Review

Graphite and carbon powders for electrochemical applications☆

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Abstract

Graphite and carbon powders occur in many different forms and emanate from many different sources. A systematic classification is given based on the degree of crystallinity, from low-structured coke and coal products to macro-crystalline graphite and nanocarbons. In the family of 'carbon black' the highly conductive products acetylene black and PUREBLACK® Carbon are discussed. Graphite can be either natural or synthetic. Depending on the formation of the natural graphite, the morphology can vary from micro-crystalline to macro-crystalline. Expanded graphite is described as a chemically treated, extremely thin, flake graphite. Synthetic graphite products are classified as either primary material or secondary. The latter is defined as a byproduct of graphite component- and electrode-manufacturing.

The characterization of graphite and carbons is also discussed. A very useful characterization tool – the 'PSTP' model (purity, structure, texture, particle size) – has been formalized. These four basic parameters determine most of the application parameters such as conductivity, lubricity and elasticity. The influence of these four basic parameters on the application properties is examined.
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Keywords: Natural graphite; Synthetic graphite; Expanded graphite; Carbon black; PUREBLACK® Carbon; Desulco® and Acheson processes

Contents

1. Introduction

In this review, graphite and carbons are defined as powders unless mentioned otherwise. Both are used in a variety of electrochemical applications. In the late 19th century, the Leclanché primary battery used carbon black in the positive manganese dioxide electrode as a conductive additive and for electrolyte retention. In alkaline batteries (primary or secondary), graphites are used not only as electrical conductors but also as lubricants to protect manufacturing tools, and as compaction additives. Bipolar plates in polymer electrolyte membrane (PEM) and phosphoric acid fuel cells are made out of graphite for reasons of electrical and thermal conductivity, as well as chemical inertness in both acidic and alkaline environments. In zinc-air batteries, carbon black serves as an oxygen absorber and in fuel cells as a catalyst support. Initially, hard carbons and, later, graphite have acted as a host for lithium ions in lithium-ion batteries. Graphitic carbons have made it possible that very reactive lithium can be 'domesticated' so that it can be placed next to the ears of mobile phone users.

Reviewing the various applications of graphite and carbons in electrochemical systems, a number of very useful properties can be identified, e.g.;

- electrical conductivity;
- thermal conductivity;
- inertness (but specific reactivity under certain conditions);
- thermal resistance up to 3000 °C;
- gas and liquid absorption;
- lubricity;
- compressibility;
- elasticity.

In chemical terms, graphite and carbons are simply composed of elemental carbon and it is surprising that so many totally different forms are exhibited by this element.

2. Classification of carbon and graphite

Carbon is positioned in the fourth group of the Periodic Table. The element, with the atomic number 6, exhibits a density which depends on its crystalline modification, melting point > 3550 °C and boiling point 4827 °C. The mass of the isotope ^{12}C was designated in 1962 as the standard for the determination of atomic mass. Furthermore, carbon occurs in the form of diamond, graphite, carbon black, coke, activated carbon, fibres, fullerenes, nanotubes, etc. In the following, carbon and graphite are classified with respect to their use in electrochemical systems.

2.1. Unstructured carbons

Unstructured carbons generally consist of graphene layers (planes of carbon atoms linked in a hexagonal array) that are stacked like a 'house of cards' [1] with many distortions in the structure. The several types of unstructured carbon may be classified as follows:
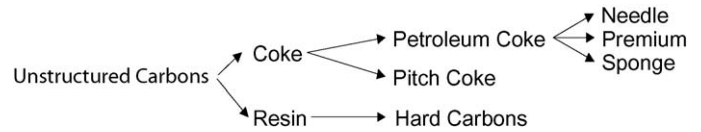
Classification of unstructured carbons.

Petroleum coke derives from oil as the heaviest fraction at the end of the fractional distillation process. Depending on purity, the conditions in the coker and the subsequent calcinations, a variety of different coke types can be produced. Typical representatives of the petroleum coke family are (Fig. 1):

- *Needle coke* which consists of highly structured graphene layers.
- *Regular coke* with irregularly oriented graphene layers.
- *Sponge coke*, which is coke with a highly porous structure.

Pitch coke is the solid residue produced by the devolatilization and carbonization of tars and pitches.

Coke products are mainly used in the graphite electrode and aluminum industries, as carbon raisers,[1] or as fuels. Coke products as such are not suitable for electrochemical applications. They do, however, play an important role in the manufacture of the synthetic graphite powders that are used in several battery systems.

Hard carbons consist of highly unstructured graphene layers. The starting materials are resins or similar products, which are carburized and baked. Hard carbons were used in the first lithium-ion batteries in the negative electrode as host for the lithium ions. Today, graphitic carbons are preferred due to their higher capacity.

2.2. Carbon black

Carbon black is also known as channel black, furnace black, lamp black, thermal black and acetylene black. These designations refer to the method of manufacturing. All these products have in common that liquid or gaseous hydrocarbons are decomposed at elevated temperature under a reduced presence of oxygen [3]. Nowadays, the most important carbon black is the 'furnace black' in which hydrocarbons are partially combusted and immediately quenched with water. The primary particle consists of several graphene-like layers, which combine to form spherical or oval particles as shown in Fig. 2. The particle diameter can vary according to the processing conditions and generally lies between 5 and several 100 nm.

Primary particles combine to form aggregates of different size and structure. The following four basic forms of aggregate structures can be defined [4] in terms of increasing degree of aggregation:

- spherical;
- elliptic;

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[1] Carbon raisers are added to the cast iron melt so as to adjust the carbon content.

Fig. 1. Various forms of coke: microscopic pictures in polarized light (by courtesy of Prof. Crelling [2]).

- linear;
- ramified.

In this sequence, the electrical conductivity and oil absorption characteristics increase and the bulk density decreases.

In electrochemical applications two carbon blacks are of special importance, namely acetylene black and PUREBLACK® Carbon.

Acetylene black is produced by partial oxidation of acetylene gas at high temperature. Due to the processing conditions, acetylene black shows a high aggregate structure and crystal orientation. The combination of these two characteristics makes acetylene black very valuable as an electrical conductor and as an electrolyte absorber in battery systems.

PUREBLACK® Carbon represents a new generation of carbon black. Furnace black is graphitized in a second step by a continuous graphitization process in a fluidized bed [5]. The graphitization organizes the graphene layers to a graphitic structure, as can be seen in Fig. 3, and makes them highly conductive and very pure. In addition, due to the 'healed' surface, moisture pick-up is very limited and solvent absorption is small. These features make the product an easy-to-handle conductive additive for lithium-ion batteries and other electrochemical systems.

2.3. Graphite

Due to its unique properties graphite is by far the most important carbon in electrochemical applications. It is the most

Fig. 3. Graphitic structure of PUREBLACK® Carbon before and after heat treatment at 2000 °C (by courtesy of Columbia Chemicals).

crystalline form of carbon, apart from diamond and fullerenes. It is soft and grey to black in colour. It exhibits the properties both of a metal, such as thermal and electrical conductivity, and of a non-metal such as inertness, high thermal resistance and lubricity.

Graphite consists, with very few exceptions, of polycrystalline particles or granules regardless of whether it is synthetic or natural. In other words each particle is a composition of numerous single crystals. In the case of natural flake graphite these single crystals are oriented in a preferred direction, while in synthetic graphite this orientation is more random.

The crystalline structure of graphite can be described as parallel layers of hexagonal rings (Fig. 4). The layers are
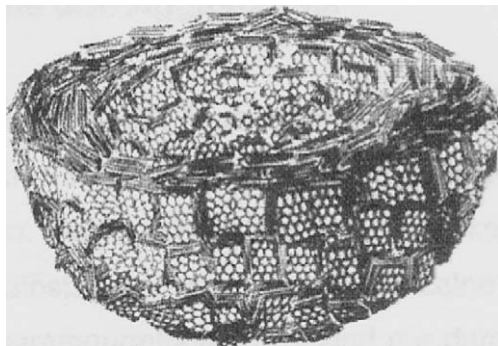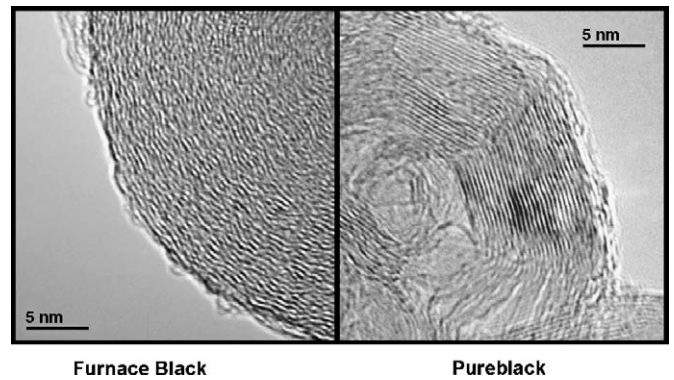

Fig. 2. Model showing cutaway of single carbon black particle with concentric layers. Parallel orientation of ordered layer groupings and diminishing graphitic order near particle center.
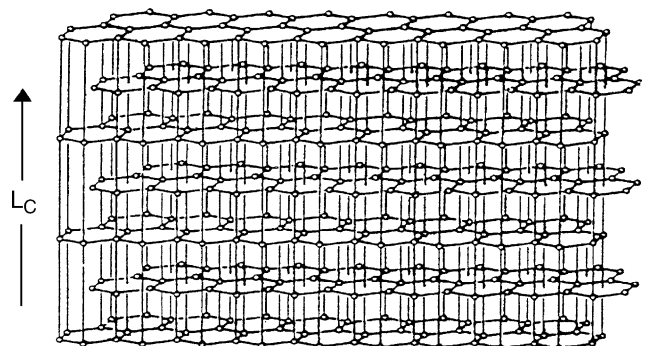

Fig. 4. Crystalline structure of graphite, hexagonal form with the layer sequence ABAB.

mostly positioned in a hexagonal structure, with the stacking sequence ABAB. A rhombohedral form with the layer sequence ABCABC, occurs in small amounts in highly crystalline natural graphite or after intensive grinding in ball mills.

At first sight, graphite appears to be a well-defined homogeneous product with a known chemical and physical structure. The reality however is different. Different varieties of graphite (flake graphite, vein graphite, amorphous graphite, synthetic graphite, etc.) exhibit quite different properties. In a first step towards classifying graphites, the following is proposed:
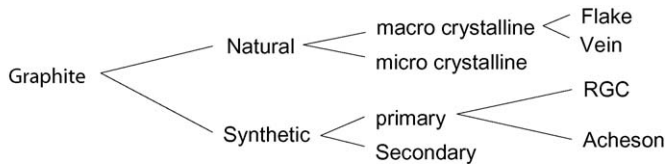
Classification of graphites.

The first differentiation refers to the origins of graphite.

2.3.1. Natural graphite

Natural graphite is described, according to the International Union of Pure and Applied Chemistry as a mineral found in nature. It consists of graphitic carbon regardless of its crystalline perfection [6]. At present, China is the principal producer of natural graphite, and provides 40% of the world's output. Other producers are located in Brazil, India, Korea, Canada and Sri Lanka. Natural graphite can be further sub-divided in micro-crystalline and macro-crystalline forms which are delineated in terms of crystallite size. Micro-crystalline graphite or amorphous graphite is extracted and processed in China, Korea, Mexico and elsewhere. Since its purity and crystallinity are rather low its conductivity and lubricating properties are modest. This graphite is not thought to be suitable for electrochemical applications.

Macro-crystalline graphite: basically the following two forms have to be distinguished.

Vein graphite occurs mainly in Sri Lanka. It occurs as large crystals, that are condensed into lumps and chips. Although equipped with excellent characteristics such as high electrical conductivity and lubricity and high purity, the product is only rarely used in electrochemical systems. The reason is that only two mines in Sri Lanka are producing commercially and these have limited capacity. Long-term availability is questionable.

Flake graphite is found to a great extent in China and also in Brazil, Canada, Africa and elsewhere. It consists of large crystals, that are uniformly oriented into scaly or lamella forms (see Fig. 5). The graphite content in the ore from which this material is obtained varies between 5 and 40% by weight. By means of mechanical separation and flotation, the purity can be raised to 99.5% carbon. In order to achieve purities >99.9% carbon, which are normally a prerequisite for battery applications, chemical (either acid or alkaline), or high-temperature, treatment is required.

High purity flake graphites, milled and classified to various sizes, are used in a number of battery systems. The main
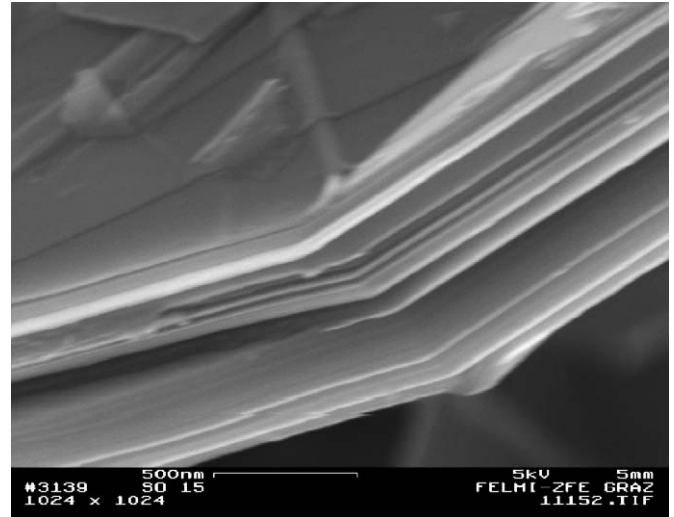

Fig. 5. SEM picture of lamella structure of flake graphite (by courtesy of the University Graz).

application is as a conductive and process-facilitating additive in primary alkaline batteries.

Expanded graphite is a special form of flake graphite produced by intercalation of strong acidic anions. By immediate exposure to temperatures up to 800 °C, the anions evaporate and break the crystals into very thin sheets of 100 nm thickness or less (see Fig. 6). The graphite expands by a factor of 300 or more. Milling the expanded graphite into fine powders make them usable for a number of battery systems [7,8]. Expanded graphites are characterized by a very high bulk volume and high electrical conductivity.

2.3.2. Synthetic graphites

Synthetic graphites are all basically prepared by heating unstructured carbon at temperatures above 2500 °C. This heat treatment orients the disordered layers into the graphitic structure. Depending on the raw material used and the heat treatment process, the characteristics of the synthetic graphite differ. Furthermore, the term 'synthetic graphite' also includes the rejections and turnings of graphite electrodes and graphite parts. Such graphite varies to a great extent with regard to purity, crystallographic structure, surface area, etc., since not only the selection of the precursor is different from one electrode type to another, but also the amount and type of pitch binder and graphitization temperatures varies. In order to differentiate such types of graphite from that produced for the sole purpose of manufacturing highly consistent graphite powders, the terms 'primary synthetic graphite' and 'secondary synthetic graphite' have been introduced [9].

Primary synthetic graphite powders are made by high-temperature processes under strictly controlled conditions so as to achieve characteristic properties with high consistency and predictability. Industrially relevant are the Acheson graphitization and the Desulco® processes.

Although originally designed for the manufacture of SiC the Acheson furnace can be adapted to produce graphite powders with high purity and crystallinity. The basic concept is shown in
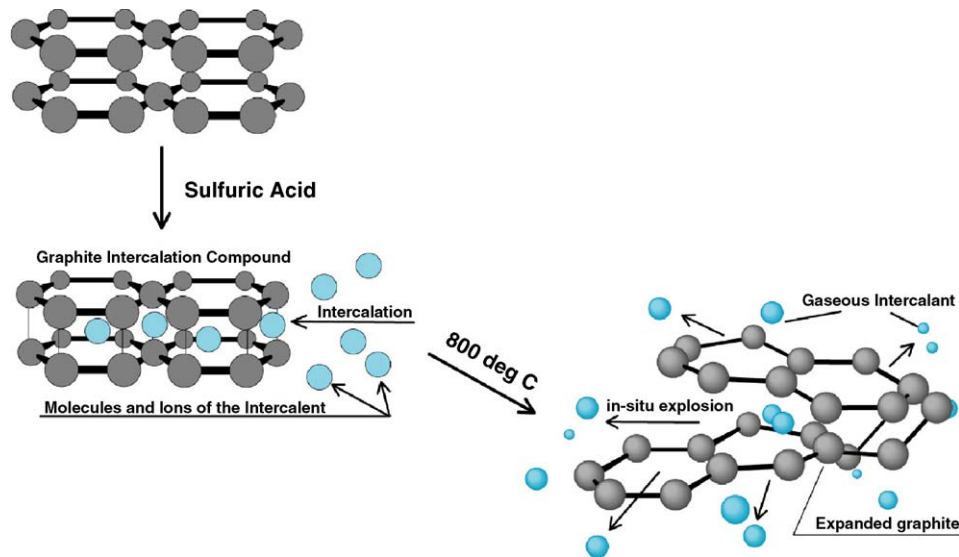
Fig. 6. Illustration of the chemistry to fabricate expanded graphite.

Fig. 7. A conductive core is placed between two large electrodes and is surrounded with petroleum coke or similar carbonaceous granules. The furnace surface is covered with sand or SiC so as to prevent the carbon from oxidation. The graphitization cycle lasts for several days. After cooling, the centre of the furnace is carefully separated and milled to the desired particle size.

The Desulco® process was originally designed for the desulfurization of petroleum coke so as to make it suitable as a carbon raiser, predominantly for ductile iron and other metallurgical applications such as steel-making, and also as a melt cover for copper. The graphite emanating from this process shows a combination of valuable properties including high purity, lubricity,

	Unreacted mixture of SiO$_2$ and C with additions
	Carbon core with electrodes.
	Walls
	Graphite.
	"Amorphous" SiC.
	SiC mass with voids containing larger crystals.

Fig. 7. Schematic figure of the Acheson furnace according to Knippenberg [10].

electrical conductivity, toughness and extremely high resiliency. Therefore, the name resilient graphitic carbon (RGC) has been created [11]. An important characteristic of the product is that, in contrast to all other graphites, the resiliency remains high over a large number of pressure cycles. The main applications of RGCs are in friction linings, carbon parts, engineering plastics, etc. In energy applications RGCs are added to high purity flake graphite to improve conductivity and provide electrodes with a controlled and predictable porosity.

The Desulco® process [12] consists of a vertical cylindrical tube made of graphite that encloses a central graphite electrode. A fluidized bed is formed around the electrodes with the product to be graphitized. The fluidized bed is heated by the internal resistance. Fresh material is added at the top and graphitized carbon migrates through the bottom into the outlet (see Fig. 8). The same process, slightly modified, is also used to purify natural flake graphite.

2.4. Nanocarbons

In order to complete the survey of the graphite and carbon powders, so-called fullerenes have to be mentioned, although they have no industrial relevance today. Such products are attractive, however, and may open new avenues for electrochemical applications if, in the future, a means may be found to produce fullerenes in large quantities at reasonable prices. The fullerene C60 has the form of soccer balls (Fig. 9). By rolling a graphene sheet (a single layer from a 3D graphite crystal) into a cylinder and capping each end of the cylinder with half of a fullerene molecule, a 'fullerene derived tubule' or 'nanotube', one atomic layer in thickness, is formed. Most studies of these materials have been made on multiwall carbon nanotubes and bundles of nanotubes [13].

Pure fullerenes are not electrically conductive but when they are doped with alkali metals the conductivity becomes as high as metals [14].

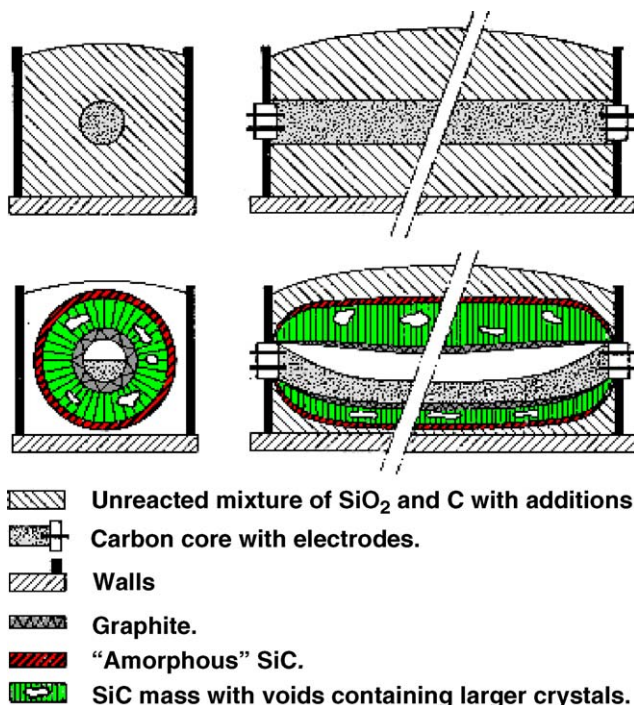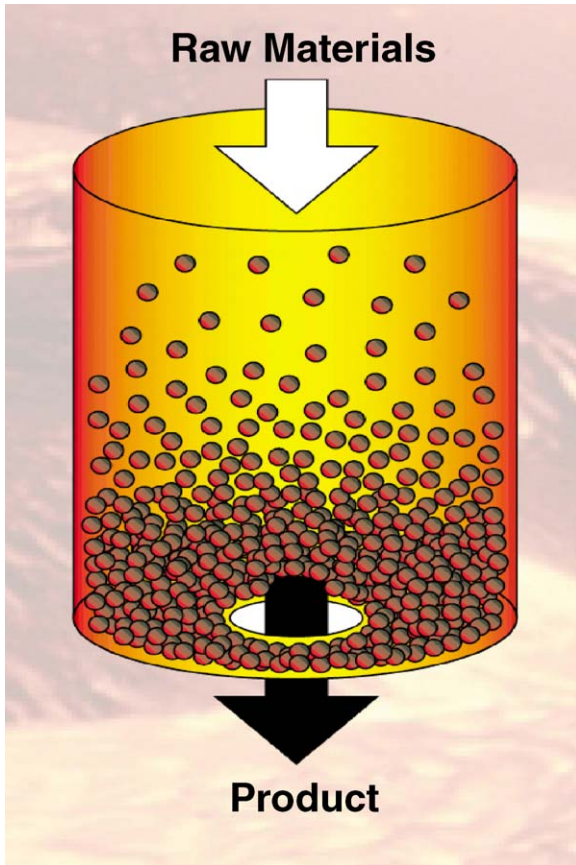The particles are extremely strong and very elastic.

Fig. 8. Scheme of the Desulco® process (by courtesy of Superior Graphite).
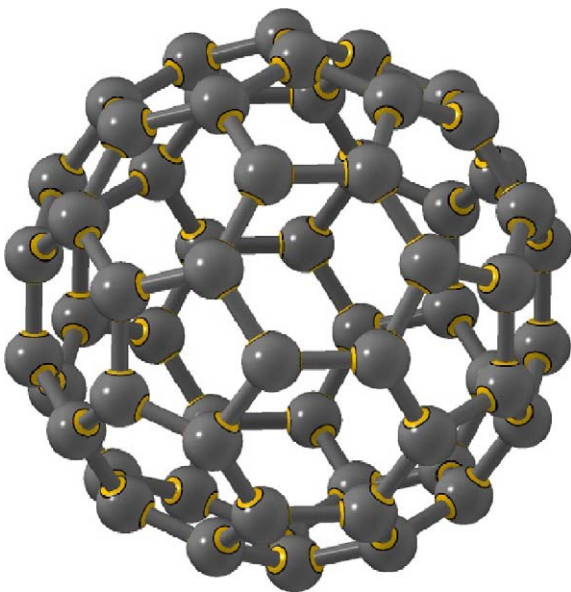

Fig. 9. Fullerene C60.

3. Characterization of graphite and carbons

3.1. The PSTP model

Since graphite and carbons show such a variety of properties, a systematic description of the characteristics is desirable.

In the early 1970s, the PSTP characterization model (in which the letters stand for P: purity, S: structure crystalline, T: texture, porosity and P: particle size) was developed and propagated [15]. These four parameters are defined as primary properties. They can be directly influenced by the raw material selection and manufacturing process. Furthermore, they are the fundamental characteristics that determine almost all other properties such as electrical and thermal conductivity, lubricity and compactibility. Therefore, these latter properties are defined as secondary or application properties. It can be shown, for example, that the electrical resistivity of compacted graphite powders primarily correlates with the negative logarithm of the particle size, d_{50} (the particle diameter in the particle-size distribution that corresponds to the 50% point by weight), and the crystallite dimension in the c-axis direction, L_c. Purity, surface area and bulk density or porosity exercise less influence on the electrical resistivity [16]. The four basic parameters need to be further explained since they underlie all the more detailed characteristics of carbons and graphites.

3.2. Definition of purity

The term purity comprises the parameters

- ash content [17,18];
- moisture content [19,20];
- superior graphite;
- impurities.

For energy applications, it is mandatory to know the level of impurities in the ppm, and in some cases even in the ppb, range. Impurities can shorten the lifetime of a battery, cause side reactions, or can even lead to explosions.

Today, several methods are available for the determination of trace elements such as atomic absorption, inductively coupled plasma (ICP), emission spectroscopy, X-ray fluorescence and others. A very suitable new method is electrothermal vaporization combined with inductively coupled plasma (ETV/ICP) [21]. The trace elements are determined with high precision directly from the solid powder without a long leaching or ignition procedure. The limit of detection is <1 ppm.

The purity level normally required for electrochemical applications is in the range of <0.1% ash with the accepted level of heavy metals <10 ppm per element or, in some cases, <1 ppm.

Table 1
Crystallite height L_c and interlayer distance $c/2$ of various graphite powders

Graphite	L_c (nm)	$c/2$ (nm)
Natural flake graphite	>300	0.3354
Microcrystalline graphite	20	0.3356
Primary synthetic graphite (Acheson)	>100	0.3358
Primary synthetic graphite (RGC)	40	0.3372
Secondary synthetic graphite	60	0.3365

Table 2
Density and surface area of various graphite powders

Graphitic carbon	True density (g cm^{-3})	Bulk density (g cm^{-3})	Surface area (sm g^{-1})
Natural flake graphite, d_{50}[a] = 9 μm	2.255	0.07	9
Expanded graphite, d_{50} = 9 μm	2.255	0.043	24
Primary synthetic graphite (Acheson), d_{50} = 9 μm	2.25	0.1	12
Primary synthetic graphite (RGC), d_{50} = 10 μm	1.97	0.16	17
Secondary synthetic graphite, d_{50} = 9 μm	2.1	0.1	14

[a] d_{50} defines the particle diameter in the particle size distribution which corresponds to the 50% point by weight (50% finer and 50% coarser than d_{50} by weight).

3.3. Definition of crystalline structure of graphites and carbons

As already discussed, graphite and graphitic carbon particles are, with a few exceptions, polycrystalline. In practice, the following parameters; crystallite size, L_c (crystallite dimension in the c-axis direction), and, L_a (crystallite dimension in the a-axis direction) and the interlayer spacing, $c/2$ (half the hexagonal lattice c-axis), describe the structural properties of graphitic crystals adequately.

The crystallite size is usually determined and calculated from the broadening of the reflection profile of the XRD pattern and the interlayer spacing from the corresponding position of the peaks.

In Table 1, several graphite powders are compared with respect to crystallite height, L_c, and interlayer spacing, $c/2$. As can be seen, natural flake graphite has by far the largest crystallite size and the most perfect graphitic order, as reflected in the interlayer distance, $c/2$. Graphite with a crystallite size of >100 nm is considered as macro-crystalline, whereas graphite with a crystallite size <30 nm is micro-crystalline. The crystalline conditions of secondary synthetic graphite can vary from macro-crystalline to micro-crystalline depending on the history of the product.

The crystalline structure is one of the most significant parameters. Macro-crystalline graphites show a high electrical conductivity and lubricity due to the crystallite size. The specific capacity of the negative electrode of lithium-ion batteries is directly dependent on the crystallite size: the larger the crystals, the higher is the specific capacity. Also, the corrosion resistance of graphite as a conductivity additive in the positive electrode increases with the size of the single crystals.

3.4. Definition of porous texture

The porous texture comprises the characteristics:

- true density [22,23];
- apparent density [24] and tap density [25];
- BET surface area [26];
- pore-size distribution, determined by means of mercury intrusion [27,28] and gas absorption (BJH—Barrett, Joyner, Halenda pore distribution) [29];
- particle shape.

The true density gives information on the presence of closed pores provided the carbonaceous product has a high purity.

The apparent density and the tap density indicate the intergranular porosity, i.e. the porosity between the particles in a powder. This porosity is mainly responsible for the absorption of liquids such as electrolytes or binders, much more so than the surface area.

The active surface area and the intra-granular pore-size distribution (intra-granular pores are the pores that are inside the particle and are normally micro-pores and meso-pores) are of minor importance for regular graphite. Chemical and mechanical treatments can create surface areas of >100 m^2 g^{-1}. These graphite products are suitable as catalyst supports.

Particle shape is a result of the manufacturing technology, the milling process and the anisotropy of the particles. The particle shape can vary from spherical (graphite and graphitic carbons applied in lithium-ion batteries) to thin sheets like tissues (expanded graphite).

The porous texture has a strong influence on the compressibility, the three-dimensional thermal, and electrical, conductivities and the chemical stability such as corrosion resistance. Some data on texture are given in Table 2.

3.5. Particle size

Particle size is a difficult parameter to quantify meaningfully for some forms of carbon. The problem is illustrated in Fig. 10a and b. For spherical graphite (Fig. 10a) the particle size is relatively easy to determine – the diameter of the sphere. In the case of synthetic graphite (Fig. 10b), however, there is no explicit definition of the size of one particle. The problem is potentially troublesome because particle size and particle-size distribution are two of the factors that exercise an important influence on application properties.

Industry has found a pragmatic way to get around the problem. Instead of discussing the most appropriate diameter to consider for a particle-size distribution, the sieving or analytical method is defined and must be applied in specifications.

The most common methods are shown in Table 3.

Table 3
Analytical methods for determining particle-size distribution

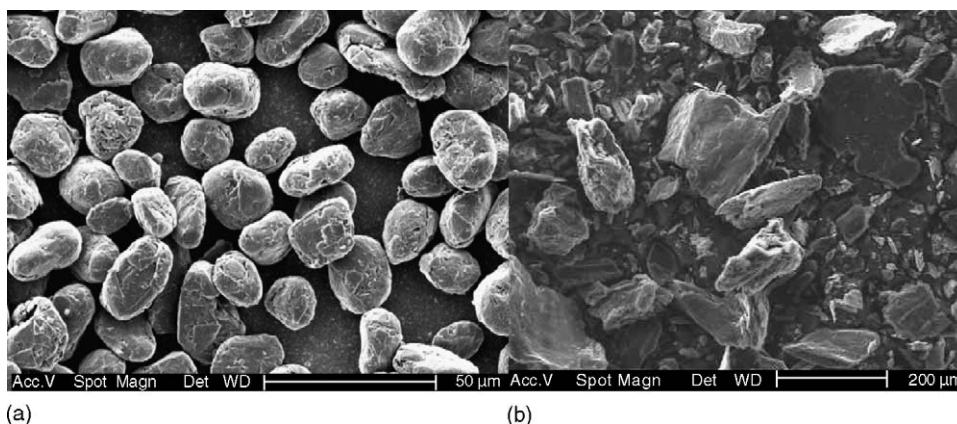| Method	Recommended range of particle size
Vibrated screening [30]	0.063–30 mm
Air jet screening	0.045–0.25 mm
Laser diffraction	0.1–500 μm

Fig. 10. (a) Natural graphite spherodized and (b) synthetic graphite.

Particle-size analysis by means of laser diffraction determines the particle size by using the diffraction or diffusion angle of the laser beam on particles. This is the most widely used technique for particle-size analysis of fine powders. The instruments are easy to use and can provide analysis over a broad range, in a variety of dispersion media. There is, however one disadvantage, namely, the method applies complicated mathematical models to calculate particle size from the laser light distribution.

4. Conclusions

One of the fascinations of graphites and carbons is the wide variety of forms of appearance and the huge variety of properties. With the product classification and the approach to characterization proposed in this article, it is hope that the materials will be better understood and more manageable in use. This classification and characterization is by no means complete. More parameters, such as surface conditions or colour, could be added but industry experience has shown that, in more than 90% of the cases, four basic parameters are sufficient to explain and predict the behaviour in energy applications. The classification and characterization model provide a certain systematic approach to the structuring of product descriptions (data sheets) and specifications. The characterization model is equally applicable to carbonaceous solid materials and to carbon blacks. In the case of such materials, however, we must add the very import characteristic of the aggregate structure to the four basic parameters. With the discovery and development of the nanocarbons the characterization model probably needs to be widened further.

Today graphite and carbons are used in a variety of electrochemical applications on account of their high purity, electrical and thermal conductivity, and specific surface properties, and also thanks to their attractive price/performance ratio. Lithiumion, zinc primary cells, alkaline batteries and fuel cells would all be unthinkable without graphite and carbon. Also the traditional lead-battery has recently been improved by the use of graphitic carbons in the negative plate so as to meet the today's high demanding requirements.

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