Synthesis and Characterization of C-Ag Nanomaterials for Battery Electrode Application

Ruby Michelle N. Buot, Gil Nonato C. Santos

Abstract — Carbon-Silver composite nanomaterials were grown on the surface of a carbon rod using the Horizontal Vapor Phase Crystal Growth technique. The effect of flame annealing to the source materials before HVPC treatment and the varying ratio of the C-Ag materials were studied and was investigated using the SEM and EDX analysis. Results showed that after subjecting the annealed materials with flame, it produced a high yield of deposited nanomaterials compared to the unannealed ones. The improvement of the battery's characteristic was evident with the integration of the C-Ag nanomaterial composite and was confirmed through a comparative study of a 24 hour-voltage discharge profile against the other existing batteries and measurement of its internal resistance using dataStudio and PASCO V-I sensor.

Index Terms— HVPC, composite materials, nanomaterials, battery electrode, carbon and silver

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1 INTRODUCTION

Abundance of carbon in nature made it an easy target as a prominent material utilized in different fields not only in daily mundane tasks but also in science. It even served a very popular subject in research that one author of a book (Harris 2009) wrote that "by the 1980s carbon science was widely considered to be a mature discipline, unlikely to yield any major surprises, let alone Nobel Prizes". However, a quick search on the library and on the worldwideweb will instantly depict an irony of such a statement for it is evident that due to the discovery of carbon nanotubes carbon science is once again fashionable.

The bulk of the research involves carbon nanotubes(CNTs) and its similar form. Though most of the research focuses on understanding its growth mechanisms, some have geared towards investigating its technological applications.

Recent studies include investigations of CNTs and its role in harvesting and storing energy. Though these researches are considerably at their infancy stage, results showed an improved performance in solar cells and better reversible cycles in lithium-ion battery when CNTs and related structures are incorporated in the system.

All these provide an interesting avenue of investigation from synthesis of carbon nanomaterials to its evaluation for application, and this is what the current investigation will be. Manipulation of parameters will be carried out to lead to a better understanding on the nucleation and growth mechanisms of carbon nanomaterials and to further confirm its role in enhancing an electrode's electrochemical performance.

This study is focused on the characterization of carbon-based nanostructures synthesized through Vapor Phase Crystal (VPC) Growth Technique for electrode application.

Specifically, the study aims to investigate the effects of the doping of silver on the graphite powder in the following characteristics of carbon-based nanostructures: surface topography and morphology, elemental composition, and electrochemical performance.

2 EXPERIMENTAL SECTION

2.1 Sample Preparation and HVPC Fabrication

Bulk materials namely graphite powder, silver powder and commercial carbon lid were subjected to EDX analysis showing 99.94%, 98.72% and 98.41% purity respectively.

These materials were prepared by batch, with each batch containing two sets of samples. Each set contains 35 mg commercial carbon lid and a 35 mg graphite powder plus a varied percent by mass addition of sil-

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ver powder that is 0.0175 mg(5%) and 0.175 mg (50%). Each of these samples was loaded into an amorphous silica tube with 8.5 mm inner size diameter, 11 mm outer size diameter and 220 mm length.

The first batch of samples labeled U-0%, U-5% and U-50% were directly subjected to HVPC treatment without having been subjected to flame annealing while the second batch labeled F-0%, F-5% and F-50% was subjected to a flame annealing procedure before HVPC treatment.Using a programmable Thermolyne Tube Furnace, all samples are baked at the optimum growth parameter initially determined elsewhere [1] that is 1200°C growth temperature with a ramp time of 80 minutes for 8 hours. The tubes were then allowed to gradually cool to~100°C and are then drastically cooled by immersing their one end in ice cold water with temperature ~10°C.

Applying nanomaterial retrieval techniques, the HVPC treated materials are then prepared for SEM and EDX analysis [2] and battery fabrication.

2.2 Battery Preparation and Fabrication

The fragments of the HVPC treated amorphous silica tube were submerged in a sodium hydroxide (NaOH) solution and sonicated for 90 minutes. The solution is then filtered, first using a gauze cloth then an ashless filter paper. The filtered NaOH solution is used as an electrolyte, the HVPC treated commercial carbon lid as an electrode and the filter paper as the battery electrode separator. A single electrochemical cell is fabricated out of each HVPC treated carbon lid.

The single cell voltage profiling of each electrochemical cell was done at a 24-hour continuous discharge using PASCO V-I sensor and DataStudio software. The cell with the best discharge profile was reproduced for a 3v battery internal resistance profiling and was subjected to an actual load application test using blinking light emitting diodes (LEDs) as load.

3 RESULTS AND DISCUSSION

SEM and EDX analyses confirmed the HVPC fabrication of silver and carbon nanomaterial to be successful especially on zone 2 and 3. Nanomaterials are formed by promoting nucleation sites through vaporization and condensation of source material by providing a thermal gradient inside the tube as tubes are inserted halfway into a furnace programmed for increasing the temperature at very high level.

The optimum sample setup was found to be the F-5% followed by the U-5% as compared to other sample setup preparation.

The maximum ratio of the source material (i.e. 50%) was observed to produce bigger structures of the material which are mostly in micrometer scale with lesser nucleation site.

Lower surface energy ensures stability of material, thus lowering the surface energy would promote nanomaterial nucleation. Surface energy can be lowered in an overall system, by using concave surfaces as substrate since concave surface has a lower surface energy than a convex surface. Thus Ag nanomaterials were found to nucleate best in the concave groove of the carbon rod rather than on the other regions. [3].

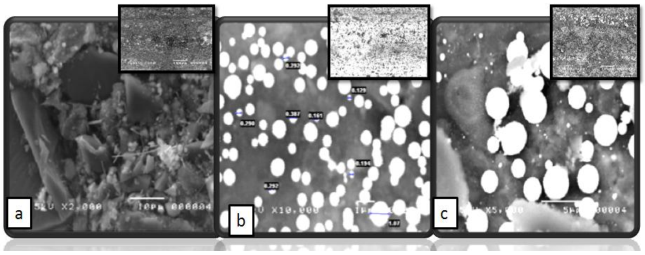

Figure 1. SEM micrographs of nanomaterial formed on flame annealed carbon lid, (a) F-0% (b) F-5% (c) F-50%. Inset shows distribution of silver on carbon lid.

The self-discharge rate is a measure of how quickly a cell will lose its energy while sitting on the shelf due to unwanted chemical actions within the cell. The rate depends on the cell chemistry and the temperature. However, a battery discharge prompted by a connection of a load depends on the load the battery has to supply. The load was therefore held fixed upon comparison of discharge performance among cells.

A flat discharge curve depicts the ideal discharge performance of a battery since the supply voltage stays reasonably constant throughout the discharge cycle. A sloping curve facilitates the estimation of the state of charge of the battery since the cell voltage can be used as a measure of the remaining charge in the cell. Figure 2a shows the curves of the different voltage discharge profile of a battery. The X axis was based on discharge time; the length of each discharge curve would be proportional to the nominal capacity of the cell thus the F5% single cell set up which is represented by the red line shows a better discharge profile than the U-5% cell represented by blue line.

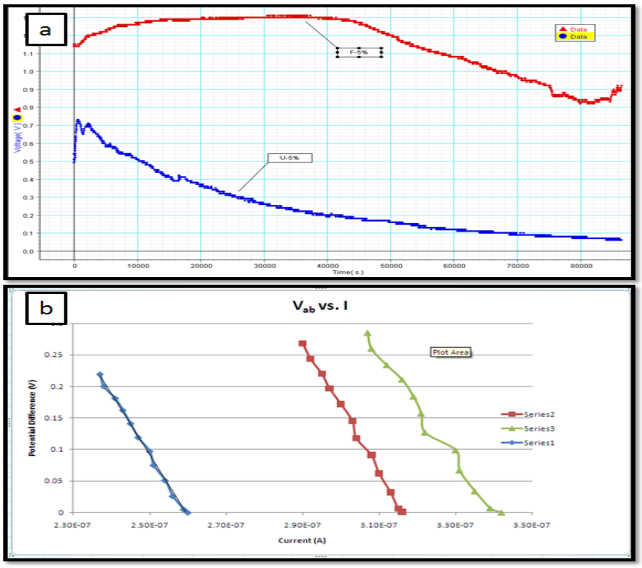

Figure 2. (a) 24-hour single cell voltage profile at constant discharge with constant load (b) Graph of terminal voltage vs. current of three battery setup- commercial, U5% and F5%.

An electrochemical cell's internal resistance determines the operating current it delivers. A comparison on the internal resistance of a battery with nanomaterial to that of a battery without a nanomaterial was therefore undertaken.

Figure 2b illustrates the comparison of the slopes of the three batteries showing the prototype battery with nanomaterial to have the smallest internal resistance which is 8 MΩ as compared to both batteries labeled as commercial and raw which are found to have an internal resistance of 10 MΩ. The blue color represented the raw battery, red the commercial battery and green the battery with nanomaterial.

In the actual load testing, the ~3V battery with nanomaterial was able to power all colors of the two blinking RGB LEDs connected in parallel while the ~3V battery without nanomaterial was limited to lighting up only one red LED. This confirms that battery with nanomaterial has a better voltage source than those without nanomaterial since the blue LEDs have a higher forward voltage than the red ones. The battery with nanomaterial was also observed to have a longer shelf life than the battery without nanomaterial.

4 CONCLUSION

Flame annealing before HVPC treatment promotes nucleation of material rather than growth thus Carbon and silver nanomaterial composite of high purity can be successfully synthesized using HVPC technique when optimum growth parameters and flame annealing treatment are applied.

The HVPC technique is effective in direct surface modification of battery electrode. However, a rod with concave grooves should be used as a substrate to enable direct deposition of nanomaterial.

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REFERENCES

[1] J. Feraer, Fabrication of Carbon Nanomaterials using Horizontal Vapor Phase Growth Technique 2009.

[2] W. Espulgar, Characterization of Silver Nanomaterials, Synthesized by the Horizontal Vapor Phase Crystal (HVPC) Growth Technique, for Antimicrobial Purposes 2010.

[3] G. Cao, *Nanostructures& Nanomnaterials Synthesis, Properties and Applications*, Imperial College Press, London 2004.

[4] D. Linden, T. Reddy *Handbook of Batteries*, McGraw-Hill, Two Penn Plaza, New York 2002.