Preparation sheet and Characterization of Carbon Nano Tube /Phenolic resin Nanocomposite for Fuel Cell Bipolar Plate

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Abstract

Proper variation in phenolic resin content is an efficient means to reduce apertures or foramens during manufacture process and decrease cracks occurred under high temperature circumstance.

Furthermore, above-mentioned method also successfully fabricates and increases the mechanical properties and electrical conductivities of nano-composite of ultra-thin fuel cell bipolar plate (thickness around 1.2mm) with micro-fluidic channel.

1 Introduction

Graphite plate is the most general material for preparation of fuel cell bipolar plate. Advantages of graphite plate including great electrical conductivity, excellent anti-corrosiveness, stable chemical property, low contact resistance and great durability which lead to high output of electrochemical power .Due to brittleness of graphite, it is a major difficulty on assembly process. The thickness of graphite bipolar plate around   millimeters is a problem to cause   high-weight, big-volume and high-cost, which limits out-put power of full cells.

Nano-composite for fuel cell bipolar plate possesses several advantages such as grate electrical conductivity, low-cost, light-weight, excellent mechanical properties, anti-corrosiveness and easy-molding.For nano- composite bipolar Plate, Bulk Molding Compound (BMC) is an efficient means for fabricating plate because the bipolar plate made by BMC possesses higher electrical conductivity than injection-molding method.Economizing the use of material, an advantage of BMC leads to low-cost. Except the advantages quoted in the previous column, it also includes high accuracy on size and quality of products.In this article, we explore the preparation and manufacture for fuel cell bipolar plate by BMC.

Because of great thermal stability CNT/ phenolic resin processes than others, which can reduce the apertures during manufacture process and decrease the crakes or flows resulted while material processed under high temperature.

2 Experimental

2.1 Experimental Matierials

1. Phenolic Resin
    Chang Chun Group . Taiwan
2. poly   ehtylene oxide
    Chang Chun Group. Taiwan
3. Graphite powder
    Wei Chang Co.Ltd, .Taiwan
4. Carbon fiber
    Grafil Inc. U.S.A.
5.   TPT (tetra-isopropyl titanate)
    DuPont. Taiwan
6. Carbon nano tube   MWNTs
    Desunnano Co.,Ltd ,Taiwan

2.2 Mold Preparation

Ultrathin mold is required for the conductive bipolar plate design ($3 \times 3 \times 1.2mm^3$, the thickness at center of plate is about 0.95mm) and the surface of mold is plated with high-entropy multi-component alloys which are created first by professor doctor Jien-wei Yeh, Dept. of Material Science, National Tsing Hua University,Taiwan, R.O.C. The ultrathin mold with alloys is presented in Fig. l.

2.3 Samples Preparation

1. The accurate percentage of phenolic resin, tetra-isopropyl titanate (TPT) and CNT were poured into a beaker and dispersed by mechanical stirring, then place well-dispersed resin at ultrasonic oscillator and keep shaking for a while.
2. Pouring the accurate percentage of well-dispersed resin and carbon material into BMC stirrer and mix them by clockwise and counterclockwise stirring.
3. The well-dispersed resin added with carbon fiber is mixed well by clockwise and counterclockwise stirring again. After that, the resin was stored at a freezer.
4. The bulk taken from freezer was divided into several proper weight bulk before hot press.
5. Flate mold was fixed at a hot-press machine. Warming up bulk was placed at the center of mold under preheating temperature at 165℃ around 300 seconds without pressure,and then exerting pressure (3000psi) upon mold keeps around 1500 seconds for creating a specimen.

After completing previous five steps, according to the standards of ASTM D257, ASTM D-3801, ASTM D790 and ASTM D256, the test specimens were cut as rectangular plates to fit in with the preceding standards.
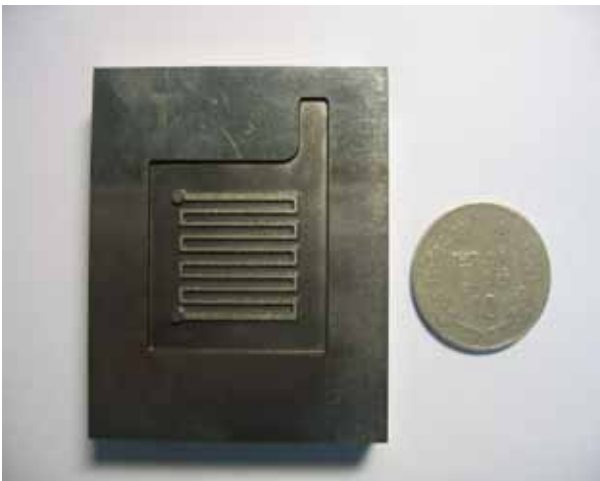

Fig.1 Fuel cell Bipolar Plate of the mold

2-4 Experimental Method

2-4-1 Flexural Strength Test

According to the standards of ASTM D790, the process control must be adopted during test conducted on Universal Test Systems, Tnstron -

4468,for controlling the chuck to descend at a rate of 2mm/min and to obtain the flexural strength. At least six specimens must be prepared for individual different test. A mean value of all data recorded on flexural strength must be adopted.
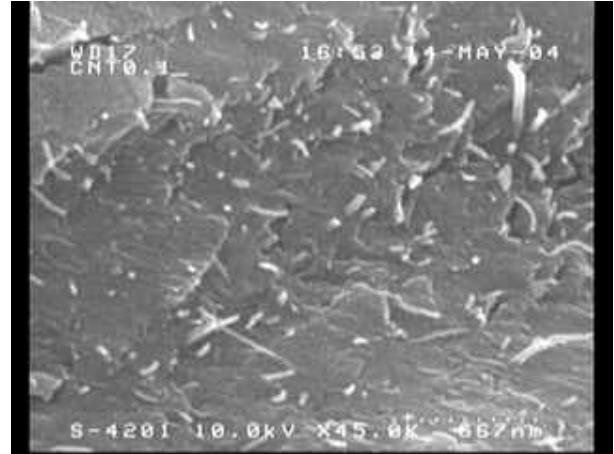

Fig. 2 SEM of composite bipolar plate with 75wt% Graphite and 0.3phr Carbon Nano Tube contents (x45,000)
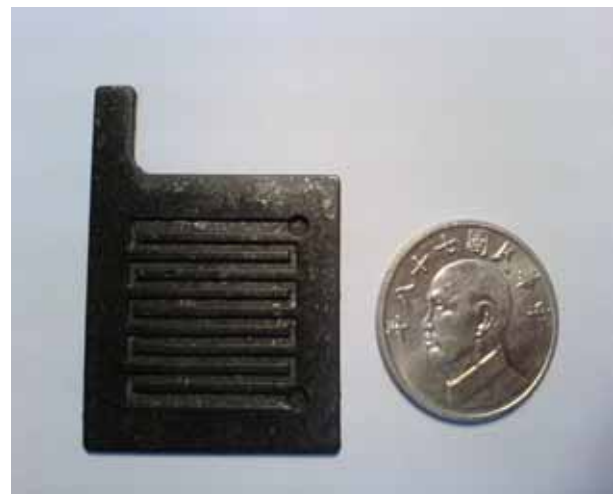

Fig.3 Sheet of composite dipolar plate (3×3×1.2mm)

2-4-2 Impact Strength Test

According to the standards of ASTM D256, at least six specimens were cut as rectangular plates ($63*15*3mm^3$)by a precision diamond saw, and then conduct test by Pendulum Impact Testing Machine.Actual impact momentum = experimental impact momentum(ft-lb) thickness of specimen(in) Actual impact momentum = a mean value obtained from the values by the

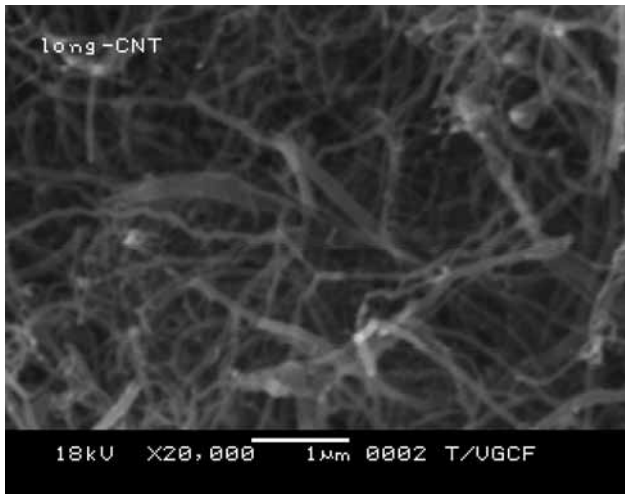values presented on an indicator minusing the calibrated values.

Fig. 4 SEM of Carbon Nano Tube   x 20,000

2-4-3 Electrical Conductivity Test

According to the standards of ASTM D257, The test specimen applied with valtage and current, and the both values are measured at one end. All formulas are expressed as follows

Volume Resistivity $\rho = \pi \times \left(\dfrac{D1}{4t}\right) \times Rv$ (1)

Surface Resistivity $\sigma = \pi \times \dfrac{(D1 + D2)}{(D1 - D2)} \times Rs$ (2)

Major Electrode Diameter   D1   cm
Minor Electrode Diameter   D2   cm
Thickness of Specimen   t   mm
The Measured Volume Resistivity   Rv   Ωcm
The Measured Volume Resistivity   Rs   Ω

2-4-4 Vertical Flame Test

According to the standards of ASTM D-3801,the Flame-Retardance tests conduced on different specimens are 94V-0, 94V 1and 94V 2, the test procedures as follows

1. First step, vertically clip at the point 6mm from upper end of the test specimen and spray a square layer of cotton with approximate domensions of 50× 50× 6 mm 6 mm thick ,which is placed 300 mm lower from the end of the test specimen.
2. Electrical current was adjusted at 150ml/min, and pressure must be kept less than 10mm water column.
3. Igniting and adjusting the flame of the flame chamber to be blue, 20± 1mm.

4. Moving flame chamber to aim the flame at the center of the lower end of the test specimen. If the test specimen will be melted, the flame chamber must be inclined at bevel of 45 degrees.
5. Removing the flame 150mm away from the spec-imen after burning for 10 seconds and recording the first combustion time,t1 second.
6. The flame must be placed immediately 10mm lower away from the base end of the specimen when the burning stops. After burning for 10 seconds, the flame must be moved 150mm lower away from the specimen and the second combustion time t2 and t3 without flame must be recorded.

2-4-5 Limit Oxygen Index   L.O.I.

According to the standards of ASTM D2863 3min or 50mm must be required for burning at least. The L.O.I. can be expressed by the following equation

$$L.O.I = \frac{O_2}{(O_2 + N_2)} \times 100 \qquad (1)$$

L.O.I   21   flammable
L.O.I   22~25   selfextinguishing
L.O.I   26   flame-retardance

3. Results and Discussions

3-1 Electrical Conductivity

Figure 5 shows the electrical conductivities (19s/cm, 24s/cm, 70s/cm, 171s/cm   S/cm   )of composite bipolar plates with constant carbon fiber contents(2wt%)and different graphite contemts (65wt%, 75wt%, 80wt%, 85wt%). From figure 9, the electrical conductivity increases to 70 s/cm when the graphite contents increases to 80wt% . On the other hand, the electrical conductivity decreases to 24 s/cm   when the graphite contents decreases to 75wt%. For comparison of contents of composite bipolar plates, the graphite content increases progressively when the resin content decreases gradually.According to adove-mentioned result, the composite bipolar plate is regarded as a great conductive channel.

Figure 6 shows the electrical conductivities of composite bipolar plates with a constant graphite content(75wt%) and a constant carbon fiber content (2w%) and different CNT cortents. The conductivity is at 137   s/cm   when the CNT of composite bipolar plate is added to 0.01phr.

From figure 6, the conductivities increases progressively with increasing CNT content, and the graphite content added to 75wt% makes composite bipolar plates as a great conductive channel and to decrease the resistance. The electrical resistance apparently downgrade after adding a specified ratio of CNT which also raises electrical conductivity.The varied trend of the previous statement implies that the conductivity of surface of test specimen will occurs when the conductive additive added to a specified ratio must be required. Additionally, the conductivity of whole bulk material is the major test of Volume Resistivty. Therefore, adding the conductive additive and making some conductive paths or channels among some parts of the test specimen will result in decline of Volume Resisivity.

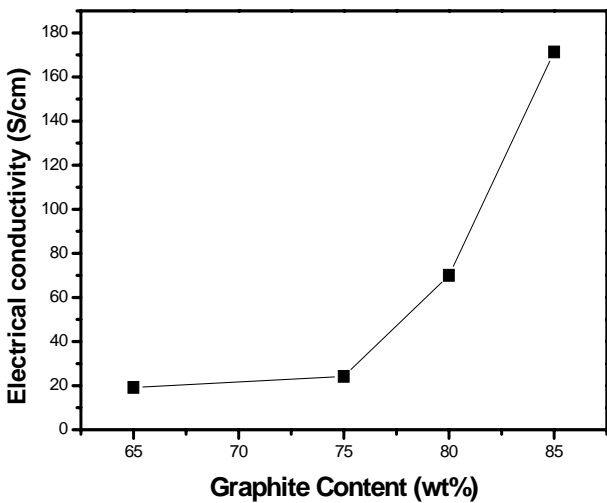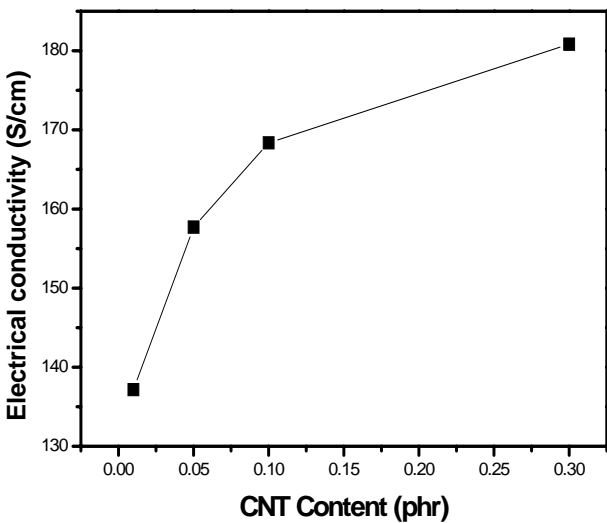Fig. 5Electrical conductivity of composite bipolar plate with different graphite contents

Fig.6 Electrical conductivity of composite bipolar plate with 75wt% Graphite and different Carber Nano Tube contenr

3-2 Flexural Strength

Figure 7 shows flexural strength of composite bipolar plate with a constant carbon fiber content(2w%) and different CNT contents. Flexural strength reduces progressively with increasing graphite powder content. In general BMC process, the stuff or filler contents are added from 40% to 60% . Comparing to the general BMC process, the higher conductive additive content causes the resin content to decline. Due to the great stalemate of resin in BMC, the flexural strengths of higher resin content materials are better than that of lower resin content materials. Consequently, on the basis of the previous phenomenon, the flexural strength reduces with increasing graphite powder content.

Figure8 shows the flexural strength of composite bipolar plate with a constant carbon fiber content (2wt%), a constant graphite content (75wt%) and different CNT contents. The flexural strength reduces little by little with the increase of the CNT contents, the decline trend in flexural strength related to the CNT contents is more alleviative in comparison with that related to the graphite contents and nickel-plating graphite contents. The above-mentioned results occur because of nano-material with powerful Van der Waals and high ratio of the insufficient surface atom coordination which are the major reasons to increase the mechanical properties of nanocomposite   materials.The high specific surface area and the insufficient surface atom coordination lead to the higher absorbility of CNT in comparison with that of bulk materials. The great absorbity of CNT results in the insufficient absorbity of rasin on the graphite surface. The flexural strength reduces progressively with
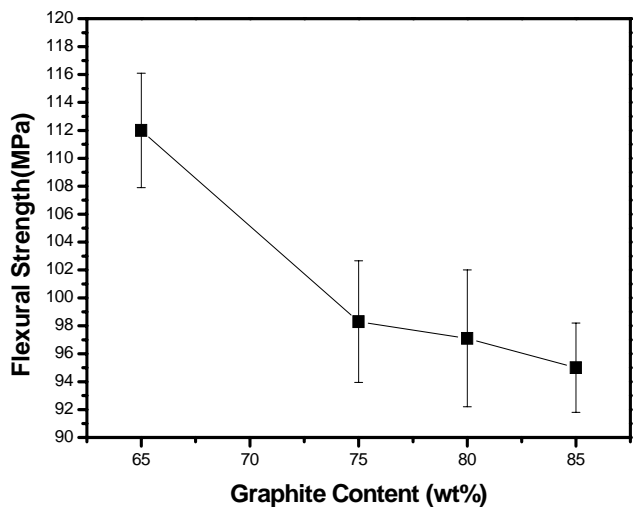

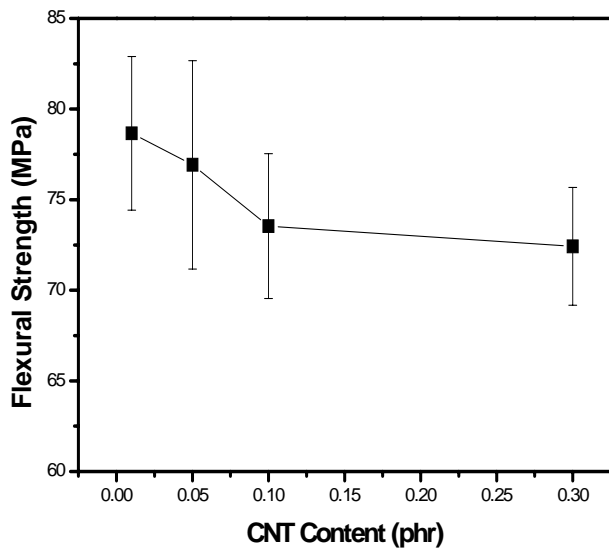Fig. 7 Flexural strength of composite bipolar plate with different graphite contents

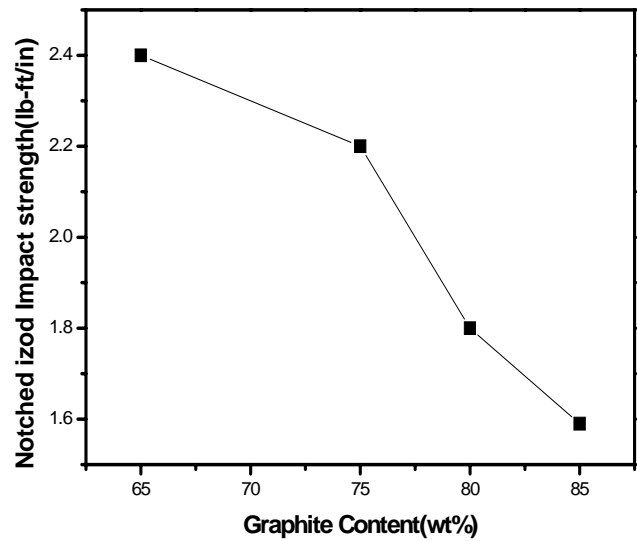Fig .8 Flexural strength of composite bipolar plate with 75wt% Graphite and different Carbon Nano Tube contents

Fig. 9 Notched Izod Impact Strength of composite bipolar plate with different graphite contents

Fig.10 Notched izod impact strength of composite bipolar plate with 75wt% Graphite and different Carbon Nano Tube contents

3.3 Impact Strength

Figure 9 shows the Notched Izod Impact Strength of composite bipolar plate with a constant carbon fiber content (2wt%) and different graphite contents. The resin content increases with the decrease of the graphite con tent. Because resin can act as a adhesive, the mechanical strength of material decreases with the decrease of the resin content. The conductive additive content (65%) of BMC in this study is higher than that of the general BMC. The impact strength declines with increasing the graphite powder content.

Figure 10 shows the Notched Izod Impact Strength of composite bipolar plate with a constant carbon fiber content (2wt%) , a constant graphite content (75 wt %) and different CNT contents. The Impact Strength reduces progressively with increasing CNT content . Moreover, the high specific surface area and the insufficient surface atom coordination lead to the higher absorbility of CNT in comparison with that of bulk materials. Due to the great absorbility of CNT, the increase of CNT content results in a shortage of resin content and leads to the descent on impact strength.
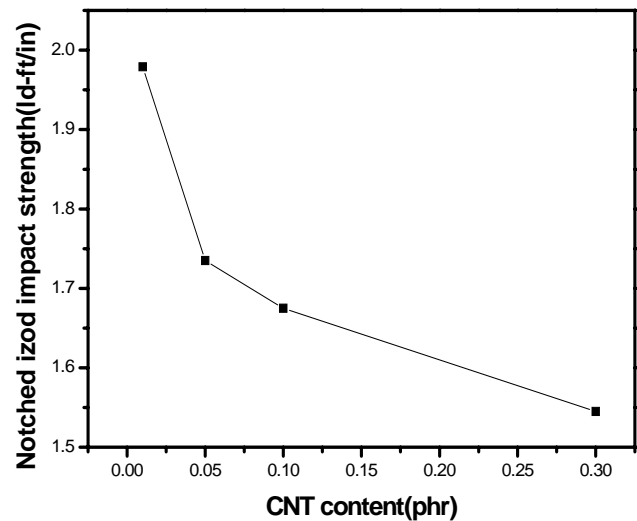
3.4 Flame-Retardace Property

In this study, the composite bipolar plates fabricated by utilizing the BMC are applied in the polymer electrolyte full cell bipolar Plates. Due to the flow of hydrogen and oxygen through between the bipolar plates, the explosion because of burning of both hydrogen and oxygen will be a concerning on the operation of bipolar plates. Hence, the fire

proof flame-retardance of composite bipolar plates is required for preventing the burning occurred due to both external force and flame because the flame-retardance can stop from the explosion of fuel cell bipolar plate.

Table 1 indicates the UL-94 and L.O.I test results of phenolic resin/graphite composite systems with a constant carbon fiber content (2wt%) and different graphite contents (65wt%, 75wt%, 80wt%, 85wt%). Table2. shows the UL-94 and L.O.I test results of phenolic resin/graphite composite systems with a constant carbon fiber content (2wt%), a constant graphite content (75wt%) and different CNT contents (0.01wt%, 0.05wt%, 0.1wt%,0.3wt%). All

L.O.I test results are bigger than 50 and consistent with 94V-0.

Table 1 The UL-94 and L.O.I test results of henolic resin/ graphite composite systems with different graphite contents

Graphite Contents (wt%)	Flaming drops	Cotton ignited	UL-94 standard	L.O.I
65	N/A[a]	N/A	94V-0	>50
70	N/A	N/A	94V-0	>50
80	N/A	N/A	94V-0	>50
85	N/A	N/A	94V-0	>50

Table 2 The UL-94 and L.O.I test results of henolic resin/graphite composite systems with 75wt% graphite and different carbon nano tube contents

CNTs Contents (phr)	Flaming drops	Cotton ignited	UL-94 standard	L.O.I
0.01	N/A[a]	N/A	94V-0	>50
0.05	N/A	N/A	94V-0	>50
0.1	N/A	N/A	94V-0	>50
0.3	N/A	N/A	94V-0	>50

3.5 Thermal Conductivity Test

In the composite bipolar plates, not only the heat but also the electrical surrent of MEA occurred in Electrochemical reaction must be released. if the heat can not be released, it will influence on the power efficiency. Hence, the great thermal conductivity is required for stabilizing the system and preventing the increase of temperature.

Figure11. shows the thermal conductivity of composite bipolar plate with a constant carbon fiber content (2 wt%) and different graphite contents. The different graphite contents are 65wt%, 75wt%, 80wt% and 85wt%, and the thermal conductivity values produced are 58w/m-k, 61w/m-k, 62w/m-k, 62.9w/m-k. The thermal conductivity increase with the increase of graphite content because graphite is a great conductive material, and the thermal conductivity of graphite increase with increasing the density. In contrast, resin possesses lower thermal conductivity, the thermal conductivity of resin do not increase apparently increasing the different graphite contents.

Figure 12 depicts thermal conductivity of composite bipolar plates with a constant carbon fiber content (2wt%) , a constant graphite content (75wt%) and different CNT contents as 0.01phr, 0.05 phr, 0.1 phr and 0.3 phr. The thermal conductivity of CNT is not much different from that of the polymer composite bipolar plate with mixed purified graphite powder because of the greater axial properties of CNT.

Figure 4 shows the SEM image of the web-like entanglement of CNT. After fabrication process, CNT mixed into the composite still presents web-like chaotic dispersion in fig.5,6.

The thermal conductivity 62.9w/m-k of composite bipolar plate with the CNT content of 0.1 phr is close to that (62.9w/m-k) of bipolar plate with the graphite content of 80wt% .
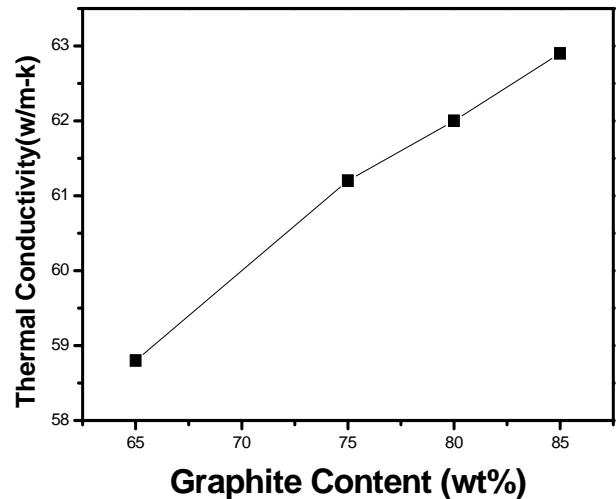

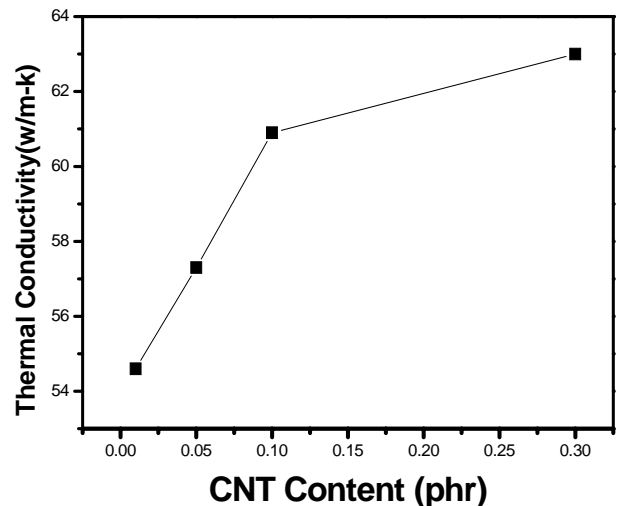Fig. 11 Thermal Conductivity of composite bipolar plate with different graphite contents

Fig. 12 Thermal Conductivity of composite bipolar plate with 75wt% Graphite and different Carbon Nano Tube contents.

3.6 Gas Permeability

The bipolar plate in fuel cell acts as a gas flow field plate. A lot of the complicated fluidic channels within the bipolar plate let oxygen

delivered from anode and gas delivered from cathode disperse well in fluidic channels and diffuse to MEA through the gas diffusion layers. In order to prevent the instability of power efficiency of system caused by gas flowing among the internal and external parts of the bipolar plate, the gas permeability of bipolar plate is far important.

During the hot press with condensation reaction of phenolic resin, not only a large amount of water and methanol but phend and aldehyde leak out or drain simultaneously.

The evaporation rate and quantity are the major points to influence on the aperture size and number of composite bipolar plate. As a result, hot press temperatures, hot press conditions, operating conditions, processing time and the BMC material position are the significant influences on the gas permeability.

Figure 13 shows oxygen permeability of composite bipolar plate with a constant carbon fiber content (2wt%) and different graphite constant, the test results of oxygen permeability are 2.02×10^{-6}, 1.14×10^{-6}, 1.10×10^{-6}, 1.09×10^{-6} (mL/cm^2-sec) respectively. The gas evaporation produced from condensation reaction of phenolic resin occurred during hot press of composite bipolar plate with graphite content at 65wt% and resin content at 35wt% results in the apparently porous phenomenon which causes high gas evaporation. In contrast, the gas permeability decreases gradually with the increase of graphite content. The gas permeability value is at lowest point when the graphite content added at 85wt% ,and not much different from that with graphite content added at 80wt%.

If the graphite content increases, the rasin content will decrease, witch will insult the decrease of porous number produced from condensation reactionduring hot press of composite bipolar plate. Gas existed among graphite's during BMC kneading process by hot press leaks out from material.

The internal situation of composite bipolar plate can be seen through OM microphotograph. The number of the porous existed by above-mentioned process is less thanof the composite bipolar plate with the praphite content at 65

wt% and the layer structure of graphite is batter for Gas Barrater.

The gas permeability reach's top when the graphite content at 65 wt%, and gas permeability declines with increasing the graphite content. Figure 14 shows oxygen permeability of composite bipolar plate with a carbon fiber content (2 wt%) and a fixed amount content of graphite (75 wt%) and different CNT content. The teat results of oxygen permeability are 9.68×10^{-8}, 1.18×10^{-7}, 1.07×10^{-6}, and 1.13×10^{-6} (mL/cm^2-sec).

The oxygen permeability apparently increase with the increasing the CNT content. The high specific surface area and the insufficient surface Aton coordination lead to the higher adorability of CNT in comparison with that of bulk materials. The great absorily of CNT results in the insufficient absorily between graphite and rasin on the graphite surface , which leads increading porodting moreover the leakage of gas among the graphite during not press process also leads to the porosity.

As a result , the porodity of composite bipolat plate increase little by with increasing the CNT content.

3.7 Dimensional Stablilty

In order to prevent the gas leakage and system stability during single cell series connection of the high voltage power apparatus supplied with fuel cell sets, the dimensional stability of bipolar plate is far important. The dimensional stability of composite bipolar plate fabricated by BMC processing is not as good as that of the graphite plate or metal plate, but the low shrinkage requirement of dimensional stability achieved by concerning the great prescription including resin, low shrinkage agent and conductive additive contents is significant.

Figure 15 shows molding shrinkage of composites bipolar plate with a constant carbon fiber content (2 wt %) and different graphite contents. The test results are 0.39%, 0.38%, 0.37% and 0.37%. The resin shrinks during cooling process. In contrast, the graphite expands slightly during cooling process. The shrinkage decreases with increasing the graphite content. The tendency of molding shrinkage can be seen in Figure 15 The major reason of the slight
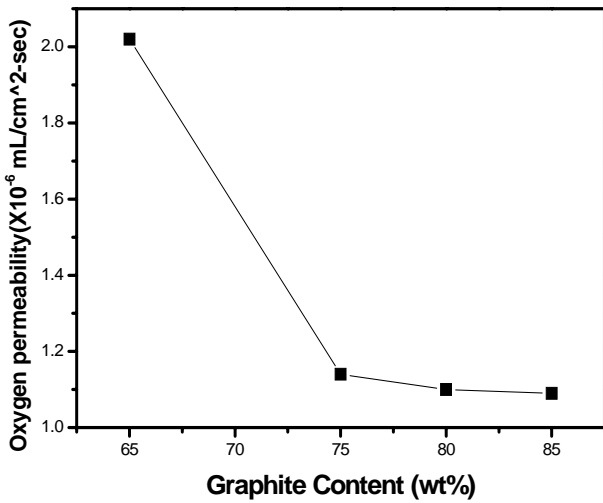
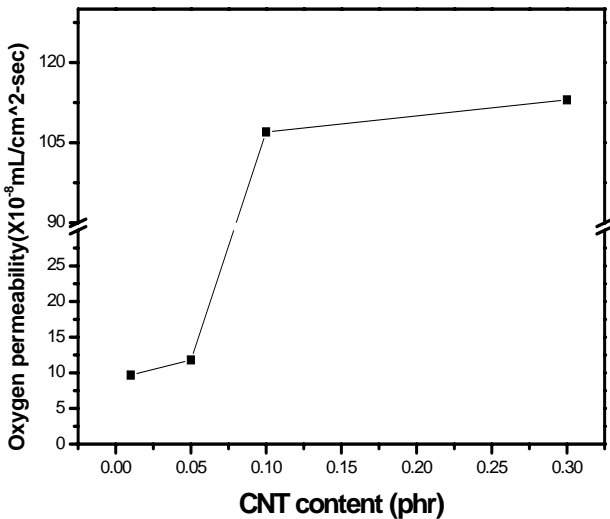Fig. 13 Oxygen permeability of composite bipolar plate with different graphite contents

Fig.14 Oxygen permeability of composite bipolar plate with75wt% Graphite and different Carbon Nano Tube contents

difference between shrinkages is to vary the graphite powder content of prescription. If both resin content and graphite powder content are varied simultaneously, the much difference will occurs between test shrinkages.

Figure 16 depicts the molding shrinkage of composites bipolar plate with 2 wt% carbon fiber content, 75 wt% graphite content and different CNT contents . The test results are 0.3%, 0.38%, 0.32% and 0.28%. Because the total surface area of CNT is bigger than that of graphite powder, the resin content of CNT is less than that of graphite. Hence, the resin among graphite and CNTs not disperse well to lead to the porous existed within the bipolar plate and porous existence slowdowns the expansion-

shrinkage effect of material during temperature variation process. Resin expands when not and shrinks when cold during the process of cooling to the room temperature after not process. In contrast, graphite shrinks when not and expends when cold. But the resin effects can counteract the graphite effects. Therefore, graphite acts as a low shrinkage agent to leak dimensional stability.
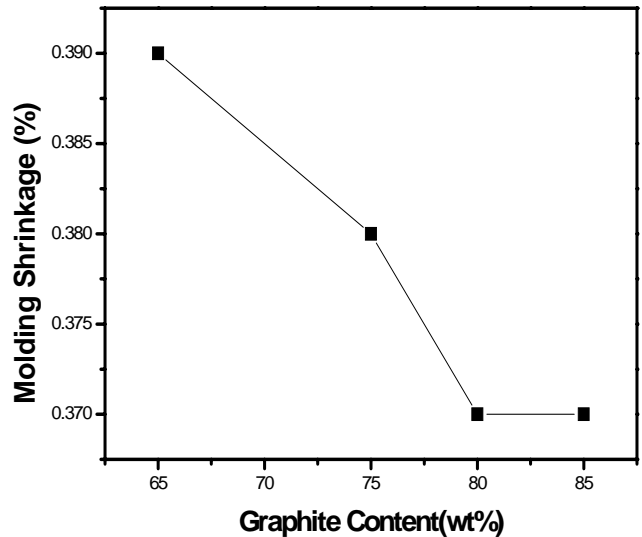

Fig. 15 Molding shrinkage of composite bipolar plate with different graphite contents
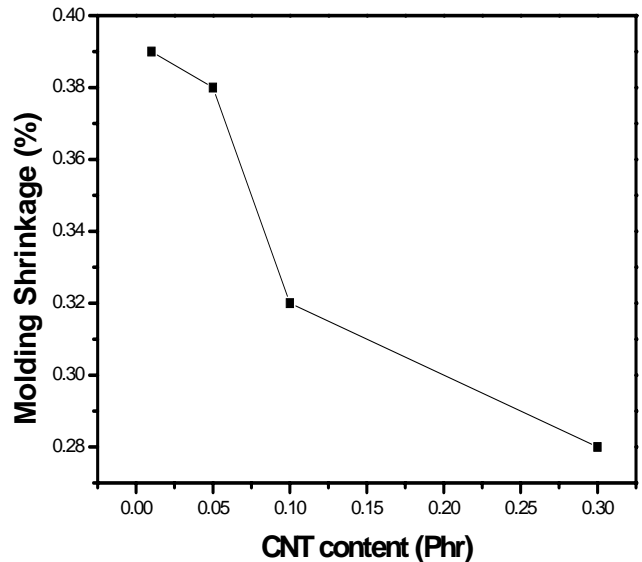

Fig. 16 Molding shrinkage of composite bipolar plate with 75wt% Graphite and different Carbon Nano tube contents

3.8 Corrosion Resistance

In order to keep the fuel cell efficiency at steady state, the chemical stability of bipolar plate

immersed in the powerful sulfuric acid solution is far important, which, a crucial problem of medal bipolar plate, must be solved.

Figure 17 shows anodic polarization curve, the upper part of curve is associated with anodic polarization carve and lower part of curve is related to catholic polarization curve, and the corrosion current can be obtain by Tafel method. Figure 21 also shows the corrosion currents of composite bipolar plate with a constant carbon fiber content (2 wt%) and different graphite contents (65 wt%, 75wt%, 80wt% and 85 wt%). The corrosion currents are 2.84×10^{-5} , 1.77×10^{-5}, 9.9, $\times 10^{-6}$, 1.70×10^{-5}(Amps/cm^2).

The corrosion current of composite bipolar plate with different graphite contents is very law. The composite bipolar plate with graphite content high than 65 wt% can be regarded as a anticorrosive material. Figure18 shows the corrosion current of composite bipolar plate with 2 wt% carbon fiber content, 75 wt% graphite content and different CNT contents(0.01, 0.05, 0.3phr) and depicts the different corrosion currents as 4.1×10^{-5} , 3.47×10^{-5}, 2.81×10^{-5} , 4.43×10^{-5} (Amps/cm^2). There is no a large variation on the corrosion current with different CNT contents, and bipolar plates possess a great corrosion resistance. Therefore, no corrosion occurs. Composite bipolar plate possesses a great corrosion resistance with different CNT contents.
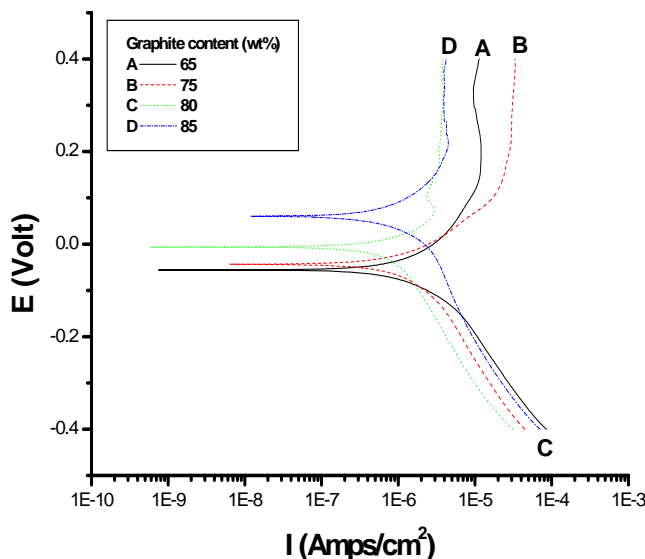

Fig. 17 Corrosion current of composite bipolar plate with different graphite contents

bipolar plate is anisotropic web-like entanglement after BMC molding. Hence, the thermal expansion effect of CNTs with in composite bipolar plate may

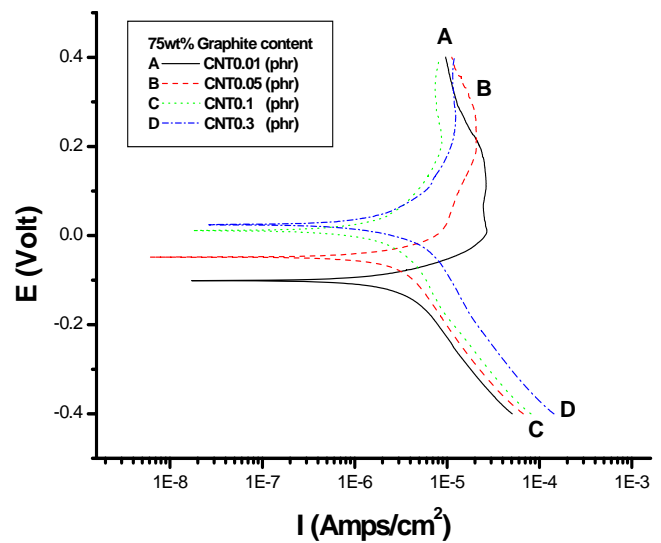counteract mutually to decrease the thermal expansion.

Fig. 18 Corrosion current of composite bipolar plate with 75wt% Graphite and different Carbon Nano Tube contents

Resin expands when hot and shrinks when cool, carbon fiber and graphite shrink when hot and expand when cool. Moreover, the weight percentage of graphite is higher than that of carbon fiber and resin. Therefore, the above-mentioned situations lead to the decreage of the thermal expansion with increasing CNT conte

4. Conclusions

In this study, we successfully fabricate nano-composite fuel cell bipolar plate mixed with phenolic resin and conductive carbide by BMC. Adding proper content of CNTs is an efficient means to increase the physical properties such as electrical conductivity and thermal conductivity.

But previous means does not apparently elevate the mechanical properties, even decrease it .Therefore , in macro appearance, CNTs regarded as impurity may decrease mechanical properties. In micro appearance, the dispersion of CNT within metrix may influence on the material performance. If the dispersion of CNT can be further improved, the entire properties of composite bipolar plate should be increased.

Proper variation in phenolic resin content is an efficient means to reduce apertures or foramens during manufacture process and decrease cracks occurred under high temperature circumstance.

Furthermore, above-mentioned method also successfully manufactures and increases the mechanical properties and electrical conductivities of nano-composite of ultra-thin fuel cell bipolar plate (thinness around 1.2mm) with micro-fluidic channel.

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