

## Observations on the Coke Air Reactivity Test

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Keywords: Air Reactivity, Desulfurization, Calcined Coke

### Abstract

The ISO Coke Air Reactivity test has been used by the smelting industry for many years but its use as a calcined coke specification is decreasing. This paper presents a review of previous work published on this test and presents experimental data on a wide range of cokes currently being used for anode production. Coke air reactivities are strongly dependent on coke calcination levels and it is possible to drive air reactivities lower by increasing calcining temperatures. With the general increase in sulfur level of high sulfur cokes used in anode coke blends, higher calcining levels are not desirable due to their negative influence on coke porosity as a result of thermal desulfurization. Many smelters are now adopting lower real density specifications, which runs counter to achieving the low coke air reactivities required to meet coke air reactivity specifications.

### Introduction

The air reactivity of calcined coke as determined by the ISO 12982 method [1,2] is a calculated result based on the ignition temperature of the coke. A coke sample prepared to 12x16 mesh (1.4-1.0 mm) is heated in air at a rate of 0.5°C/min or 10°C/min until the coke ignites which causes a sudden and measurable increase in temperature. A calibration curve based on experimentally derived data calculates the air reactivity based on the ignition temperature.

Coke air reactivity was a widely specified property by the aluminium industry for many years beginning in the early 1990's. It was considered an important property for optimizing calciner operation and as a predictor of the airburn performance of baked anodes [1,3]. This situation has changed significantly and most smelters have now abandoned coke air reactivity specifications.

There are many reasons why coke air reactivity is no longer used as a specification and the objective of this paper is to review previous work published on the air reactivity test and present data which shows the strong correlation between coke air reactivity and calcination level. For most cokes, it is possible to drive coke air reactivities lower by increasing calcination temperatures.

Increasing calcination temperatures can be counter-productive to coke quality and anode performance due to thermal desulfurization problems [3,4]. As the sulfur level of high sulfur cokes continues to increase, the impact of this problem becomes more pronounced and it is one of drivers for the trend towards the use of under-calcined coke [5,6,7,8]. The adoption of lower real density specifications for under-calcined coke runs counter to meeting historical coke air reactivity specifications.

### Lab-Calcined Data

Rain CII used over 10 different sources of green coke in the US for anode blends in 2012. Different cokes are used at different calciners based on the proximity of the calciner to the refinery and the typical number of cokes used in a blend is 3-5 but it can be

higher or lower depending on coke availability and quality. The range of qualities is large has been reviewed previously [9].

Coke air reactivities have been measured on most of these cokes through laboratory calcination trials and in many cases, through full scale kiln trials. The agreement between the two is generally good and laboratory calcination allows air reactivities and other properties to be measured rapidly over a wide temperature range.

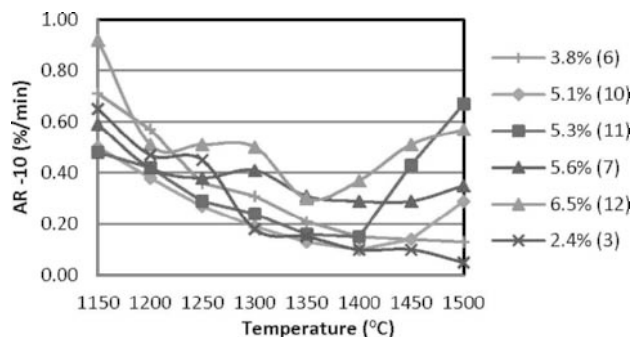
In the lab tests, green coke samples were prepared by screening to obtain naturally occurring 4x14 mesh (4.75mm-1.18mm) material and then crushing the remaining +4 mesh material and re-screening at 14 mesh. The 4x14 mesh fractions were combined and heated in open top crucibles at a rate of 30°C/min to approximate the heating rates found in rotary kilns (typically 30-50°C/min). Samples were calcined to final temperatures of 1150°C-1500°C and held for 15 minutes at the final temperature.

Table 1 shows air reactivity (AR) results for a wide range of coke samples calcined at approximately the same level (as indicated by the consistent Lc results). The fast method (10°C/min) was used for these tests.

**Table 1: Air Reactivity of Different Calcined Coke Samples**

Sample	S (%)	V (ppm)	Na (ppm)	Lc (Å)	AR (%/min)
Coke 1	1.55	122	41	29.0	0.07
Coke 2	1.76	131	42	29.4	0.12
Coke 3	2.38	368	54	29.8	0.18
Coke 4	2.47	259	63	29.8	0.37
Coke 5	3.07	449	55	30.1	0.29
Coke 6	3.49	647	81	29.9	0.31
Coke 7	4.45	385	93	28.7	0.41
Coke 8	4.67	431	67	28.4	0.43
Coke 9	4.69	626	77	30.1	0.51
Coke 10	4.70	471	94	29.0	0.27
Coke 11	4.71	540	70	29.7	0.29
Coke 12	5.63	606	77	28.7	0.51

Figure 1 shows temperature vs air reactivity (10°C/min) data for six different coke samples from Table 1, identified by their green coke sulfur content and identification numbers from Table 1.



**Figure 1: ISO AR at 10°C/min (fast method)**

Table 1 shows the low sulfur cokes have a relatively low AR and the higher S cokes show a higher AR. The  $R^2$  correlation for sulfur and AR is 0.66 and for vanadium and AR is 0.49. The equation developed by Hume [3] which includes S, V and Na does not show an improved  $R^2$  value for the fast method, but it does for the slow heating method (0.5°C/min), with an  $R^2$  similar to what was reported for the equation.

Figure 1 shows that the air reactivity decreases as the temperature increases as has been previously reported [10,11,12], and then increases as porosity opens due to desulfurization, confirming what has been reported previously [3]. The data in Table 1 and Figure 1 highlight the wide range in coke air reactivities for cokes that are all currently used routinely in anode blends. Many of these cokes could not be used today if historical coke air reactivity specifications were in place.

Temperature vs air reactivity curves using the slow method (0.5°C/min) are shown in Figure 2. The general shapes of the curves are similar to the fast method curves for all of the cokes shown. The air reactivity results for the 6.5% sulfur coke using the slow method are not shown in Figure 2 because all the results were out of the equipment measurement range (i.e. >1.8%/min).

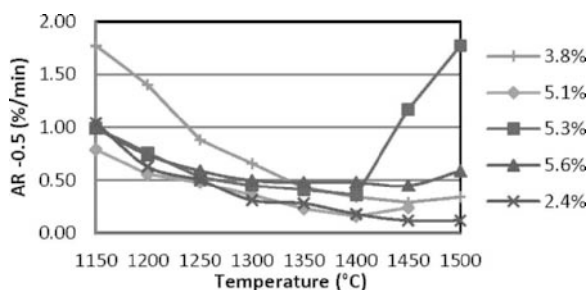


Figure 2: ISO AR - 0.5°C/min (slow method)

The slow method was developed in response to earlier work [13] to provide an air reactivity result that was more indicative of the air reactivity of an anode when considering diverse impurities in the coke, and although there was some improvement, the correlation was not good in practice. Several papers have been published using data that was developed using the slow method [12,14,16,18].

Figure 3 shows the sulfur level as a function of temperature for the same six cokes shown in Figures 1 & 2. The effect of thermal desulfurization for the high sulfur cokes at high temperatures is obvious. The temperature range in the lab experiments is higher than those used in a rotary kiln and the very high levels of thermal desulfurization would not be expected during normal production. The low sulfur coke shows almost no desulfurization which is typical of low sulfur cokes.

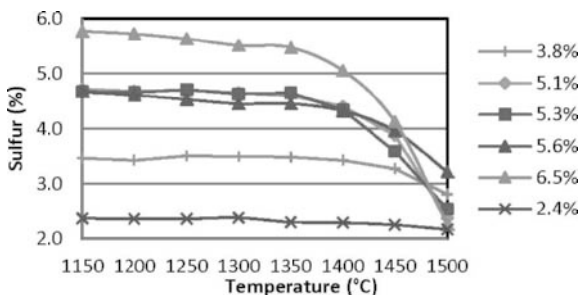


Figure 3: Desulfurization in High Sulfur Cokes

Figure 4 shows the trend in real density for the same cokes. The low sulfur coke shows a steady increase in real density with temperature whereas most of the higher sulfur cokes show a decrease in real density with the onset of thermal desulfurization. The highly isotropic coke with a sulfur level of 5.6% shows quite a different temperature-real density relationship as a result of its significantly different texture or microstructure.

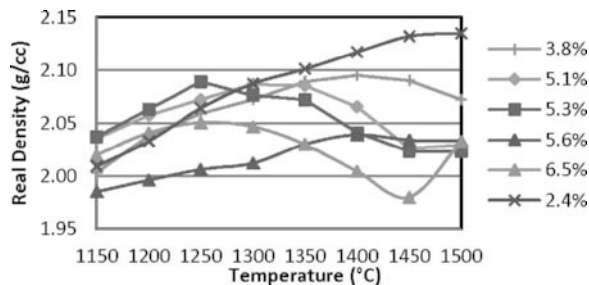


Figure 4: Real Density (-200 microns fraction)

Figure 5 shows the change in the average crystallite size or Lc as a function of temperature. The Lc is not affected by coke desulfurization because it is a measure of the degree of ordering of the carbon structure. At higher temperatures, the carbon structure becomes more ordered and the average crystallite size increases. It is not affected by changes in the micro-porosity of the coke like real density.

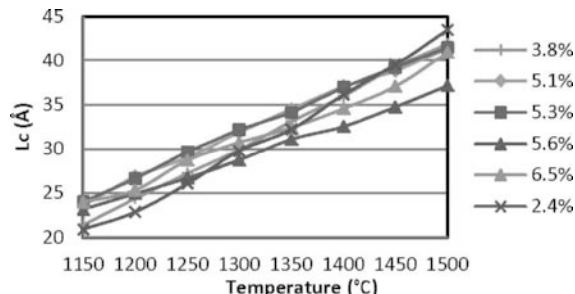


Figure 5: Crystallinity (Lc) of Calcined Coke

The specific electrical resistivity of coke is also unaffected by thermal desulfurization and it shows an almost linear decrease with temperature for all cokes, Figure 6.

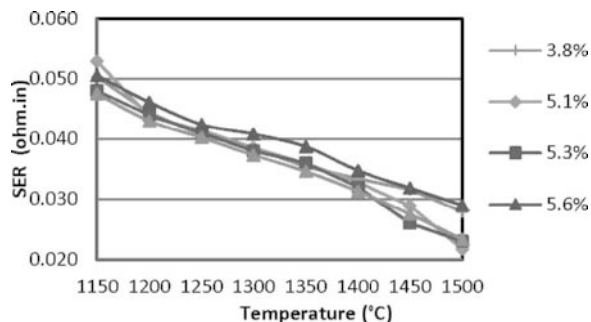


Figure 6: SER of Coke as a Function of Temperature

The data in Figures 3-6 highlight the importance for calciners to be aware of the calcination behavior of different green cokes in blends. The data in Figures 4-6 in particular, highlight the importance of measuring both the real density and Lc or the real density and electrical resistivity when calcining cokes with widely varying sulfur levels. If a calciner is only measuring real density

to control the calcination level, it is very easy to start desulfurizing a blend without being aware of it.

If the calciner is trying to achieve a high real density target of 2.07 or 2.08 g/cm<sup>3</sup> for example, it may not be physically possible to achieve such a target with a blend containing high sulfur cokes due to thermal desulfurization and the presence of isotropic cokes with lower average real densities. An operator's natural inclination to increase the calcination temperature will typically make the problem worse and drive additional desulfurization and a further decrease in real density. The Lc test is useful for avoiding this problem.

#### Kiln Trial Data

The above physical changes can be illustrated with data from a full scale kiln trial. In this case, a blend of green cokes with sulfur levels ranging from 1.4-6.5% was used to give a blend with an average sulfur level of 4.0%. Figure 7 shows the change in real density and Lc of the calcined product as the calcination temperature was increased.

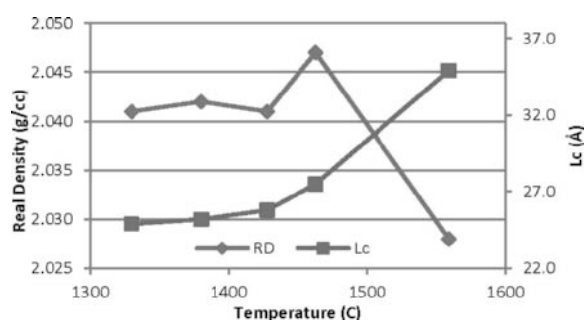


Figure 7: Real density and Lc for Kiln Trial

The kiln temperature was changed over a relatively short period during this trial and the refractory brick temperature did not reach a steady state level at each temperature, which is the primary reason the coke real density and Lc did not change significantly over the temperature range of 1330 – 1430°C. The real density and Lc showed a more significant increase for the next data point and then a much larger change at the final temperature of 1560°C. Thermal desulfurization of the coke at the final temperature is very apparent from the decrease in real density. The Lc on the other hand, shows a steady increase consistent with the insensitivity of this test to desulfurization.

Figure 8 shows the air reactivity and vibrated bulk density (VBD) of the calcined blend as a function of temperature. The ASTM D7454 VBD results shown are measured on samples prepared to 20x35 mesh (0.85-0.50 mm).

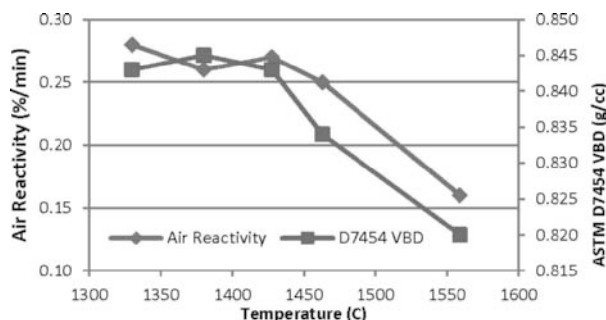


Figure 8: Air Reactivity and VBD

The air reactivity shows a strong correlation with the temperature and decreases to the relatively low level of 0.13%/min (fast heating rate) at the highest temperature. The VBD on the other hand, shows a significant decrease at the final temperature illustrating the negative effects of desulfurization on coke porosity. If the calciner was trying to meet a coke air reactivity specification of 0.20%/min, this could easily be achieved by increasing the calcining temperature but it would negatively affect coke porosity and would not allow the VBD specification to be met.

This example illustrates why care must be taken when calcining cokes with different sulfur levels and structures, and when real density is used as the control measure. Most coke specifications are based on real density and it has become much more difficult to achieve high real density values in a rotary kiln with the range of green cokes used today.

In an ideal world, it would be beneficial to calcine cokes separately at different levels to avoid desulfurization of high sulfur cokes. This is not practical however given the higher number of cokes used in blends and the need to store each separately to allow blending after calcination. Calciner SO<sub>2</sub> permit limits also typically prevent high sulfur cokes being run separately. High sulfur green coke typically loses 12-15% sulfur during calcination and this would significantly increase SO<sub>2</sub> emissions relative to a blend of high and low sulfur cokes.

The data in Table 2 show the quality of some high sulfur cokes that were calcined separately in a rotary kiln, and are currently being used in anode blends. These blend cokes are typically used in lower percentages due to the high sulfur and vanadium levels, but their high air reactivity values would have disqualified them from use a blend coke in the past. Note the difference in real density results for cokes C and D even though they were calcined to a similar level.

Table 2: High Sulfur Cokes Calcined in Rotary Kiln

Parameter	A	B	C	D
Lc (Å)	35.0	28.6	29.7	29.5
Real Density, -200µm (g/cm <sup>3</sup> )	2.09	2.04	1.99	2.05
VBD, -28+48 (%)	0.91	0.85	1.01	0.88
Air Reactivity – Fast (%/min)	0.53	0.43	0.47	0.25
CO <sub>2</sub> Reactivity (%)	3.6	5.9	4.0	3.2
Calcium (%)	0.003	0.011	0.016	0.002
Iron (%)	0.008	0.017	0.033	0.017
Nickel (%)	0.038	0.020	0.026	0.017
Silicon (%)	0.017	0.025	0.024	0.005
Sodium (%)	0.008	0.010	0.011	0.011
Sulfur (%)	4.00	3.72	4.66	5.77
Vanadium (%)	0.074	0.049	0.062	0.050

#### Air Reactivity – A Brief History

It was reported in 1988 [1] that a simple apparatus and corresponding method was developed to determine the air reactivity of calcined coke by measuring the ignition temperature of the coke. The authors concluded that the air reactivity results from this test method had a high potential to be used for, among other things, selection of appropriate raw materials. For an aluminium industry looking for ways to reduce anode consumption, this methodology was well received and resulted in

the ISO 12982-1 air reactivity test [2] being a specified requirement by many aluminium producers.

In 1993, it was reported [3] that in order to maintain a good air and CO<sub>2</sub> reactivity of coke it is necessary to control the quality of the cooling water by removing alkali and alkaline earth metals. Not long after, some merchant calciners installed relatively expensive equipment to remove impurities from the water used to cool the coke exiting the kiln to improve the air and CO<sub>2</sub> reactivity of the coke.

Others [14] concluded in 1993 that the use of deionized water to quench calcined coke offered no advantage over well water when considering the steady-state air reactivity of coke, and that the air reactivity of calcined coke as determined by ignition temperature was not a true measure of the steady-state air reactivity of the coke. They also concluded that the only valid way to measure the steady-state air reactivity of coke was by thermogravimetric analysis, and recommended a temperature of 525°C.

At the same time that stringent air reactivity specifications were evolving, others warned in 1993 [13] that it is dangerous to choose an air reactivity method to approve or reject a raw material, and stated that up to that time, there was no coke air reactivity test that could predict the anode air reactivity behavior obtained by different technologies. They concluded that the safest way for a smelter to select a raw material would be to perform a controlled plant test to estimate the behavior of the coke in the potlines.

Work conducted [11] on calcined coke and lab anode samples in 1993, showed that measurements of coke air and CO<sub>2</sub> reactivity did not correlate with electrolytic consumption results for lab anodes. In past studies, laboratory consumption figures correlated well with plant data, so the authors concluded that coke reactivity measurements did not seem to provide any useful information for the prediction of anode performance. The paper also provided data on the relationship between coke calcination levels (as measured by the Lc method) and coke air reactivities. The data show quite clearly that it is possible to reduce coke air reactivities by calcining cokes to higher levels as reported in this work. Although this was not widely understood throughout the industry, it was well understood by coke calciners and was one of the drivers for the relatively high calcination levels used by some calciners in the industry at that time.

Air reactivity was not the focus of work [15] in 1993, but the authors concluded that the vibrated bulk density was negatively impacted by heat up rate and desulfurization of high sulfur cokes which were being used in blends at the time. They noted the importance of establishing real density specifications based on need and substantiated by improved anode performance rather than being arbitrarily selected. The paper cautioned against the trend by aluminum companies to request higher real density specifications and higher calcination levels. In a follow up paper in 1994 [10] the authors included coke reactivity data and stated that higher calcination temperatures were an effective tool for decreasing coke air reactivities as measured by thermogravimetric analysis, but for high sulfur cokes, thermal desulfurization can cause reactivities to increase and negatively impact coke VBD and other properties.

To better understand the impact of higher coke calcination temperatures and increasing real densities, authors in 1994 [12] prepared lab anodes from cokes with two different sulfur and real density levels. They concluded that it was necessary to bake the

anodes at a minimum temperature such that the real density of the binder coke is equal to that of the base coke in order to obtain minimum binder coke/base coke differential reactivity. The use of coke with higher real density therefore requires anode baking at higher temperatures. This conclusion came at a time when calciners had to calcine to a higher temperature in order to meet air reactivity specifications required by many customers.

In 1997 [16], calcined coke data were compared with the resultant plant anode data for a period of several years and the authors concluded that no correlation existed between coke air reactivity and anode air reactivity residue for any of the time periods studied (1992-1995). That same year, it was reported [17] that the removal of oil that was applied to calcined coke to reduce dusting had a significant impact on the air reactivity result, depending on which removal method was employed. At that time many laboratories removed the oil by thermal treatment instead of solvent extraction due to the published negative health effects of solvents. The authors concluded that thermal treatments altered the surface characteristics of the calcined coke and resulted in abnormally high or low air reactivity results, depending on the method used.

In 2001 [18], lab anodes were prepared from one coke source that had been calcined two different ways, one in a rotary pilot plant kiln and the other in a stationary lab furnace in covered graphite crucibles. The two calcined cokes had significantly different ISO air reactivities, but the resultant lab anodes had almost the same air reactivity although all other parameters were similar. The authors concluded that coke air reactivity tests do not provide a reliable indicator of anode air reactivity and the evaluation of calcined coke for anode suitability need not include coke air reactivity testing.

As a result of all of the work done throughout the 1990's, calcined coke consumers began to realize that, although the ISO air reactivity of the coke can be lowered by increasing the temperature in the kiln, it is merely a measure of the reactivity of the surface of the coke and not a steady state measure of the carbon reactivity. Further, much data has been published that shows that there is little correlation between the ISO air reactivity of calcined coke and the air reactivity of the resultant anodes.

#### **Impact of Coke Calcination Level on Anode Quality**

It has been previously published that the sulfur level of high sulfur cokes used in anode blends continues to rise [9]. This work showed that high sulfur cokes can be used successfully in blends without negative consequences but care must be taken to avoid overcalcining these cokes and over-baking anodes made with these cokes, because both have the potential to negatively affect anode properties.

In 2001, authors [19] studied bench scale anodes made with coke calcined in the lab to different temperatures, all of which were below the usual calcination levels for industrial cokes at that time. They concluded that an increase in coke calcination temperature is unfavorable to anode reactivity and thermal shock resistance, but favorable to anode density (although not at high baking temperature). Their conclusions were verified in a plant trial where coke calcined to a lower temperature led to reduced carbon dust due to a reduction in the difference of the reactivity of the pitch and anode coke grains of the matrix. Additionally, they reported that anode density was not changed and there was no noticeable increase in sensitivity to thermal shock.

Two papers presented in 2009 studied the impact of using under-calcined coke to produce anodes. In one paper, authors [5] concluded that results obtained on cokes and pastes showed that the coke reactivity does indeed decrease with increasing calcination temperature (up to desulphurization), but that under-calcination is beneficial to decrease the reactivity of anode pastes. Others [6] concluded that the use of under-calcined coke in the production of anodes provided a decrease in the overall reactivity of the anode, a reduction in carbon consumption and a lower risk of anode problems. These benefits were seen despite a slight decrease of anode density related to the lower apparent density of undercalcined coke and partly compensated for by a higher shrinkage rate during baking.

The aluminium industry has continued to study the effect of using under-calcined coke, and in 2011 authors [7] reported results from a study that used a single source green coke calcined to two different levels for which anodes were made and then used in a smelter. Although air reactivity was not the focus of the paper, they concluded that anodes produced from under-calcined coke showed an improvement in density and CO<sub>2</sub> reactivity.

In 2012, others [8] published the results of a study that involved comparing under-calcined coke with standard calcined coke. Under-calcined coke was defined as coke having an RD <2.05g/cc and Lc <25.3Å and standard calcined coke defined as coke with an RD of 2.05 - 2.09g/cc and Lc of 25.3 - 32.7Å. The study included green anode production, baked anode production, and anode performance in the pot room. The authors concluded that the air reactivity of anodes made with under calcined coke was better than those made with standard calcined coke despite the fact that the air reactivity of the under calcined coke was almost twice the air reactivity of the standard coke. Net carbon consumption for under-calcined coke anodes was also slightly lower compared to standard calcined coke.

One of the authors followed up the above paper with a presentation at the GAC Carbon Conference in September 2012 [20]. Based on the positive results reported in the above paper, the Alba coke calciner and smelter have converted the entire operation to use under-calcined coke. The calciner is typical of a modern merchant coke calciner that is not attached to a refinery and which uses multiple green coke sources with low and high sulfur contents.

### Discussion

The ISO coke air reactivity test was developed at a time when the industry had a ready supply of high quality anode grade coke. During the 1990's, a high sulfur coke was regarded as one with a sulfur level of 3.0 - 3.5% and vanadium levels in the range of 250-400 ppm. Cokes with sulfur levels up to 3.5% can be calcined to high levels without significant desulfurization and it was relatively straightforward to calcine cokes like this, and to calcine lower sulfur cokes to high levels to achieve air reactivity specifications of 0.10 - 0.15%/min (fast method). Real density specifications in the range of 2.08 - 2.10 g/cc were very common during this period reflecting the general industry view that higher calcination levels were advantageous to anode performance.

This situation has changed dramatically as a result of the much greater range of green coke qualities used in anode blends today. It is detrimental to calcine many of these cokes to high calcination levels and for many of these cokes, maximum real densities in the range of 2.04-2.06 g/cc are possible. The move toward lower calcination levels is becoming more widespread and it is a logical

progression for the industry. Although this is commonly referred to as "under-calcining" the definition can be a little misleading. As long as coke is calcined to a high enough level to remove volatile matter, complete the bulk of the shrinkage and transform the structure into an electrically conductive one, coke can be considered to be calcined.

Although most smelters have abandoned coke air reactivity specifications for the reasons cited in this paper, it is still a specified requirement at some smelters. Test results are easily influenced by coke surface chemistry and there is no published data which shows a good correlation between coke air reactivity and anode consumption. Coke blends with air reactivities in the range of 0.3-0.4%/min are now being used routinely in anodes with excellent potroom performance. Excess carbon consumption is driven by many factors, but among the more critical are anode baking levels, the presence of sodium from poorly cleaned butts, coke and anode sulfur levels, and cell cover practices.

Many smelters today place more emphasis on anode CO<sub>2</sub> reactivity rather than anode air reactivity or airburn potential. Airburn of anodes can be controlled successfully through cell cover practices, anode temperatures and cell stability but CO<sub>2</sub> burn of anodes is a more fundamental problem due to the continuous evolution of CO<sub>2</sub> gas from the anode electrolytic surface. The positive benefits of higher sulfur levels for controlling anode CO<sub>2</sub> reactivity are well known [21] and a growing trend for smelters is to specify both a minimum and maximum sulfur level for the coke supply.

### Conclusions

Many papers have been published on the ISO coke air reactivity test over the last 20 years and a significant number of these have been cited in this review. The lack of correlation between coke air reactivity results and anode performance is perhaps the most significant shortcoming of the coke air reactivity test. The measurement of coke ignition temperatures is a transient, rather than steady state measurement which is readily influenced, for example, by the presence of surface contaminants from cooling water and different methods for removing oil used for control of coke dusting.

Coke air reactivities are strongly influenced by coke calcination levels and the trend towards lower calcination levels and the use of "under-calcined" coke makes sense for the industry given rising sulfur levels in high sulfur cokes. This runs counter to achieving low coke air reactivities but many smelters are now using coke calcined to lower levels quite successfully and it avoids the problems associated with over-calcination and desulfurization of blends containing high sulfur cokes.

Rain CII discontinued running the coke air reactivity test at the end of 2011 when the specification was dropped by a few remaining smelters. This was not an easy decision to make but the information presented in this paper provides some of the background and rationale behind this decision.

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