

Determination of the crystallinity of calcined and graphitic cokes by X-ray diffraction†

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Although the Scherrer equation has been the basis of the XRD method for the determination of the crystallinity of calcined coke, the most accurate interpretation of coke crystallinity involves profile analysis. In this paper, the general background related to coke crystallinity (L_c) determination is described, Alcan and ASTM standard methods are compared and line breadth as a function of crystallite size is characterized. At present, it is generally understood that the coke graphitization process occurs during calcination of coke and is related not only to the calcination temperature, but also to other parameters (composition, process conditions). As coke graphitization affects electrolytic cell performance, it should be monitored and quantified. The methods used for the determination of the degree of graphitization (DOG) and described in the literature are useful for cases involving calcination temperatures above 1800 °C. These methods are critically examined. Because known methods do not apply to the low-temperature graphitization processes (1200–1500 °C), a new approach was conceived. It is based on profile line analysis and an estimate of the graphitic contribution in coke specimen can be made. The new approach involves a commercial X-ray diffractometer and a corresponding software package and relies on profile fitting of the (002) coke peak. A calcined coke specimen or the presence of a graphitic portion in calcined coke can then be clearly recognized. During the profile fitting operation it is possible to resolve the graphitic portion from the calcined peak profile mathematically, and then to express it quantitatively. Among several profile functions that were tested, the split Pearson VII was found to give the best fit to data corresponding to calcined coke. A novel equation by which the DOG can be estimated in calcined coke is given. Selected examples of the DOG determination are described.

Keywords: Calcined coke; graphitic coke; crystallinity; degree of graphitization; profile line analysis; X-ray diffraction

Over the past several decades, X-ray diffraction (XRD) has generally been used for the determination of crystallinity (known as the parameter L_c) in green and calcined petroleum coke. Most industrial laboratories involved in L_c determination apply their own or the ASTM method (D 5187) which appeared in 1991. The methods cover the determination of the mean crystallite thickness of a representative, pulverized sample of calcined coke by interpretation of an XRD pattern produced using conventional X-ray scanning techniques. The XRD pattern is obtained in a scan of the carbon (002) reflection

covering the ranges 14–34 °2 θ (Cu tube) or 19–39 °2 θ (Co tube). In the far past, the data were recorded in the form of a strip chart. Initially, the interpretation of the scan was carried out manually; it was based on a graphical procedure. Later, computer software designed to read and store the angular and intensity measurements became involved in the task. Whether manual or computer aided, the determination of a baseline is done first, followed by peak height, half peak height and the half peak height angles 2 θ_1 and 2 θ_2 .

Differences between Alcan and ASTM methods

Sample preparation

The ASTM method allows the use of any of the following techniques for packing the coke sample into the diffractometer specimen holder: back filling, front filling, side loading and even briquetting. Probably the document needed to include the practices of various users. On the other hand, the Alcan method recommends that a cavity slide sample (a packed powder mount) be prepared. The point is that the way in which the coke specimen is 'packed' affects the results. The packing influences X-ray penetration of the specimen and this clearly affects the peak broadening mechanism. In the case of a typical matrix the X-ray beam penetrates only approximately 20 μm , so that the specimen appears thin. However, for carbon (which is a light matrix) the X-rays penetrate the whole sample. Consequently, diffracted beams from underlying layers cause line broadening. This means that the resulting L_c values are artificially deflated, which can only be tolerated for cokes. When analyzing anthracite and graphite a very thin smear should be used. This practice does not add broadening due to X-ray penetration, but it does lower measured intensity.

In general, there is an inverse correlation between the thickness of the specimen used and the L_c value obtained. Therefore, although results obtained with different preparation methods are acceptable for process control, they should not be compared with those from other places. In other words, any inter-laboratory comparison should involve results obtained using the same sample preparation technique.

Interpretation of measured data

There is a difference in the calculation procedure in the ASTM method and Alcan standard method. Both methods are based on Scherrer's equation.¹ In the Alcan method the Scherrer equation employed is

$$L_c = \frac{0.89\lambda}{\beta \cos\theta} \quad (1)$$

where 0.89 = Scherrer's constant, λ = radiation wavelength, θ = angular position of the peak of interest and $\beta = 2\theta_1 - 2\theta_2$ (in °2 θ); pure diffraction broadening represented in

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the case of coke by FWHM (full width at half maximum). In the ASTM method, the Scherrer equation has been replaced by

$$L_c = \frac{0.89\lambda}{2(\sin\theta_1 - \sin\theta_2)} \quad (2)$$

This approximation is valid only when $\theta = (\theta_1 + \theta_2)/2$ and β are both small. Most of the calcined coke peaks are asymmetric and some are very asymmetric. Therefore, $\theta \neq (\theta_1 + \theta_2)/2$ and $2\theta_1 - 2\theta_2$ is not small. Moreover, it seems that because the original Scherrer equation itself is simple, there is no need for the approximation.

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.

Line breadth of coke as a function of its crystallite size

With powder samples, a peak observed at some particular diffraction angle, 2θ , is generally due to diffraction from several symmetry-equivalent planes. A pure diffraction maximum produced by a crystalline powder has a natural profile which is determined largely by the crystallite-size distribution.² Crystallite size is defined as the size of a microdomain that causes X-ray diffraction. Diffraction is more sensitive to the microdomains and less to the particle size. A particle, even if it looks like a perfect crystal, typically is composed of many crystallites. They feature numerous lattice imperfections and small mosaic blocks. An (hkl) reflection is caused by crystallites with (hkl) planes parallel to the specimen surface. A calcined coke is considered as a two-dimensional, random-layered structure. On a diffraction pattern such a structure is revealed by the presence of only $(hk0)$ and $(00l)$ reflections. In the case of graphite-type materials, crystallites are stacks of graphitic carbon platelets located parallel to one another.

The shape of a diffraction peak is important in the measurement of lattice distortions, whereas its breadth is significant in the determination of crystallite size. The geometrical properties of the diffractometer introduce aberrations into the pure diffraction profile which cause it to be more or less asymmetric, broadened and displaced from its theoretical 2θ angle. As a result, the profile shapes obtained with a conventional powder diffractometer are not easily described. In order to estimate the magnitude of the peak broadening the Scherrer equation was used first. In the Scherrer equation the β parameter represents the pure diffraction broadening by the sample contribution alone. In reality, what is measured is the 'observed' peak breadth $B = \text{FWHM}$. This is because the true sample contribution β is superimposed by broadening (b) caused by the instrument itself. Determination of the pure diffraction breadth β constitutes a major effort associated with crystallite-size analysis. Scherrer's original postulate was that the peak breadths are strictly additive² so that $B = \beta + b$. This has since been found not to be generally applicable. Warren³ derived the relationship between integral breadths that $B^2 = \beta^2 + b^2$ where the pure diffraction and instrumental broadening profiles are both assumed to have a Gaussian shape. However, it has been shown by various other investigators that the instrumental profile follows other functions more closely.

Depending on the class of diffractometer and corresponding resolution, the b contribution is in the range 0.07–0.15 $^\circ 2\theta$. For example, using a Philips PW 1700 diffractometer the measurement of the 3.35 Å graphite line gives a value of about 0.13 $^\circ 2\theta$. A similar result (0.12 $^\circ 2\theta$) can be obtained from a (100) quartz line. By contrast, more recent diffractometers contribute broadening of about 0.05–0.1 $^\circ 2\theta$, depending on the diameter of the focal circle and applied slits. For the calculations carried out below, a value of 0.10 $^\circ 2\theta$ was assumed. Since values of k , λ , b

and 2θ are given, a calibration curve of L_c versus B can be constructed. For a Co tube, assuming after Warren³ that $\beta^2 = B^2 - b^2$ and substituting the given values in eqn. (1), we have

$$L_c = \frac{0.89 \times 1.789 \times 57.3}{\sqrt{(B^2 - 0.10^2)} \times 0.966} \quad (3)$$

where the factor 57.3 is used to convert the value of β from degrees to radians. Hence the line width is given by

$$B^2 = \frac{94.44^2}{L_c^2} + 0.10^2 \quad (4)$$

This relationship is presented in Table 1 for crystal dimension of various materials.

The contribution of the instrumental broadening to the sample profile is stable. However, its significance increases rapidly with crystal dimension. In calcined coke measurements, the instrumental contribution (parameter b) is negligible and almost never considered. The measured breadth (FWHM) is assumed to represent the sample contribution. In the case of anthracite the contribution due to the instrument must be taken into account. For crystals as large as a few μm (alumina, gibbsite), the instrumental broadening is dominant. The sample contribution to the measured peak width is too small to be measured.

Modern determination of the FWHM is based on a procedure, called profile fitting. In this procedure, measured data are stored in the form of a digital scan. Next, a computer simulation produces a mathematical representation of the entire line profile $f(\epsilon)$. Background is compensated for automatically. Smoothing (by means of a third-degree polynomial method) is optional, but improves the fit quality remarkably when activated. Computation leads to the following results: (002) peak angular position; peak intensity (at the top, in counts s^{-1}); peak net area (normalized); FWHM; integral width; and centroid (centre of gravity).

The normalized peak area is peak intensity (in counts s^{-1}) multiplied by the measuring step in degrees. Such areas are not dependent upon the measuring conditions. The FWHM is the overall width of the line profile at half-maximum intensity measured above the background. The FWHM used in the past was measured manually (with a ruler) based on peak scan. The integral width is defined as the integrated intensity of the line profile above background (peak net area), divided by the peak intensity:⁴

$$B_{\text{IW}} = \frac{1}{I_p} \int I(2\theta) d(2\theta) \quad (5)$$

To calculate the integral breadth one needs digital data corresponding to the peak profile. The centroid is a measure of peak location.

Using the Scherrer equation the L_c parameter was calculated next for each FWHM value. Table 2 gives an example of data which were generated using the DIFFRAC-AT software and a Siemens D5000 diffractometer.

The profile fitting provides not only the L_c parameter but also valuable additional information. Moreover, a graphical repre-

Table 1 Variation of line width ($^\circ 2\theta$) with crystal dimension

Material	Crystal dimension $L_c/\text{\AA}$	Linewidth, B ($^\circ 2\theta$)
Coke	30	3.15
	50	1.89
	100	0.95
Anthracite	500	0.214
Boehmite	1 000	0.138
Alumina, gibbsite	10 000	0.100
	100 000	0.100

sensation (XRD scan) of the coke peak is also available. In contrast to methods based on intensity measurement in a few predetermined places on the coke peak, profile fitting requires a much longer measuring time (now 14 min) which is dictated by the need to complete the (002) peak scan. Fortunately, most of the time the measurement is carried out automatically for a series of samples.

As far as calcined coke is concerned, the Scherrer equation (based on the determination of the FWHM) will remain the basis of the XRD method for crystallinity determination. However, for graphitic cokes this approach is largely inefficient and cannot be applied.

Determination of degree of coke graphitization (DOG)

Graphitic cokes

Calcined coke specimens are clearly defined by their profile shape and angular range. Graphitic cokes mark a gradual transformation from random structure to crystalline graphite. They tend to be marked by an unusual, asymmetric shape and specific range of the integral width and centroid. Even when a graphitic coke displays a symmetric profile, such a profile appears narrower than for a calcined coke, and it is shifted towards a larger diffraction angle (smaller d value).

The possibility of finding graphite in an ordinary calcined coke has been recognized for some time. However, if the L_c determination procedure has not been programmed to provide coke diffractograms, then they will not be available for inspection. In such a case, coke analyses are carried out automatically, and numbers corresponding to L_c are computer generated for each sample. The danger is that an automatic procedure may miss a graphitic coke.

Fig. 1 shows a graph which correlates coke calcination temperature with L_c for a series of samples. One sample calcined at 1300 °C indicates a much higher L_c value than

Table 2 Parameters obtained for coke samples

Parameter	Sample				
	1	2	3	4	Alcan standard
Peak position ($^{\circ}2\theta$)	29.92	30.02	29.76	29.0	29.96
Peak intensity/ counts s^{-1}	1611	1592	1424	1370	1256
Peak net area/ counts $s^{-1} \text{ } ^{\circ}2\theta$	5813	5677	5262	5578	5181
FWHM ($^{\circ}2\theta$)	2.970	2.958	3.069	3.510	3.560
Integral width	3.72	3.67	3.82	4.23	4.31
Centroid ($^{\circ}2\theta$)	29.58	29.57	29.52	29.47	29.42
$L_c/\text{\AA}$	31.78	31.91	30.76	26.52	26.9

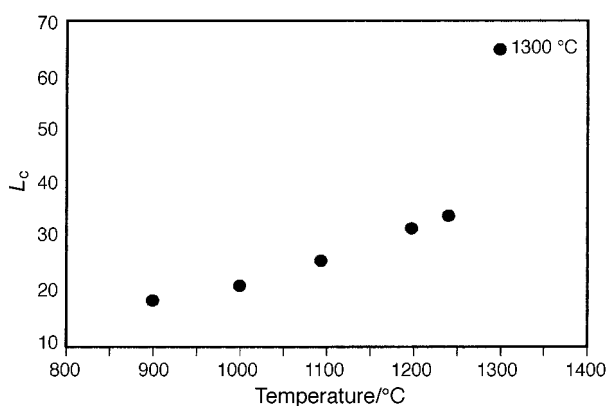


Fig. 1 Calibration curve of L_c versus temperature.

expected. In a subsequent XRD study this sample resulted in a diffractogram which did not resemble the rest (see Fig. 2). Fig. 2 is a superposition of three diffractograms covering the 20–40 $^{\circ}2\theta$ range that were acquired using identical recording conditions. The diffractograms correspond to calcination temperatures of 1200, 1250 and 1300 °C. The most intense, highly asymmetric and simultaneously the narrower peak is related to the sample calcined at 1300 °C. For this peak profile, the Scherrer equation simply does not apply. Clearly, it is a composite peak which is made of two distinct superimposed peaks. One peak corresponds to ordinary calcined coke and it shapes the left part of the peak shoulder, at a smaller 2θ angle. The other peak is shifted to the right (larger 2θ angle). It corresponds to the graphitic part of the coke material. The degree of coke graphitization (three-dimensional crystalline ordering) influences the amount of this graphitic portion in the sample, and consequently affects the angular position of the second peak and its intensity. Ultimately, under favorable conditions (temperature), the entire sample might turn into graphite. Fig. 3 illustrates XRD scans of two peaks measured using identical scanning conditions. The broad, weak peak is from a calcined coke specimen; the sharp and intense peak is from pure graphite. The distinction is clear.

If the initial analysis using profile analysis software, such as DIFFRAC-AT, indicates that the investigated coke peak is composed of two contributions, then a line profile analysis is required. Further analysis is carried out in two separate stages: (i) profile fitting (separation) of the two contributions and (ii) deconvolution of the instrument contribution from the resolved profiles and determination of their crystallinity and crystallite

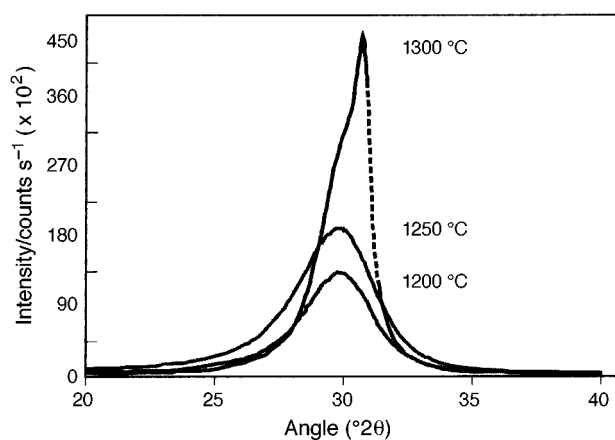


Fig. 2 Superposition of three diffractograms.

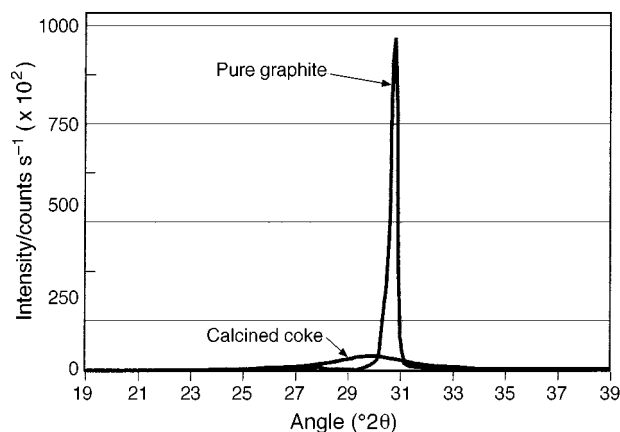


Fig. 3 Comparison of diffractograms for calcined coke and graphitic coke.

size distribution. The second subject will not be discussed in this paper.

Theoretical considerations

Mathematically, each profile of the observed maximum $h(\epsilon)$ is the convolution, or fold, of the pure diffraction profile $f(\epsilon)$ and the weight function of the apparatus $g(\epsilon)$:⁵

$$h(\epsilon) = \int_{-\infty}^{\infty} g(\eta)f(\epsilon - \eta)d\eta \quad (6)$$

where the variables ϵ and η have the same units as 2θ . The process for obtaining f from h and g is called deconvolution. In order to adjust a mathematical profile function to the measured data, least-squares refinement techniques are mostly used. Computer simulation produces a mathematical representation of the diffraction curve. To represent the expected profile shapes many mathematical models have been tried with varying degrees of success. Most models employ 12 intrinsic parameters to describe the instrument aberration and wavelength-dependent contributions to the profile, and three parameters to describe the sample-dependent variables of line position, height and line broadening.^{6,7} The number of parameters needed to define a line varies with the selected profile shape function and whether it is assumed to be symmetrical or not. Among the most important functions one can select are Lorentz (Cauchy), Gauss, Voigt (convolution of Cauchy and Gauss functions), pseudo-Voigt, Pearson VII.

The first three of these functions are symmetrical. Since the actual profiles are never pure Lorentz or pure Gauss, the corresponding equations have only limited practical value. The remaining functions are more complex as they require four parameters. When a line is not assumed to be symmetrical, it is treated as two half lines sharing the same location and height, but not the same shape—it is said that the function is split.

The problem with graphitic cokes is that the pure diffraction profile $f(\epsilon)$ is composed of two contributions, coke and graphite. Consequently, the two contributions must first be separated to obtain two separate line profiles. Only then can the instrumental contribution be eliminated by deconvolution. In practice, any of the above functions could be used to separate the superposed coke peak. The selection of an appropriate model function for the deconvolution is important, because errors incurred in this operation are transmitted to the next stage.

In order to confirm unequivocally the presence of a graphitic portion in the specimen and to resolve the (002) diffraction profile, a PROFILE program is run first. PROFILE is the new profile fitting program of the Siemens DIFFRAC-AT package which helps obtain accurate line locations, intensities and widths from resolved and non-resolved X-ray diffraction lines. The number of peaks to be resolved could be either identified automatically by PROFILE or set by the operator.

Experimental results

Data collection was achieved using a Siemens D5000 automated powder diffractometer. The instrumental details are given in Table 3.

The Siemens D5000 diffractometer was not equipped to work with monochromatic radiation ($K\alpha_1$), and therefore all measurements were carried out with a $K\alpha_1$ plus $K\alpha_2$ doublet. The presence of the α_2 component enhances the line broadening and introduces asymmetry into the profile. As a consequence, profile fitting has to be performed using $K\alpha_1$ plus $K\alpha_2$ profiles.

The α_2 elimination is achieved automatically by computation during profile fitting.

The initial experimental effort was aimed at selecting the best mathematical model to represent the (002) coke peak. A calcined coke characterized by a highly asymmetric profile shape was selected for the test. Fig. 4 illustrates examples of four different fits obtained with Gauss, Lorentz, Voigt and split Pearson VII mathematical models. Various individual parameters of the model were tried. Clearly, the split Pearson VII model provides the best fit between theoretical and measured line profiles. Consequently, the split Pearson VII function with undefined parameters was selected for profile fitting runs. If employed for a calcined coke, it needs eight adjustable parameters.

Next, a graphitic coke was used. Fig. 5 shows the outcome of the profile fitting in a graphical form. Two separate peaks were resolved from the original line profile. Selected numerical data corresponding to this profile fitting is given in Table 4. Significant new parameters are: angular (2θ) and d position of both peaks (\AA), peak height (counts s^{-1}) and integrated intensity (area) and FWHM ($^\circ 2\theta$). The goodness of profile fitting is estimated by reliability (Rel) and theoretical reliability (TR) parameters (%). The reliability is related to the difference between observed and calculated profiles. Obviously, the lower the number the better. The profile fitting results are saved as a DPF file. Using the data in Table 4, one can estimate the proportions of both contributions in the sample or the degree of graphitization.

Degree of graphitization (DOG)

In the past, it was generally assumed that the graphitization process depends on the calcination temperature, which is

Table 3 The Siemens D5000 measuring conditions used at Alcan

Radiation	Co- $K\alpha$
Detector	KeveX, Peltier cooled
Monochromator	No
Generator	40 kV, 40 mA
Divergence slit	Fixed, 2 mm (coke), 1 mm (graphite)
Step size	0.01 $^\circ 2\theta$
Scattering slit	2 mm (coke), 1 mm (graphite)
Receiving slit	0.6 mm (coke), 0.1 mm (graphite)
Scanning range	20–40 $^\circ 2\theta$ (coke), 25–35 $^\circ 2\theta$ (graphite and graphitic coke)
Measuring time	1 s per step (coke), 5 s per step (graphite and graphitic cokes)
Wavelength	1.7889 \AA
PHD window	20–80%
Sample changer	40 position
Sample spinner	On

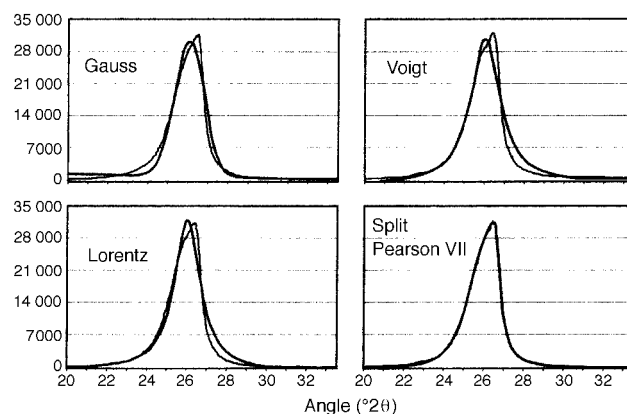


Fig. 4 Example of four different fits.

assumed to be high. At present we know that the graphitization process is related not only to the calcination temperature, but also to other parameters (composition, process conditions). Sørli and Gran⁸ proved that the cell operating temperature is sufficiently high for catalytic graphitization of the carbon lining to take place. With increasing graphitization, the size of the graphitic layers and the total number of such layers (contributing to the XRD process) increase continuously, whereas the apparent interlayer spacing decreases. In the open literature, the degree of graphitization (DOG) has been measured using one DSC and two different XRD methods.

The DSC method involves the specific heat capacity and is based on the two-dimensional model of phonon transport. By this method a single parameter, the in-plane Debye temperature, θ_D , characterizes the quality of graphene layers.⁹ The Debye temperature is the temperature at which all phonon modes are excited, and the specific heat capacity reaches a nearly constant value. The degree of graphitization, g , was defined as a function of the Debye temperature:

$$g = \frac{\theta_{D, \text{graphite}}}{\theta_{D, (T)}} \quad (7)$$

where g approaches 1.0 at high temperatures and is much less than 1.0 at low temperatures. The authors claim that the numerical values of g obtained by this technique are very close to those obtained by the XRD method involving the (002) interplanar spacing. However, the specific heat capacity is an indicator of bulk lattice quality and not of the planar stacking in one direction.

For typical non-graphitic carbons the interlayer spacing represented by the (002) peak is constant at 3.44 Å and in graphite it is 3.354 Å. Maire and Mering¹⁰ defined a degree of graphitization (g) as

$$g = \frac{3.44 - d_{002}}{3.44 - 3.354} = \frac{3.44 - d_{002}}{0.086} \quad (8)$$

where d_{002} is the average interlayer distance (Å) measured by XRD. Statistically, the degree of graphitization is the probability of parallel orientation for two consecutive graphite layers.

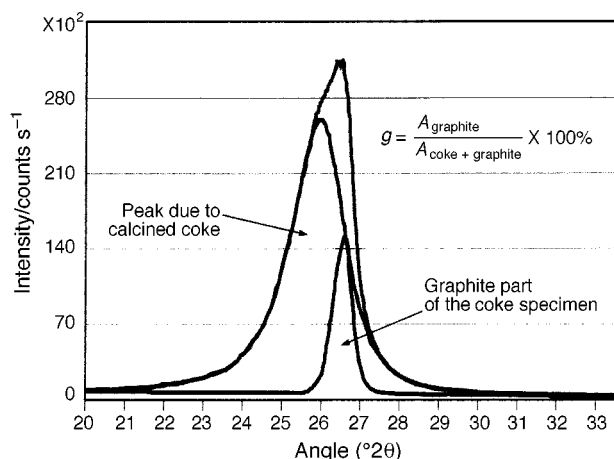


Fig. 5 Outcome of the profile fitting: new method.

Table 4 Example of profile fitting results

Peak No.	Function	Position		Height (counts s ⁻¹)	Area (normalized)	FWHM (°2θ)
		°2θ	d/Å			
1A	Split P VII	25.88	3.439	12 827	27 125	1.602
2A	Split P VII	26.50	3.359	7 374	4 768	0.569

Another XRD method is based on the intensity ratio of the (002) diffraction peak of the carbon specimen relative to a defined graphite standard:¹¹

$$g = \frac{I_s}{I_r} \quad (9)$$

where I_s and I_r are the X-ray intensity/mass ratios for the specimen and reference, respectively.

Eqns. (7)–(9) are considered useful for cases involving calcination temperatures above 1800 °C. This is because above this temperature the graphitic portion of the peak doublet becomes dominant and the error of the peak position corresponding to the graphitic portion becomes tolerable. None of the above methods provides an accurate estimate of DOG for the low-temperature calcination ranges. In contrast, using the data obtained from the PROFILE software one can now attempt to calculate DOG for samples calcined at low and high temperatures. This can be done in two ways.

First, knowing the d positions of two resolved peak profiles, left (L) and right (R) (Fig. 5), eqn. (8) can now be employed for profile on the right. However, we do not feel that eqn. (8) provides an accurate or adequate description of DOG. This is because various amounts of the graphitic material in the sample may feature the same d_{002} interlayer distance. In contrast, a ratio of the normalized surface area A of the graphite and carbon portions represents a better choice:

$$g = \frac{A_{\text{graphite}}}{A_{\text{coke} + \text{graphite}}} \times 100\% \quad (10)$$

We feel that the last definition represents reality most appropriately. For example, using the data in Table 4 (the area intensity), one can estimate the proportions of both contributions in the sample. Consequently,

$$g = \frac{4768}{4768 + 27125} \times 100 = 15\% \quad (11)$$

The degree of graphitization defined by eqn. (10) can be used to study differences among samples in a group. However, it has to be realized that the two resolved peak profiles have not yet been corrected for instrumental broadening. This needs to be done next in order to obtain results on an absolute scale. From the data in Table 4, it is apparent that the FWHM values for the two profiles are 1.60 and 0.569 °2θ, respectively. If the profiles are considered individually, the instrument contribution in each is represented by approximately 0.1 °2θ. Then, especially for the second profile, the specimen and instrument contributions are of the same order of magnitude. It has been suggested that the h profile should be a factor of 1.2 broader¹² than the g profile for successful deconvolution. Based on the limited initial data, this seems to be the case for graphitic cokes.

Discussion

Modern XRD software allowing profile analysis is a necessary tool in the analysis of coke crystallinity. When compared with the Scherrer equation the profile analysis constitutes a new benchmark in coke crystallinity determination. Additional new parameters such as integral width, peak position and centroid are important and should be collected and compared over a long run. Using profile analysis software, coke (002) graphical representation becomes available for every sample so that a simple visual examination allows first a quick evaluation of a potential specimen graphitization. If either from a visual observation of the (002) peak profile or from the software analysis of the numerical data there is no confirmation of calcined coke, then the Scherrer equation simply does not apply. In such a case the Scherrer equation cannot be used to calculate

crystallinity from the FWHM parameter measured for the (002) coke peak. Consequently, a different analytical procedure must be used. The new procedure involves profile fitting and provides a tool for clear recognition of a calcined coke specimen, or the presence of a graphitic portion in calcined coke. Using software such as DIFFRAC-AT (PROFILE), it is not only possible to resolve the graphitic portion from the calcined coke peak profile mathematically, but also to express it quantitatively. The numerical data generated by PROFILE can be used in an additional stage to deconvolute the instrumental contribution from both profiles, and to evaluate the respective crystallite size and crystallite size distribution. The details will be covered in a technical communication.

Development of analytical methodology related to the characterization of graphitic cokes could be continued even further. Using the Rietveld XRD approach and a full diffractogram analysis rather than a single peak, it should be possible to obtain additional information such as the probability P of finding a 3R stacking fault, the strain parameters, the preferred orientation and the L_c/L_a anisotropy ratio.

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