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Fabrication of Photo-Capacitor Electrode with Carbonaceous Materials

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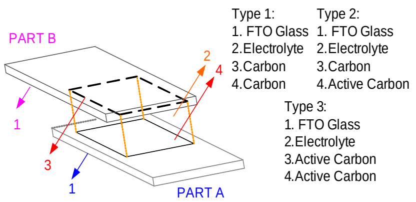
Abstract - 최근 광 캐패시터 전극 분야는 고효율과 넓은 응용 분야로 인해 주목 받고 있다. 본 연구에서는 carbon, 활성 carbon을 사용하여 캐패시터샘플을 제작 하였고 간단한 샌드위치 구조에 각각의 캐패시터 전극은 20 x 15nm의 셀을 사용하였다. 각 셀들은 제작방법에는 졸겔법이 사용되었다. 각 셀들의 수분 및 기타물질의 제거를 위해 120℃에서 1시간동안 건조시켰고 500℃에서 2시간동안 소결처리 하였다. 소결처리로 인한 carbon의 특성변화를 알아보기 위해 XRD분석을 실시하였다. 본 연구에서는 캐패시터의 특성을 파악하기 위해 임피던스 특성을 분석하였고 그에 따른 부수적인 결과들을 기술하였다. active carbon을 사용한 캐패시터의 충방전 특성을 측정하였고 최종적으로 평균 두께 32μm, 입자사이즈 1~4.5μm의 캐패시터전극용 샘플(20 x 15nm)을 제작하였다.

1. INTRODUCTION

Photo-capacitor electrode with carbonaceous materials was fabricated with simples and wich method using 2 different materials. Carbon and active carbon as carbonaceous materials was used and it will be compared to know the better efficiency and fabricated using the same method. Capacitor electrode was fabricated using several methods, such as evaporation, sputtering, chemical vapor deposition, sol-gel method, thermal method and the fabrication process will be explained in detail. In this process, carbon and active carbon powder must be changed from powder to paste to be assembled later on conducted glass plate. Carbonaceous materials were made from heating the material below its melting point until its particles adhere to each other. In this work, 450°C~520°C in 2 hour sintering process was analyzed and annealing treatment was needed. This fabrication process is simple preparation method of capacitor electrode and the discussion was focused on fabrication

2. EXPERIMENTAL

Carbonaceous materials will be assembled to conducts glass to make capacitor electrode with simple sandwich type. Capacitor electrode was fabricated using simple sandwich method and made in 3 different varieties. This fabrication method was shown in fig.1. Carbon and active carbon paste was used in this material as a cell which size 20 x 15 mm. Capacitor is separated in two parts and electrolyte is used as a separator.



<Fig 1> Photo-capacitor electrode illustration

2.1 Carbonaceous material preparation

Carbon/active carbon powder was immersed in 15ml Titanium Iso-propoxide and 100ml 2-propanol for 1 hour, mixed by using magnetic stirrer. In this process, Carbon/active carbon powder of 0.4 g was added to this solution and 10~15 ml NH_4OH+H_2O (1:1) was added drop by drop under constant stirring until it turned to a sol at ambient condition. Then, in sol condition, carbon or active carbon was heated at 120°C in 2 hour to make a dried gel to perform sintering process. Sintering process is needed to obtain a very high purity of carbon/active carbon. Sintering temperature in carbon will analyzed with 450°C~520°C in 2 hour and it shown in Table 1. Particle sizes of carbon and active carbon were investigated by field emission scanning electron microscope.

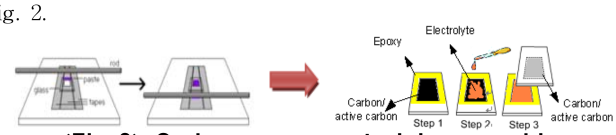
<Table 1> Carbon paste manufactures condition

Carbon	A	B	C	D
Dry temperature	120°C	120°C	120°C	120°C
Time	120 min	120 min	120 min	120 min
Sintering temperature	450°C	480°C	500°C	520°C
Time	120 min	120 min	120 min	120 min

In 1 gram dry carbon, 2 ml α-terpineol with 0.7 g ethyl cellulose was immersed in 100 ml Ethanol for 1 hour and put in ultrasonic cleaning. Then, using evaporation process, carbon/active carbon paste can be made in 8-10 min with 50°C~75°C in water heated chamber. The carbon paste, used for comparison, was prepared in exactly the same manner as that of the active carbon.

2.2 Carbonaceous materials assembly

The cells were doctor bladed onto conducting glass plates using a glass rod or a rod coater. The method is illustrated in fig. 2.

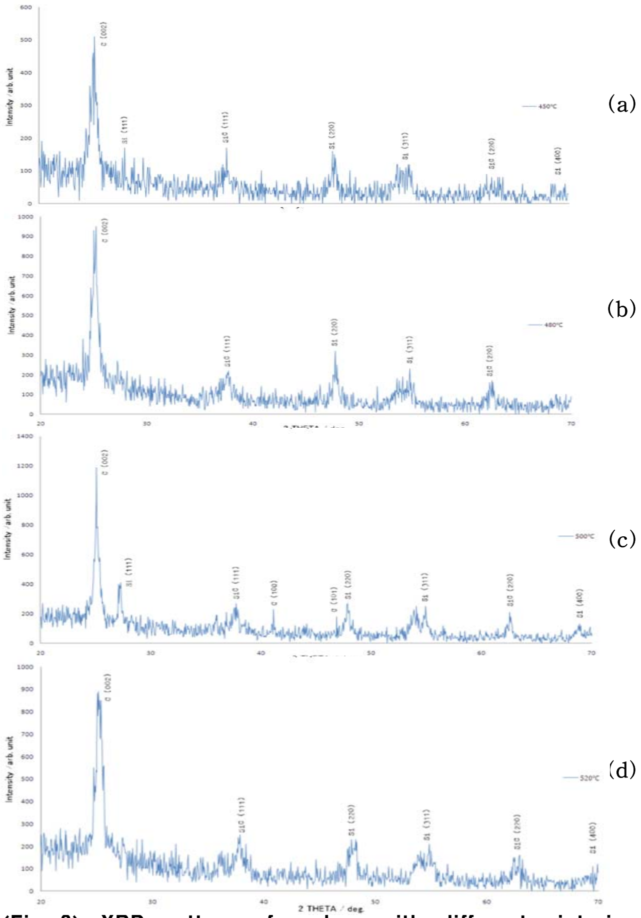


<Fig 2> Carbonaceous materials assembly

A cast frame was cut out of tape and it was laid over the glass substrate. Mold was made from rectangular aluminum pipe to achieve some consistency in the carbonaceous area. A pattern size of the cast (cell) was cut using a scalpel on a cardboard and the tapes were then laid on the cardboard and cut using the pre-cut pattern. Additional tapes were required to cover the sides of the glass to prevent the paste from staining the 25 x 45 mm sized glass. Paste was initially dispensed using a small spoon and aluminum pipe as a rod coater. After the paste was spread, conducting glass was left to dry for 5 minutes before the tapes were removed and then it left to dry at 70°C for 10 minutes. Capacitor electrode was made using two electrodes with carbon or active carbon, were attached together with electrolyte as a separator.

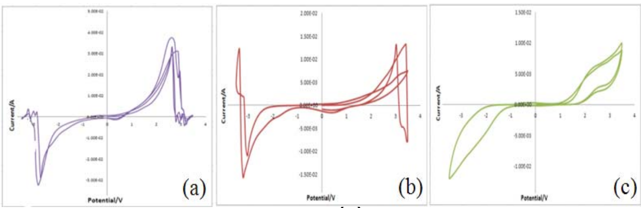
3. RESULTS AND DISCUSSION

The structural and physical characterization of capacitor electrode was characterized by X-ray diffraction techniques (XRD). Alpha step is used to know the carbon cell thickness which 4 carbon sample were analyzed with mean thickness 32 μm. Active carbon paste must have the same thickness with carbon because it used for comparison (thickness range 30-40 μm). High purity of carbon has become an important research to get the best sample. Carbon powder with 500°C sintering temperature in 2 hour is the better temperature to get the high purity of carbon and the better efficiency in discharge process. In fig. 3 which using XRD, it's also shown the comparison in each sintering temperature.



<Fig 3> XRD pattern of carbon with different sintering temperature (a) 450° C (b) 480° C (c) 500° C (d) 520° C

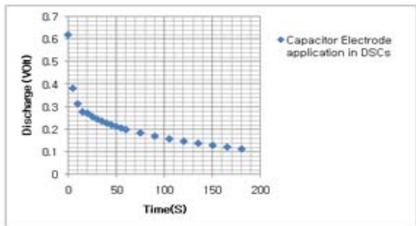
The XRD patterns of carbon powders prepared by co-precipitation and calcined at 450°C-520°C. Carbon powder at 500°C show very sharp diffraction peaks and very broad diffraction lone, indicating good crystalline. It Carbons in general show a 002 diffraction maxima at 2Θ – 25° and overlapped 100 and 101 maxima (usually identified as the 10 peak) at 2Θ – 42°. The stack height Lc, of the graphitic micro-crystallites is determined by the width of the 002 peak. Carbon heated found that both diffraction peaks near 25 and 42° grow with the increase of heating temperature. They also proposed a geometric relationship between the microcrystalline structures and specific surface area of carbon.



<Fig 4> Cyclic voltammograms (a) carbon-carbon (b) active carbon-carbon (c) active carbon-active carbon

Cyclic voltammetry as different electrode material was used to determine the electrochemical properties of electrode capacitor and thus to quantify the specific capacitance for each capacitor. Fig. 4 shows the cyclic voltammetry of carbon and active carbon as electrode for electrode capacitor. The electrode potential was scanned between -3.5 and 3.5 V at 50mV/s with different electrode material. The voltammogram shows a resolved peak at 2.645 V and -2.815 V in carbon-carbon electrode material, 3.415 V and -3.205 V in active carbon-carbon material and 3.48 V and -3.48 V. It can be seen that the two traces are almost similar. There is a drop in current peak between the electrodes from 3.258e-02 to 3.719e-02 for carbon material, from 1.325e-02 to 6.966e-03 for active carbon-carbon material and from 9.917e-03 to 8.785e-03 for active carbon material which increasing as the potential goes past the critical limit. In c-v characteristic, active carbon is more constant on the surface of the electrode then the other electrode material which then detaches from the electrode and allows the current to increase as a function of increased potential.

In this work, electrode capacitor focused to be used as photo-capacitor. In principle, charging reaction is initiated by the light-induced charge separation of dye molecules at the hetero-junction interface and the electron injection from photo-excited dye molecules to the semiconductor conduction band following the mechanism of dye sensitization. Electron and holes of dye transfer to electrode capacitor which positive and negative charges will be accumulated on the microporous surface of carbon or active carbon. Fig. 5 exhibits photo-capacitor electrode behavior application in the discharge process and voltage decrease in the discharge process which a main component as DSCs was eliminated. The voltage approximately decreases in constant condition.



<Fig 5> Discharging characteristic photo-capacitor

4. CONCLUSIONS

Photo-capacitor electrode using simple sandwich method works well with 20 mm x 15 mm size carbonaceous electrode material was analyzed with 3 different samples. Based on this experiment, simple sandwich capacitor 500°C sintering temperature in 2 hours has the highest purity of carbon and the most efficient in the discharge process which using sol-gel method with mean thickness in each type of capacitor is 32 μm. In c-v characteristic, active carbon is more constant on the surface of the electrode then the other electrode material which then detaches from the electrode and allows the current to increase as a function of increased potential.

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