Study on Purification and Modification Processing Technology of Microcrystalline Graphite

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Abstract. With the continuous development of new energy and new material technology, social industries pay more and more attention to graphite resources, and the related new graphite materials and technologies emerge as the times require in scientific research and practical application. With the continuous development of high-tech graphite industry, the general high-carbon graphite products cannot meet the requirements of all walks of life, so it is necessary to further improve the purity of graphite. Therefore, in the research and development process of new graphite materials and technologies, the first step is to realize the purification and processing of graphite raw materials, and then the functional modification process of graphite can be completed, so that the new graphite materials can meet the application needs of various industries in society in terms of particle size, morphology or performance. Based on this, the exploration of various purification methods of microcrystalline graphite and the processing technology of functional modification of graphite have practical and theoretical significance.

Keywords: Microcrystalline Graphite, Purification Process, Functional Modification Technology.

1. Introduction
Graphite, as the most widely used nonmetallic ore in the present and even in the future, has excellent properties such as high temperature resistance, radiation protection, conductivity, sealing and so on. In graphite materials, there are some composite materials that can be directly used, which are mainly used in mechanical equipment, metallurgy, chemical industry and other fields. With the further development of society, energy resources have become an important factor restricting the development of graphite. Graphite has been widely used in different areas because of its high-quality performance. It can be said that graphite has become an important raw material in the 21st century and plays an irreplaceable role in promoting economic development and improving people's living standards. Natural graphite and cryptocrystalline graphite can be divided into two types according to their application. The crystal that can be observed directly by vision becomes crystalline graphite, while the crystal that needs to be seen under SEM becomes microcrystalline graphite. At present, there are few researches on microcrystalline graphite, and lack of systematic research theory and preparation technology equipment. Therefore, the application process of microcrystalline graphite has become the focus of current academic research. The specific research is as follows:
2. Analysis of Purification Process Technology of Microcrystalline Graphite

2.1. Overview of Microcrystalline Graphite
Graphite is a mineral with crystalline structure, which has lubricity and oil-bearing properties. Its molecular structure makes graphite rich in characteristics. ① Excellent chemical stability. Graphite can be oxidized only when the temperature is higher than 500 °C. The overall chemical activity of graphite is low, and it will not react with various organic / inorganic acids and bases. Therefore, graphite can be used to prepare process reaction containers or storage containers for various special drugs. ② High thermal conductivity. The outer structure of graphite belongs to layered structure, which makes it has higher thermal conductivity than ordinary metal materials. And when the graphite is heated at higher temperature, its thermal conductivity is lower, so it can be used in various high temperature environments. ③ Good conductivity. Graphite has a large number of easily moving electrons, so it can be used as a negative material for batteries. ④ High plasticity. Graphite presents two states. When it is soft, it can stretch into various shapes; when it is hard, its strength is higher than that of diamond, so it can be well adsorbed on the solid surface, and used as an anti-corrosion material. For example, the production process of graphite gasket, with high sealing. ⑤ High temperature resistance. The boiling point of graphite can reach 4250 °C and the melting point is about 3800 °C. Therefore, graphite is one of the most widely used non-metallic materials.

2.2. Study on Purification Technology of Microcrystalline Graphite
It can be seen that microcrystalline graphite has excellent structure and properties, which makes it has a wide range of applications in various fields. Therefore, it is very important to explore the energy-saving and environmental protection, high-speed purification technology of microcrystalline graphite. According to the relevant research data, the current purification methods of graphite are shown in Table 1.

| Purification method | Equipment requirements | Carbon content of product | Advantages | Disadvantages |
|---------------------|------------------------|--------------------------|------------|---------------|
| Flotation method    | Simple equipment       | Generally 80% - 90%, high up to 95% | Low energy consumption, low cost | Limited purity improvement |
| Alkali acid method  | Simple and versatile equipment | 99%-99.9% | Low one-time investment, high taste | Large energy consumption, long process flow, large water consumption |
| Hydrofluoric acid method | Simple equipment | 99%-99.9% | Simple process, low cost | Highly toxic, serious environmental pollution |
| Chlorination roasting method | Complex equipment | About 98% | Low energy consumption, high efficiency | Complex equipment, limited purity of product, unstable process, toxic chlorine gas, serious environmental pollution |
| High temperature method | Complex equipment needs to be specially designed | Over 99.99% | High carbon content of product | Large equipment investment, high energy consumption |
3. Study on Purification Technology of Microcrystalline Graphite

3.1. Experimental Preparation for Purification Process of Microcrystalline Graphite

Different purification processes have different test methods and results. This paper takes flotation as an example. ① Preparation for the experiment. Preparation of microcrystalline graphite ore raw materials, single cell flotation machine, box type resistance furnace, etc. ② Experimental methods. The purification of microcrystalline graphite by flotation method needs to go through slurry mixing, flotation and post-treatment. The process flow is shown in Figure 2. After purification, it is necessary to test the carbon content, element content, laser particle and other performance parameters of the extracted microcrystalline graphite.

![Figure 1. Purification process flow chart of microcrystalline graphite by flotation](image)

3.2. Experimental Results and Discussion on Purification of Microcrystalline Graphite

① Discussion of experimental results. Through the observation and test of the relevant parameters of professional instruments, it can be seen that the raw ore particle size of microcrystalline graphite purified by flotation method is large and effective, with an average distribution of 0.2-1.2m with poor floatability. Therefore, secondary chemical purification is needed. It is found that the fixed carbon content of graphite ore is about 82.08%, and the ash element content of raw graphite ore is mainly composed of Si, Al, Fe, Ca. In a word, the flotation method has higher requirements for the performance of microcrystalline graphite raw ore. In the future, if you want to realize the wide and efficient application...
of flotation method, it is necessary to fine-tune the relevant parameter in the experiment, so as to reduce the impact of flotation experimental factors on the experimental results.

② Analysis of influencing factors of the experiment. For the flotation purification, the mass fraction of slurry mixing, the inhibitor and the diesel oil used in the experiment will affect the flotation purification rate. For example, the inhibitors used in the flotation process are sodium hexametaphosphate, sodium silicate and sodium carboxymethyl cellulose. The inhibition mechanism of these three inhibitors is different, and their formation and effect are also different. For example, sodium silicate acts as a mineral inhibitor in dispersing raw materials, while carboxymethyl cellulose nanoparticle acts as electrostatic adsorption, chemical adsorption and hydrogen bonding. Therefore, in the process selection of microcrystalline graphite, it is necessary to select the best purification method according to the specific parameters of microcrystalline raw materials and the performance requirements of the industry for new microcrystalline materials.

4. Functional Modification Process Technology of Microcrystalline Graphite

The functional modification of microcrystalline graphite mainly refers to the modification and preparation of purified microcrystalline graphite, so as to further optimize the function of microcrystalline graphite. However, the functional properties of microcrystalline graphite needed in different industries are different, which makes the functional modification process of microcrystalline graphite different. At present, the common functional modification of microcrystalline graphite mainly includes inorganic functional modification and organic functional modification technology. Inorganic functional modification mainly involves the oxidation modification of microcrystalline graphite to prepare two-dimensional nanostructured materials to improve its chemical activity; and nitrogen doping modification technology, by doping nearly a large number of nitrogen atoms in graphene, thus the performance of graphene materials is affected, making it have better electrocatalytic activity than other electrocatalysts. Organic co functionalized modification can be divided into two categories: covalent modification and non covalent modification. The method of covalent bond modification is to oxidize graphene and attach carboxyl and other oxygen-containing functional groups on the edge of graphene to produce nucleophilic ring opening reaction and other covalent reactions.

5. Application Research of Functional Modification of Microcrystalline Graphite

5.1. Experimental Preparation for Functional Modification of Microcrystalline Graphite

① Experiment and device preparation. Raw materials and reagents such as purified oxidized microcrystalline graphite, ammonia, potassium permanganate and hydrogen peroxide are prepared. Electronic balance, ph reagent and freeze-drying oven are used.

② Experimental scheme and steps. In the experiment, the oxidation microcrystalline graphite solution is prepared, ammonia water was added, and its ph value is controlled between 10-12. The sample was added to the PTFE reaction tank for temperature and time control. After reaction, the samples are characterized and tested. Multi function surface electron spectrometer and X-ray gun are used to test.

5.2. Results and Discussion of Functional Modification Process Technology of Microcrystalline Graphite

① Analysis of experimental results. Through the test and analysis of the related characterization of the samples, it can be seen that the function of microcrystalline graphite is different with different preparation process, different ammonia water and ph value settings. For example, the influence of ph on the nitrogen doping, different ph on the dispersion of microcrystalline graphene is different. For the functional modification of microcrystalline graphite external structure analysis, it can be found that the macro and micro morphology of the samples are different under different ph values. Under different ph conditions, the macrostructure is a three-dimensional columnar structure with light weight and high structural strength. Even in the case of minimum ph, small pieces of sample are broken, and there are many holes in the sample, which shows the disordered stacking of graphene. Under different ph values,
the structure of microcrystalline graphite oxide presents different patterns. For example, after hydrothermal reaction, microcrystalline graphite presents disordered stacking. At the same time, the reaction temperature also affects the nitrogen doping. When the temperature is 140-160 °C, the samples after the reaction show compact stacking of small pieces, forming an orderly structure, no pore structure, and small specific surface area. When the reaction temperature is 170-180 °C, the sample presents a porous network structure, showing a disordered structure. The pore structure formed by interlacing lamellae can be seen, which is nano scale holes. The lower the temperature, the slower the reduction of the reaction system. This is because the charge balance between layers of the sample is broken, so there is a disordered network structure.

In a word, more parameters and reagents will affect the external structure and function of microcrystalline graphite under the functional modification process of microcrystalline graphite. In the future, in order to realize the wide application of microcrystalline graphite in more fields and improve the parameter performance and functional stability of microcrystalline graphite, it is necessary to grasp the purification method and functional modification processing technology of microcrystalline graphite comprehensively and scientifically, and adopt the correct temperature and ph value when doping nitrogen. Only in this way can we improve the various performance characteristics of microcrystalline graphite. For example, the modified graphite and microcrystalline graphite oxide grafted with pei can make the graphite have high supercapacitor performance and oil-water separation performance after functional modification.

6. Conclusion
To sum up, the energy shortage and environmental pollution treatment will become a difficult problem for human beings. In view of this development contradiction, it is imperative to develop and apply new energy. Microcrystalline graphite, as a new material made of modified raw materials, has great application potential in various high-tech industries. The functional modification of microcrystalline graphite processing technology is not a long-standing, but through continuous experimental preparation. This paper finally obtained that microcrystalline graphite ammonia nitrogen doping, microcrystalline graphite microstructure has undergone a high-quality change, after pei grafting reaction, although the overall strength of the modified material prepared is not high, it still has high value for application. Therefore, for the application and research process of microcrystalline graphite in the future, based on the existing parameter data and experimental experience, continuous parameter optimization and technological innovation are needed. Only in this way can the extensive and energy-saving development of microcrystalline graphite be realized.

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