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ARTICLE

Effect of Parameter Describing Pore Structure on Properties of Carbon and Graphite Materials

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Abstract: With the constant upgrading of traditional and emerging industries, carbon and graphite materials (CGM) have become an important strategic resource and essential mineral material to support the development of high and new technology. The current situation of report for the CGM still remains at macro-mechanical property. Seldom is reported about pore structure of CGM, which may affect mechanical properties. It is well known that properties of CGM depend on its micro-structure, and the pore structure plays an important role in microstructure. Therefore, this paper reviews the development and application of CGM, and summarizes the parameters of pore structure in traditional CGM. Meanwhile, the internal differences of the pore structure between the traditional CGM and the porous graphene are compared and discussed. Moreover, the effect of parameters characterizing of pore structure for CGM on comprehensive properties are described and the correlation between them are also discussed. It provides a theoretical basis for material scientific description of physical and chemical properties, material modification, performance optimization and new process design.

Key words: carbon and graphite materials; porous graphene; pore structure; classifications of pore; comprehensive properties

CGM is a nonmetallic mineral with potential strategic value, especially in high-tech industries. CGM not only has excellent conductivity and thermal conductivity, but also has excellent chemical and high temperature stability[1-5]. However, with the rapid development of traditional and emerging industries[6,7], CGM is confronted with more stringent performance standards, including Young's modulus, stiffness, high temperature resistance, corrosion resistance, frost resistance, strength, etc; furthermore, it shall be able to be used repeatedly in various extreme environments.

It is well known that any properties of the material are determined by its micro-structure, especially pore structure in essence. Therefore, if these problems are to be understood and solved radically, researchers should follow the basic idea that material science depends on research and design of micro-structure in a great extent. Moreover, study on pore structure is an important part of the microstructure research. Actually, effects of the pore structure on macroproperties of CGM under the normal and extreme environment have been a hotspot of material science in recent years.

This paper reviews the development and application of CGM, and the parameters of pore structure in CGM. Moreover, the effect of parameters characterizing of pore structure for CGM on comprehensive properties are described and the correlation between them are also discussed. It provides a theoretical basis for material scientific description of physical and chemical properties, material modification, performance optimization and new process design.

Since the members of CGM are extremely numerous, in the present paper, several representative types are selected, and several kinds of properties are closely related to their pore structure. It is preliminarily divided into traditional carbon materials (graphite, activated carbon, exfoliated graphites, etc) and new graphene materials (three-dimensional (3D) porous graphene, porous graphene nanocomposites, etc), and they are compared and analyzed.

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The U. S. Geological Survey (USGS) released important data into the public domain[8]. In 2016, the world's proven reserves of natural graphite have reached 250 million tons. It is remarkable that 95% graphite resources are concentrated in Turkey, Brazil, China, Mozambique and India[9], and the output of graphite is 90, 72, 55, 13 and 8 million tons, respectively.

The resources of graphite raw materials in China are very rich. At present, some graphene products have been commercialized. Three-dimensional porous graphene and porous graphene nanocomposites are expected to be the ideal material for preparation of a storage device, and the market prospects are unlimited.

Since the discovery of graphene, great progress has been made on the application of graphene and its derivatives. The proper functional treatment and chemical modification for graphene will result in better performance of nanomaterials.

The recent study[10-12] reported that porous graphite materials not only overcome defect of graphene, which exhibits easy stacking or aggregation due to the effect of strong van Edward force between π-π , but also is better than the graphene in performance.

On account of this, it can be concluded that researchers must focus on the development of new industries in the work of CGM in the future[13-15]. Therefore, we should fundamentally understand the influence of pore structure of CGM on its macroscopic properties. It is well known that the macroscopic properties of CGM depend heavily on the microstructure which is manifested by the pore structure.

1 Pores in Carbon and Graphite Materials

Most of carbon and graphite materials are porous materials, which have many pores with different sizes and shapes[16-18]. The pore structure of CGM has an important influence on the macroscopic properties. It can be concluded from results that its macro-performance such as mechanical properties, depends on the pore structure characteristic parameters, including porosity, distribution of pore size, specific surface, pore shape, surface fractal dimension of pores, etc[19,20]. Therefore, in the present paper, it is necessary to explain the relationship between the characteristic parameters of pore structure of CGM and its comprehensive properties.

1.1 Classification of pores

Porosity is the main characteristic of porous materials, and is the dominant factor of macroproperties of porous materials[21-24].

The expression of porosity can reflect not only the size of voids but also the spatial characteristics pore structure of CGM, which are the key to solve the CGM penetration and other problems[16]. In addition, the macroscopic properties of CGM including deformation property and strength characteristics are affected by characteristic parameter of pores such as porosity and spatial characteristics of pore structure in varying degrees.

1.2 Size of pores

The products of CGM are used in different application fields. Among them, the pores size of graphite products is also different. Therefore, it is necessary to study the relationship between the pore size and the mechanical properties of CGM.

According to International Union of Pure and Applied Chemistry (IUPAC), the pore size of graphite products can be roughly divided into three categories including micro-pores, meso-pores, and macro-pores[25-27]. The size of CGM can range from cm-scale of pores such as graphite electrode for electric furnace steel-making to meso-scale of pores such as meso-porous composite materials of graphene, and then slightly larger than atomic scale of pores such as highly dense graphite materials.

1.3 Specific surface area

To some extent, the specific surface area of porous materials truly reflects the area of contact with the outside environment. The specific surface area is also an important parameter in the physical and chemical properties of CGM.

Because the pore geometry and surface morphology of CGM are complicated, the data can only be obtained by an indirect technical method. The specific surface area of porous materials has been tested by these methods such as mercury intrusion porosimetry (MIP)[28], gas adsorption method and small angle X-ray scattering (SAXS)[29] over the years.

1.4 Surface fractal dimension of pores

With the development of fractal theory, it provides a new method for the study of pore structure and macro-properties of porous materials[30]. Some scholars[31,32] have found that the structure of some porous materials such as graphite, concrete and other porous materials with natural rock structure has obvious fractal characteristics. Therefore, fractal dimension can be used to find out the essential relationship between macro-properties of porous materials and their microstructure pore characteristics.

When pore volume fractal dimension increases, specific surface area becomes larger, the total porosity becomes lower, but the number of small diameter increases based on the fractal model and fractal dimension theory[33]. This also complicates the spatial structure of the pore, and the inner surface of the pore is roughened. Therefore, fractal dimension can be used to make up for defects that the mercury injection method cannot directly determine the structural characteristics of porous materials (pore geometry, pore space distribution and specific surface area of pores) under certain conditions.

However, the quantitative relationship between the fractal dimension of porous materials, the pore structure characteristics and macro mechanical properties is still insufficient, and the future research will be carried out around it.

2 Effect of Pore Structure on Properties of CGM

2.1 Effect of pore structure on mechanical properties of CGM

No matter what kind of CGM is applied in traditional industry or emerging industry, all of them cannot be separated from mechanical properties of CGM, because CGM is subjected to external forces such as single force and integrated force in the process of application in the conditions of room temperature or high temperature or high pressure or vacuum or corrosion. Therefore, it is necessary to discuss the effect of pore structure on mechanical properties of CGM.

The influence of pores in CGM on mechanical properties can be summarized as the following two aspects. On the one hand, the number of pores is directly related to the area of the load acting on the graphite material, and the stress of the material decreases with the increase of the number of pores; On the other hand, the existence of pores will make the stress concentration at the hole, and provide the space needed for the deformation of the material, thereby affecting the strength and fracture performance[34].

The latest research[35,36] investigated that mechanical behavior and fracture of graphite nanomeshes, and the results show that the ultimate tensile strength and materials toughness decreases with increasing porosity.

2.2 Effect of pore structure on adsorption properties of CGM

Frequent oil spill accidents[37], coal burning exhaust emissions[38] and industrial oily waste water emissions[39] have become the major culprits of global ecological environment pollution. In order to solve these problems, there are three kinds of governance methods for the pollution. First of all, the application of traditional methods such as activated carbon is used to remove oil from water[40]. Although good governance effect of activated carbon has been achieved, there are still some problems: the adsorption capacity of activated carbon is low, which will sink after adsorption, and the high cost and difficulty to recycle and reuse also limit application of activated carbon[41]. In addition, there are other adsorbents including cotton, wool, polypropylene, nylon synthetic fibers and perlite, etc. They adsorb oil and water at the same time, which brings difficulties to the post treatment[42]. Therefore, they are not ideal adsorbents for the treatment of oil spills and oily waste water.

The second treatment method is a new porous adsorption material: exfoliated graphite[43]. The researchers[44] found that exfoliated graphite is an excellent adsorbent. In particular, the exfoliated graphite has a large amount of oil adsorption and floats on the water surface after suction. In addition, the exfoliated graphite as absorbent is easy to recover and reuse. Common oil absorption materials and their oil absorption properties are shown in Fig.1[45-48]. Why the adsorption of exfoliated graphite is so good? On the one hand, the pore structure of activated carbon is mainly micro-porous and meso-porous[49], and the difference is that the pore structure of exfoliated graphite is mainly macro-porous and meso-porous[50]. It is the kind of pore structure with large pore and middle pore,

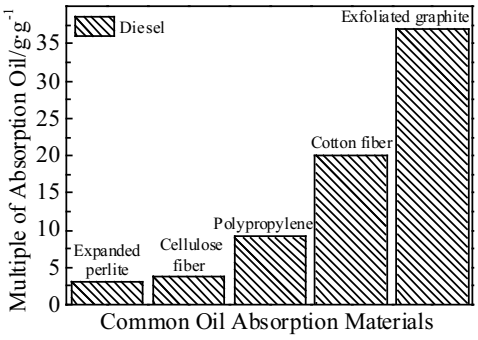

Fig.1 Common oil absorption materials and their oil absorption properties[45-48]

and most of the pore size concentrated at 1 nm to 100 nm, that offer the unique adsorption characteristics of exfoliated graphite. On the other hand, the surface pores of exfoliated graphite are usually opening pores, and they are used as the channels that the outside connects to the inner space. Therefore, it can be concluded that microstructure of exfoliated graphite determines that it is easy to absorb macro-molecules.

The last solution is to use a new material: porous graphene materials, which originate from graphene, but the performance of porous graphene materials is superior to that of graphene due to the introduction of nanopores. For example, meso-pore and macro-pore in porous graphene materials can promote permeation and transport of substance; the micro-pore in porous graphene material can improve its surface area. In addition, the introduction of different sizes of pores can make porous graphene materials avoid the adverse effects[51,52].

2.3 Effect of pore structure on thermal properties of CGM

Because of the disadvantages of traditional thermal conductive materials such as copper and aluminum, they are unable to meet the demand of modern industry. For example, the propellant grain of jet engine produces more than 3000 °C of high temperature, and produces instantaneous thermal shock of 2500 °C and 6 MPa of high pressure. Without exception, properties of CGM are affected by high temperature and ultra-temperature during the using process. Therefore, the thermal properties of CGM are very important for their applications.

The heat conduction of CGM is different from that of metal material which depends on the free carrier, and the heat conduction of carbon and graphite materials is determined by phonon. Phonon is a quasi-particle, which is a collective manifestation of lattice vibration[53,54]. Therefore, the thermal conductivity of CGM is closely related to lattice vibration and its propagation. Moreover, it can be concluded that the propagation of lattice vibration will be seriously attenuated at the pores or at rough hole-wall when the CGM contain pores.

Maybe, it is caused by the following two reasons. The first one is that the air is a poor conductor of heat, and the time and distance of phonon propagation in air increases with increasing porosity, which also leads to a decrease in total phonon momentum; the second one is that solids that participate in thermal vibration propagation are reduced. Therefore, it can be concluded that thermal conductivity of CGM decreases with increasing porosity or surface roughness of pores.

The relationship between thermal conductivity and porosity of graphite size containing carbon refractories is illustrated in Fig.2. It can be seen that the thermal conductivity of graphite decreases with the increase of porosity from 300 °C to 800 °C[55]. Meanwhile, it must be pointed out that the thermal conductivity of graphite declines with the increase of porosity and smaller pore (diameter <0.1 μm) volume as well as the decrease of the mean pore diameter[55].

2.4 Effect of pore structure on electrical properties of CGM

At present, the carbon based electrode materials involved in super capacitor mainly include: activated carbon, graphene, carbon nanotubes, porous graphene, etc. For example, the previous study shows[56] that activated carbon has the advantages of well-developed pore structure, friendly environment, high stability and large specific surface area. Therefore, the activated carbon is widely used as electrode materials. However, the defects of activated carbon as conductive materials cannot be ignored. On the one hand, the conductivity of activated carbon is poor. On the other hand, the single pore structure of activated carbon also limits the use of activated carbon as conductive material. For example, it may hinder the transport of electrolyte substances in all directions and slow down delivery rate of electrolyte substances[56]. These problems have seriously restricted application of activated carbon as electrode material, especially in the field of super capacitor.

Therefore, people's perspective moved to the graphene research. The research found that the graphene has the advantages of high conductivity, unique micro-structure and high specific surface area, especially carrier mobility of suspended graphene, which is 200 000 $cm^2 \cdot V^{-1} \cdot s^{-1}$ and 140 times higher than the carrier mobility of commercial silicon materials[57]. These advantages determine that it is suitable for electrode materials. However, graphene still has the following deficiencies at current stage: the strong Edward force between layers of graphene sheets is the main reason why the actual capacitance of graphene is far below its theoretical value. Therefore, the future work for graphene is to solve the problem of easy agglomeration and stacking.

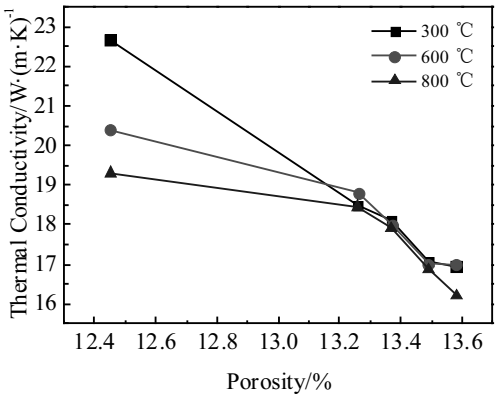

Fig.2 Relationship between thermal conductivity and porosity of different graphite size containing carbon refractories[55]

Fortunately, researchers found an effective way to solve the defects of graphene. Porous graphene not only inherits the advantages of graphene, but also makes up for the defects of graphene due to introduction of nanopores. From the view of electrolyte transport, nanopores can enhance electrolyte transport efficiency and improve exchange of substances. From the view of sieving and isolation, nanopores can sieve different sizes of ions and molecules. Finally, band gap of graphene can be opened by nanopores based on the application of electronic device[58]. For example, El-Kady et al[59] reported the preparation of graphene foam prepared by chemical reduction. Graphene foam is an opening three-dimensional porous structure which is composed of different sizes of pores from hundreds of nanometers to tens of nanometers. Graphene foam can achieve high conductivity (up to 1738 $S \cdot m^{-1}$).

There is no doubt that the electrical properties of graphite materials are affected by its pore size.

2.5 Effect of pore structure on chemical properties of CGM

It is well known that the chemical properties of materials are closely related to the development of modern industry, and catalytic performance is one of the important components in the field of chemistry[60]. The composition of most catalysts can be divided into the following three categories: active component, carrier and co-catalyst.

These carriers often provide relatively large specific surface areas or have developed pore structure, which provides a good contact surface with the outside world. To accelerate the diffusion of reaction gas and generation gas in the pores, the purpose of accelerating catalytic reaction has been achieved.

The traditional catalyst carrier cannot satisfy the development of chemical industry. For example, with silica as catalyst carrier, it has good chemical stability and does not react with general acid besides fluorine and hydrogen fluoride. However, the defects of silica as catalyst carrier cannot be ignored, because the raw material of silica contains a trace of impurities, which may lead to poisoning of catalyst and decrease activity of catalyst[61].

The previous study showed[62,63] that CGM is very suitable to be used as catalyst carrier due to its unique properties. For

example, activated carbon as catalyst carrier has a large specific surface area and developed pore structure, which determine catalytic performance of activated carbon. In addition, these parameters of carbon carrier can be modified by physical and chemical methods. This will cause that the function of activated carbon is more powerful and has a wider application range. However, the application of activated carbon is restricted due to its low selectivity, uneven pore size distribution, different pore sizes, poor chemical stability and catalytic performance with low efficiency. Therefore, the urgent task is to find more excellent CGM to deal with the development of modern chemical industry.

Recently, graphene porous materials have attracted much attention. The design essence of porous micro-structures is mainly based on graphene which is easy to meet π-π stack, and the theoretical specific surface area has not reached the actual value, the results will restrict graphene application[64]. In general, defects in graphene will reduce its performance, especially conductivity. However, the formation of pores defects will reduce the performance; on the contrary, pores defects will improve performance or obtain new functions. Why the introduction of nanopores and defects of pores produce such an ideal result?

On the whole, on the one hand, the micro-structure of porous graphene and higher specific surface area provide channels for molecular or ion transport; on the other hand, they can also act as carriers for functional nano-particles[65].

2.6 Effect of pore structure on electrochemical performance of CGM

The pore structure is also related to electrochemical performance of CGM. For example, the effective specific surface area of the traditional activated carbon is low, because of its smaller pore size. In addition, Putz[66] combined graphene materials with polyvinyl alcohol (PVA), and the result shows the composites have mechanical properties two times higher than the theoretical value. But the composites are restricted as energy storage electrode materials due to little pore distribution.

To address the question, it is important to notice that the electrochemical capacity and cyclic stability of the electrode material should be maintained, when the surface area of the material is increased. It can effectively expand its pore size and shorten the axial length of the channel, thus promoting the rapid transmission and effective diffusion of the electrolyte at the interface of the electrode material.

The recent studies[67] have found that 3D graphene is modified by adding a transition metal compound or a conductive polymer, the composite formed by the two element 3D structure has excellent electrochemical performance. Compared with carbon materials, transition metal compounds have higher energy density and cycle stability. The combination of 3D graphene and transition metal compounds can achieve complementary advantages. Through the continuous study of graphene, transition metal compound and conductive polymer, it is found that the application of three element 3D graphene composite can be realized. For example, Nguyen[68] prepared the composite structure of 3D graphene/$NiCo_2O_4$ by chemical vapor deposition (CVD) and electrochemical deposition methods, and the result shows the characteristic of rich micro-pore and mesoporous can guarantee the high electron/ion conductivity and large capacitance performance.

Another important application of porous graphene is the chemical sensor. The unique 3D structure of graphene can accelerate the diffusion and transfer of molecules in the electrochemical reaction, improve the electron transfer rate, and further enhance its conductivity.

3 Conclusions

1) There are many pores with different sizes, shapes and uneven distribution in CGM. These pores are an important part for the micro-structure of CGM. It can truly reflect the comprehensive properties of carbon based materials. Characteristic parameters of pore structure of CGM include porosity, geometry size of pore, pore distribution, surface fractal dimension of pore, specific surface area, etc. Based on the characteristic parameters of these pores, the relationship between pore structure parameters, pore formation mechanism and comprehensive properties should be deeply understood.

2) Understanding these internal connections can not only provide strong support and theoretical basis for the development of traditional fields and emerging industries such as catalysts used in low-temperature fuel cell, electrode materials for super-capacitors, field effect transistor, chemical sensors, seawater desalination, molecular sieve, DNA molecular detection, but also has pioneering significance in the aspect of the CGM development, modification, improvement and new technology.

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炭、石墨材料孔结构特征描述及其对综合性能的影响

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摘　要：随着传统行业、新兴产业的不断升级，炭、石墨材料（CGM）已成为支撑高新技术发展的重要战略资源和基础矿物原料。目前针对 CGM 的研究主要围绕宏观性能，对其孔结构的系统报道较少。众所周知，CGM 的宏观性能取决于其微观结构，而孔结构的研究又占据了微观结构的重要一环。因此，本文回顾了 CGM 的应用与发展，总结传统 CGM 的孔结构特征，在此基础上，对比讨论了传统 CGM 与多孔石墨烯及 3D 多孔石墨烯复合材料孔结构的内在差异，并进一步分析孔结构特征对其综合性能的影响和内在联系。为阐述 CGM 物理和化学性能、材料改性、性能优化及新工艺的设计提供理论依据。

关键词：炭、石墨材料；多孔石墨烯；孔结构；孔分类；综合性能

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