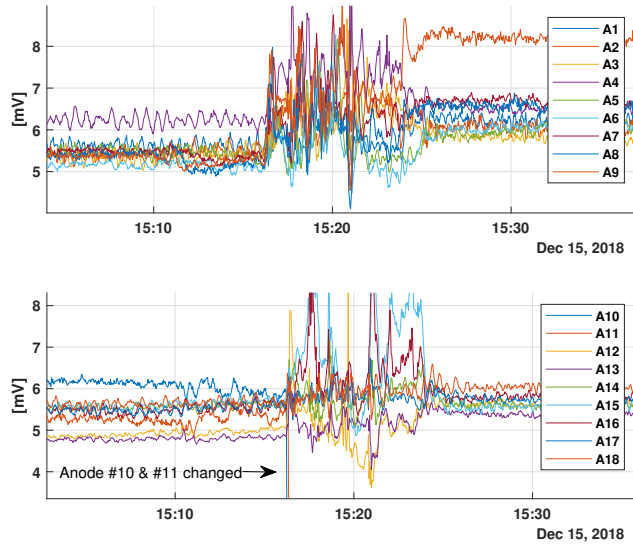


Figure 4.7: Current distribution during change of anode #10 and anode #11.

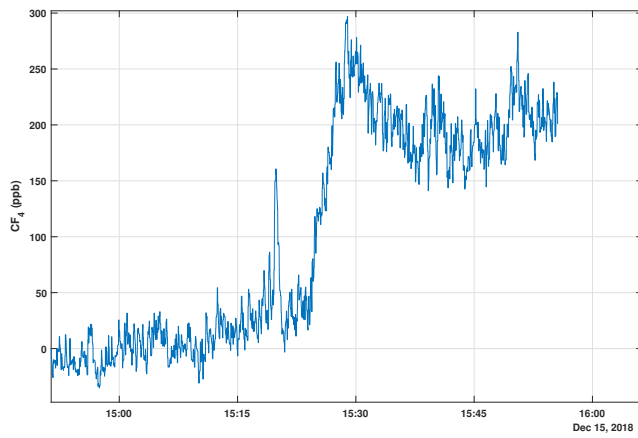


Figure 4.8: Response of CF_4 concentration during anode change.

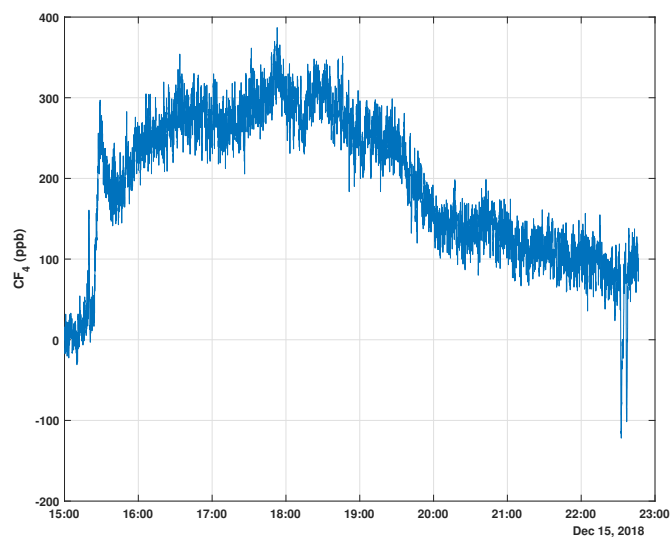


Figure 4.9: Response of CF_4 concentration during change of anode #10 and anode #11.

Conclusion and further work

This simple study has shown that co-evolution of PFC gases are present when introducing a change in anode current density or alumina concentration in a prebake cell operated at $175kA$. From the result it appears that the formation of PFCs are highly sensitive to imbalances in anode current but also to starving of alumina in the cell. This indicates that the model used for estimating PFC emissions based on only the anode effect is outdated and a new method should be developed in order to handle smelters with this specific technology.

For future work, a more detailed plan for new experiments should be developed. Clearly the individual current distribution in partnership with the QCL can be used as a tool to early detect formation of an anode effect. To make use of this information, algorithms to detect this state on-line should be developed. In addition this equipment can be of help to get information of how the alumina distribution in the cell are at a given time. This could help prevent sludge formation in the bottom of the cell, giving a more efficient production.

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