DETERMINATION OF COKE CALCINATION LEVEL AND ANODE BAKING LEVEL – APPLICATION AND REPRODUCIBILITY OF L-SUB-C BASED METHODS

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Abstract

The average crystallite size (L-sub-c or L_C) is an important property of carbon materials for aluminium electrolysis; L_C is used for characterizing the petroleum coke calcination level and sometimes also to estimate the baking level of anodes. This paper discusses problems when comparing L_C results from different laboratories using precision statements from ASTM and ISO standards. The main cause is peak broadening errors introduced by the XRD instrument and sample preparation. The L_C standards ASTM D5187 and ISO 20203 neglect these errors. Two ways are demonstrated to minimize the peak broadening effect to improve the standards, 1) by using thin sample thickness and 2) by embedding the coke in a high absorptive medium. Using L_C to determine the anode baking level is discussed and three practices are discussed; measurement on the anode directly or two methods for using a reference coke that is baked with the anode. It is shown that precision is better for the latter methods. Especially for underbaked anodes a baking level estimated from measurement of the anode L_C can be misleading.

Introduction

One of the earliest models of the structure of disordered carbons, based on X-ray diffraction, was published by R.E. Franklin in 1950 [1] and 1951 [2]. She proposed that graphitizable carbons were built up of hexagonal sheets of carbon rings, which formed small individual stacks, connected using cross-links. The stacks in the graphitizable carbons tend to pack with a preferential orientation, enabling them to merge into bigger stacks upon further heat-treatment. Petroleum coke, which is used for production of carbon anodes in aluminum production, belongs to this category. There are several other models that also have been proposed for the description of the structure of disordered carbon. But for the structure of petroleum coke, Franklin's model seems adequate and is currently the most commonly used.

The parameter used to describe the quality of the coke structure from this model is the crystallite size, described by the distances L_C and L_a. These are estimations of the size of each individual crystallite (= "graphite stack") contributing to the diffraction of the X-rays. L_C is the distance along the c-axis, perpendicular to the graphitic planes (= "crystallite height"); while L_a is the distance parallel to the planes (= "crystallite diameter"). These distances are drawn upon **Figure 1**. Of these two parameters, L_C is the one that is most relevant to carbon properties, and is also the easiest one to measure. It can be calculated by measuring the broadness of the main graphite peak in the XRD pattern. The L_C distance is usually expressed in ångström (Å) and increases upon heat-treatment.
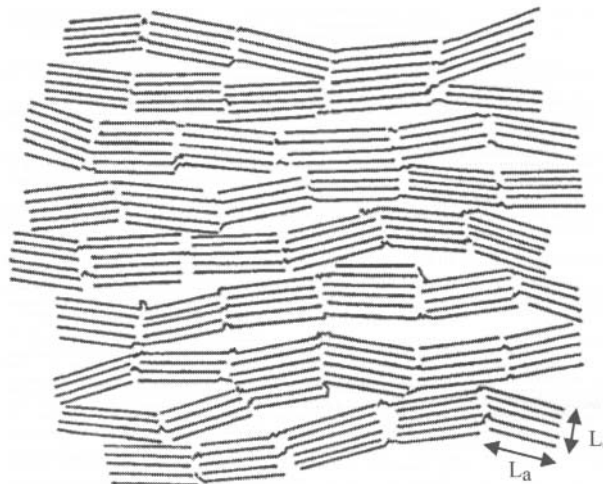

Figure 1: Illustration of Franklin's model of a graphitizable (but non-graphitic) carbon, from [2]

Application of L_C Measurements

The most common expression for crystallinity in calcined coke used in anodes for aluminium metal electrolysis today is the L_C value. The development of L_C with calcination temperature is illustrated in **Figure 2** for five cokes with different sulfur levels. The L_C growth is nearly linear in the temperature range of interest for green coke calcination. Figure 2 also illustrate that L_C growth differs between cokes, with cokes D and E reaching an L_C of 30 Å at 1200 to 1210°C calcination temperature and the lower sulfur cokes A, B and C reaching 30 Å at 1250 to 1260°C.
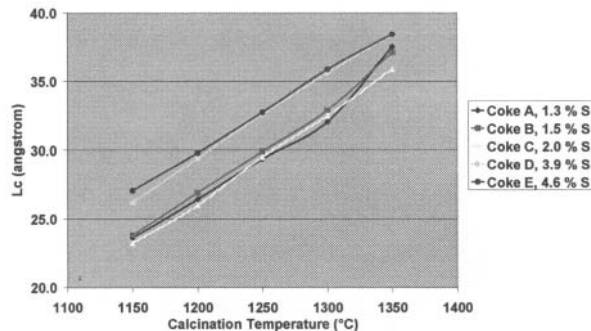

Figure 2: L_C development with calcination temperature for five cokes with different sulfur levels [3].

L_C is also useful for checking uniformity of heat treatment of a coke. Due to the nature of the process, rotary kilns will cause different L_C development for fine, medium and coarse grains as the tumbling action sends coarse grains closer to the fire [4,5]. The coarse grains then undergo higher L_C growth e.g. reaching an L_C of 34 Å while medium grains are 32 Å and fine 30 Å [4].

These aspects of coke quality illustrate the importance of having accurate and precise L_C measurements. This paper shows that there are several sources of error in the ASTM and ISO standards for measuring L_C in coke.

Precision of Current Standards

ASTM and ISO method precision is expressed by the within-laboratory repeatability, r, and the between-laboratory reproducibility, R, usually at 95 % confidence level. This is the r&R statement. It is obtained through an interlaboratory study (ILS), also termed a round robin (RR). It should be noted that the precision values obtained tends to be the best case as the voluntary participation attracts a good quality class of laboratories.

ASTM D5187
The method **ASTM D5187-91 (2010) - Standard Test Method for Determination of Crystallite Size (L_C) of Calcined Petroleum Coke by X-ray Diffraction** was recently revised with a new precision statement.[1] The revised precision limits are

Repeatability, r = 0.5 Å
Reproducibility, R = 1.9 Å

The earlier r&R[2] precision used in ASTM D5187-91 (2007) and previous revisions was L_C dependent, with r equal to $0.021*L_C$ and R equal to $0.11*L_C$. Especially between-laboratory comparison precision was improved with the 2010 revision. For example, for an L_C of 30.0 Å, the old R value of $0.11*L_C$ was 3.3 Å, significantly higher than the new R value of 1.9 Å. In a comparison between two laboratories, the R value states that determination on two samples of the same material the difference in L_C should be within 1.9 Å for 95 out of 100 such comparisons

ISO 20203-2005
The method **ISO 20203-2005: Carbonaceous materials used in the production of aluminium — Calcined coke — Determination of crystallite size of calcined petroleum coke by X-ray diffraction** was based on ASTM D5187-91 (2002) and had the same r&R precisions statement with r of $0.021*L_C$ and R of $0.11*L_C$ described in the preceding section. A 2010 revision with new r&R precision limits is being voted on.[3] The new r&R limits are

Repeatability, r = 0.5 Å
Reproducibility, R = 1.8 Å

The ISO r&R is comparable to the ASTM r&R. Five laboratories (Hydro & Slovalco) participated in both the ISO and ASTM round robins.

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[1] ASTM 2010 r&R: The research report is available from ASTM as RR: D02-1690 (D5187). Eleven laboratories analyzed two duplicates of each of seven samples spanning 21 to 34 Å (ångström).

[2] ASTM 1991 r&R: Ten laboratories analyzed two duplicates of each of six materials.

[3] ISO 2010 r&R being voted on: Eleven laboratories analyzed three duplicates of each of ten materials.

Precision Discussion
As a control on calcination, the L_C within-laboratory repeatability of 0.5 Å is sufficient. For the same coke it corresponds to control of the calcination level within 8°C, estimated from Figure 2. However, the between-laboratory reproducibility of 1.8 Å or 1.9 Å is not satisfactory as a precision limit for comparisons and a control of a specification. With this level of precision, the coke buyer's laboratory will have problems running a reliable monitoring program on coke shipments. The uncertain precision for the between-laboratory reproducibility is problematic.

A Critical Review of the Current Standards

The current paper will in the following section argue that there are many problems with the current ASTM and ISO standards for measuring L_C in coke. Since the relevant sections of these standards are identical, a reference to "the standard" in this paper will apply to both ASTM D5187-91 and ISO 20203. Quotations are emphasized in italics with section number from the ISO 20203 standard.

9.1.2 Determine the average low and high backgrounds (Points A and B, respectively) on the diffraction scan and connect them with a straight line.

This is not as easy as it sounds, and the illustration to how to do this (**Figure 3**) is not accurate. The starting point A should have been set at a lower angle to include the entire peak, perhaps at 15° in this case. The broader the peak is (lower L_C), the harder it is to decide where the peak actually starts. A better way to do this would be to use a mathematical algorithm to curve-fit the background and use either a derivative of the background-subtracted curve to determine where the peak starts and stops, or use a fixed angle window (which probably is a more reproducible approach).

9.1.3 Construct line CD parallel to line AB, and going through the apex of the peak at point G [(hkl (002) at 0,335 nm]. Draw the line such that, if the peak is irregular, it will pass through the average of the irregularities.

It is not mentioned in the standard what an "irregular" peak is, which leaves it to the analyst to subjectively determine good data from bad, and allows a possible arbitrary correction in the analysis. For FWHM determination, the three lines AB, CD and EF should be parallel, in the illustration they are not. Also, the theoretical d_{002} peak of graphite at 0.335 nm corresponds to an angle 2Θ at 27.38° (for the typical Cu Kα radiation at λ = 1.54056Å commonly used in XRD equipment). This angle is illustrated by the authors of the current paper as a dotted line in Figure 3. Petroleum cokes usually have the main peak shifted a couple of degrees lower, because Franklin's model is not exact for cokes. An alternative model [6] is where the structure of disordered carbons is viewed as graphite sheets with a various amount of interstitial carbon atoms. From this model, a soft carbon where all possible interstitial sites are present has an interlayer spacing of 0.344 nm. Petroleum cokes have a d_{002} spacing fairly close to this. The illustration in the standard show the usual peak which is shifted, but the standard do not explain why the peak is shifted nor explain how to deal with the shifted peak.
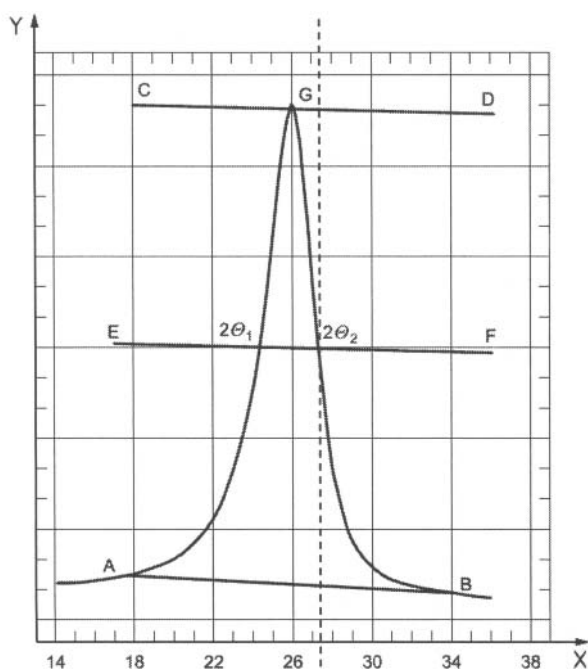
Figure 3: Calculation of the graphite peak broadness by the ASTM D5187-91 / ISO 20203 method. Y axis is intensity and X axis is 2Θ angle.

9.1.4 Determine the full-width half maximum (FWHM) of line AB. Construct line EF such that it intersects the peak at half of its maximum value. The points at which EF intersects the peak are 2Θ₁ and 2Θ₂, respectively.

This instruction is good for its simplicity, but modern computerized X-ray software will usually already have a built-in function for determining the half-width of a peak automatically, which will be easier to use and much more accurate. A potential problem with this approach is that the actual curve-fitting procedure used inside the software may be unknown and may vary between software packages. The standard should ideally specify how to do this mathematically or algorithmically.

9.1.5 For computer simulation based on the intensities recorded at 0.2° intervals, produce a mathematical representation of the diffraction curve. Determine the baseline, peak, peak height, and half-peak height to produce the half-peak height angles, 2Θ₁ and 2Θ₂, as above.

The 0.2° step size is a hangover from older generation diffraction equipment, where low count rates required that compromises in resolution were made to allow data collection at a reasonable speed. Modern PSD detector systems offer in the order of 100-200x the counting rate of old point detectors, making such compromises unnecessary. The choice of such a large step size is also very problematic when collecting data on highly carbonized materials, which may exhibit a peak width as narrow as 0.05°.

9.2 Determine the mean crystallite height L_C (derived from Scherrer equation)

$$\overline{L}_c = \frac{0.89\lambda}{2(\sin\theta_2 - \sin\theta_1)}$$

This equation is a derivative of the Scherrer equation. It is easy to use, but not accurate. Frank R. Feret has described [7] the modifications Alcan has applied to the ASTM method, and explains why. Feret's comment on the ASTM method's use of the derivative equation is as follows: *"This approximation is valid only when Θ = (Θ₁ + Θ₂)/2 and β are both small. Most of the calcined coke peaks are asymmetric and some are very asymmetric. Therefore, Θ ≠ (Θ₁ + Θ₂)/2 and 2Θ₁ - 2Θ₂ is not small. Moreover, it seems that because the original Scherrer equation itself is simple, there is no need for the approximation."*

The original Scherrer equation (which Feret prefers) is given as

$$\overline{L}_c = \frac{0.89\lambda}{\beta\cos\theta}$$

where β is the peak integral breadth (IB) or full width at half maximum (FWHM) and Θ is the angle at the peak position. The authors of the present paper assumes that the reason for using the derived equation in the standard is that it is slightly easier to use, since the input is Θ₁ and Θ₂, which is measured manually on the XRD scan. However, modern XRD software using a built-in function for the FWHM measurements would instead report β and Θ directly, which can then be input to the more correct Scherrer equation. Hence, there is no reason to use the derivative equation instead of the original equation.

It is mentioned in the standard that the use of the constant 0.89 is "arbitrary". Feret also comments on this: *"The Scherrer constant depends largely upon the crystallite shape, the (hkl) indices and the definitions taken for β and L_C. Various investigators have assumed values from 0.70 to 1.70 for this constant. For cokes it is set equal to 0.89 for the sake of uniformity in published results."*

The value of 0.89 is not truly arbitrary, as the standard suggests – it actually derives from the assumption that the sample is comprised of spherical particles of cubic symmetry. Further, it is related to the method used to measure the peak width. The value of 0.89 is actually derived for the case where the integral breadth of the peak is used, whereas the standard stipulates that the FWHM is used. In this case a value of 0.94 should be used. While this makes little difference to the repeatability and reproducibility of results, it does lead to a systematic error in the absolute values measured and their comparability with values obtained by other techniques.

The ISO standard has this closing remark in section 9.2:

The above equations make the assumption that the true line width is equal to the measured width, the contribution of instrumental line broadening is negligible.

This assumption is manifestly wrong. Every diffractometer has an inherent linewidth which is a function of many variables, including the measuring radius, detector system, the radiation spectrum used and the choice of beam collimating slits and optics. The differences between instruments are not insignificant and ignoring inherent resolution characteristics results in a number of errors to the reproducibility of L_C values. The most significant contributors are the radiation spectrum and the instrument optics.

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However, one could argue that the petroleum cokes usually have a FWHM much larger than the instrument contribution, so that the latter can be neglected. A potential problem with the "instrumental line broadening" is that XRD software usually has a function for removing the secondary ($K\alpha_2$) X-ray peak, which will be present unless the instrument is equipped with a monochromator. This function may be undocumented, and its use can have unpredictable results on the very broad coke peaks. The instrumental broadening is also influenced considerably by the setup of divergence slits in use, so two different instruments can have a considerably different resolution.

Earlier Proposals to Improve Methods for Measuring L_C

The paper by F. R. Feret [7] provides important understanding of the relation of the d_{002} peak shape to the degree of graphitization of carbon, and suggest to use computerized profile fitting methods to calculate the L_C values. Iwashita et.al. [8] published in 2004 a proposal for a new standard procedure of X-ray diffraction measurements on carbon materials. Currently this is by many scientists regarded as "state of the art" for L_C measurements. The main differences to the ASTM and ISO methods are 1) Silicon is added as an internal reference to the peak position and 2) The X-ray pattern is corrected for angle dependent factors. These include the Lorentz factor (L), Polarization factor (P), Absorption factor (A) and Atomic Scattering factor (Fc).

The effect of using an internal reference is largest for high crystalline carbons. The effect of the angle dependent corrections is largest for low crystalline carbons, and is therefore relevant for cokes. The application of the angle dependant corrections to the X-ray pattern will increase the measured L_C value. Whether or not the application of the angle dependant corrections will improve the between-laboratory precision has not yet been investigated. It is relatively straightforward to perform such a study since it only requires a purely mathematical treatment of the collected X-ray patterns and no instrumental changes.

The Effect of Sample Preparation

Carbon has a very low X-ray absorption coefficient[4]. A consequence of this is that the X-rays will penetrate relatively deeply into the sample used for X-ray diffraction, giving diffracted reflections also from a volume below the sample surface. This results in a broadening of the diffraction lines, so that the FWHM calculates to a lower L_C value than the actual value. The amount of broadening (and consequent lowering of the measured L_C value) will depend on the sample preparation method and the sample holder in use.

There are two ways to reduce the absorption effect:

1) Prepare a very thin sample where the penetration depth can be neglected
2) Mix the carbon with something that absorbs X-rays better than carbon, without introducing extra peaks in the graphite peak interval of carbon

For 1), a commonly used method to prepare a very thin sample is to suspend the powder in a volatile solvent (e.g. isopropyl alcohol) and place some droplets of this mix onto a polished surface of a

silicon single crystal, which is oriented in such a way as to give no X-ray reflections in the scanned angle range. Sample holders made for this use are commercially available. Upon evaporation of the solvent, a very thin layer of sample powder will be distributed across a surface which has no X-ray reflections. The lack of reflections from the silicon crystal will ensure a good signal-to-noise ratio, which may be necessary because of the lower X-ray intensity from a sample prepared this way.

For 2), when mixing the carbon with something of higher X-ray absorption, these requirements must be met:

a) the mixing compound must have a much higher X-ray absorption than carbon
b) the mixing compound must not have reflections near the d_{002} peak of carbon
c) the mixing compound must be inexpensive and not be hazardous to the environment (since it would be used as a disposable)
d) the compound must have a similar grain size/shape so that it mixes well with the carbon powder

Preliminary investigations has shown that while it is relatively easy to find compounds that satisfies requirements a), b) and c), this is not the case for requirement d). Carbon powder consists of flaky particles that form a low density powder. The best candidates satisfying both a), b) and c) are pure metals and their borides and carbides. These compounds have high densities, and tend to form rounded grains when milled. This makes it difficult to mix such powders homogeneously with carbon, lowering the reproducibility of the procedure.
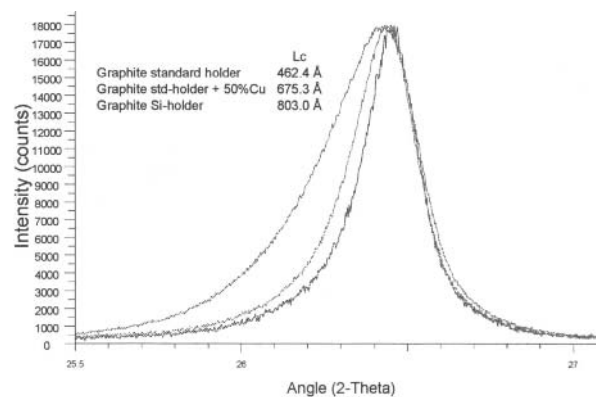

Figure 4: The effect of X-ray absorption

The effect of the X-ray absorption and the improvements proposed in this paper are illustrated in **Figure 4**. The patterns shown are of a graphite material, where the relative contribution from the absorption broadening is large. This example represents therefore a worst case situation. The red line shows the pattern using a standard sample holder, where the depth of the sample is about 1 mm. The blue line shows the pattern using a thin sample on a single crystal sample holder. The green line shows a pattern collected from an absorptive mix of 50% carbon and 50% copper powder in a standard sample holder. The absorption of the copper[5] reduces the X-ray penetration depth into the sample, giving a narrower peak. But the peak is still not as narrow as with

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[4] 4.576 cm²/g at 8 keV, data from http://physics.nist.gov/

[5] 52.55 cm²/g at 8 keV (11 times higher than carbon)

the silicon sample holder. The FWHM of the pattern collected on the silicon sample holder is about 50% of the normal holder, resulting in a much higher L_C value.

Using a silicon sample holder seems to be the best method, but may be less reproducible, as the distribution of the powder of the carbon-solvent mix may be too unpredictable to be used as a standard procedure for quantitative measurements of L_C in carbons. At the time of writing, no comparative studies have been performed on this issue.

The Anode Baking Level – Methods Using L_C

Three methods will be discussed:

1. The direct measurement of the anode L_C
2. An indirect method using a reference coke that is calcined with the anode during baking
3. The indirect method using the reference coke, with the addition of a calibration linking the reference coke to a temperature scale

It has been shown that between-laboratory precision for L_C is 1.8 to 1.9 Å in the ISO and ASTM methods. This means that laboratories can report L_C-values that are systematically 1.5 Å off each other even when using exactly the same raw materials. For this reason, between-laboratory comparisons based on methods 1 and 2 can be troublesome to interpret. However, successful between-laboratory comparison is possible, and can be achieved if the laboratories use a common reference, a calibration. This is the basic assumption of the equivalent baking level method. By using a common temperature scale, the equivalent scale, the L_C analysis for anode baking level can be harmonized at any laboratory. The key is the use of the same reference coke and its calibration.[6]

The method is standardized as **ISO 17499 (2006) — Carbonaceous materials used in the production of aluminium — Determination of baking level expressed by equivalent temperature**. Central to the method is establishing the analytic relationship between the individual laboratory L_C measurements and the temperature scale for the reference coke heat treatments.

Between-laboratory Precision
The major advantage of this method compared to using an uncalibrated reference coke is the improvement in comparisons between laboratories. Note that, as the equivalent scale value is a calculation from the L_C of the reference coke, the repeatability, or within-lab precision, will not be better than for the L_C measurement itself. The use of the calibration flushes out most of the interlaboratory differences in the L_C part of the analysis giving a between-lab precicion closer to the within-lab precision. The precision statement of ISO 17499 is based on a 2003-2004 interlaboratory study.[7] The r&R limits are

Repeatability, r = 9°E
Reproducibility, R = 14°E

The gain in precision is considerable, see Table 1, which presents the relative precision of the three methods as the ratio of R versus the expected range of measured values.

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[6] A good reference coke is a low sulfur single source sponge petroleum coke such as cokes A and B in Figure 2.
[7] Inter laboratory study with ten laboratories; three duplicates of each of ten materials.

Table 1: Comparing the between-laboratory precision for the three methods for estimating anode baking level.

Method	Range of values	Between-lab precision	Rel%
1. L_C anode	28 to 34 Å	1.8 to 1.9 Å	31
2. L_C reference	17 to 37 Å	1.8 to 1.9 Å	9
3. Equivalent temperature	1000 to 1400°E	14°E	3.5

The Equivalent Baking Temperature Scale
The scale is based on a calibration set of eleven calcined reference coke samples heat treated from low (underbaked) level to high (overbaked) level. This ensures a range for all baked anodes. On the scale, normal baking level is around 1230°E, underbaked anodes are below 1150° and overbaked above 1330°E. The unit °E instead of °C is meant to emphasize that the measured temperature is an *equivalent* heat treatment of the reference coke.

Measuring L_C on Anodes Directly
A reasonable criticism of the equivalent method is that establishing and using the method is more complex and costly than method 1 (the anode L_C). Analyzing anode L_C on the anode cores is simpler and less expensive than other methods and would be a method of choice. Hydro has investigated this possibility, but extensive testing showed the relationship given in **Figure 5**.
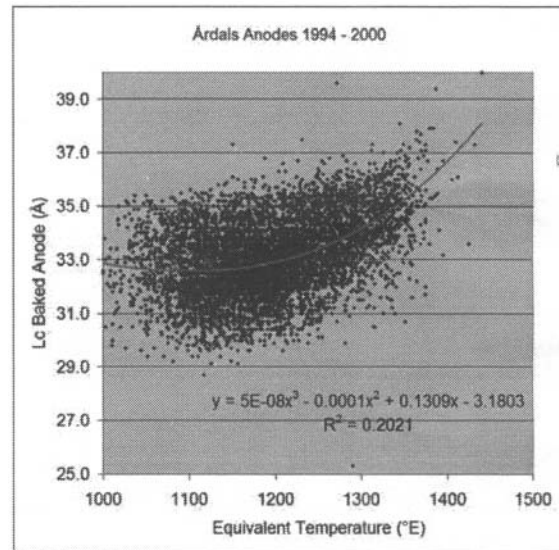

Figure 5: Anode L_C development when plotted versus the equivalent baking temperature method.[9]

The most obvious weakness is the horizontal part of the curve, which will make the detection of underbaked anodes difficult, if not impossible. The normal baking level is 1230°E[8], and the chart indicates poor detection from that baking level and downwards. That is problematic, and adding to that uncertainty is the spread in values for one L_C measurement, given the within-laboratory repeatability of 0.5 Å. Taking a measurement of 33.0 Å as an example, the baking level can be any value from 1050 to 1300°E. And then comes additional noise such as incidents of

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[8] Based on acceptable specific electrical resistance, strength and carboxy dusting levels.

highly under- or overbaked butts, the calcination level of the cokes, the coke blends, recipes and type of pitch.

To illustrate this issue in a more controlled study, a series of designed pilot anodes were made with three types of coke. Each batch was baked to three baking levels, slightly underbaked (1150°E), normal coke calcination level and slightly overbaked (1330°E). The L_C values are plotted in **Figure 6**. The development of the anode L_C is difficult to quantify below baking level 1230°E – the change in L_C is small compared to the repeatability of the L_C analysis method.
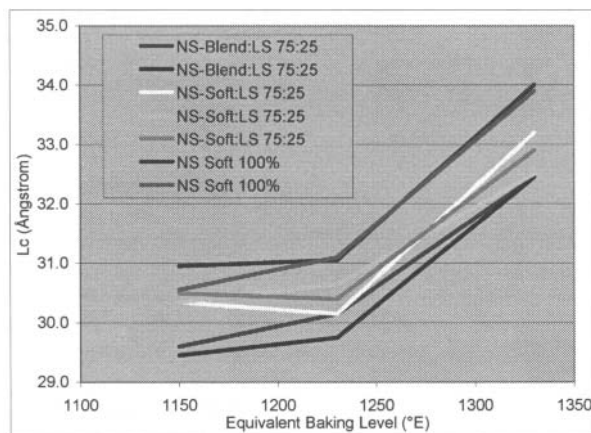

Figure 6: Anode L_C plotted versus the equivalent baking level, for pilot anodes made using different types of coke.

Conclusions

The L_C value development during calcination of coke is suitable for quantifying the heat treatment of the coke. However, L_C analysis as described in ASTM D5187 and ISO 20203 has some errors and weaknesses leading to poor between-laboratory reproducibility. The paper discusses the improvements and also potential drawbacks:

• Correct the errors in ASTM / ISO standards – *a drawback is that new values cannot be compared directly to old*

• Improve sample preparation using a Si sample holder or an absorptive mix – *it will be a challenge to gain acceptance for this across labs*

• Harmonize the computerized curve-fitting and calculation of L_C – *it will be a challenge to have software vendors standardize these methods*

It is suggested to ASTM Committee D02.05 and ISO Technical Committee 226 that they look into these proposals for improving the L_C analysis, thereby improving its commercial relevance.

The paper also discusses use of L_C analysis to determine the anode baking level. Three practices are presented; measurement of L_C on the anode directly and two methods for using L_C of a reference coke that is baked with the anode. It is shown that between-laboratory comparison is best for the equivalent baking level method, ISO 17499, as it avoids some of the current weaknesses in D5187 and ISO 20203.

For the direct anode L_C method it is shown that precision is comparatively poor, and especially for underbaked anodes there is an inherent risk in the method of reporting with low accuracy.

For Hydro, the gain in precision and the necessity of comparing anodes from different baking furnaces is sufficient reason for using the somewhat more complex equivalent method. And it has been shown that the equivalent temperature yield baking level information that is significant for anode quality in electrolysis, specifically carboxy dusting and current efficiency [10].

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