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Spectroscopic Analysis of Spherical Graphite from Xinjiang Altai

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1 Introduction

In recent years, the study has found that spherical graphite has a high charge and discharge capacity and electrochemical stability, is the ideal lithium battery anode material and important ultra-high capacitor material, with a high cost performance (Chuan et al, 2012). Spherical graphite is also an important raw material for synthetic diamond (Gao et al, 2009). Spherical graphite is a rare natural graphite, in recent years, Xinjiang in China we have found a natural formation of spherical graphite. In the industry mainly through the natural flake graphite ballification treatment (Dong et al, 2000; Shen et al, 2005) or the mesophase carbon microspheres were prepared by thermal polycondensation method, and coal tar pitch was used as raw material (Wang et al, 2000). At present, people know little about spherical graphite, some high-value spherical graphite is used as ordinary graphite mining, a large degree of waste of resources. Therefore, the microstructure and properties of spherical graphite have important material significance and geological significance.

In this paper, phase analysis of the original samples, the purified samples and the oxidized samples were analyzed by X-ray diffraction.

2 X-ray diffraction analysis

2.1 Sample handling method

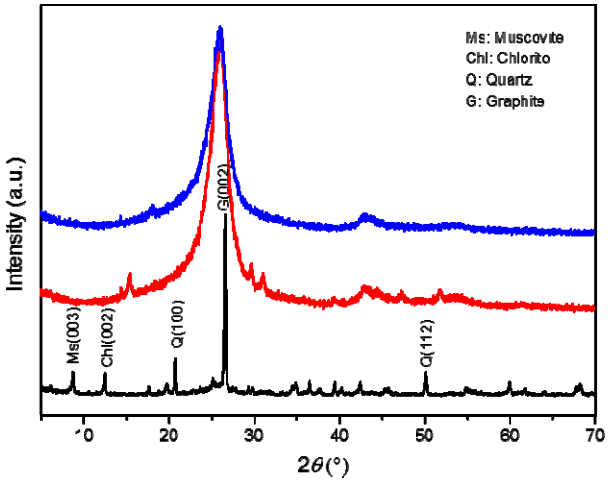
The samples were mainly subjected to two steps of purification and oxidation. The spherical graphite was purified by hydrochloric acid-hydrofluoric acid method. The oxidized spherical graphite condition is: concentrated nitric acid as intercalation agent, potassium permanganate, phosphorus pentoxide as oxidant, spherical graphite, potassium permanganate and phosphorus pentoxide mass ratio 20: 7: 25, nitric acid mass fraction 68%, room temperature using magnetic stirring 80 min, washed to neutral, centrifuged filtration, and then fully dried at 60℃ for 12h.

2.2 X - ray diffraction analysis

The samples were tested using a Rigaku D / MAX-2400PC automatic powder X-ray diffractometer using a Cu Kα ray with a scanning wavelength of 0.154056 nm, a tube pressure of 40 kV, a flow rate of 100 mA, a scanning step width of 0.02 °, DS = SS = 1 / 2º, RS = 0.3 mm, scanning speed 2 (º) / min, scanning range 5 ° ~ 70 °.

According to the half-width β of the diffraction peak of (002), the sizes of the crystal axes of the a and c-axis directions are calculated by the formulas (1) and (2), respectively, La and Lc (002)(Chuan et al, 2012; Li et al, 1999):

$$La=(1.77\times\lambda)/(\beta\cdot\cos\theta) \quad (1)$$



$$Lc(002)=(0.9\times\lambda)/(\beta\cdot\cos\theta) \quad (2)$$

Fig.1.X-ray diffraction spectra of the original samples, the purified samples and the oxidized samples
The black line on behalf of the original spherical graphite,the red line on behalf of the purification of spherical graphite, the blue line represents the oxidation of graphite

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Table 1 Structural parameters extracted from the XRD spectra

Sample	Crystallization Factor		FWHM(β)/°	2θ/°	d_{002}/nm	μ
	La/nm	La/nm				
Original spherical graphite	89.22	45.37	0.18	26.539	0.3356	0.85
Purified spherical graphite	18.88	9.60	0.85	26.161	0.3403	0.25
Oxidized spherical graphite	16.81	8.55	0.93	26.041	0.3419	0.13

Where λ is the X-ray wavelength, 0.154056 nm; β is the half width of the analytical peak; θ is the Bragg angle R. Flanklin (1951) argues that the degree of graphitization can be obtained from the distance d002 (Å) of the (002) (Å) web in the X-ray powder diffraction pattern.

$$d_{002}(nm)=0.3440-0.0086\cdot\mu(2-\mu) \quad (3)$$

It is generally believed that with the increase of the degree of graphitization, that is, the μ value increases, the structure of graphite is also more perfect (Xian et al, 2015).

According to the analysis of x-ray diffraction data, there are quartz, muscovite, chlorite and other mineral impurities besides the graphite in the Altaic spherical graphite ore. After purification by hydrochloric acid-hydrofluoric acid, most of the mineral impurities are removed, resulting in a relatively pure spherical graphite(Fig.1).

Based on these data, the crystallization factor, layer spacing, and graphitization degree of each sample can be calculated. The results are shown in Table 1. The original sample μ value is 0.85, indicating that the spherical graphite graphite degree is higher. After the oxidation and purification of spherical graphite, in which the removal of mineral impurities on the basis of the structure also changed: spherical graphite d_{002} from the original 0.3356nm growth to 0.3403nm and 0.3419nm. Also, the crystal grain size becomes smaller, indicating that the entire graphite crystal was destroyed. It can be seen that the spherical graphite can be used for lithium-ion batteries, super capacitors and so on, But the performance of its excellent yet to be further proof. It is worth noting that the spherical graphite after purification in the layer spacing has become larger, Indicating that the spherical graphite reacts with hydrofluoric acid during the purification stage. It can be seen that the spherical graphite and ordinary graphite is different, worthy of our further study.

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