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RELATIONSHIPS BETWEEN COKE PROPERTIES AND ANODE PROPERTIES – ROUND ROBIN 19

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Abstract

This paper discusses the preparation and production of bench scale and pilot scale anodes with five different calcined coke samples prepared for a world-wide calcined coke round robin (RR). A key objective of the RR and anode testing was to look for relationships between calcined coke properties and anode properties, particularly coke bulk density/apparent density results and anode densities. The calcined coke RR was the 19th organized by Rain CII Carbon, but this time it was a collaborative effort with Hydro Aluminium and R&D Carbon. Calcined coke results for RR19 are discussed in greater detail in another paper in these proceedings. Bench scale anodes with the five cokes were prepared at R&D Carbon and used to select optimum pitch levels for production of pilot scale anodes. This is the first time a RR with such a broad scope has been coordinated and published and it has provided some useful data for the industry.

Introduction

During the 2010 TMS meeting in Seattle, the ASTM Standards Committee convened a special meeting to discuss coke bulk density testing. At least four different bulk density or apparent density test methods are used within the industry and there is no agreement on which method provides the best predictor of coke performance in anodes. Coke bulk densities have decreased on average over the last 10 years [1] and there is renewed interest in the relevance of the different test methods and their ability to predict anode densities. One of the actions from the meeting was to organize a dedicated session on coke bulk density testing at the 2011 TMS meeting. Seven papers were published in that session [2,3,4,5,6,7,8] and it was followed by a panel discussion on what could be done to reach better consensus within the industry.

One of the recommendations from the panel was to conduct an industry wide, calcined coke round robin comparing the various bulk and apparent density test methods. A round robin (RR) was organized by Rain CII Carbon and Hydro Aluminium and is the subject of a companion paper in these proceedings [9]. It was the nineteenth RR organized by Rain CII and is hereafter referred to as RR19.

The preparation of the five coke samples is well described in the companion paper along with all the test results including within-lab repeatability and between-lab reproducibility data. Results reported in the paper include chemical analysis (S, V, Ni, Fe, Si, Ca, Na, P), real density, Lc, mercury apparent density, tapped bulk density (ISO 10236) and vibrated bulk density (ASTM D4292 and ASTM D7454). Bulk densities for the different preparation methods were also measured using the GeoPyc instrument [6]. A total of 28 labs participated in RR19.

In a significant expansion of the RR19 work, 150kg samples of each of the five cokes were sent to R&D Carbon for the preparation and testing of bench scale and pilot scale anodes. The bench scale anodes were prepared to determine optimum pitch levels prior to the production of larger, 150mm diameter pilot scale anodes at the optimum pitch level.

The primary objective of the above work was to look for correlations between the various coke bulk density and apparent density tests and anode properties. Correlations with baked anode density were of special interest but the work provided a good opportunity to look at correlations between all calcined coke properties and baked anode properties. The five calcined cokes selected for the round robin were chosen quite deliberately to represent extremes in terms of bulk density, structure and chemical analysis (primarily S and V). Three of the five cokes were single source, straight run calcined cokes (A, B and S) and cokes C and HB were blended calcined cokes.

Calcined Coke Properties and Anode Production

The properties for the five coke samples are shown in Table 1. Most of the results are based on measurements made at R&D Carbon. The results for 28x48 mesh (2.36-1.18mm) VBD, 20x35 mesh (0.85-0.425mm) VBD and Hg AD are all based on the industry average results reported in the companion paper.

Table 1: Calcined Coke Properties

Property	Unit	Coke A	Coke B	Coke C	Coke S	Coke HB
Grain Size +8mm	%	10.8	0.4	11.7	1.6	11.9
TBD (1-2mm)	kg/dm^3	0.73	0.98	0.81	0.86	0.83
VBD 28x48 Mesh	kg/dm^3	0.83	0.99	0.86	0.90	0.88
VBD 20x35 Mesh	kg/dm^3	0.79	0.95	0.82	0.86	0.84
Hg AD (Pechiney)	kg/dm^3	1.72	1.76	1.72	1.74	1.72
Grain Stability	%	65	77	80	76	83
Real Density	kg/dm^3	2.08	2.00	2.06	2.06	2.07
Crystallite Size Lc	Å	29.9	29.3	26.6	26.6	28.2
Spec. Elect. Resist.	µΩ.m	434	580	454	498	457
CO_2 Reactvity at 1000°C	%	8.9	4.4	6.9	6.3	9.0
Air Reactivity at 525°C	%/min	0.06	1.15	0.38	0.21	0.35
Sulfur	%	1.46	4.37	3.05	1.16	2.16
Vanadium	ppm	94	619	404	144	241
Nickel	ppm	173	263	205	64	172
Iron	ppm	167	537	295	78	195
Silicon	ppm	70	159	129	54	228
Calcium	ppm	77	120	87	24	155
Sodium	ppm	42	121	45	29	62

Some general comments on the five cokes are as follows:

- Coke A is a relatively low bulk density, low sulfur coke. It is always used in blends with other cokes.
- Coke B is a high bulk density, highly isotropic coke with high S and V. It is used at low levels (<10%) in blends.
- Coke C is a blended coke used routinely at some smelters for anode production. It has a moderately high S and V level.
- Coke S is a relatively high bulk density, low sulfur coke. It is used primarily as a blend coke.
- Coke HB is another blended coke used routinely at smelters for anode production.

Preparation of Bench Scale Anodes

Samples of each of the five cokes were sized into different fractions including preparation of a fines fraction via jet milling (details in next section). No butts were used in the recipe.

Small bench scale anodes (50mm diameter x 100mm length) were produced at 4 pitch levels using a 112°C Mettler softening point, medium QI pitch. The cores were baked over 20 hours to 1100°C. Graphs of baked anode density vs pitch content for the five cokes are shown below and were used to select the optimum pitch levels for pilot scale anodes.

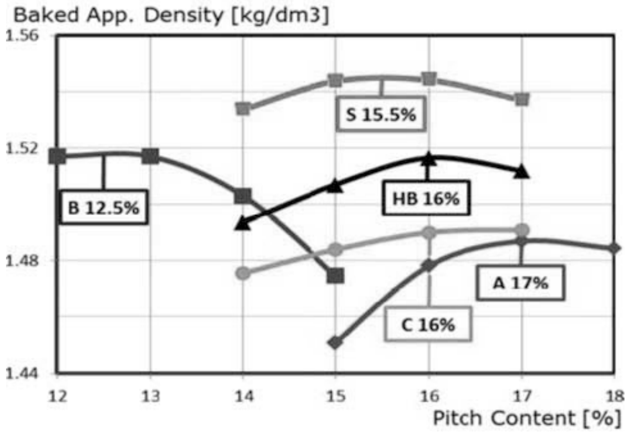

Figure 1: BAD vs Pitch Content for Bench Scale Anodes

Production and Testing of Pilot Anodes

Pilot scale anodes were prepared at the optimum pitch content for each coke according to the scheme in Figure 2 and described previously [10].

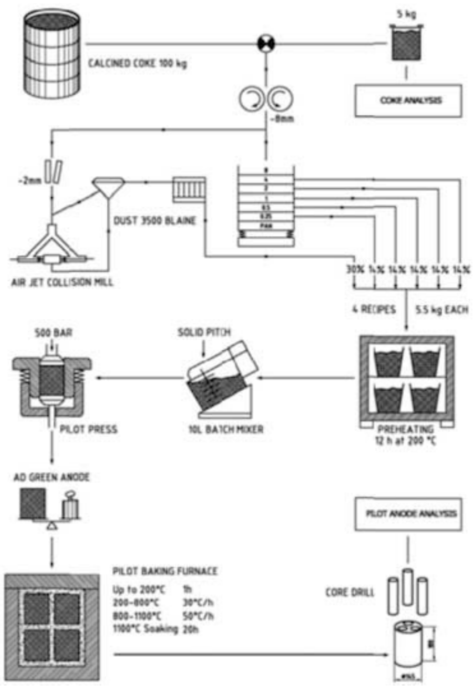

Figure 2: Production of Pilot Anodes

Four batches of 5.5kg were prepared for each coke by mixing according to the recipe and preheating at 200°C for 12 hours. The preheated coke and solid coal tar pitch were added to an Eirich mixer and mixed at 172°C and then cooled to 150°C by injecting water prior to forming. Forming was carried out via hydraulic pressing for 1 minute at a pressure of 500 bars. The green anodes were 146mm in diameter and 180-200mm in length.

The green anodes were baked to 1100°C in a pilot baking furnace which can bake up to 12 anodes at once. Three core samples were drilled from each baked anode with a diameter of 50 mm and length of 200mm. Testing was performed according to standard ISO test procedures.

Pilot Anode Results

Results from testing the pilot anodes are shown in Table 2. Without the addition of butts, densities are typically ~0.03 kg/dm^3 lower than full size production anodes. Mechanical properties, including permeability and electrical resistivity are also typically a little worse than production anodes, while thermal conductivity, thermal expansion coefficient and reactivity are similar.

Table 2: Pilot Anode Properties

Properties	Units	Coke A	Coke B	Coke C	Coke S	Coke HB
Optimal Pitch Content	%	17.0	12.5	16.0	15.5	16.0
Green Apparent Density	kg/dm^3	1.53	1.53	1.52	1.59	1.56
Baking Loss	%	6.0	4.6	5.6	5.9	5
Baking Shrinkage	%	2.6	3.9	2.5	2.0	1.7
Baked Apparent Density	kg/dm^3	1.47	1.52	1.47	1.53	1.49
Spec. Elect. Resistance	μΩm	62.1	53	62.5	65.4	62.4
Compressive Strength	Mpa	33	47	26	29	36
Flexural Strength	MPa	9.8	17	10.9	8.6	9.9
Coef. Thermal Expansion	10^{-6}/K	3.7	5.7	4.0	3.5	3.8
Air Permeability	nPm	2.7	13.2	2.5	1.8	2.6
Real Density	kg/dm^3	2.06	1.99	2.05	2.07	2.07
Thermal Conductivity	W/mK	3.06	3.19	2.85	2.91	3.12
CO2 Reactivity Residue	%	92.1	95.8	93	91.3	90.5
Air Reactivity Residue	%	93.7	65	71.2	78.5	87
Impurities by XRF						
S	%	1.3	4.19	2.36	1.05	1.86
V	ppm	84	528	302	129	207
Ni	ppm	161	232	156	62	154
Fe	ppm	205	504	328	135	309
Si	ppm	89	227	154	71	341
Ca	ppm	82	115	95	37	171
Na	ppm	50	134	42	29	61

The data generated during the RR19 study was extensive with 28 labs providing data on calcined coke properties and R&D Carbon providing anode data on both bench scale and pilot scale anodes. It is not possible to review and present all the data in this paper so only selected data and correlations will be discussed for pilot scale anodes. More details on the precision of the various coke property tests can be found in the companion paper [9].

Pitch Level and Anode Density Correlations

Of primary interest in this study were correlations between the various coke TBD/VBD results and optimum pitch levels and anode densities. Given the wide range in coke bulk densities for the five cokes (up to 35% for some tests), one might expect to see a wide range in optimum pitch levels and baked anode densities. R^2, the Coefficient of Determination, is used in the discussion; each lab's RR19 coke property result is correlated with the common anode property result. Labs with less than five cokes measured were not included in the comparisons.

The bench scale anodes were useful for selecting optimum pitch levels and the levels ranged from a low of 12.5% for coke B to a high of 17% for coke A. Since coke bulk density is intended to be an indirect measure of coke porosity, it is reasonable to expect a good correlation with optimum pitch level. This was indeed the case, and the R^2 correlations for many of the labs that participated were above 0.90 for the various VBD/TBD tests as shown in Table 3. The column titled "Count" refers to the number of results used for the correlations. Some obvious outlier results with R^2 values <0.2 were removed from the analysis.

Table 3: R^2 Correlations for Optimum Pitch Level

Test	Count	R^2 Avg.	R^2 Range
D4292 28x48	11	0.93	0.73 - 0.99
D4292 GeoPyc	6	0.97	0.96 - 0.98
D7454	6	0.97	0.93 - 0.99
ISO TBD 0.5-1mm	6	0.89	0.76 - 0.96
ISO 0.5-1mm GeoPyc	5	0.91	0.89 - 0.94
ISO TBD 1-2mm	6	0.89	0.70 - 0.94
ISO 1-2mm GeoPyc	4	0.91	0.89 - 0.93
ISO TBD 2-4mm	6	0.94	0.97 - 0.97
ISO 2-4mm GeoPyc	4	0.91	0.90 - 0.93
ISO TBD 4-8mm	6	0.97	0.90 - 0.99
ISO 4-8mm GeoPyc	4	0.98	0.96 - 1.00
Hg AD	7	0.81	0.65 - 0.94

The Hg AD test was not as good a predictor of optimum pitch level on average for the five cokes in this RR. Very few labs run this test today, and it was included in the RR for comparative purposes only.

A total of 11 out of the 28 labs that participated in RR19 measured VBD's by the ASTM D4292 method making it the most widely reported VBD result in RR19. The lab identifications are randomized so that L03 (Lab 03) for example, does not correspond to the 3rd lab listed in Table 1 of the companion paper [9]. Figure 3 shows the R^2 values for the D4292 VBD test vs. optimum pitch content and these data are the detailed numbers that account for the first row of data in Table 3.

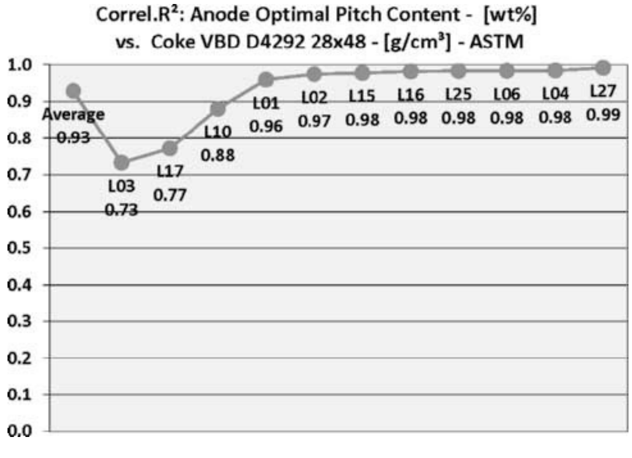

Figure 3: R^2 Values for Pitch Level vs ASTM D4292 VBD

The property of most interest in this study was baked anode density (BD) and Figure 4 shows the relative values for the five cokes. The difference in baked anode densities is around 4% which is significantly lower than the relative difference in coke bulk densities. Of particular note is the fact that the coke B anodes did not show the highest baked density despite a significantly higher bulk/apparent density by all of the test methods used.

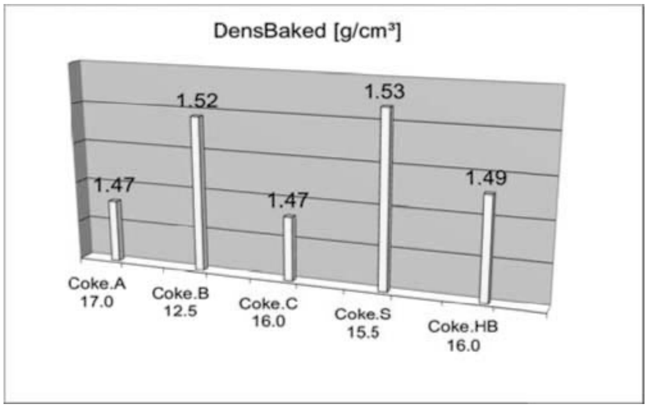

Figure 4: Baked Anode Density results with coke and the optimum pitch level [%] on the x-axis

An example of the R^2 correlations between the baked anode densities and the ASTM D4292 test are shown in Figure 5 and the correlations for baked anode density vs the ISO 10236 test (1x2mm) are shown in Figure 6.

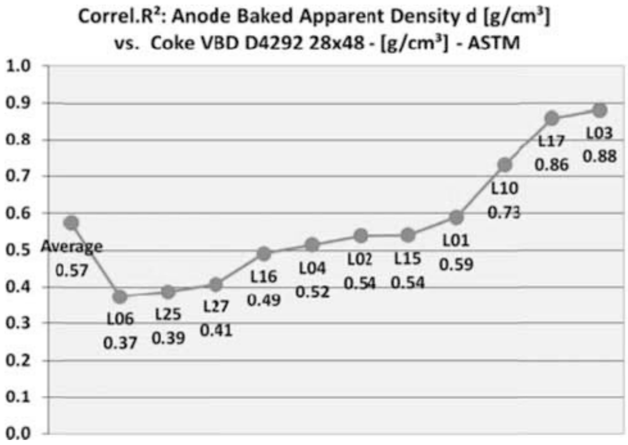

Figure 5: R^2 Values for BD vs ASTM D4292 VBD

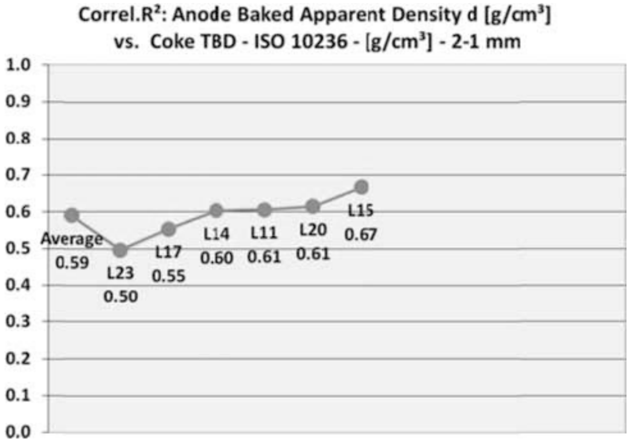

Figure 6: R^2 Correlations for BD and ISO 10236 (1-2mm)

Table 4 shows a summary of the R^2 values for the baked anode density with all of the VBD/TBD and Hg AD tests. For the Hg AD results, two lab results were eliminated from the analysis due to R^2 values below 0.2 (Lab 07 and Lab 14). Correlations between the Hg AD and BD showed a wide range, reflecting the difficulty of running this test consistently between labs. This was also the case for the D4292 VBD test.

Table 4: R^2 Correlations for Baked Anode Density

Test	Count	R^2 Avg.	R^2 Range
D4292 28x48	11	0.57	0.37 - 0.88
D4292 GeoPyc	6	0.54	0.49 - 0.59
D7454	6	0.56	0.44 - 0.67
ISO TBD 0.5-1mm	6	0.64	0.38 - 0.74
ISO 0.5-1mm GeoPyc	5	0.66	0.61 - 0.71
ISO TBD 1-2mm	6	0.59	0.50 - 0.67
ISO 1-2mm GeoPyc	4	0.61	0.59 - 0.66
ISO TBD 2-4mm	6	0.56	0.52 - 0.64
ISO 2-4mm GeoPyc	4	0.57	0.53 - 0.62
ISO TBD 4-8mm	6	0.46	0.35 - 0.69
ISO 4-8mm GeoPyc	4	0.46	0.37 - 0.58
Hg AD	6	0.63	0.39 - 0.84

Based on the results of this RR, none of the VBD or TBD tests stand out as being clearly better than the others in terms of their correlation with BD. The correlations for some individual labs were good (>0.80) but for the most part, the R^2 values were in the 0.5 – 0.6 range. When coke B is removed from the analysis, the correlations improve somewhat. Coke B is not a traditional sponge coke and has a highly isotropic structure like shot coke but an angular/irregular particle shape more like sponge coke [1]. It would not be realistic to make anodes with 100% of this coke due to its high coefficient of thermal expansion.

In principle, coke B should have given a higher BD but the results illustrate some limits of VBD, TBD and Hg AD tests for predicting anode densities. The same granulometry recipe was used for all five cokes with 30% fines milled to a Blaine Index of 3500. Coke B required significantly less pitch than the other cokes so the ratio of fines to pitch was higher with this coke. With a lower open porosity in the coke particles, this likely resulted in a greater amount of binder matrix (pitch + fines) between the aggregate particles than the other recipes.

For the other cokes, more binder matrix filled the open pores in the coke and the remainder filled the voids between the aggregate particles. Coke B contains very few large (>100µm) pores so most of the binder matrix was distributed around and between aggregate particles which resulted in more binder matrix relative to inter-particle void space. This additional binder matrix would push apart the coke aggregate particles due to the excess of fines. This is supported by the significantly higher baking shrinkage of the coke B anodes. The lower real density of the coke B fines then negatively impacted the BD of the anodes.

All of the cokes used in this study were calcined to a similar level with Lc's in the range of 26-30Å. The anodes were baked using the same heating rate and final temperature so the high baking shrinkage of the coke B anodes was almost certainly due to shrinkage of the binder matrix and not of the coke aggregate particles themselves.

A granulometry optimization of the coke B anodes would likely have given a recipe with a lower level of fines and a higher baked anode density. This demonstrates the importance of optimizing all aspects of anode production when making significant coke quality changes or other changes in an anode plant. Good examples of such optimization studies have been reported in past papers [11, 12]. In addition to granulometry changes, modern paste plants also have the capability to adjust parameters like coke and pitch pre-heat temperatures, paste mixing and forming temperatures and anode forming pressures. All of these need to be optimized if the focus of the work is to maximize baked anode densities.

The green anode densities (GAD) show no obvious correlation to the coke VBD/TBD and Hg AD results and baked anode densities. This result is unexpected and in most carbon plants, baked anode densities normally correlate quite well with green anode densities. The difference here may be the wide range of optimum pitch levels which is much higher that what would normally be encountered in a production plant. There is a strong correlation between the BD and the dry aggregate density where:

$$\textit{Dry Aggregate Density} = GAD \times (100 - \%\textit{Pitch Level}) / 100$$

This calculates the bulk density of the aggregate with the pitch removed and is a useful crosscheck to make sure anodes are correctly pitched.

Before finishing the discussion on anode densities, it is worth pointing out that coke C and coke HB are the only two cokes out of the five that are used routinely to make production anodes. Typical baked anode densities for coke C are 1.57-1.58g/cm³ and for coke HB, they are 1.57-1.58g/cm³ in one smelter using this coke and 1.58-1.59 in another smelter which has a more capable paste plant. These densities are well above those measured in this study.

This is likely due a combination of the absence of butts and insufficient material to fully optimize the pilot anode production parameters. When testing a wide range of different coke samples, several iterations of testing can sometimes be required to produce anodes at optimum conditions. For this testing, R&D Carbon had a limited supply of each coke available for the pilot anode production. It is possible to produce pilot anodes that very closely match full production anodes as reported in another paper in these proceedings [13] but it takes time to optimize production conditions. Notwithstanding this, it is believed that the results and trends reported in this study are indicative and useful for comparative purposes.

Baking Loss and Permeability

The coke B anodes showed the lowest baking loss as a result of the lower pitch level. This result is expected and can be calculated (more or less) by assuming a pitch coke yield of 66%. The permeability of the coke B anodes was much higher than the other anodes and well above the levels expected for production anodes. Past studies with isotropic cokes have also shown higher permeability and this may be further evidence of the need to re-optimize the aggregate granulometry and anode production conditions when utilizing significant percentages of isotropic cokes in anode blends. The permeability of the other anodes was also higher than those typically found in production anodes which, along with the low anode densities, suggests that production conditions were not fully optimized for the pilot anodes.

Anode Real Density

The real density of anodes produced with coke B is significantly lower than the other cokes as a result of the lower real density of this coke. This result is expected and is due to the isotropic structure of coke B. This cannot be corrected by baking anodes to a higher level so care must be taken when using anode real densities to control anode baking levels when isotropic cokes are used in anode blends.

Most of the labs participating in RR19 that measured coke real densities showed a strong correlation with the anode real densities as shown in Figure 7. A couple of the labs showed a relatively poor correlation however, due to problems measuring the lower real density of coke B. This is discussed in more detail in the companion paper [9].

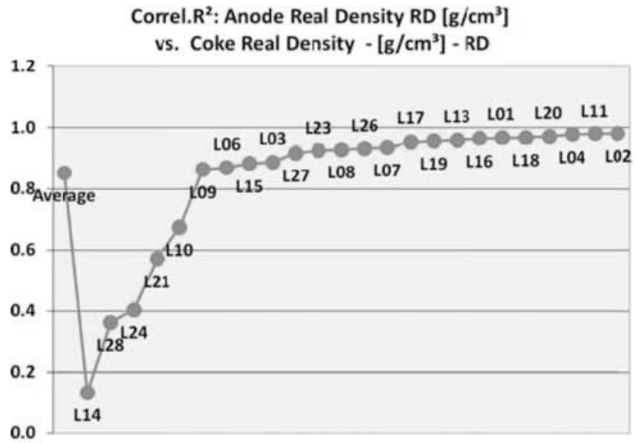

Figure 7: R^2 Values for Coke Real Density vs Anode RD

Anode Chemical Analysis

All the coke chemical analysis results from RR19 showed a strong correlation ($R^2>0.90$) with the anode chemical analysis results shown in Table 2. This is expected since only coke and pitch were used to make the anodes so the only chemical analysis change from coke to anodes was the dilution effect of adding pitch. As shown in the companion paper [9], the agreement between the 28 labs on all the chemical analysis results was generally excellent. These tests have good precision and are well established and well accepted within the industry.

The only caveat to the above, is that the agreement between analysis results deteriorated at higher levels of sulfur and trace metals. This is believed to be due to a lack of suitable high range calibration standards at some labs. The majority of labs used x-ray fluorescence (XRF) for sulfur and trace metals analysis and this method is very dependent on having a reliable set of calibration standards covering the full range of elemental concentrations being measured in unknown samples.

Electrical and Anode Mechanical Properties

One notable result from this study was the much lower electrical resistivity of the Coke B anodes, Figure 8. Similar results have been found in other unpublished pilot anode studies where significant volumes (>10%) of isotropic cokes have been used in the aggregate recipe. The reasons for this are not fully understood but it may warrant further investigation. One thing the results highlight is the lack of correlation between the coke specific electrical resistivity (SER) results reported in Table 1 and the anode electrical resistivity (ER). Coke B has a significantly higher SER but the anode ER was much lower. This indicates that anode ER is driven more by the presence of micro-cracks in the anode structure than the coke SER.

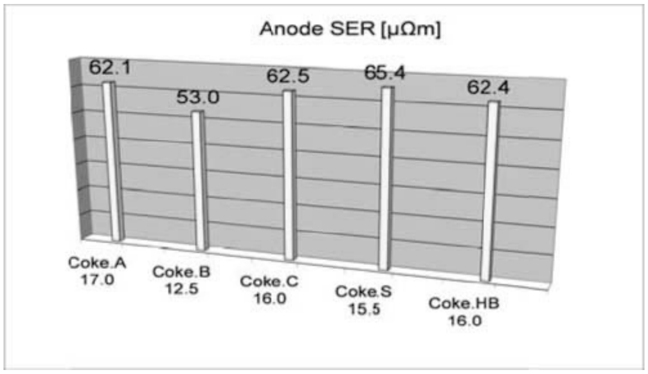

Figure 8: Anode specific electrical resistivity with coke type and the optimum pitch level [%] on the x-axis

The mechanical strength of the coke B anodes is significantly higher than the other cokes as indicated by both the compressive strength and flexural strength. Compressive and flexural strengths for the other pilot anodes all fall within typical industry ranges. This finding is also consistent with previous work on isotropic cokes – both shot cokes and non-shot isotropic cokes.

CTE and Thermal Conductivity

The CTE of the coke B anodes is significantly higher than the other anodes as expected due to the highly isotropic structure of the coke. It would not be possible to produce anodes containing a high percentage of this coke (>50%) due to thermal shock problems during anode heat-up in the cells. It works well as a blend coke and is used routinely in anode blends up to 10%. The CTE for the other anodes falls within typical industry ranges. The thermal conductivity of all the anodes also falls within normal industry ranges.

Anode Reactivities

Since the bench scale and pilot scale anodes were produced without butts material, there is limited value in drawing too many conclusions about the anode reactivity data. All the anode CO_2 reactivities are generally excellent with residues >90%. There is a good correlation between anode CO_2 reactivity residues (CRR) and coke and anode sulfur levels. There is also a good correlation between anode CO_2 reactivity dust (CRD) levels and coke and anode S levels. The R^2 for CRD and coke S level has an average value of 0.85 across all labs and for CRR, the average is 0.75.The correlation between reactivity loss (CRL) is not as strong at 0.45.

Measurement of coke CO_2 reactivities by the ISO 12981 test was not part of RR19 but the R&D Carbon results for coke CO_2 reactivity are included in Table 1. The R^2 correlations between coke CO_2 reactivity and CRR, CRD and CRL were respectively; 0.64, 0.15 and 0.78.

The air reactivity residue (ARR) of the coke B anodes was the lowest of the five cokes (65%) which is not surprising given the significantly higher V (619ppm) and Na level (121ppm) of this coke. This was followed by the coke C anodes which were a little better with an ARR of 71%. This coke had the next highest V level at 404ppm.

Measurement of coke air reactivity was not part of RR19 but the R&D Carbon results are presented in Table 1 for each of the 5 cokes. The R^2 values for coke air reactivity vs anode reactivity residue (ARR), air reactivity dust (ARD) and air reactivity loss (ARL) range from 0.59 to 0.64. Despite the lower than average anode air reactivity results for coke C, this coke is used routinely at the 100% level at a smelter in the US. Further details can be found in another paper in these proceedings [14].

Discussion and Conclusions

The primary objective of this part of the RR19 study was to establish correlations between the various coke bulk and apparent density tests and anode densities. A secondary objective was to look at correlations between other coke properties and anode properties. The study showed that all VBD/TBD procedures in common use through the industry do a good job of predicting optimum pitch levels. Their ability to predict baked anode densities is not as reliable and none of the VBD/TBD tests or the Hg AD test stood out as being any better than the others.

The scope of this study was limited so it is not possible to draw definitive conclusions about coke and anode density correlations. The study does, however, highlight the relative complexity of the anode production process. Many factors affect baked anode density and coke porosity/bulk density is one of these factors. Anode plants have gotten much more capable over the last 20 years and many anode producers are making high quality, high density anodes from cokes that would have been regarded as high porosity cokes in the past.

With the above in mind, it probably makes sense for the industry to use VBD/TBD tests with the highest level of repeatability and reproducibility. As discussed in the companion paper [9], this means tests without extensive sample preparation such as the ISO 10236 test and a new ASTM procedure being developed using natural fractions. These tests will at least allow reliable tracking of coke bulk density trends over time and between different coke sources.

The anodes produced with coke B produced the most unusual results in this study which is perhaps not surprising given the highly isotropic structure of this coke. It is likely that the granulometry was not optimum for this coke and a lower percentage of fines would likely give better results for anode density and permeability. The significantly lower electrical resistance of anodes made with this coke was of interest and highlights the lack of correlation between anode ER and coke SER. The mechanical strength of these anodes was also significantly higher than the other anodes as was the CTE.

Most of the other anode properties were in line with expectations. Anode reactivities correlate quite well with impurities such as sulfur and vanadium but care needs to be taken when interpreting these results in the absence of butts and the typical increase in sodium and calcium levels this causes.

It is unlikely that another RR on this scale will be attempted. Further RR's are planned however to support the development of a new ASTM bulk density test.

Acknowledgments

The effort required to coordinate and complete this RR study was large. The production of bench and pilot scale anodes was a major expansion of the RR and the authors wish to acknowledge the large effort and excellent work by R&D Carbon to complete this part of the RR. Thanks are also extended to the 28 labs that participated in the RR.

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