Characterization of Nanoporous Metal-Carbon Nanotube Composite Arrays

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Introduction

Carbon nanotubes (CNTs) grown in an array have a strong and lightweight nanoporous structure. With the ability to deform to almost 90% and recover, they are a pseudoelastic material with thermal and electrical properties that suggest usefulness in applications such as electrical contacts in compliant systems. CNTs, however, are not suitable for all types of environments. Metals such as nickel and copper are often easier to join to using solder and other contacts. These metals, unfortunately, deform plastically when they are in the bulk form and the elasticity of the contacts is a crucial property for such an application. Previous studies have demonstrated that thin Cu and Ni coatings can also behave in a pseudoelastic manner. Therefore, a composite nanoporous structure with CNTs coated with a thin Cu-Ni coating may be able to provide the needed pseudoelastic behavior.

Objectives

•Determine a method to plate the CNTs with Ni
•Plate samples of CNTs with Ni at different times and look at the effects of agitation

Methods

Previous studies about plating CNTs were researched to determine a proper method. It was found that electroless nickel plating has been conducted on carbon fibers at X'ian University of Technology in China. This method consists of an important six step pretreatment followed by the plating solution.

Degumming
•soaked in acetone for 30 min
•removed and cleaned with deionized water (DI) and methanol in turn

Coarsening
•soaked in 60% nitric acid solution for 30 min
•removed and cleaned with DI and methanol in turn

Sensitizing
•soaked in stannous chloride and hydrochloric acid for 10 min

Activating
•soaked in solution of silver nitrate, ammonium hydroxide, and DI for 10 min

Reducing
•immersed in 37% formaldehyde solution for 30 sec
•removed and cleaned with DI

Dispersing
•immersed in mixture of polyethylene glycol and butanol in an ultrasonic cleaner for 20 min
•removed and cleaned with DI and methanol in turn

Figure 1. A flowchart of the pretreatment steps conducted on the CNTs before plating.

The plating solution consisted of:

•Nickel Sulfate
•Sodium Hypophosphite
•Lactic Acid
•Deionized Water

This solution had a pH of 3. A solution of hydrochloric acid and DI was made to have the same pH as the plating solution to determine whether or not the acidity affected the CNTs.

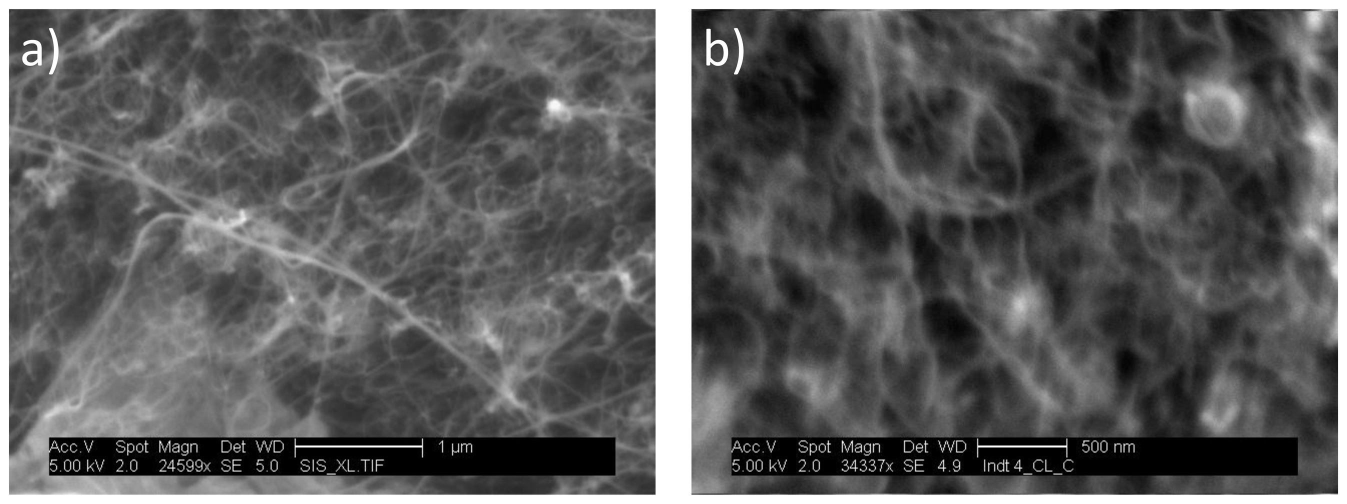

Figure 2. SEM images of CNTs. a) CNTs as they are originally. b) CNTs after soaking in an acid bath.

The plating solution was heated to 90° C in order to plate the CNT arrays. The CNTs were left in the solution for various conditions. The conditions tested include 10, 20 and 60 minutes, and with and without stirring. The plated foams were characterized using scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS), which confirmed nickel was successfully plated onto the CNTs.

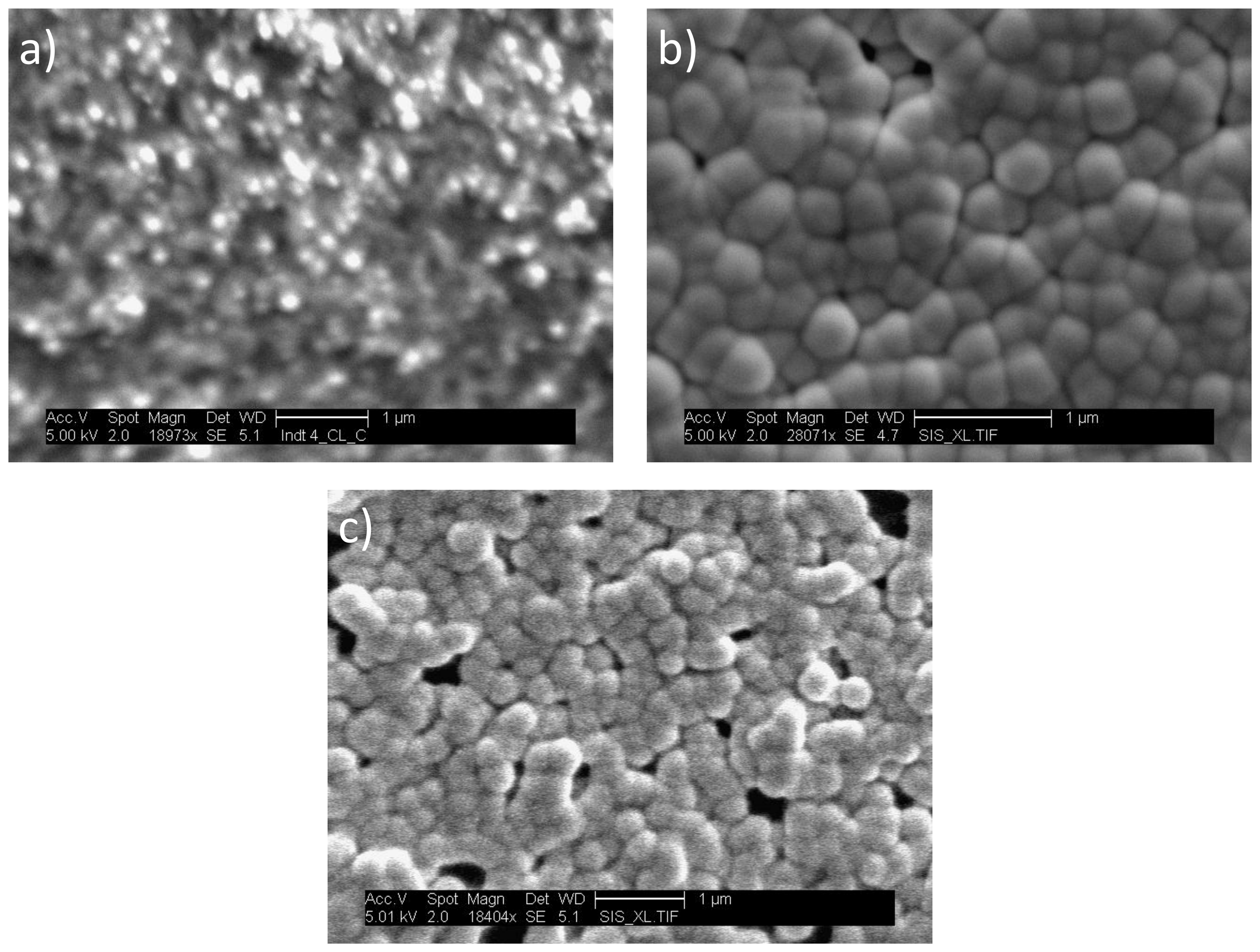

Figure 3. SEM images of the CNTs. a) after being plated at 90° C for 20 min without stirring. b) after being plated at 90°C for 60 min without stirring. c) after being plated at 90° C for 60 min with stirring.

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Results

Table 1. EDS analysis results

EDS results	Wt% Ni	At% Ni
10 min	0	0
20 min	14.07	6.41
60 min	69.09	52.83
60 min w/stirring	21.82	11.78

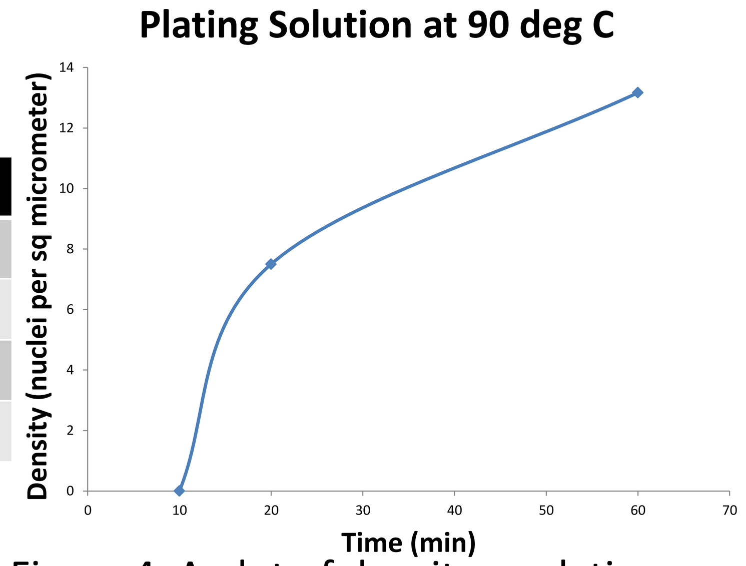

Figure 4. A plot of density vs plating time, without stirring.

Table 2. Average nuclei diameter for each plating time.

Average Nuclei Diameter	
20 min	152 nm
60 min	317 nm
60 min with stirring	252 nm

Table 3. Average distance between nuclei, measured from center to center for each plating time.

Average Nuclei Distance	
20 min	213 nm
60 min	263 nm
60 min with stirring	256 nm

Table 4. A Student's t-test is a statistical test. Here it compares the distance between nuclei from center to center for the 60 and 20 min plating times, both without stirring.

Student's t-test	60 min	20 min
Count	20	20
Mean	263.15	213.35
Variance	3762.56	2483.5
Std. Dev	61.3397	49.8348
Std. Err	13.716	11.1434
Mean Difference	49.8	
Deg of Freedom	36	
T Value	2.818	
T Probability	0.007758	

Table 5. Student's t-test comparing the distance between nuclei from center to center for the 60 min plating times with and without stirring

Student's t-test	60 min	60 min stirring
Count	20	20
Mean	263.15	256.5
Variance	3762.56	2339
Std. Dev	61.3397	48.3632
Std. Err	13.716	108143
Mean Difference	6.65	
Deg of Freedom	36	
T Value	0.38073	
T Probability	0.7056	

Conclusions

The plating density suggests that new nuclei are formed throughout the plating process, so in addition to an apparent incubation timed needed to deposit Ni, new Ni clusters deposit throughout the process. This is validated by the student t-test which shows that the nuclei distances from the 60 min plating and the 20 min plating are statistically different. This suggests that to create a uniform thin coating future tests may have to attempt to increase nucleation rates. Furthermore, the student t-test comparing agitation shows that there is no difference caused by stirring and plating is controlled by the reaction, and not diffusion to the interface.

Future Work

In the future, different plating temperatures will be tested to see the effect it has on the process. The proper time and temperature will be determined to produce a thin layer of nickel. Then copper will additionally be plated on top of the nickel in an effort to achieve the pseudoelastic behavior by alternating layers of nickel and copper to create core shell structures.

References:
1. Zhang, Yunpeng, Hang Zhou, and Chunlan Ren. "Research on Surface Metallization of Carbon Fiber Based on Electroless Plating." *Advanced Materials Research. 189-193. (2011): 1301-1306. Print.*