

CONTINUOUS PFC EMISSIONS MEASURED ON INDIVIDUAL 400KA CELLS

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Abstract

PFC emissions from the primary aluminium industry have traditionally been linked to anode effects (AE), which are most broadly defined as when the voltage on a cell exceeds 8 Volts for more than 3 seconds. Recent studies however have revealed the existence of continuously generated PFCs, which are not related to a conventionally defined AE. In a study focusing on individual 400kA cells, the continuous generation of PFCs was measured on several pots, was found to vary from one cell to another, and to vary within a number of hours. Generation of these PFCs also varied from one localised region to another within a cell and may be linked to instabilities in local current distribution, as suggested by 20Hz continuous monitoring of individual anode currents. While the study provides further understanding on the characteristics of these PFCs, further work is required to determine the fundamental causes of this emission.

Introduction

Perfluorocarbon (PFC) emissions are a considerable greenhouse gas emission from the primary aluminium smelting industry. The two forms of PFC commonly found in the smelting process are tetrafluoromethane (CF₄) and hexafluoroethane (C₂F₆). Both PFC gases have very long atmospheric lifetimes (50,000 and 10,000 years, respectively) and very high global warming potentials, i.e. 7,390 and 12,200 times that of carbon dioxide (CO₂), respectively [1]. As such, the industry has made considerable efforts to understand and mitigate the mechanisms behind PFC generation, resulting in numerous studies on the subject [2, 3].

Traditionally, PFCs have been directly linked to ‘anode effects’ (AE). These occur when: (i) dissolved alumina levels in the molten electrolyte become low/depleted (e.g. below 2% wt) and (ii) current draw on one or more anodes exceeds a certain critical current density [4, 5]. At these conditions, normal electrolysis ceases and cryolite is decomposed at anode surfaces in favour of alumina, forming CF₄ and C₂F₆ gases. These PFCs wet anode surfaces and form an insulating gas film, forcing current flow to shift to adjacent anodes. Soon after, these adjacent anodes also reach critical current density and PFC generation rapidly propagates to all remaining anodes in the cell [2].

PFC emissions continue to be generated until the AE is ‘terminated’. This requires a replenishment of alumina to the bath and dispelling of PFC gas films by short-circuiting anodes with the metal pad, either through *manual* (with a wooden pole) or *automatic* (by lowering the anode beam) means [2, 5]. Because of the direct link between AEs and PFC emissions, the industry has placed considerable focus and effort towards minimizing both the frequency and duration of AEs. Since AEs are often characterized by an exponential increase in cell voltage, often from 4-5V up to 20-50V [3], the most commonly accepted definition for an anode

effect is when cell voltage *exceeds 8V for more than 3 consecutive seconds* [6]. This definition forms the basis of AE frequency (AEF) and duration (AED) statistics, which are often used to estimate total PFC emissions for a smelter site.

In the past, AEs have been widely viewed as the only dominant mechanism of PFC generation. However in recent years, PFC generation in the *absence* of a conventionally-defined AE have been reported in operating smelters, most notably in studies of Chinese facilities by Li *et al.* [7, 8] and Marks and Bayliss [9]. As such, these PFCs have been termed as *non-AE (NAE) PFCs*. Due to their appearance in measurements as low level, continuous background signals, they are also known as *background or continuous PFCs*.

The contribution of NAE-PFCs to total PFCs appears to vary considerably from one smelter to another, from practically zero to as high as 92% [8]. While smelters in China appear to be particularly affected by NAE-PFCs, these emissions are by no means limited to the Chinese smelting community. In a recent survey of 13 Chinese and 17 non-Chinese facilities [9], NAE-PFCs were attributed to a median contribution of 70% and 22% of total CF₄ emissions respectively, the remainder being attributed to anode effects. Clearly, this new emission pathway cannot be ignored nor discounted. While work to reduce AE frequencies and durations must continue in order to reduce AE-related PFCs, smelters with significant NAE emissions will *not* be able to reduce their total PFC burden unless NAE components are also mitigated. To do this, we must first understand the mechanisms by which NAE emissions are generated.

The objective of this current work was therefore to determine some of the causes or characteristics of NAE or continuous PFC emissions, at the *individual cell* level. Previous studies have largely focused on emissions at scrubber inlets, which are contributed by large numbers of cells. Li *et al.* [7, 8] have conducted single cell studies, involving anode current distributions (discrete mV ‘fork’ measurements, not continuous) alumina concentrations, bath temperatures and feeding cycles as factors for NAE-PFC generation; however, such studies have so far been inconclusive.

Hypothesis

It has been well documented that PFC generation occurs locally on individual anodes *prior* to the onset of an AE, resulting in a redistribution of local anode currents [5]. The current study was therefore framed around a similar hypothesis, i.e. that NAE-PFCs are generated by a process of *localised* PFC generation under *individual* anodes in specific reduction cells. To test this, the study measured PFCs from individual cell exhausts at a Chinese 400kA technology smelter, simultaneously with monitoring of individual anode currents.

Two pathways for continuous PFC generation were suggested, i.e.: (a) PFCs are generated continuously under individual anodes but remain localised (i.e. do *not* propagate to other anodes, which would result in an anode effect); or alternatively, (b) PFCs are generated intermittently under individual anodes, but cycle continuously from being generated, to being dispelled under anodes, to being generated again.

The first pathway (PFCs not propagating to other anodes) is more likely to occur in large cell technologies, with numerous anodes and low anode current densities (less tendency to hit critical current density, resulting in PFC propagation and AEs) [10-12]. These are characteristic of modern high amperage Chinese cell technologies and may explain why NAE contributions appear to be higher in Chinese facilities. Many of these large cell technologies are also characterized by low ACD operations. This can result in difficulties transporting alumina around the cell which in turn promotes localised PFC generation.

Differences in bath chemistry might be another factor behind the higher levels of NAE-PFCs found in China. LiF, MgF₂, KF and other bath modifiers are becoming increasingly common in Chinese bath chemistries, often as a result of alumina impurity inputs than as controlled additions to the process [13]. Compared to conventional bath, highly-modified baths have lower alumina solubilities [13], which further promote the generation of PFCs.

Methodology

Monitoring of PFC Emissions

PFC gases CF₄ and C₂F₆ were measured from cell exhausts using a *Temet DX4000* Fourier Transfer Infrared (FTIR) spectrometer (sample cell with path length of 9.8 m, volume of 0.5 L, resolution 7.8 cm⁻¹). Sample gases were pumped continuously from (i) a stainless steel (SS) probe fitted with a 15 μm pore size fritted SS filter, inserted into the exhaust header of individual cells, then (ii) through 3/8" PFA grade Teflon transfer tubing to (iii) another 15 μm fritted SS filter, then (iv) through gas scrubbers and (v) a rotometer to monitor sample gas flow, and then (vi) to the heated gas sampling pump. The sample gas stream was scrubbed with Drierite® to remove the majority of water and with activated alumina to remove any HF. The water was removed for a more unambiguous visual identification of compounds from infrared spectral features and the HF was removed because of its high corrosiveness. After exiting the pump the gas entered a 1 m controlled temperature transfer line before entering the FTIR sample chamber. The gas sampling pump, transfer line and gas sampling cell were all maintained at 120 ± 0.5 °C. Complete spectral scans were measured at the rate of 10 scans per second. Measurements were then averaged over 5 s increments for the majority of data collection. The flow rate of the gas through the cell was approximately 2 L/min. The typical composition of the measured smelter exhaust gas is shown in Table 1.

Gas components that have spectral interferences with CF₄ and C₂F₆ must be measured for the Calcmet calculation software to accurately correct the gathered spectra and calculate the concentration of the two target compounds, CF₄ and C₂F₆. Specific interferences for which corrections were applied for CF₄ included water, carbon dioxide, methane, sulphur dioxide and hexafluoroethane. For C₂F₆ spectral corrections were included for water, carbon dioxide, sulphur dioxide and tetrafluoromethane.

Table 1 - Typical smelter exhaust gas content.

Compound	Typical Concentration
H ₂ O	0.1 - 5 % vol
CO ₂	0.35 - 1.5 % vol
CO	0.035 - 0.15% vol
CH ₄	1 - 5 ppmv
SO ₂	15 - 250 ppmv
COS	0.5 - 15 ppmv
CF ₄	<0.01 - 150 ppmv
C ₂ F ₆	<0.01 - 3.5 ppmv

Monitoring of Anode Currents

PFC measurements were conducted alongside a continuous, high frequency monitoring of individual anode currents. This was to determine whether continuous PFCs were related to fast moving, localised phenomena on individual anodes.

Current draws on 48 anodes were measured using DC-voltage drop 'fork' probes (150mm between forks); these were fixed to anode rods below the anode beams and above pot hooding. Voltage drop signals (in the range of 0-10 mV) were then wired to a data acquisition (DAQ) box, consisting of (i) *Dataforth 8B* conditioners for electrical isolation and 500x signal amplification and (ii) a National Instruments *cRIO-9076* controller (with *NI 9205* analogue input modules) providing high frequency data acquisition (at 20 samples/s) and storage. Average cell voltage was also measured with the DAQ system.

Single Cell Studies

Single cell studies with monitoring of PFCs and anode currents were conducted on three individual cells (37, 39, 41) over 12 to 72 hours. Auxiliary measurements (bath temperature and chemistry, metal/bath levels) and observations (alumina feeder condition, anode age) were also taken for each pot. Short surveys of continuous PFCs across multiple cells were also made.

Results & Discussion

Continuous PFCs Vary from One Pot to Another

Surveys of continuous or NAE-PFCs were made by moving the emission sampling probe from cell to cell to determine the variability of emissions among cells. The results of this are shown in Figure 1 and demonstrate that for five cells, CF₄ concentrations varied from below instrument detection limits (<0.01 ppmv), to as much as 0.65 ppmv. These were all NAE-PFCs as no AEs were

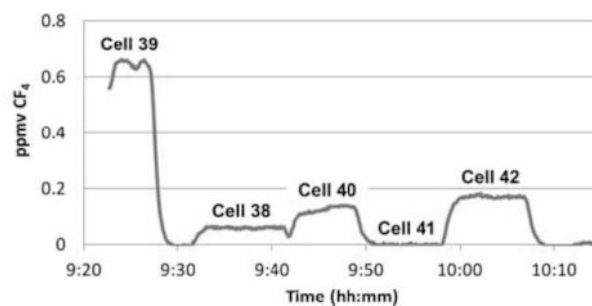


Figure 1 – NAE-PFC emissions (levels ppmv CF₄) from a survey of five cells in series; note the dips in CF₄ denote the switching of the sampling probe from one cell to another.

detected on any of these pots during the survey. The survey confirmed that at any point in time, NAE-PFCs vary from one pot to another, i.e. some cells are high emitters (e.g. pot 39), while others are low/non-emitters (e.g. cell 41). As such, the majority of continuous or NAE-PFC emissions from a potline may well originate from only a small number of cells at any one time.

Emission Signatures: AE-related vs. Continuous PFCs

As noted in previous studies [8], continuous PFCs display an emission signature that is very distinct from AE-related PFCs. Figure 2 illustrates this contrast. PFCs related to anode effects are characterized by a rapid rise in PFCs, i.e. sharp emission ‘peaks’, such as the one occurring at 16:43 hours on pot 39 (Figure 2-A). This corresponds with the rapid rise in average cell voltage on pot 39 (Figure 2-B). Note that the time-delay between voltage and PFC signals for the AE represents the time required for sampled gas to travel from the cell to the FTIR. Anode effects are defined by a sharp, sudden rise in cell voltage above a ‘trigger’ voltage. While 8 V (for ≥ 3 s) is the most commonly used ‘trigger’ for an AE, this may vary at local facilities.

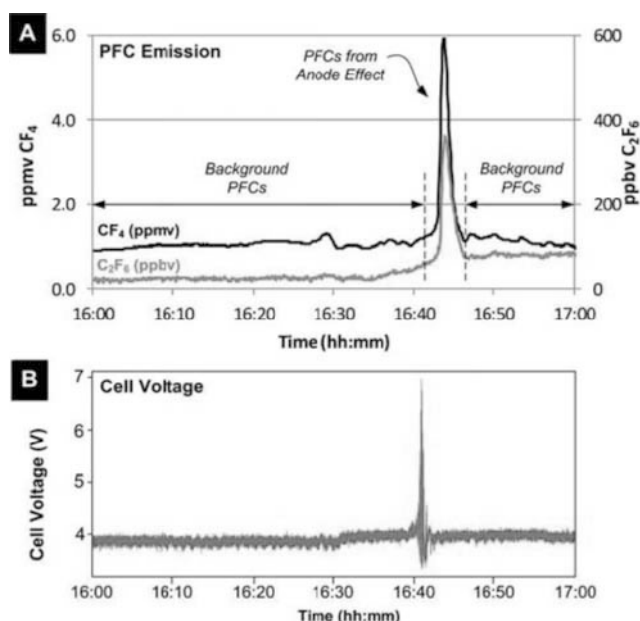


Figure 2 – [A] Contrast in emission signatures for anode effect-related PFCs vs. background/continuous PFCs on one cell, and [B] corresponding cell voltage on pot 39.

In the example shown in Figure 2-B, it is highlighted that cell voltage *never* exceeds the 8 V threshold (maximum of 7 V from 20 Hz measurements); therefore, an anode effect is never officially ‘declared’. However, for all intensive purposes (judging by the PFC emission signature), the event is an ‘anode effect’. A closer look at anode currents (shown later in Figure 5) confirmed that PFC propagation in the cell did occur, being initiated at 1-2 under-loading anodes (as described in [2]) and then spreading to perhaps two-thirds of anodes in the cell. However, PFCs did not appear to propagate across the entire cell, i.e. the AE remained localised, and may explain why cell voltage never exceeded 8 V.

After the ‘localised AE’ was terminated, cell voltage returned to normal and PFC emissions returned to background levels. Figure 2-A shows that this background level is *not* zero, but is typically a

small fraction of the emission rate at anode effect. Figure 2-A also illustrates that the background emissions contain both CF₄ and C₂F₆. These represent the typical emission signature for continuous PFCs.

The example in Figure 2 raises two further points of interest. Firstly, the ‘AE’ self-terminated, i.e. *without* manual or automatic intervention (AE was never officially declared). This might point to the generation of metal pad waving/instability (from anode current imbalances during the AE) that act to dispel PFCs from anode surfaces, thereby terminating the AE. Similar short-lived, ‘self-terminating’ AEs (durations <10-15 s) represented a large fraction of officially declared AEs (using an 8 V trigger voltage) on this cell technology; such phenomena might also be observed in other high amperage cell technologies. Secondly, low-voltage localised AEs (e.g. the AE in Figure 2) are not officially ‘declared’ and therefore are not included in AEF and AED statistics; this can result in an under-accounting of AE-related PFCs from a smelter site.

Continuous PFCs Vary with Time for Each Pot

Another characteristic of continuous PFCs is that they vary *with time* for each pot. This is illustrated in Figure 3, showing the variation in CF₄ and C₂F₆ emissions over a 24 hour period from ‘high emitting’ pot 39. Relevant pot events (anode change, metal tap, two ‘localised’ AEs) are also shown on the diagram. Two periods of emissions (as labelled in Figure 3) are discussed.

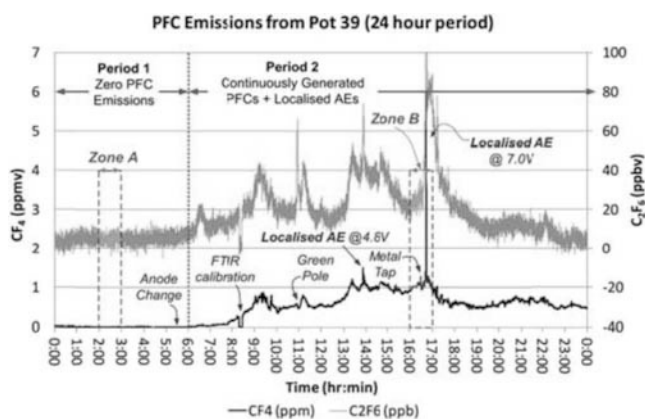


Figure 3 – PFC emissions over 24 hours on one cell, with relevant pot events and 1 hour periods of interest (Zone A and B) labelled.

Period 1: The 24 hour emission trace shows that at the start of the day (0:00-06:00 hours), PFC emissions were essentially zero or below detection limits. A 1 hour period of interest (Zone A) is highlighted for further analysis with anode current data.

Period 2: From 06:30 hours onwards, i.e. approx. 1 hour after anode change, continuous PFCs rose slowly from zero to elevated levels (as much as 1 ppmv CF₄), and remained elevated over the rest of the day. Generation of continuous PFCs therefore appear to be correlated with anode changes (associated effects on anode current draw are discussed later). It is worthwhile to note that there were no anodes changed on this pot the previous day.

During the rise in continuous PFC levels, an FTIR calibration was carried out at 08:20 hours (i.e. purging the sampling chamber with clean gas, as shown by CF₄ and C₂F₆ levels dropping to zero),

confirming that measured background PFC levels were valid. Several hours later (at 10:50 hours), an attempt was made to dispel continuous PFCs from anodes by introducing wooden 'green' poles into the tap and duct ends of the cell, i.e. to simulate a manual AE termination; however this had practically no effect on reducing continuous PFC levels.

Two localised AEs were detected at 13:50 and 16:43 hours (the second was presented in Figure 2). In both cases, AEs were not officially declared (max cell voltages of 4.8 and 7.0 V, respectively) and 'self-termination' of these AEs did not reduce background PFCs to zero. Classical understanding of AE termination is that metal pad shorting removes/disperses PFC films adhering to anodes, allowing the cell to return to normal electrolysis and stopping further PFC generation [2]. However in this case, the termination of AEs does *not* stop continuous PFC generation. Indeed, continuous emissions of PFCs continued until 8:00 hours the following day (not shown), when levels dropped back down to zero.

Over the 24 hours shown in Figure 3, the ratio of C_2F_6 to CF_4 varied from non-measurable levels of C_2F_6 , up to a ratio of about 12 wt% at the peaks of CF_4 emission. A 12 wt% ratio of C_2F_6/CF_4 is typical for classical PFPB anode effects.

Link between Continuous PFCs vs. Anode Currents

To determine whether continuous PFCs were related to issues with localised anode current draw, two 1 hour periods of interest –

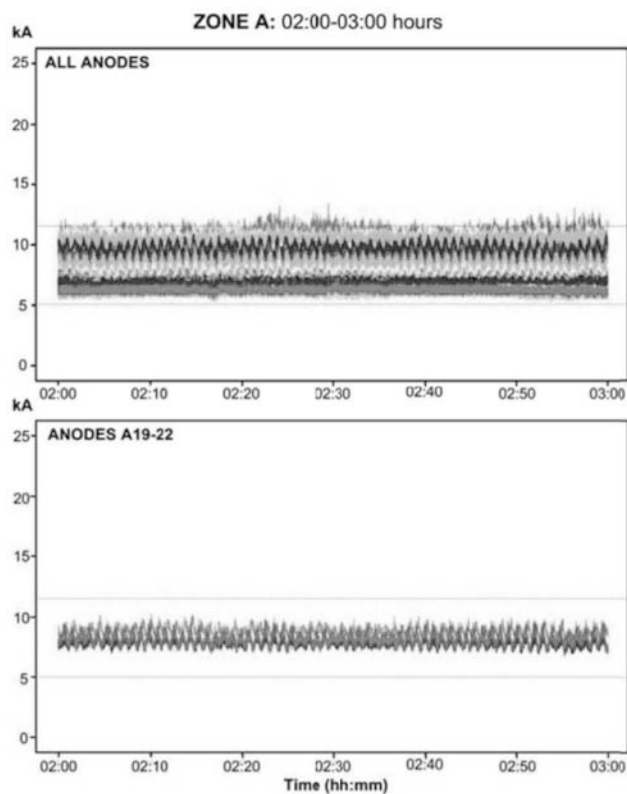


Figure 4 – Anode current distributions for [Top] all anodes and [Bottom] only anodes A19-22 on pot 39, during Zone A (zero continuous PFC emissions).

Zones A and B from Figure 3 – were selected for further analysis with anode current data. *Zone A* (2:00-3:00 hours) represented a period where continuous PFC levels were zero, whereas *Zone B* (16:00-17:00 hours) represented a period of high background PFCs and a localised AE occurring at 16:40 hours.

Anode currents for Zones A and B are illustrated in Figure 4 and Figure 5, respectively. Each figure shows current draw for all anodes in the top graph, as denoted by different coloured lines; a close up of four specific anodes (anodes A19-22) is shown in the bottom graph. Dotted lines at 5 kA and 12 kA represent 60% and 140% of average anode current loading.

During Zone A (Figure 3), background PFC levels were essentially zero. This corresponded with fairly stable anode currents throughout the whole cell (Figure 4), with all anodes within the 60-140% current draw range. It is again noted that no anodes were changed the previous day.

At 05:30 hours (Figure 3), two anodes A19-20 were changed. New anodes generally take up to 24 hours to reach thermal equilibrium with the cell and draw their full share of electrical current. As such, anodes adjacent to those newly changed (anodes A21-22) increased significantly in current draw, picking up the 'slack' in current (current draws immediately after anode change are not shown, but are similar to those represented in Figure 5). However, continuous PFC generation does not occur until 1 hour after the anode change, i.e. at 06:30 hours. This corresponds with the removal of temporary voltage additions for anode change, i.e.

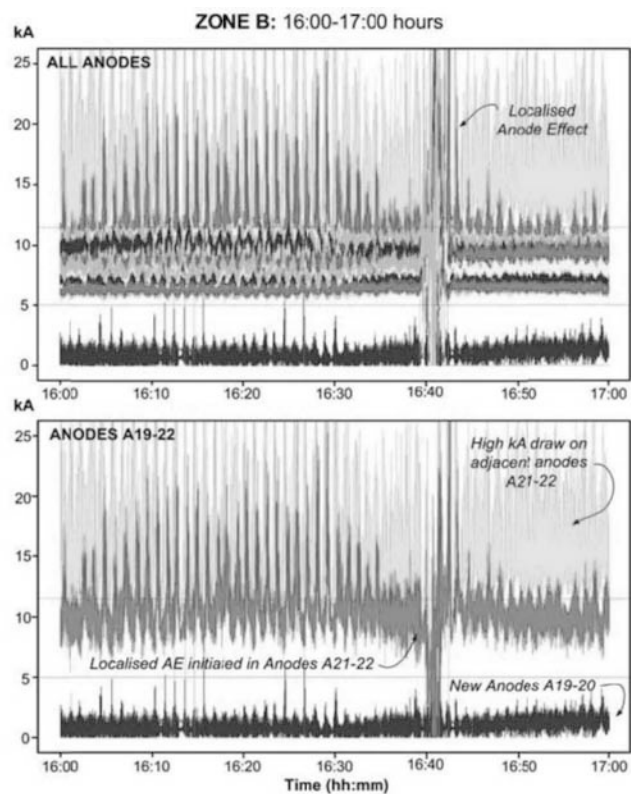


Figure 5 – Anode current distributions for [Top] all anodes and [Bottom] only anodes A19-22 on pot 39, during Zone B (high background PFCs and localised AE at 16:40).

a reduction in ACD, which hinders alumina transport. It is only after this point that continuous PFCs start being generated, ramping up to significant levels over the course of the day.

At Zone B (Figure 3), representing 10-11 hours after the anode change, continuous PFCs are close to their highest background levels for the day (~1 ppmv CF₄). As illustrated in Figure 5, the two high current draw anodes A21-22 (adjacent to new anodes) are still drawing significant levels of current, i.e. at an average of 170% of 130% of normal current, respectively. As high anode current densities increase the propensity for PFC generation, it is suspected that these anodes are those responsible for the majority of continuous PFCs measured in the 24 hour period of study.

The two high current drawing anodes (A21-22) are also the ones responsible for initiating the localised AE at 16:40 hours (Figure 2 and Figure 3). This is indicated by the 'under-loading' of current on anodes A21-22 perhaps 1-2 min prior to the anode effect, due to increased PFC generation and gas film adhering on anode surfaces.

During the localised AE, anode current instabilities were limited to anodes towards the duct end (where anodes A21-22 were situated); anodes towards the tap end registered only minor instabilities in current draw. This indicates that PFC generation did *not* propagate to the entire cell but remained localised. This also explains why maximum voltages in these AEs can be lower than the conventional 8 V 'trigger' used for declaring an AE.

After the AE (after 16:42 hours), high current draw on anodes A21-22 continue. This correlates with the ongoing generation of background PFCs. Individual anode current signatures (Figure 5) during AEs vs. non-AE periods correlate well to the differences in emission signatures (Figure 2) for AE-related PFCs vs. continuous PFCs.

Regular cycles or fluctuations in anode current draw – at frequencies of approx. 1 min – were another visible feature from anode current monitoring. Some fluctuations were initially observed on some anodes during Zone A (Figure 4, zero PFCs), but increased significantly in amplitude after the anode change, i.e. as illustrated during Zone B particularly on anodes A21-22 (Figure 5, high background PFC levels). Fluctuations may relate to metal pad waving under these anodes. It is suggested that continuous PFCs could have been generated intermittently on problem anodes (anodes A21-22), during periodic spikes in current draw, i.e. periodically exceeding critical current density but not at the point at which PFCs propagate to multiple anodes. In the example presented in Figure 5, anode A21 (closest to the new anodes) periodically peaks at 300% of normal current draw.

During tests on another cell (low emitting pot 41), similar magnitudes of anode current instabilities (as was found in pot 39 above) were also observed. Despite these 'symptoms' however, background PFC levels were *not* measured on this pot. This suggests that while continuous PFCs might be related to high or unstable anode current draws under specific anodes, there are other factors that contribute to the continuous PFC phenomenon. Nevertheless, the above findings on pot 39 do provide evidence that continuous PFCs are indeed a localised pot phenomenon. Anode changes appear to be one event that can initiate continuous emissions of PFC.

Continuous PFCs are a Localised Phenomena

A survey of PFC emissions from each of six feeders was conducted on one cell (high emitting pot 39), during a period when background or NAE-emissions were measured in the exhaust gas. The sampling probe was placed in the cell above feeder holes for 5-10 min, and then moved from one feeder location to another. Average CF₄ levels at each feeder and corresponding feeder conditions are shown in Figure 6.

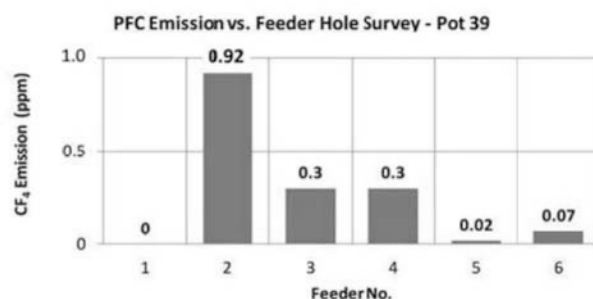


Figure 6 – Measured CF₄ emissions at six feeder hole positions on cell 39.

The majority of continuous PFCs from the cell were found to originate from one feeder hole, i.e. Feeder #2, with CF₄ levels up to 0.92 ppmv. Lower levels of PFCs were measured at Feeders #3 and 4 (each <0.3 ppmv CF₄), whereas PFC levels were zero or very low on the remaining three feeder holes.

High PFC generation at Feeder #2 might have been due to a number of factors. During the survey, it was observed that Feeder hole #2 was blocked; this may impede the transport of alumina into the bath, resulting in depletion of alumina concentrations locally in the bath. Furthermore, there were two newly changed anodes next to Feeder #2. As a result, older, adjacent anodes may have been experiencing high current densities, as was demonstrated in the previous example. Both factors would increase the propensity for PFC generation.

The feeder survey therefore confirmed that generation of continuous PFCs is a *localised phenomena* and indicated that the factors that govern continuous PFCs are likely to be similar to those governing classical AEs, i.e. low alumina concentrations and high current densities (exceeding critical current density) on specific anodes.

Correlations between PFCs and Other Factors

Comparisons were made between high emitting vs. low emitting cells (e.g. pot 39 vs. 41) in the attempt to discern any other factors behind continuous PFCs. However, no obvious correlations could be found (with respect to bath temperature, chemistry and alumina content). There remains much scope for further investigation.

Cursory examination of daily bath samples showed that total alumina content varied from 1.5-2.1% wt (LECO), with no particular trend between high and low emitting pots. Furthermore, bath samples from both cells contained LiF and KF modifiers (both in the range of 1.5-2.0% wt, from XRD); MgF₂ was not detected but was thought to be below levels detectable under XRD (<2% wt). As discussed previously, the use of highly

modified baths are suspected to be another factor behind the generation of continuous PFCs.

Conclusions

The current study determined that continuous or NAE-PFCs vary from one cell to another, and vary within a number of hours or days. Continuous PFCs consist of both CF₄ and C₂F₆ and have a distinct emission signatures, appearing as background levels as opposed to emission spikes for AEs. The mechanism of continuous or NAE-PFC generation differs from AEs in that they are not stopped after AE termination. More importantly, the study determined that continuous PFCs are localised phenomenon, being generated at specific anodes within a pot, particularly those with disproportionately high current draw and especially after anode changes. The study also observed another category of AEs, i.e. 'localised AEs' with low AE voltages (<8V), where PFC propagation is limited to a local pot region.

This work has therefore provided an initial understanding on the characteristics and underlying factors for continuous PFCs. However, there is still much work to be done to understand the fundamental mechanisms behind continuous PFCs. Such an understanding is essential if the smelting industry is to make further reductions in total PFC emissions.

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