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Original Research Article

Preparation technics and application of expanded graphite

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ABSTRACT

The research of application, preparation technics, structure and properties for expanded graphite were summarized, and their developing trends were also expected in this paper. The preparation technics of low-temperature expansible graphite, non-sulfur expansible graphite and expanded graphite compound materials synthesized by chemical oxidation, electrochemistry, microwave, detonation or gaseous volatilization were introduced mostly. The investigation actuality and application foreground of expanded graphite materials were summarized and analyzed in their applied fields of air-proof, flame retardant, lubricant, environment, catalysis, military affairs and medicine.

Keywords: Expanded Graphite; Preparation Technics; Research of Application; Summarize

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1. Introduction

Graphite is an important non-metallic mineral, which belongs to hexagonal system and has a special layered structure. In the early 1860s, Brodie heated natural graphite with chemical reagents such as sulfuric acid and nitric acid, and found Expanded Graphite (EG)[1]. It is a new type of carbon material on atomic and molecular scales, showing unique physical and chemical properties. However, its application began a hundred years later. In the past 20 years, many countries have carried out the research and development of expanded graphite, and have made major scientific research breakthroughs. As an important inorganic non-metallic material, expanded graphite materials are widely used in environmental, chemical, metallurgy, dynamic machinery, aerospace and atomic energy industries, showing strong vitality and market application prospects

1.1 Basic properties of expanded graphite

Expanded graphite crystal still belongs to hexagonal crystal system. Its shape looks like a worm and its size is between a few tenths of a millimeter and a few millimeters, so it is also called vermicular graphite crystal, as shown in **Figure 1a**[2]. The apparent volume of expanded graphite is 250–300 ml/g or more, and there are a large number of unique network microporous structures inside (see **Figure 1b**)[3]. The new carbon material of expanded graphite not only has the excellent characteristics of heat resistance, corrosion resistance, radiation resistance, conductivity and self-lubrication of natural graphite, but also has the properties of light, soft, porous, compressible and rebound that natural graphite does not have. In particular, the new composite expanded graphite material synthesized by functional modification of expanded graphite has better properties than carbon fiber asbestos, rubber and other materials have more excellent properties and wide applications. Various plates, strips, sheets and other profiles made of expanded graphite and various electronic and mechanical devices have been widely used, and show the advantages of low cost, long service life and good effect.

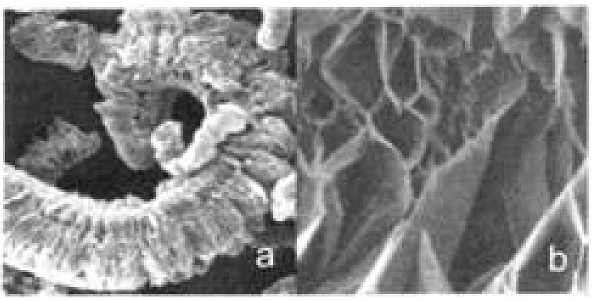

Figure 1. SEM images of expanded graphite.

1.2 Preparation principle of expanded graphite

Graphite crystal has a typical layered structure. Covalent bonds are formed between its carbon atoms in one layer, and the bond energy is 586 kJ/mol. In the interlayer, it is combined with weak van der Waals force, and the bond energy is only 16.7 kJ/mol. Therefore, other kinds of molecules or atoms can be inserted into their layers to form graphite interlayer compounds. After high temperature heat treatment, these atoms, molecules or ions inserted between layers will generate thrust due to instantaneous vaporization and volume expansion. The vaporization thrust overcomes the weak van der Waals force between layers and expands rapidly along the C-axis, pushing the layers of graphite away from each other and rapidly increasing the layer spacing, so that the volume of graphite expands tens, hundreds or even thousands of times, forming a material with light, soft and excellent resilience–expanded graphite.

2. Preparation process of expanded graphite

At present, the process methods for preparing expanded graphite are based on the basic principle of intercalation expansion. Among them, chemical oxidation method and electrochemical method are the most important methods, and have been applied in industry. In addition, according to the different insert agent introduction methods and the differences of expansion methods, in addition to chemical oxidation method, electrochemical law, there are microwave method, explosion method and gas phase volatilization methods.

2.1 Chemical oxidation

Chemical oxidation is the most widely used and mature method in industry. Because graphite is a non-polar material, it is difficult to intercalate with organic or inorganic acids with small polarity alone, so oxidants need to be used in the preparation process of chemical oxidation method. The chemical oxidation method generally immerses the natural flake graphite in the solution of oxidant and intercalation agent. Under the action of strong oxidant, the graphite is oxidized to turn the neutral network planar macromolecules of graphite layer into positively charged planar macromolecules. The distance between graphite layers increases due to the repulsion of isotropic positive charges between the plane macromolecular layers of charges. At the same time, due to the loss of electrons in graphite to form carbon positive ions, anionic interpolator enters the graphite layers and combines with carbon positive ions to form graphite interlayer compounds to become expandable graphite. The solid oxidants used in the chemical oxidation method are $KClO_4$, $KMnO_4$, $(NH_4)_2S_2O_7$, etc., and the liquid oxidants are HNO_3, H_2SO_4, $HClO_4$, H_2O_2, etc. Solid oxidants generally react violently, are dangerous, pollute the environment and have high prices. Liquid oxidants such as HNO_3 and H_2SO_4 have high requirements on the operating environment and pollute the water body, while H_2O_2 has mild reaction and little pollution[4].

For expandable graphite, the expansion volume and sulfur content are two important product index. It is generally hoped that the expansion volume is high and the sulfur content is low. Therefore, in recent years, low sulfur expandable graphite, especially sulfur-free expandable graphite, has become an important direction of research and development. By using organic acid and organic solvent as auxiliary intercalator and reducing the amount of sulfuric acid as main intercalator is the most effective way to reduce the sulfur content of expandable graphite products and expanded graphite products[5]. The use of metal halides, especially ferric chloride as auxiliary intercalation agent, has also become a way to reduce the sulfur content of expanded graphite products. Sulfur-free expandable graphite is prepared by using nitric acid, phosphoric acid, perchloric acid or their mixed acids as oxidant and intercalation agent, or solid oxidants such as potassium permanganate and potassium dichromate, or organic acids such as formic acid, glacial acetic acid, acetic anhydride and oxalic acid as auxiliary intercalation agent[6]. In addition, due to the application of multi band smoke agents and stealth shielding agents in the military field, low-temperature expandable graphite (more than 200 times in volume) has recently become a new research and development direction of expandable graphite. Low temperature expandable graphite mainly uses substances with low decomposition temperature to insert into graphite sheets to form graphite intercalations, which can achieve the purpose of low temperature expansion. Wang Ling *et al.* used $HNO_3/HBrO_3/KMnO_4$ oxidation intercalation system to prepare low-temperature sulfur-free expandable graphite. The initial expansion temperature was 130 ℃, and the expansion volume was 350 mL/g at 600 ℃.

2.2 Electrochemical method

Electrochemical method is based on the mechanism of electron acceptor in the preparation of expandable graphite. Compared with chemical method, the amount of oxidant is greatly reduced, the electrochemical reaction insert is evenly distributed between layers, and the expandable performance of the product is stable, which becomes the main goal of new process exploration[8]. The quantitative flake graphite is installed into an anode, the decomposable salt solution such as ammonium nitrate or H_2SO_4 aqueous solution is used as the intercalation agent and electrolyte. The lead plate, platinum plate or titanium nail mesh are used as the cathode and anode, and the expandable graphite is prepared by electrolysis with constant current[9,10]. In the whole production process, there is no intervention of strong acid, strong alkali and strong oxidant, which not only greatly reduces the production cost and prolongs the service life of the equipment, but also has less pollution, the prepared products have low or no sulfur, and their operability is also significantly enhanced. The preparation of expanded graphite by this method has simple process, high requirements for equipment, and many influencing factors. Sometimes the instability of ambient temperature can lead to the decrease of product expansion volume[11].

2.3 Microwave method

The expanded graphite prepared by traditional high-temperature expansion method takes a certain time to rise to high temperature, and the electric energy consumption is large in the expansion process. By using microwave to expand graphite, it is easy to operate, to control the process, and has the advantages of high efficiency and energy saving[12]. Reich *et al.* successfully prepared expanded graphite by microwave heating method, investigated the effects of microwave power, expansion time and graphite particle size on expansion volume, and found that the sulfur content of expandable graphite products prepared by microwave heating method was lower than that of traditional heating method[13,14]. Shen Jianyi and others prepared magnetic nano metal cobalt expanded graphite composites (Co-EG) by microwave heating. Meter metal cobalt particles are evenly dispersed in the unique network microporous structure layer inside expanded graphite[3].

2.4 Explosion method

For the preparation of expanded graphite by explosive method, $KClO_4$, Zn $(NO_3)_2 \cdot 2H_2O$, $HClO_4$, etc. are usually used as expansion agents to make mixtures or pyrotechnics with graphite. After heating or ignition, the heat generated by low-speed explosion of pyrotechnics is used to produce oxidation phase and intercalation at the same time, so as to make the graphite expand "explosively" to prepare expanded graphite. When $HClO_4$ is used as expansion agent, only expanded graphite is in the product, while when metal salt is used as expansion agent, metal oxide will be generated in the product, so that the surface of expanded graphite can be modified[15].

2.5 Gasphase diffusion method

The gas phase diffusion method is to place graphite and intercalation at both ends of the vacuum sealed tube respectively, heat at the intercalation end, and use the temperature difference at both ends to form the necessary reaction pressure difference, so that the intercalation enters the flake graphite interlayer in the state of small molecules, so as to prepare the graphite interlayer compound. The number of product layers produced by this method can be controlled, but its production cost is high[16].

3. Application of expanded graphite

Expanded graphite and functional composite expanded graphite materials have a wide range of applications. They can be used as flexible graphite, flame retardant, oil absorbing material, multi-band smoke agent, stealth shielding material, catalyst, medical dressing, microbial carrier and nano conductive filler. In recent years, people are sealing a lot of application research on expanded graphite has been carried out in various fields such as flame retardant, environment and military, especially in the field of sealing materials. These application studies are of great significance for the expansion and extension of expanded graphite.

3.1 Sealing material field

Expanded graphite has large specific surface area and high surface activity. It can be compressed without any binder and sintering. Graphite paper, coil or plate made by molding or rolling, is called flexible graphite[17]. Flexible graphite not only retains a series of excellent properties of natural graphite such as high temperature resistance, corrosion resistance and sealing, but also has the flexibility, resilience and low-density properties that natural graphite does not have. Compared with traditional sealing materials (such as asbestos, rubber, cellulose and their composites), flexible graphite is a sealing material with better performance, which can be used for sealing facilities in petrochemical, machinery, metallurgy, atomic energy, electric power and other industries, and is known as the "king of sealing"[18].

Flexible graphite also has some weaknesses that cannot be ignored, such as its porosity, low strength and poor wear resistance. It is not ideal to use it directly as the sealing material of some pumps, cylinders and valves. Therefore, in recent years, researchers at home and abroad are trying to research and develop flexible graphite composites to enhance their application properties[17]. The main studies on flexible graphite composites are metal-flexible graphite composites[19], polymer-flexible graphite composite[20], and inorganic-flexible graphite composites[21].

3.2 Study on application of flame retardant materials

Polyethylene, polypropylene, polyurethane and other plastics are widely used in various fields of industrial production. Because of their low oxygen index, flammability and high heat release, they are very easy to cause large fires. Therefore, the treatment of flame retardant of these materials is particularly important[22–24]. At present, flame retardant materials show the development trend of low smoke, less toxicity and no halogenation. Intumescent flame retardants are considered to be one of the promising ways to realize non halogenation of flame retardants. Expandable graphite (EG) expands rapidly under high temperature to form a worm like stable carbon layer and is non-toxic. Therefore, as a typical physical expansion flame retardant, it has become a research hotspot in the field of flame retardant. At the same time, because of its high flame retardant effect, it has been well applied in thermosetting plastics. Research shows that EG alone can effectively improve the flame retardancy of polyurethane elastomers, polyurethane foam and polyurethane coatings. However, adding EG to thermoplastic alone is not ideal to improve the flame retardant effect, so it is necessary to add flame retardants such as red phosphorus, ammonium polyphosphate, magnesium hydroxide and metal oxides for synergistic use[24,25]. In addition, by adding fine particles of expandable graphite to ordinary coatings, a better flame retardant and antistatic coating can be prepared[26]. Adding expandable graphite to APP/PER/MEL fireproof coating can effectively improve the microstructure of expanded carbon layer, reduce the thermal conductivity of carbon layer and greatly improve the thermal stability of the coating[27].

3.3 Lubricity of expanded graphite

The original lubrication properties of expanded graphite are improved because the interlayer distance is enlarged. The oil-bearing resin material with good friction and wear resistance can be made by mixing expanded graphite filled with lubricating oil with tetrafluoroethylene and polyacetal. The addition of the expanded graphite to the grease improves its shear strength, viscosity, and colloidal stability. The good polar adsorption effect and high temperature masking effect of expanding graphite have produced obvious efficiency effect in antifriction resistance of lubricating oil[18]. Li Chunfeng *et al.* treated worm graphite by ultrasonic to obtain expanded graphite lubricating oil additive of worm graphite and nano graphite flake mixture, and modified it in situ in an 10 oil with ethyl cyanoacrylate. The results show that expanded graphite additive can effectively improve the anti-wear performance and bearing capacity of lubricating oil and reduce the friction coefficient[28].

3.4 Application of expanded graphite in environmental field

Expanded graphite is a loose and porous vermicular material, which forms a large number of network pore structures, with large specific surface area, high surface activity and strong adsorption performance. In recent years, its research and application as environmental materials have attracted extensive attention. The pore structure of expanded graphite is mainly macroporous and mesoporous, so it is different from microporous materials such as activated carbon and molecular sieve in adsorption characteristics, and it is more suitable for liquid phase adsorption. Expanded graphite is hydrophobic and lipophilic, and can selectively remove non-aqueous components in water, such as removing oil slick pollution from sea, rivers and lakes[29]. Expanded graphite can form a certain "oil storage space" when absorbing oil, which can store oil substances that much larger than its total pore volume[30]. After adsorbing a large amount of oil, the expanded graphite can be aggregated into blocks and float on the liquid surface, which is easy to recover and can be recycled after renewable treatment. And expanded graphite is composed of pure carbon, non-toxic and chemically inert, so it will not cause secondary pollution in water. In addition, expanded graphite can also be used to remove oil and pollutants from industrial oils and wastewater and harmful substances, such as pesticides and dyes. In addition to selective adsorption in the liquid phase, expanded graphite also has a certain removal effect on SO_x and NO_x gases in industrial exhaust gas and automobile exhaust gas causing air pollution[31]. Fu Meng and others modified the surface of expanded graphite with CTAB as modifier. The modified expanded graphite has good adsorption performance for indoor harmful gas formaldehyde at room temperature[32].

3.5 Application of expanded graphite in military field

Millimeter wave detectors are widely used in military affairs. The United States and other western countries have 3 mm and 8 mm guided weapons. In order to counter the threat of millimeter wave guided weapons, countries all over the world have carried out research on jamming millimeter wave technology. Qiao Xiaojing and others achieved rapid preparation and dispersion of expanded graphite by pyrotechnic explosion. The expanded graphite formed by instantaneous explosion was dispersed in the predetermined airspace to form aerosol interference cloud smoke agent. The test shows that its attenuation rate to 8 mm wave is large[15]. Guan Hua *et al.* found that the expanded graphite smoke screen can better attenuate 3 mm wave and 8 mm wave radiation, so the smoke screen generated by the expandable graphite smoke agent can be used to interfere with the detection of millimeter wave radar[33]. In addition, expanded graphite powder has strong scattering and absorption characteristics for infrared waves and is a good infrared stealth material[26]. Magnetic metal expanded graphite composites have good electromagnetic shielding effectiveness in a wide frequency range. For example, Co-EG and Fe_2O_3-EG are excellent electromagnetic wave shielding materials. Expanded graphite plays the role of reflecting electromagnetic radiation, and nano magnetic metal plays the role of absorbing electromagnetic radiation[33,34].

3.6 Application of expanded graphite in electrochemical field

Expanded graphite not only has excellent conductivity and adsorption, but also has good chemical stability. In early 1998, it was proposed that the alkaline zinc manganese battery made of porous graphite in the positive electrode has a discharge performance 50 higher than that of the same type of battery[35]. Shu Dechun *et al.* added expanded graphite to the positive electrode to improve the conductivity and liquid absorption of the positive powder, and improve the manganese carbon mass ratio of the positive electrode, so as to increase the battery capacity, reduce the internal resistance of the battery and improve the high-power discharge performance of the battery[36]. Adding expanded graphite to the zinc anode of rechargeable zinc manganese battery can also reduce the polarization of zinc anode during charging, enhance the conductivity of electrode and electrolyte, inhibit anode dissolution and deformation, and prolong battery life. In addition, lithium can form graphite interlayer compounds with graphite through gas, liquid, solid and lithium salt electrolysis, which has low electrode potential and good intercalation reversibility[26]. Guo Chunyu and others prepared expanded graphite/activated carbon composites under ultrasonic oscillation mixing conditions and assembled them into aqueous electric double-layer capacitors, which can still maintain high specific capacitance and low specific capacitance decline rate under high current discharge condition[37]. In addition, expanded graphite electrodes were prepared for electrochemical detection of electroactive amino acids by Zhao Wei *et al.* The electrode is both an electrochemical sensor and enriching the molecules to be measured, which shortens the time of mass transfer process, and the electrode has good selectivity for tryptophan[38].

3.7 Application of expanded graphite in catalysis

Expandable graphite can catalyze some chemical reactions. It is found that expandable graphite has high catalytic ability for the synthesis of n-butyl acetate, benzyl acetate, dimethyl fumarate, pentaerythritol bisbenzaldehyde and methyl acrylate[39,40]. Expanded graphite has rich pore structure, large specific surface area and good adsorption of organic pollutants. Carrying TiO_2 or ZnO, composite expanded graphite photocatalytic materials can be prepared to adsorb harmful substances such as oil and dyes in water and realize photocatalytic degradation[41,42].

3.8 Other areas

Expanded graphite is a kind of very important biomedical materials because of its good biocompatibility, non-toxic, tasteless and no side effects. Based on the excellent adsorption and drainage performance, air permeability and water permeability, small adhesion with the wound, non-black wound performance and adsorption inhibition of a variety of bacteria, expanded graphite composite can be used as external wound dressing with excellent performance[43]. Expanded graphite plate has good electrical and thermal conductivity, high electrothermal conversion rate and can produce far-infrared ray. It can be used as a new heating material. Expanded graphite based phase change energy storage materials such as paraffin/expanded graphite and polyethylene glycol/expanded graphite prepared from expanded graphite have the advantages of high energy storage density, high heat conduction and heat transfer efficiency, safety and stability, green environmental protection and so on[44].

4. Conclusion and prospect

Different intercalation processes have a great impact on the preparation of expanded graphite and its physical and chemical properties. Its mechanism should be deeply analyzed and the effects of factors such as reactant functional groups and surface potential on pore structure and surface properties should be studied, so as to realize the controllable operation of the structure and surface properties of expanded graphite in the preparation process. The study of composite expanded graphite materials has endowed expanded graphite with new functions and characteristics, which greatly expands the application range of expanded graphite materials. In the future, we should not only strengthen the research on high-quality and high-performance expanded graphite materials, but also further deepen the application research of expanded graphite in electrical, magnetic and thermal components, polymeric materials, biochemical industry and military, so as to make it popularized and applied in these fields.

Conflict of interest

The authors declare that they have no conflict of interest.

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