

## Effect of Nickel Catalyst Layer Thickness and Grain Size Prepared by Electroplating Method to the Growth of Carbon Nanostructures by Chemical Vapour Deposition

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### ABSTRACT

*A simple method of growing carbon nanostructures (CNS), a mixture of carbon nanotube (CNT) and carbon nanofiber (CNF), directly on a nickel catalyst layer electroplated on the copper substrate at low reaction temperature and atmospheric pressure via chemical vapor deposition (CVD) was investigated in this study. The nickel catalyst was prepared using electroplating methods and the current density was varied to give the nickel catalyst layer with different thicknesses and grain sizes prior to the growth of CNS which was carried out at 600°C and under a mixture of 25 sccm: 100 sccm of acetylene to nitrogen for 40 min. A nickel catalyst layer electroplated at 1 mA/cm<sup>2</sup>, which possess a smaller grain size and thinner layer of nickel catalyst, enables the synthesis of high quality and dense CNS as well as high ratio of CNT over CNF.*

*Keywords: Carbon nanostructures; chemical vapor deposition (CVD); electroplating; nickel catalyst layer*

### ABSTRAK

*Kaedah mudah untuk pertumbuhan terus nanostruktur karbon adalah gabungan antara nanotub karbon dan nanoserabut karbon di atas substrat kuprum terelektrosadur lapisan nikel pada suhu tindak balas yang rendah dan tekanan atmosfera dikaji dalam penyelidikan ini. Pemangkin nikel disediakan dengan menggunakan kaedah elektropenyaduran dan ketumpatan arus yang berlainan, bagi menghasilkan lapisan pemangkin nikel dengan ketebalan dan saiz bijirin yang berbeza. Pertumbuhan terus CNS melalui kaedah pemendapan wap kimia (CVD) di atas elektrosadur lapisan pemangkin nikel telah dijalankan pada suhu tindak balas 600°C dengan campuran 25 kepada 100 sccm bagi kadar pengaliran asetilena kepada nitrogen selama 40 minit. Lapisan pemangkin nikel elektrosadur pada ketumpatan arus paling rendah mengandungi saiz bijirin yang kecil dan ketebalan lapisan pemangkin nikel yang nipis menghasilkan pertumbuhan CNS yang berkualiti dan tumpat di samping nisbah CNT yang tinggi berbanding CNF.*

*Kata kunci: Elektropenyaduran; nanostruktur karbon; pemangkin nikel; pemendapan wap kimia*

### INTRODUCTION

Generally, carbon nanostructures (CNS) mainly consist of carbon nanotube (CNT) and carbon nanofiber (CNF). The unique properties of CNT and CNF, including high thermal conductivity of up to 6000 W/mK (Zhang et al. 2010) and extraordinary mechanical and electrical properties make them valuable for electronics, optics and other field of materials (Lin & Wong 2010; Srivastava et al. 2008). Various methods can be used to synthesize the CNS, including laser ablation, electric arc discharge and chemical vapor deposition (CVD) (Jourdain & Bichara 2013; Kumar & Yoshinori 2010a, 2010b; Su & Zhang 2015; Tripathi et al. 2014). Among these methods, CVD is the most popular method due to its simplicity and lower cost as compared to others. Besides, it enables the synthesis of dense and uniform deposit of CNS, gives good reproducibility with adjustable deposition rates, ability to control crystalline structure of CNS and offer a wide scope in selecting the

chemical precursors (Hashempour et al. 2013; Zhao et al. 2011).

The quality of the grown CNS is governed by the types of catalyst and substrates used. Fe, Co and Ni are the most common metal catalysts used due to the high carbon solubility and high carbon diffusion rate in these metals at high temperature for the growth of CNS (Kumar & Yoshinori 2010a, 2010b). Nanometer-sized metal particles are required to allow hydrocarbon decomposition at lower temperature than the actual decomposition temperature of the hydrocarbon due to the catalytic effect that lowers down the decomposition temperature (Kumar & Yoshinori 2010a, 2010b). Several methods have been used to deposit nanosize catalysts on substrate such as physical vapor deposition (PVD) method (thermal evaporation and sputtering) and solution based precursor method (dip coating, spin coating, spray coating and microcontact printing) as it is widely reported that the size of catalyst will affect the diameter of the tubes/fibers produced

(Kuljanishvili et al. 2009; Singh et al. 2002; Xiang et al. 2013). Both methods have its limitations; where PVD is a costly method and involved highly complex equipment, whereas for solution based precursor method, it is hard to confine the catalyst from the solution within small patterns and some processes require longer time for the catalyst preparation (Meyyapan & Srivastava 2007). Thus, the electroplating method is found to be an alternative method, which is more simpler, low cost and able to produce uniform, dense and adherent coating on the metal or metal alloy surface, which can be used as a catalyst for the direct grow of CNS.

In this paper, we report the result of the direct growth of CNS on the nickel catalyst electroplated on the copper substrate via CVD. This paper proposed a way to grow CNS at low reaction temperature and atmospheric pressure. Besides, the suitable current density which can produce a thin nickel catalyst layer with a smaller grain size and higher surface roughness which can help to increase the efficiency of the nickel catalyst towards the growth of dense CNS with a smaller diameter is also investigated in this study.

## EXPERIMENTAL DETAILS

### NICKEL PLATING

A self-made Watts electrolytes was prepared by adding basic Watts components of nickel sulphates,  $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$  (275 g/L), nickel chloride,  $\text{NiCl}_2 \cdot 6\text{H}_2\text{O}$  (60 g/L), boric acid (50 g/L) and 0.1 g/L sodium dodecyls sulphate (SLS). SLS was added as a surfactant to form more uniform nickel deposition (Badarulzaman et al. 2009) and prevent pin holes formation when subjected to high current density. All chemicals were supplied by Merck. The electrolyte was stirred overnight in order to dissolve all chemicals and give a homogeneous solution prior to DC electroplating process (Badarulzaman et al. 2010). Pure nickel foil was used as the anode and pure copper plate with dimension of  $2 \times 3$  cm was used as the cathode. The electrodeposition was conducted at ambient temperature with the pH of the electrolytes is maintained in the range of pH3-4. The current density was varied from 1, 5, 10, 20 and 30 mA/cm<sup>2</sup> for 20 min plating time to study the effect of current density on the grain size and thickness of the nickel catalyst layer deposited on the copper substrate. The surface morphology of the electroplated nickel catalyst layer, were analyzed using scanning electron microscopy (SEM) model *FEI Quanta 450 FEG* (HV: 5 Kv, WD: 10 mm, magnification: 30 k) while atomic force microscopy (AFM) model *Park System XE-10* with *XEI* Program Software (scan area:  $5 \times 5$   $\mu\text{m}$ ) is used to determine the average grain size, surface roughness and surface topography of the electroplated nickel catalyst layer. The thickness and the presences of Ni were analyzed using X-ray fluorescence (XRF) model *Micro Pioneer* with program *XRF 2000 R Series* and energy dispersive X-ray (EDX) with *INCA* software (HV: 20 kV, WD: 10 mm, maginification: 30 k), respectively.

XRF is a non-destructive analysis that commonly used in plating industries for measuring coating thickness i.e. involved the emissions of fluorescent or secondary x-rays from the material (nickel catalyst layer) that has been excited by bombarding with high energy x-rays that is generated from the X-ray tube (4-50 Kv, 10-15  $\mu\text{A}$ , 50 W). This fluorescent radiation will be evaluated by the detector and the multistage electronics circuit will process the measurement signals. Then, the thickness of nickel catalyst layer will be computed and analyzed by the *XRF 2000 R* software. The XRF method needs no sample preparation and a fast measurement as compared to cross section method (Fischer 2015).

### SYNTHESIS OF CNS

The CVD method was carried out in a horizontal quartz tube reactor. The nickel catalyst layer electroplated on a copper substrate was placed directly in the middle of the quartz tube. The reactor was heated up from room temperature to 600°C at the ramping rate of 10°C/min and under 100 sccm of purified nitrogen ( $\text{N}_2$ ). After reaching 600°C, the temperature was maintained for 30 min under the flow of 100 sccm hydrogen ( $\text{H}_2$ ). After 30 min, acetylene ( $\text{C}_2\text{H}_2$ ) with purity 99.6% which acts as carbon precursor was mixed with  $\text{N}_2$  as diluent gas was flown in to initiate the reaction. The reaction time was 40 min and the volumetric flowrate ratio of  $\text{C}_2\text{H}_2:\text{N}_2$  was set at 1:4 for total flowrate of 125 sccm. After the reaction completed, the  $\text{C}_2\text{H}_2$  flow was turned off and the reactor was allowed to cool down to ambient temperature under the flow of  $\text{N}_2$  gas. The surface morphology of CNS grown on the electroplated nickel catalyst layer was analyzed using SEM (HV: 5Kv, WD: 10mm, magnification: 10k), meanwhile the types of CNS and diameter of CNS produced was determined using a transmission electron microscopy (TEM) model Philips CM 12 which system operating at 80 kV. The purity of CNS growth was analyzed using a thermogravimetric analyzer (TGA) TA instrument SDT Q600 equipped with TA Thermal Advantages Software for data analysis. For this characterization, the amount of sample used was about 3-7 mg loaded into an alumina crucible. The temperature of this analyzer was raised to 900°C with a ramping rate of 10°C/min under the flow of 100 mL/min purified air. Besides, the quality of CNS is determined by using Raman Spectroscopy, model Reinshaw via Raman Microscope with laser excitation at 532 nm of argon laser at room temperature in the range of 100-3200  $\text{cm}^{-1}$ .

## RESULTS AND DISCUSSION

### NICKEL PLATING

SEM images in Figure 1 shows the surface morphology of the electroplated nickel catalyst layer on the copper substrate prepared at different current densities. From Figure 1, as the current density increased, the shape of the particles changed from a needle-like to spherical shape

particles. A similar trend was observed in the growth of the grain size of the electroplated nickel catalyst layer, a larger grain size was formed at higher current density. This observation was similar to the findings reported by Uhm et al. (2015). From the images, low current density formed smaller and more uniform grain particles, whereas high current density formed nickel particles of random sizes. This is because the electroplating process conducted at lower current density contributes to more stable and uniform deposition (Hili et al. 2015). A smaller grain size of electroplated nickel catalyst layer was formed at lowest current density, 1 mA/cm<sup>2</sup>, as compared to other

current densities due to the slower depletion of the Ni<sup>2+</sup> ion concentration at cathode electrolyte interface that contribute to higher nucleation rate at cathode surface. This leads to a better distribution of smaller grain particles (Bakonyi et al. 1996; Nayeb Sadeghi et al. 2012; Ul-Hamid et al. 2012). A smaller grain size and uniform distribution of nickel catalyst is important in producing smaller diameter CNS, CNTs in particularly (Hashempour et al. 2013; Nayeb Sadeghi et al. 2012) during the CNTs growth, lead to the production of CNTs decorated with about 6 nm Cu<sub>2</sub>O nanoparticles. We used SEM to study the surface characteristics of Ni catalyst films and characteristic of

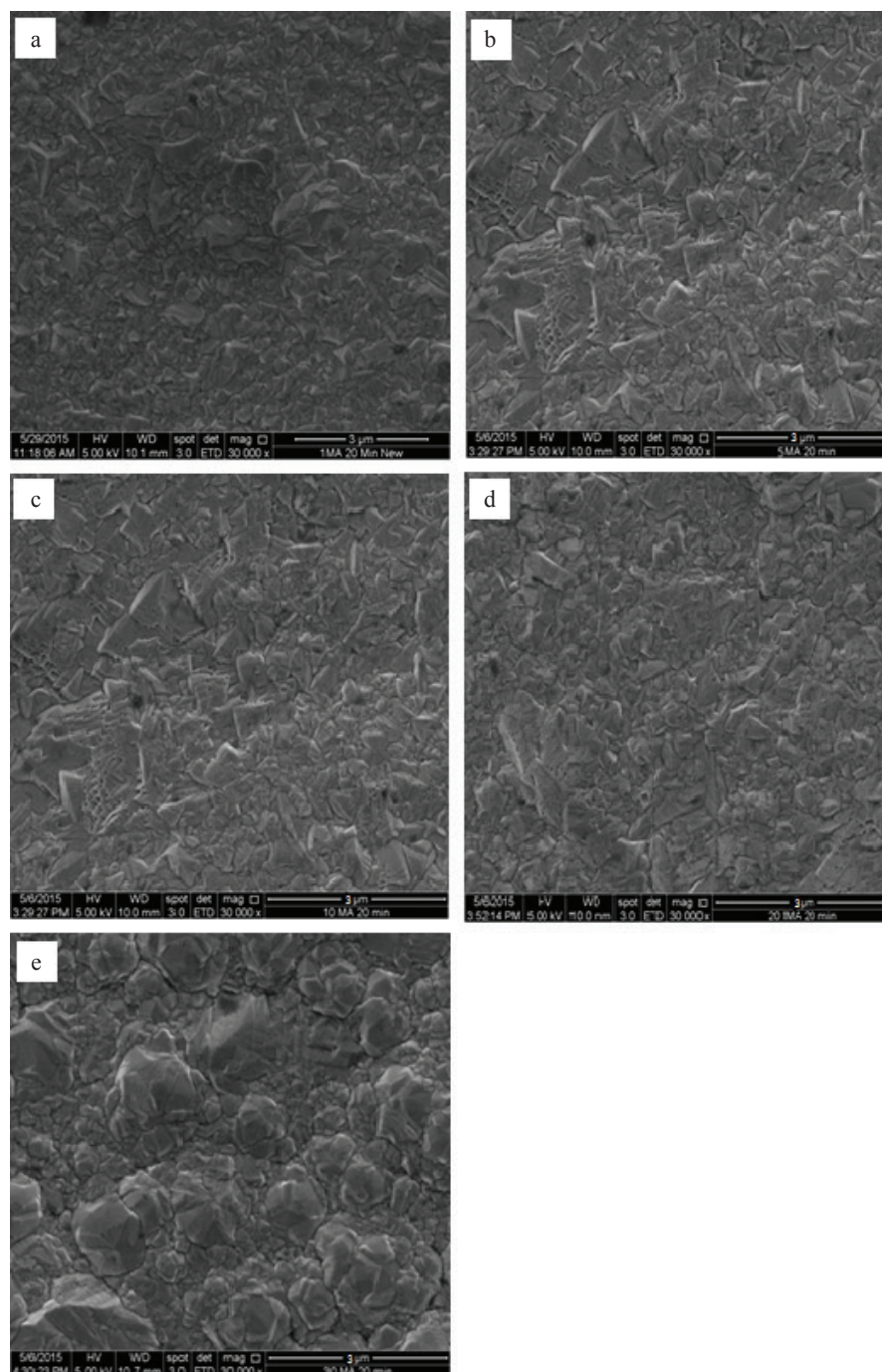


FIGURE 1. SEM images for nickel catalyst layer electroplated at different current densities at 30,000 magnification a) 1 mA/cm<sup>2</sup> b) 5 mA/cm<sup>2</sup> c) 10 mA/cm<sup>2</sup> d) 20 mA/cm<sup>2</sup> and e) 30 mA/cm<sup>2</sup>



TABLE 1. Elemental composition of nickel catalyst layer electroplated on the copper substrate at different current density

Current density (mA/cm <sup>2</sup> )	Element (wt. %)	
	Ni	Cu
1	51.50	48.50
5	65.30	34.70
10	73.50	26.50
20	92.10	7.90
30	96.40	3.60

grown CNTs. Raman spectroscopy, transmission electron microscopy (TEM). In order to verify the presence of nickel and weight percent (wt. %) of nickel over the plated area, the nickel catalyst samples were analyzed by EDX as shown in Table 1. The results showed that, as the current density increased, the weight percentage of nickel also increases. Even though 1 mA/cm<sup>2</sup> contains less weight percentage of nickel as compared to other current densities, but at this weight percentage, over 50 wt. % of the nickel plated catalyst layer, it is able to grow CNS as reported by Makris et al. (2005). The authors reported that over 40 wt. % of nickel is able to form CNS and contains high conversion of carbon precursor to CNS (Makris et al. 2005).

The thickness and percentage of non-uniformity of electroplated nickel catalyst layer were determined by the XRF characterization. The thickness of the nickel catalyst layer increased when the current density increases for both the edge and center as shown in Table 2. When high current density (above 20 mA/cm<sup>2</sup>) was applied to the process, the rate of the reduction process occurs at the cathode surface for Ni<sup>2+</sup> ions to form Ni metal increased as stated in Faraday's law, the rate of deposition is proportional to the current density (Luo et al. 2006), thus leads to the greater deposition rate and thicker deposits. Besides, Table 2 shows that with the same current density, the edge of the electroplated nickel catalyst is thicker than center of the copper substrate and the difference becomes larger for higher current density. This is due to the plating rate of the Ni<sup>2+</sup> ions in the solutions. At lower current density, the fluidic friction between the electrolyte and solid surface is dominant and a low crowded current is not enough to compete with the friction effect which causes the plating rate of Ni<sup>2+</sup> ions on the edge to be relatively small.

TABLE 2. Thickness of nickel catalyst layer electroplated on copper substrate at different current density

Current density, (mA/cm <sup>2</sup> )	Thickness, ( $\mu$ m)		Standard deviation, ( $\pm\mu$ m)	
	Edge	Centre	Edge	Centre
1	0.58	0.46	0.09	0.04
5	2.71	1.55	0.07	0.07
10	6.32	2.66	0.14	0.11
20	12.17	4.88	0.47	0.14
30	21.34	7.66	1.09	0.26

Meanwhile high current density which contains strong electrical field and highly crowded current contributes to the much faster plating rate at the edge. Thus, forming thicker nickel layer plated at the edge (Li et al. 2009).

The average grain size and surface roughness of the nickel catalyst layer was determined by an atomic force microscopy (AFM). The samples were heated at 600°C for 30 min under N<sub>2</sub> flow prior to the AFM characterization to study the effect of heat treatment on the grain size and surface roughness of nickel catalysts. The average grain size and surface roughness of the electroplated nickel catalyst on the copper substrate were measured using *XEI image* software and the results are shown in Table 3 while the surface topography of electroplated nickel catalyst at different current densities were shown in Figure 2. The results showed that upon increasing current density, the average grain size of the electroplated nickel catalyst was increased while the surface roughness of the sample decreased. Moshkalyov et al. (2004) related the surface roughness with the thickness of the electroplated nickel catalyst layer and reported that a thinner catalyst layer possesses higher surface roughness compared to a thick catalyst layer due to the segregation of nickel island particles (Moshkalyov et al. 2004). From the XRF result of the present study, the nickel catalyst layer electroplated at lowest current density, 1 mA/cm<sup>2</sup> contained a thinner nickel catalyst layer and the thickness of nickel catalyst layer was increased as the current density increased. After the heat treatment of nickel catalysts at 600°C under N<sub>2</sub> environment, the nickel catalyst layer electroplated at 1mA/cm<sup>2</sup> had the highest surface roughness and this phenomenon in turn affected the growth of CNS via CVD process.

TABLE 3. Grain size and surface roughness of nickel catalyst layer electroplated on the copper substrate at different current density

Current density (mA/cm <sup>2</sup> )	Grain size (nm)	Standard deviation grain size, ( $\pm$ nm)	Surface roughness (nm)	Standard deviation surface roughness, ( $\pm$ nm)
1	160	4.04	126	1.52
5	221	11.00	106	4.50
10	266	13.50	102	7.50
20	323	14.01	97	8.00
30	366	14.00	52	9.50

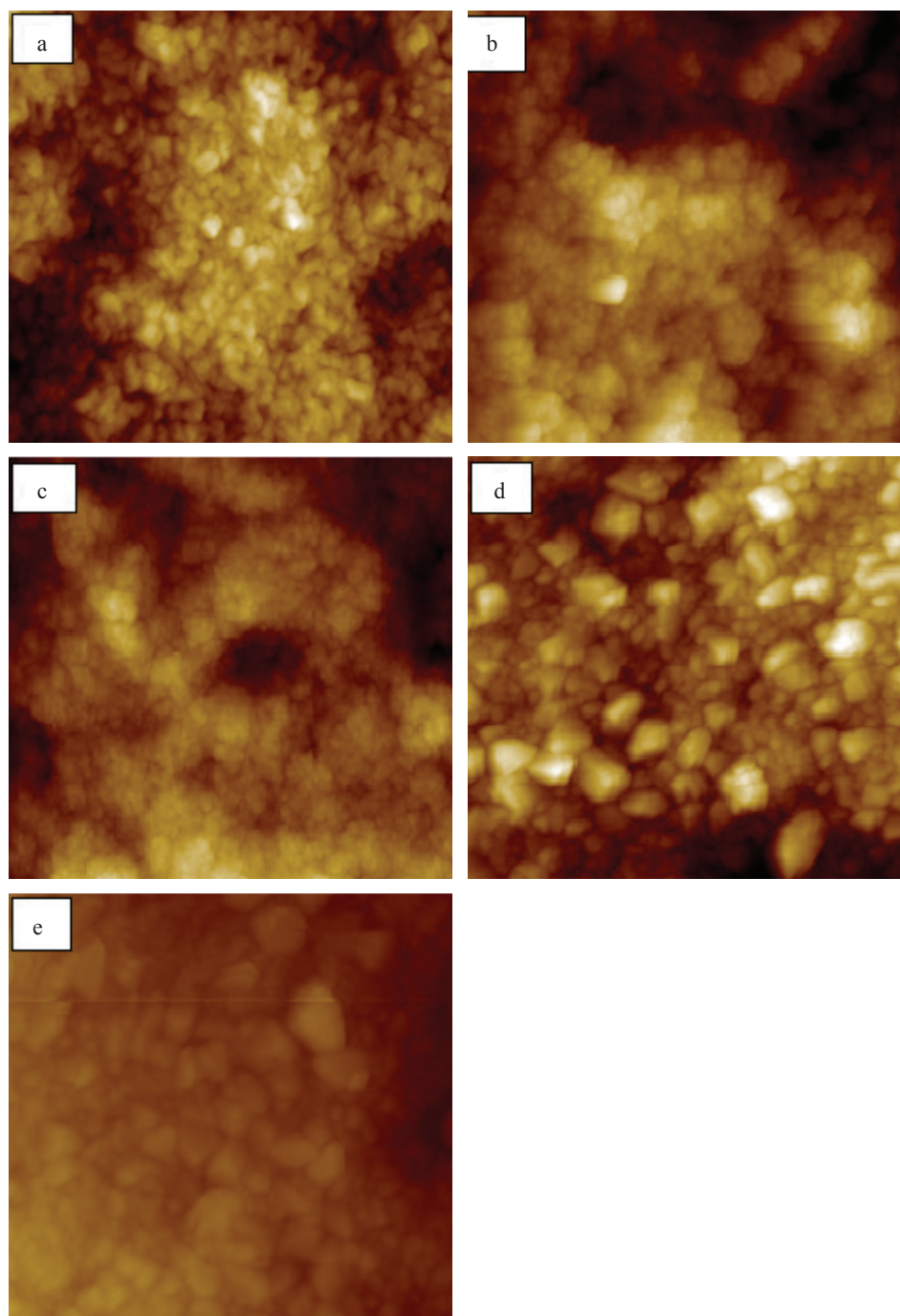


FIGURE 2. AFM images for the surface topography of nickel catalyst layer electroplated at different plating current densities a) 1 mA/cm<sup>2</sup> b) 5 mA/cm<sup>2</sup> c) 10 mA/cm<sup>2</sup> d) 20 mA/cm<sup>2</sup> and e) 30 mA/cm<sup>2</sup> (scan area of the images is 5 × 5 μm)

#### CARBON NANOSTRUCTURES (CNS) GROWTH

Figure 3 shows the SEM image of CNS growth on nickel catalyst layer electroplated at different current densities. The result shows that, all electroplated nickel catalysts were able to grow highly dense CNS arrays. The types of CNS and outer diameter of the CNS produced were determined using TEM, Figure 4 and Table 4 present the TEM results of different types and outer diameters of the CNS produced. The results show that, the nickel catalyst layer electroplated at lowest current density, 1 mA/cm<sup>2</sup>, tends to produce CNTs which have a hollow core structure. This

observation supports the findings of previous studies where the diameter of growing CNS is related to the size of catalyst nanoparticles (Hashempour et al. 2013; Nayeb Sadeghi et al. 2012) during the CNTs growth, lead to the production of CNTs decorated with about 6 nm Cu<sub>2</sub>O nanoparticles. We used SEM to study the surface characteristics of Ni catalyst films and characteristic of grown CNTs. Raman spectroscopy, transmission electron microscopy (TEM; an increased in the catalyst size will increase the diameter of CNS. This phenomenon is possibly due to the decomposition of acetylene gas on the surface of nickel

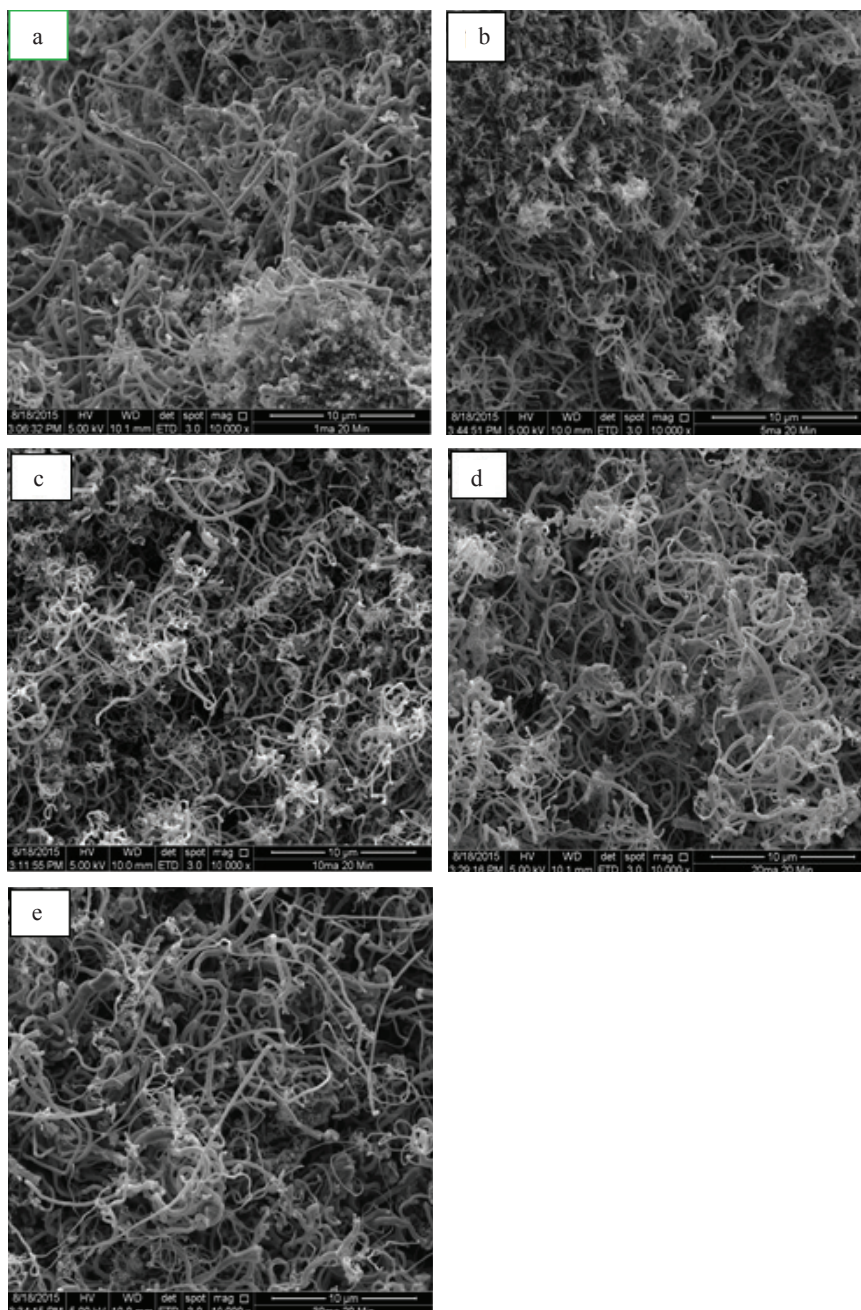


FIGURE 3. SEM images for CNS growth on nickel catalyst layer electroplated at a) 1 mA/cm<sup>2</sup> b) 5 mA/cm<sup>2</sup> c) 10 mA/cm<sup>2</sup> d) 20 mA/cm<sup>2</sup> and e) 30 mA/cm<sup>2</sup>

catalyst particles releasing hydrogen gas and carbon atoms, in which carbon atoms dissolved and diffused through the catalyst particles and finally precipitated to form the body of CNS filament. Besides, the carbon concentration gradient inside the nickel catalyst particles also allowed the CNS filaments to grow accordingly with the same diameter of the nickel catalyst particles (Teo et al. 2003). Moshkalyov et al. (2004) also relates the structure of the CNS growth with the thickness of the electroplated catalyst layer. A thinner nickel catalyst layer tends to produce long and straight CNTs due to the lower agglomeration tendency of thinner electroplated catalyst layer compare to thicker catalyst layer (Moshkalyov et al. 2004; Radhakrishnan et al. 2009). Meanwhile, the agglomeration of nickel catalyst

particles for thicker nickel catalyst layer electroplated at high current density contribute to the formation of larger diameter of nickel catalyst particles, inducing the growth of larger diameter CNS. Thus, it can be deduced that nickel catalyst layer electroplated at 1 mA/cm<sup>2</sup> in which contained smaller grain size and thinner nickel catalyst layer enable the growth of more CNS with smaller diameter as shown in Figure 4.

The overall quality and purity of CNS produced can be determined using TGA. A thermogravimetric (TG) curve and differential thermogravimetric (DTG) curve for carbon deposit synthesized by all electroplated nickel catalyst are presented in Figure 5. From Figure 5(a), the amount of residue for carbon deposit synthesized on nickel



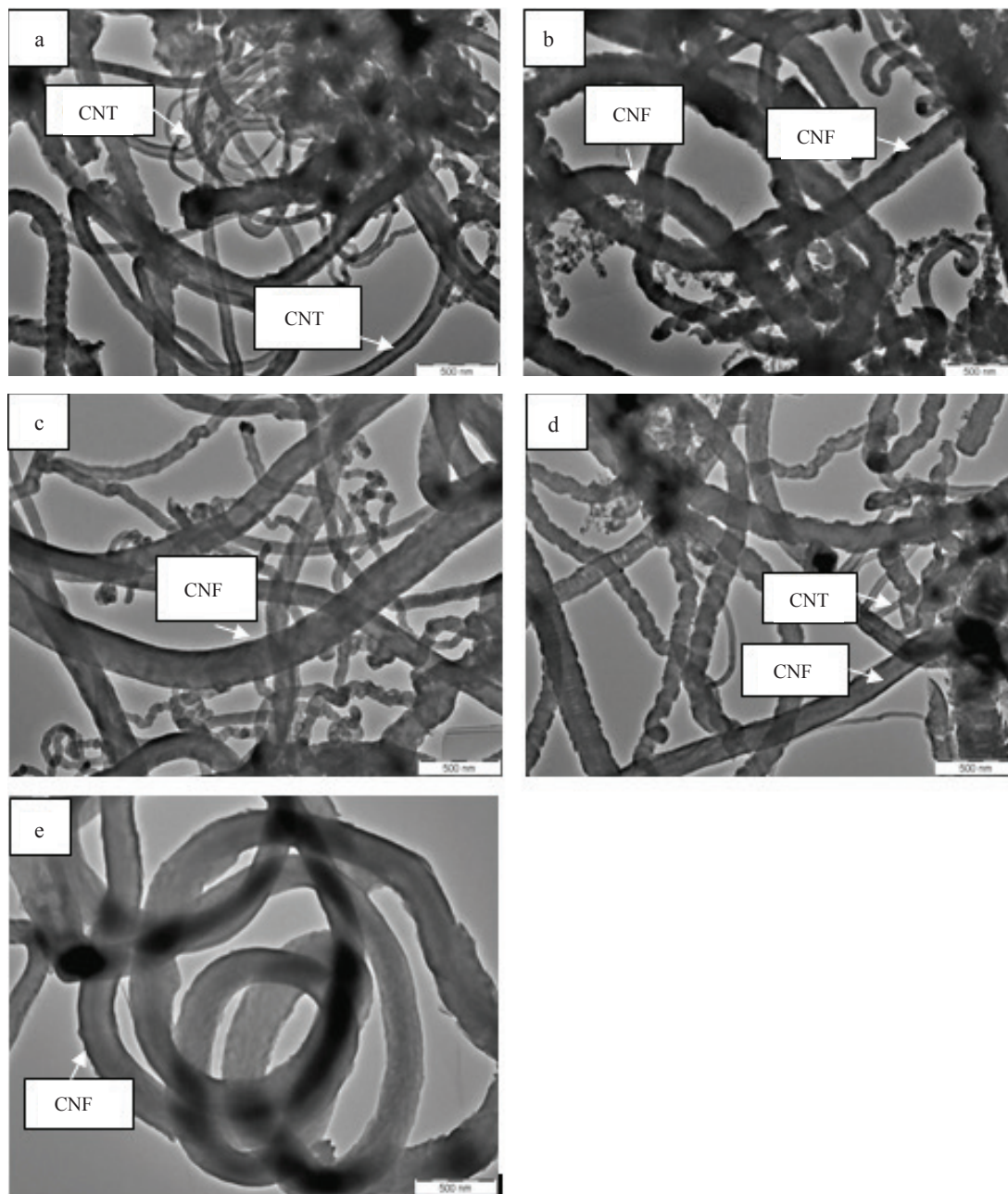


FIGURE 4. TEM images for carbon deposit on the nickel catalyst layer electroplated at different current density a) 1 mA/cm<sup>2</sup> b) 5 mA/cm<sup>2</sup> c) 10 mA/cm<sup>2</sup> d) 20 mA/cm<sup>2</sup> and e) 30 mA/cm<sup>2</sup>

TABLE 4. Range of outer diameter CNS produced on nickel catalyst layer electroplated on the copper substrate at different current density

Current density (mA/cm <sup>2</sup> )	Range of outer diameter (nm)	Standard deviation, (±nm)
1	38-103	30.35
5	71-232	45.81
10	68-258	50.48
20	41-206	61.18
30	206-310	69.37

catalyst layer electroplated at 1, 5, 10, 20 and 30 mA/cm<sup>2</sup> were 0.56, 1.02, 2.49, 2.48 and 3.93%, respectively, which equal to the percentage of purity of 99.44, 98.98, 97.51, 97.51 and 96.01%. The nickel catalyst layer electroplated at 1 mA/cm<sup>2</sup> produced the highest percentage of purity compared to other electroplated at higher current density. Meanwhile, DTG curve in Figure 5(b) helps to determine the degree of graphitization of the carbon deposit. The higher the inflection temperature and smaller difference between on-set and end temperature indicate the presence of highly graphitized CNS (Chen et al. 2006). From Figure 5(b), no significant peaks are observed at temperature between 300 and 400°C, which refer to the oxidation temperature of amorphous carbon

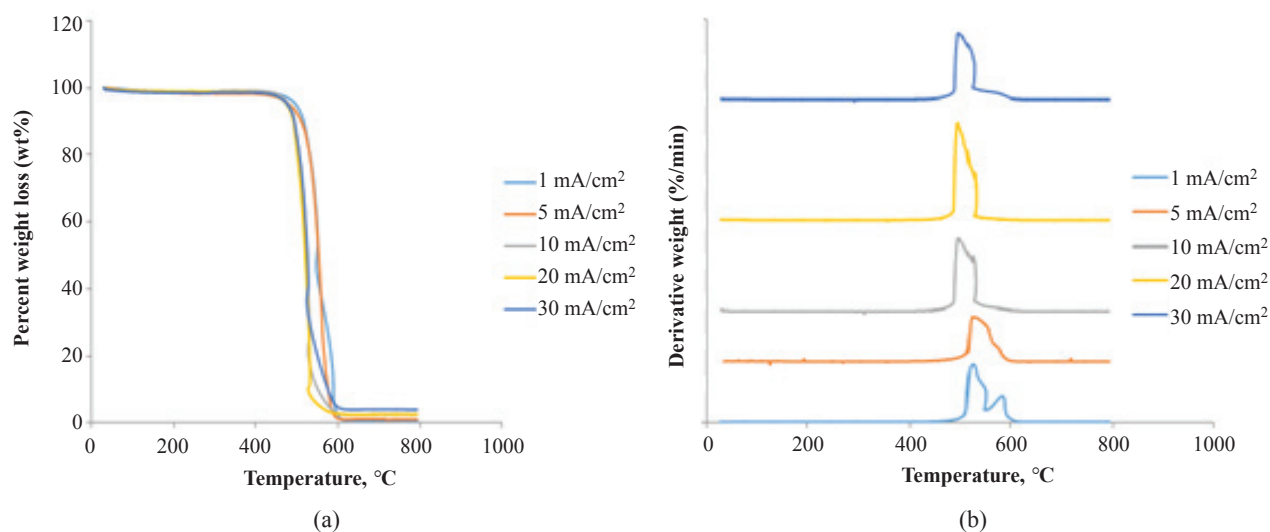


FIGURE 5. (a) TG-curve and (b) DTG curve of the carbon deposit growth on nickel catalyst layer electroplated at different current density

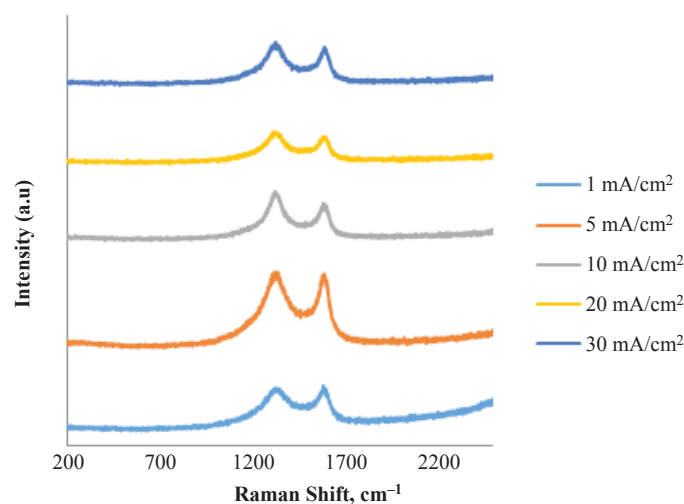


FIGURE 6. Raman spectrum of the carbon deposit growth on nickel catalyst layer electroplated at different current density

(Chen et al. 2006), indicating that the carbon deposits grown on the nickel catalyst layer electroplated for all current densities were mainly CNS and contains negligible amount of amorphous carbon. CNS grown on the nickel catalyst layer electroplated at 1 mA/cm<sup>2</sup> contained two peaks with the inflection point temperature of 528.03°C and 585.82°C. These two peaks show that there are two possible types of CNS, CNTs and CNFs where CNTs possess higher thermal stability oxidized at higher temperature, 585.82°C and CNFs oxidized at 528.03°C (Ludvig et al. 2011). Meanwhile there was only one significant peak exists for carbon deposit synthesized on nickel catalyst layer electroplated at other current densities referring to the oxidation peak of CNFs.

The quality of CNS produced on nickel catalyst layer electroplated at different current densities was further analyzed using Raman spectroscopy (Seah et al. 2013) as shown in Figure 6. Two major bands were observed, D-band (peak at 1350 cm<sup>-1</sup>) and G-band (peak at 1580

cm<sup>-1</sup>). D-band refers to the disordered graphite, wall disorder or fibers that are related to the level of disordered carbon and defects concentration whereas G-band refers to the degree of graphitization of CNS. Figure 6 shows the presence of D-peaks for CNS grown at all current densities in which possibly includes defects, amorphous and lower graphitized carbon. However, for characterization using TGA-DTG, oxidation of amorphous carbon due to the relatively higher reactivity of amorphous carbon with air as compared to CNS occurred in the temperature range of 300-400°C (Chen et al. 2006). From TGA-DTG result as shown in Figure 5(b), no peaks were present at this temperature range for all samples. Thus, by combining both TGA-DTG and Raman spectroscopy results, we speculated that the absent of amorphous carbon but the present of high amount of wall defects for all as-produced CNS samples. The overall quality of the CNS samples can be determined by the ratio of I<sub>D</sub> and I<sub>G</sub>. The lower I<sub>D</sub>/I<sub>G</sub> ratio, the less defect of the CNS produced. The I<sub>D</sub>/I<sub>G</sub>



ratios for current density of 1, 5, 10, 20 and 30 mA/cm<sup>2</sup> were 0.97, 1.03, 1.27, 1.13 and 1.10, respectively. The nickel catalyst layer electroplated at 1 mA/cm<sup>2</sup> produced relatively less defects of CNS as compared to other current density as indicated by the lowest ratio of  $I_D/I_G$  below 1.00. This is due to higher surface roughness and thinner nickel catalyst layer which reduce the agglomeration tendency of Ni particle, thus produced high purity and highly graphitized CNS (Moshkalyov et al. 2004). The agglomeration of nickel catalyst that formed very large catalyst particles diameter for growing CNS eventually caused the catalyst deactivation for the nucleation and growth of CNS and thus reduce the purity of CNS growth (Seidel et al. 2004).

#### CONCLUSION

This study showed that nickel catalyst prepared by the electroplating method using different current densities were able to grow CNS with high density. The results from this study showed that, nickel catalyst electroplated at lower current density, 1 mA/cm<sup>2</sup>, produced a thinner nickel catalyst layer with smaller grain size and higher surface roughness, which increased the efficiency of the nickel catalyst towards the growth of more CNS with smaller diameter, CNT. Besides, the CNS synthesized using nickel catalyst electroplated at lower current density were consists of high purity and high graphitization.

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