

Mechanism of Surface Modification of Carbon Black in Plasma

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Abstract

The mechanism of surface modification of carbon black in plasma has been investigated. The experimental results show that after treatment in plasma, the surface energy increased, the PH value decreased. The reason which caused the exchange mentioned above was that the chemical construction of carbon powder exchanged under plasma condition.

1. Introduction

Carbon black with diameter of several microns is a special material with fine size, deep black color and conductivity, and therefore is an important original material in print ink and rubber industry. In order to get the products with high quality, the carbon powder must be adhesive with binder and could be easily dispersed in binder. However, the raw carbon powder is lack of these behaviours. It must be treated before utilization in processing. The traditional ways of treatment were some chemical methods. But the effects were not satisfied. In order to look for a better treating method than before, an investigation on modification of carbon powder in plasma has been done in our laboratory. The experiment was finished in RF and DBD discharge system. The work gases were air, oxygen respectively. The variation of surface construction and characteristics of treated carbon black were tested by XPS, IR spectrum, and PH-Meter, etc.

In this paper the variation of chemical construction of carbon powder after treated in plasma and DBD device system will be re-

ported in detail.

2. Experimental Devices

A RF glow device and a DBD discharge device were used in our experiment. The construction and arrangement of DBD system was showed in Fig. 1. The information about our RF system was written in referene^[1].

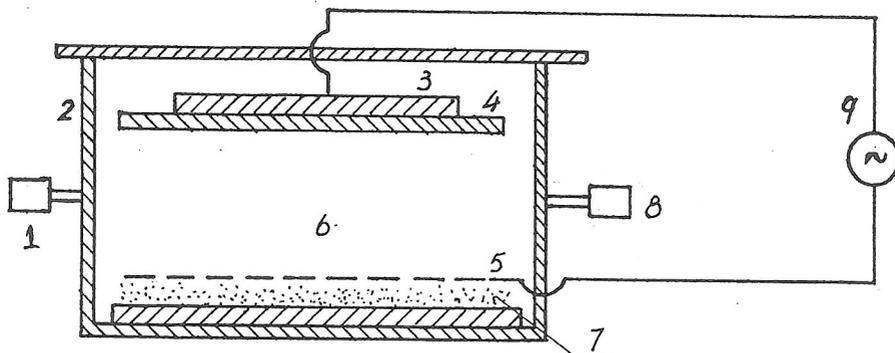


Fig. 1. The construction and arrangement of DBD system.

1. injection valve. 2. container. 3. electrode. 4. dielectric. 5. web electrode. 6. plasma. 7. carbon powder. 8. output valve. 9. power.

The RF glow discharge was supported by a source of power with frequency of 40MHz and power of 800 watts. The discharge chamber was a glass tube with height of 20 cm and radius of 4 cm. Two plate electrodes were arranged outside of discharge tube and pressed close to the wall of tube. In order to avoid the powder of carbon were pumped into pump, a vacuum transition room was used and arranged in the front of discharge tube. The work pressure was $(2-5) \times 10^{-1}$ torr. The DBD discharge device was a special experimental system. It can be operated under atmosphere condition and produce plasma with large size. A source of power with frequency of 20-40 KHZ and power of 1 KW was used in this work.

3. Experimental Results

After treatment in plasma, some behaviors of carbon powder were exchanged. The major variation included following:

a. The ability to adsorb water increased. The variation of this character was tested by means of

measurement of contact angle of water against carbon powder. In order to finish this measurement, a special measurement system was designed, and it was as showed in Fig. 2.

This testing equipment included a water container and a glass tube with fine diameter. The bottom of the glass tube was a plate with many fine holes. As reported in reference [2], the relationship of rising time to rising height of water in carbon column was written in equation (1):

$$h^2 = cr\gamma \cos\theta t / 2\eta \quad (1)$$

Here t and h were rising time and rising height respectively. r was the radius of testing tube, c was a constant, γ and η were surface tension and viscosity of water separately. θ was contact angle of water with carbon powder, the value of $\cos\theta$ represented treating effect. For a certain powder and liquid, equation (1) can be written as following:

or

$$h^2 = A \cos\theta t \quad (2)$$

$$\cos\theta = h^2 / (At) \quad (3)$$

Here $A = cr\gamma / (2\eta)$ was a constant, too. From Eq. (2) and (3) we can see that by measurement of values of h and corresponding t , the treating effect ($\cos\theta$) could be obtained. The measurement results were showed in Fig. 3.

From fig. 3 we can see that (1) After treatment in plasma the wet part of column of carbon powder increased, and the longer treating time the higher height. (2) Using different working gas, the treating effects were different. In a certain treating time the treatment result in oxygen plasma was about one time higher than that in air plasma and two times higher than that in glow plasma.

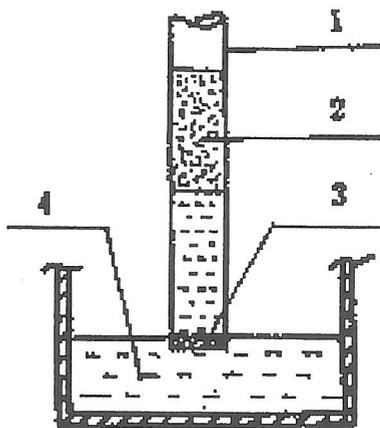


Fig. 2. The construction of measurement system of contact of water to carbon powder 1. testing tube; 2. carbon powder; 3. plate with fine holes; 4. water

b. After treatment in plasma the PH value of carbon powder were changed. The testing results were written in tables 1 and 2.

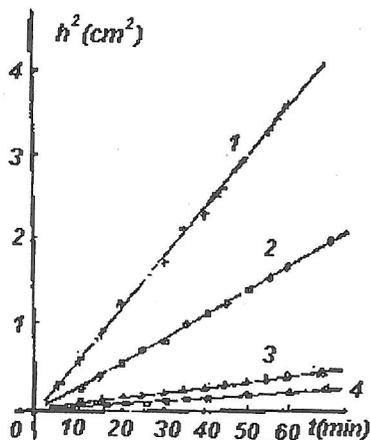


Fig. 3. The h^2-t patterns under different plasma condition
 1. DBD plasma in O_2 ; 2. DBD plasma in air; 3. glow plasma in air; 4. untreated

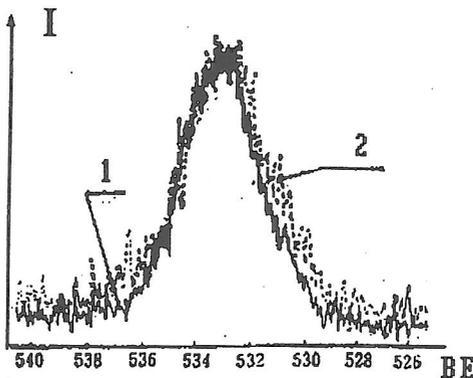


Fig. 4 O_{1s} XPS spectra patterns of carbon powder
 1. untreated; 2. treated

Table 1. The variation of PH value of different sort of carbon powder before and after treating in plasma.

	sample 1	sample 2	sample 3
untreated	5.67	8.43	6.0
treated	3.22	4.44	2.51

Table 2. The variation of PH value so same sample after treatment in plasma with different power.

treating power	0	lower	middle	high
PH value	6.06	5.35	4.61	2.51

The results in tables 1 and 2 told us that the PH values of every kind carbon powder decreased after treatment in plasma, and the higher treating powder, the greater decrement.

c. The exchange of chemical construction of carbon powder sur-

face before and after treatment were tested by using XPS. and IR spectrum.

The typical patterns of XPS pattern were shown in fig. 4.

The strength of XPS signals on energy ranges of 535.5—537.0 eV and 530—533 eV increased after treatment. As we know this range of energy on XPS pattern correspond to groups of hydroxyl, carbonyl and carboxyl. It means that after treatment in plasma the content of active groups containing oxygen increased in surface of carbon powder. These conclusion was agreeable to the testing results about PH values.

The infrared spectrum patterns were shown in Fig. 5.

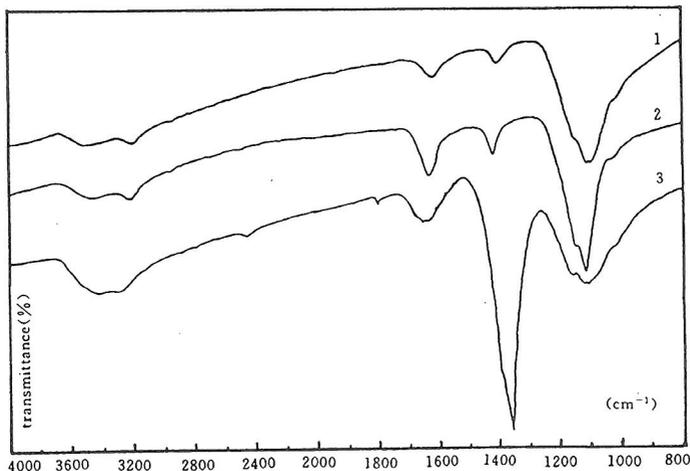


Fig. 5. IR spectrum pattern of carbon powder
(1) untreated. (2) treated in RF plasma
(3) treated in DBD plasma.

Three IR patterns in Fig. 5. corresponde to untreated⁽¹⁾, RF discharge treated⁽²⁾, and BDB discharge ereafed⁽³⁾ samples. Comparing them each other we can see. that the peak value in wave-number range of 1300 — 1500cm⁻¹ was increased after treatment and

the increment was larger by treating in DBD discharge than that by treating in RF discharge. It means. that ① Some new active chemical compounds produced. ② Oxygen play important role in modification reactions. These results were agree with the results obtained from XPS analyse.

4. conclusion

Analyse all experimental results we can get a conclusion that under plasma condition the chemical construction of carbon powder

were exchanged. some new compounds and active groups produced on the surface. these may be the reasons of variations of surface behaviours of carbon powder.

Reference

- [1]. Ge yuan jing et. al, proceeding of the 2nd Asia-pacific Conference on plasma Science and Technology p 377 (1994) Korea
- [2]. Jiang Zi-duo et. al, Chemical Bulletin 7, 31 (1987)