Modification of Multi-walled Carbon Nanotubes Surface to Increase Wettability Composite Aluminum Powder Matrix AA 7075

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Abstract - The copper coating process on multi-walled carbon nanotubes (MWCNT) has been carried out by electroless plating method to improve interfacial bonding between MWCNT and alumininium matrix. The initial step of electroless plating is to pre-treat MWCNT which includes oxidation (hydrophilic treatment), sensitization and activation. The oxidation process uses a solution of sulfuric acid and nitric acid, sensitization is carried out using tin (II) chloride dihydrate with hydrochloric acid, while the oxidation process uses a solution of palladium (II) chloride with hydrochloric acid. MWCNT which has been activated, is immersed in an electroless plating bath containing chemicals such as; Copper (II) sulfate pentahydrate, Potassium sodium tartrate tetrahydrate, Nickel (II) Chloride Hexahydrate, Polyethylene glycol 6000 and stirred with a magnetic stirrer at 200 rpm temperature of 25 ^{o}C. Deposition time is varied, ie 15, 30 and 60 minutes.

Test results with SEM-EDX show that the deposition time affects the copper content of the MWCNT. With a deposition time of 15, 30 and 60 minutes, copper content was 1.141 %, 3.227 % and 33.56 % by weight. The XRD test results showed that with 15 minutes deposition time, there was 97 % MWCNT and 3 % copper oxide compound. While cuprite compounds formed 61%, 16 % copper oxide compounds and 23% MWCNT at 30 minutes deposition time.

Keywords-- multi-walled carbon nanotubes, electroless plating, copper, deposition time.

1. INTRODUCTION

The use of Aluminum Matrix Composites (AMCs) has received much attention in the fields of aerospace, defense and automotive applications because it has a high ratio of strength to weight, increased stiffness, high temperature properties, controlled coefficient of thermal expansion, improved performance of electrical properties and adjusted, increased abrasion and wear resistance compared to monolithic alloys [1]. AMCs have been studied extensively using various types of amplifiers such as SiC, BC, Al2O3, TiB2 and graphite.

Today, carbon nanotubes (CNT) have been studied as reinforcements in metal matrix composites (MMC) for exceptional mechanical, thermal and electrical properties [2]. Researchers have sought to fabricate CNT-Al matrix composites, which can be used in sophisticated applications such as aircraft and automotive materials, and thinner electric cables with lower weights will save energy [3]. However, the fabrication of CNT-Al matrix composites faces two main problems for increasing the effective strength of composites. One of them is the CNT dispersion that is less uniform in the matrix, due to the strong CNT attachment caused by Van der Waals forces. Another problem is imperfect interface bonding in CNT / Al, which is caused by poor wettability between CNT and Al matrix.

Carbon nanotubes are known to have unique structures and properties such as length to diameter ratio, strength, flexibility and high thermal conductivity, etc. [4-6]. Several studies have been conducted to obtain potential carbon nanotube applications. Lately, studies on carbon nanotubes have focused on hydrogen storage [7-9], catalyst [10], etc. which carbon nanotubes are used as a substitute for good performance. Further studies have been conducted focusing on metal deposite or metal oxide nanoparticles on the surface of carbon nanotubes [11-12]. Carbon nanotubes deposite with smaller particles and good space show better performance by increasing the active catalyst surface area and further demonstrating high chemical and thermal stability on carbon nanotubes. On the other hand, the electrical and mechanical properties of carbon nanotubes can be changed or adjusted through coating with metal nanoparticles to obtain the desired properties. Therefore, deposition on carbon nanotubes has made an important discussion in this study.

In the past few decades, several methods have been used in CNT-Al composite fabrication, including; high-energy ball milling (HEBM), stir-processing friction, metallic flake, powder metallurgy, stir casting and nanoscale dispersion. However, most research has focused on the problem of CNT dispersion, which sometimes causes damage to CNTs or residual contamination in the matrix. Few studies have been conducted to study CNT / Al interfaces, which are essential keys to understanding the behavior of CNTs in CNT-Al matrix composites [13].

The ties and nature of the interfaces that exist between the amplifier and the aluminum matrix play an important role in the properties of AMCs [14]. The structural strength of composite materials depends largely on the chemical properties, atomic structure, and bonding on the interface, which transfers the mechanical load from the matrix to reinforcement through the interface [15]. Metal matrices should have good wettability with reinforcement material to avoid the formation of the thalamic cavity and ensure good adhesion to the interface to avoid delamination on the interface [16].

Poor wettability of non-metallic CNTs in molten metallic aluminum is caused by poor dispersion of CNTs and weak interfacial bonding, which is a major problem in CNT / Al composite fabrication. In fabricating CNT / Al composites, CNTs are not easily mixed with Al due to the large difference in surface tension between the two materials. Al surface tension is 955 mN.m-1 [17], while the surface tension of CNT is 45.3 mN.m-1. [18]. A technical obstacle to forming CNT / Al composites with high mechanical strength is the high oxidation ability of Al, which causes Al particles to be easily oxidized and lose their metallic characteristics. Because of these difficulties, the wettability of Al on the surface of the CNT is really difficult to realize.

The latest issues develop how to overcome large surface tension differences and increase Al wettability on the surface of the CNT, in a simple way in fabricating CNT-Al composites to obtain high mechanical strength. CNT surface coating with metallic coating is a good choice for forming a strong and resilient CNT / Al interface and the reaction products produced by intermetallic interphases are useful for improving interfacial properties. Various coating processes have been tried on CNTs using NiO [19], SiC[20], copper[21], Ni-P[22], and aluminum materials.[23]. All of these studies show better wettability and interfacial bonding is stronger between aluminum and coated-CNT matrices. But not all of these studies observed the contact angle formed between liquid and coated-CNT aluminum.

It is very difficult for most metals to deposite directly on the surface of carbon nanotubes. Therefore, a pre-treatment was carried out first to form functional groups such as carboxylic, carbonylic, and hydroxlic groups on the surface of carbon nanotubes preparing for metal nucleation or compounds. [24-25]. Characterization of carbon nanotubes revealed that the density of nanoparticles deposite on the surface of carbon nanotubes was low with this pre-treatment method. To increase the activity of carbon nanotube surfaces, Ang et al. [26], making the core catalytic with two-step sensitization-activation methods and a single-step activation approach. Activated carbon nanotubes are coated with copper and nickel with electroless plating methods. This pre-treatment, is very complicated and in sensitization or activation, $SnCl_2$ - $2H_2O$ used is harmful to people and the environment.

In this study, copper coating was carried out on the surface of the CNT using the electroless plating method, which aims to increase the wettability between the aluminum matrix and CNT. Copper is one of the transition metals that has strong carbon bonds [27]. Metal matrices tend to form more stable bonds with phase metal [28]. The electroless process of copper plating on the surface of the CNT is a good choice, because it is efficient and effective.

2. EXPERIMENTAL PROCEDURES

Multi Wall Carbon Nanotubes powder with size, OD: 10-20 nm, length: 10-30 m and purity > 98%, purchased from supplier Chengdu Organic Chemicals Co. Ltd., China as the main ingredient in this study. The processing method used for MWCNT coating with Cu is the electroless plating method. MWCNT coating increases the strength of the sample and wettability with the matrix. The electroless plating process of MWCNT with Cu was carried out at the Lab. University of Brawijaya Chemical Engineering.

Before the electroless plating process, pre-treatment of MWCNT included oxidation (hydrophilic treatment), sensitization and activation.

Oxidation: 1 gram MWCNT is mixed with 100 ml of H_2SO_4 and HNO_3 (1: 3) solution and maintained at 120 ° C for 10 hours (round magnetic stirrer at 100 rpm). MWCNT was rinsed with aquadest and separated from the media by centrifugation (centrifugation rotation at 4000 rpm for 15 minutes).

Sensitization and activation: like graphite, the surface of carbon nanotubes has low chemical reactivity and does not act as a catalyst for the deposition of the copper and no metal coating takes placel [29]

Sensitization: The oxidized MWCNT is mixed with 250 ml of a solution of 0.1 mol / L $SnCl_2$. $2H_2O$ - 0.1 mol / L HCl for 30 minutes by rotating the magnetic stirrer at 100 rpm at a temperature of 25 ^oC (the solution was prepared beforehand and 72 hours at 25 ^oC), followed by rinsing with distilled water and separated from the media by centrifugation (centrifugation rotation at 4000 rpm for 15 minutes).

Activation: MWCNT which has been pre-activated / sensitized was mixed with 250 ml solution of 0.0014 mol / L $PdCl_2$ - 0.25 mol / L HCl for 30 minutes by rotating the magnetic stirrer at 100 rpm at a temperature of 25 ^oC, followed by rinsing with distilled water and separated from the media by centrifugation (centrifugation rotation at 4000 rpm for 15 minutes).

Electroless plating: Activated MWCNT is inserted into an electroless plating tub which contains chemicals such as; Copper (II) pentahydrate sulfate ($CuSO_4$. $5H_2O$), Potassium sodium tartrate tetrahydrate ($KNaC_4H_4O_6$. $4H_2O$), Nickel (II) Chloride Hexahydrate ($NiCl_2$. $6H_2O$), Polyethylene glycol 6000. Volume of 250 ml solution and stirred with magnetic stirrer at 100 rpm temperature of 25 ^oC for 15, 30 and 60 minutes, followed by the addition of Formaldehyde solution (HCHO) gradually. The pH of solution 12 is regulated by the addition of Sodium hydroxide (NaOH). Furthermore MWCNT was washed and rinsed with aquadest and separated from the media by centrifugation (at 4000 rpm for 15 minutes), then dried at 105 ^oC.

To find out the molecular groups of the MWCNT samples which were oxidized were analyzed using Fourier Transform Infrared (FTIR) Spectrometer with Shimadzu Prestige 21 brand in the Lab. Central Malang State University. The surface morphology of MWCNT sample which was coated by Cu were analyzed by microscopy using electron microscope (SEM) coupled with energy dispersive X-ray spectroscopy (EDX), at the Lab. Bio Science UB and Lab. Metallurgical Mechanical Engineering UNUD with a SEM JEOL JSM-651OLA. X-ray diffraction (XRD) (Philips Panalytical X-pert, Model PW 3710) with Cu Ka radiations, was used to obtain the diffraction patterns in the Lab. Central Malang State University.

3. RESULTS AND DISCUSSION

A. Spectroscopic analysis

Based on the FTIR test results for pure MWCNT and oxidized MWCNT samples were analyzed using Fourier Transform Infrared (FTIR) Spectrometer of Shimadzu Prestige 21 brand in the Lab. Central Malang State University, obtained a graph as in Fig. 1.

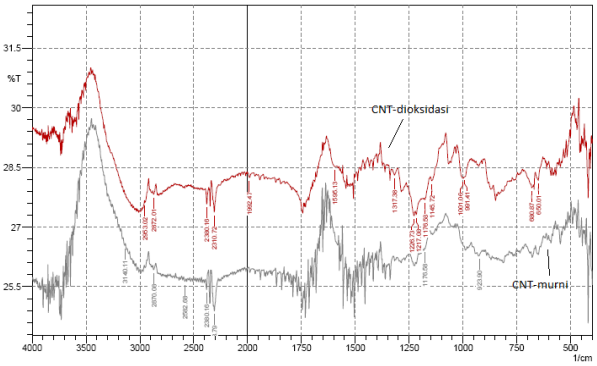


Fig-1 : FTIR test results of pure MWCNT and MWCNT oxidized

From Fig. 1, it can be explained that;

- A peak appears at wave number 2850-2970 cm^{-1} which is likely to indicate the presence of C-H Alkane groups which usually appear in wave numbers 2850-2970 and 1340-1470 cm^{-1}.
- A peak appears at wave number 1300-1370 cm^{-1} which is likely to indicate the presence of NO_2 group. Nitro compounds which usually appear in wave numbers 1500-1570 and 1300-1370 cm^{-1}.
- The peak at wave number 1180-1360 cm^{-1} appears on the bacterial cellulose CMC sample which is likely to indicate the presence of a C-N Amine / amide group which usually appears in the wave number.
- A peak appears at the wave number 1050-1300 cm^{-1} which is likely to indicate the presence of a C-O alcohol group / ether / carboxylic acid / ester which usually appears in the wave number.
- A peak appears at wave numbers 690-900 cm^{-1} which is likely to indicate the presence of a C-H group of aromatic rings which usually appear in wave numbers 690-900 and 3010-3100 cm^{-1}.
- A peak appears at wave number 675-995 cm^{-1} which is likely to indicate the presence of an Alkena C-H group which usually appears at wave numbers 675-995 and 3010-3095 cm^{-1}.

B. Microscopy analysis

The SEM test was first performed on pure MWCNT and oxidized MWCNT. What is done at the Lab. UB Bio Science and obtained the results as shown in Fig. 2 and Fig. 3. Next SEM-EDX test was carried out on the MWCNT sample which was Cu-

coated with a deposition time of 15 minutes. The results are shown in Fig. 4. The Cu content deposited in MWCNT was 1.141% by weight.

In Fig. 5 is the result of SEM-EDX test on the MWCNT sample which was coated by Cu with a deposition time of 30 minutes. The Cu content deposited in MWCNT was 3.227% by weight.

SEM-EDX test results Fig. 4 and 5 are carried out at UB's Science Lab. While the SEM-EDX test of the MWCNT sample which was Cu coated with 60 minutes deposition time, was carried out at the Lab. Mett Metallurgy Mechanical Engineering Unud. The results are shown in Fig. 6. The Cu content deposited on MWCNT is 33.56% by weight.

In the deposition process, the Cu particles from Copper (II) pentahydrate sulfate ($CuSO_4$. $5H_2O$) which are in the solution move and attach to the surface of MWCNT. The longer the deposition time, the more Cu particles will stick to the surface of MWCNT.

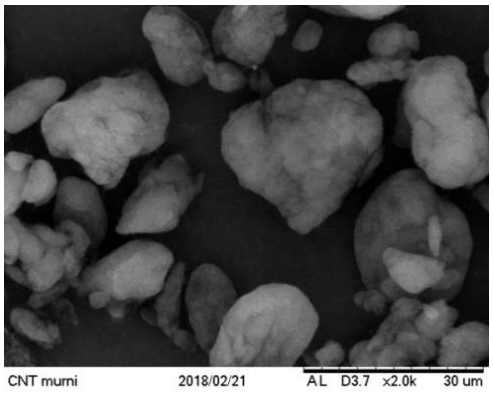


Fig - 2 : SEM test results of pure MWCNT

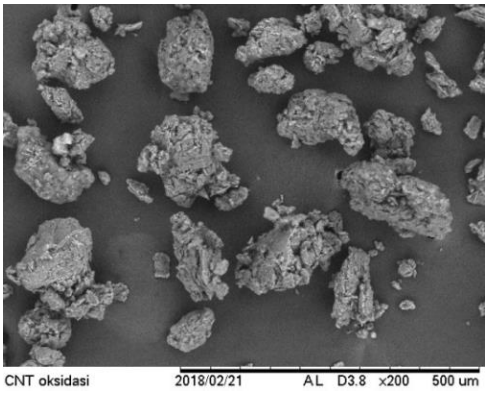
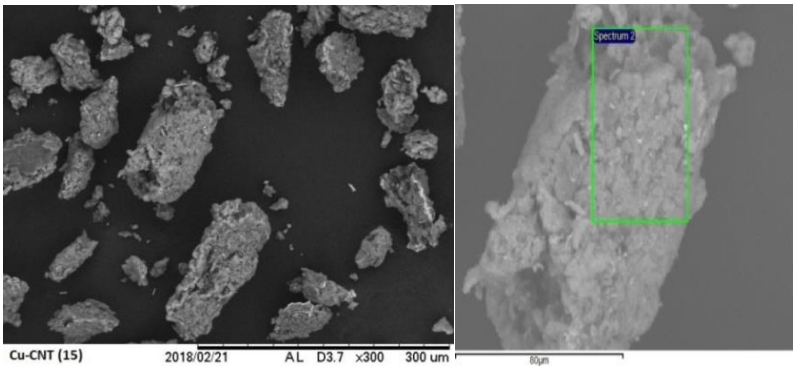


Fig - 3 : SEM test results of MWCNT oxidized



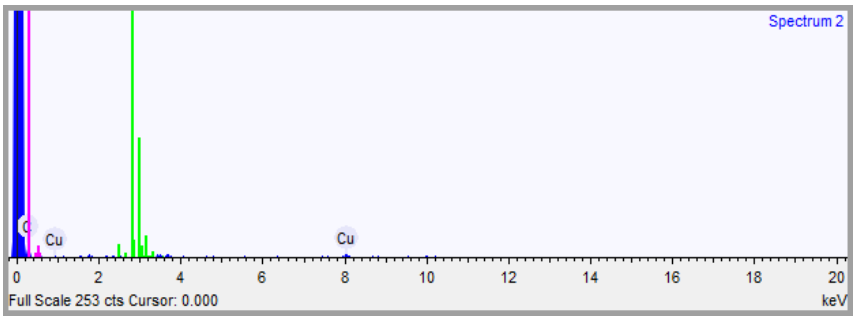


Fig - 4 : SEM / EDX test results of MWCNT coated Cu with 15 minutes deposition time

Table-1 : Composition of elements in MWCNT coated Cu with 15 minutes deposition time

Element	Weight %	Weight % σ	Atomic %
Carbon	98.859	1.107	99.782
Copper	1.141	1.107	0.218

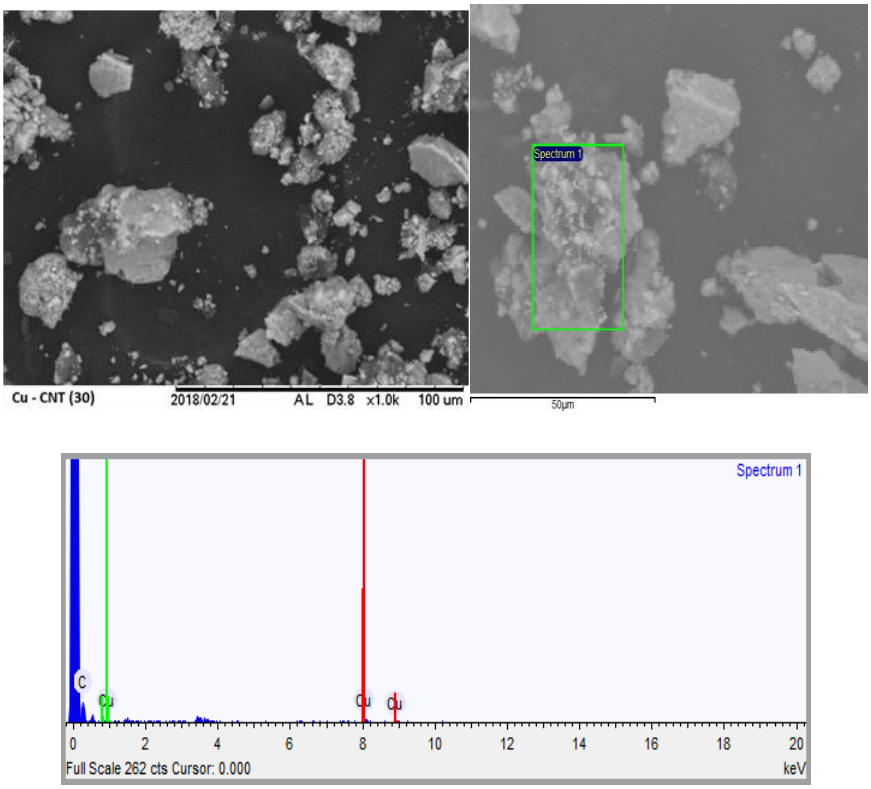


Fig - 5 : SEM / EDX test results of MWCNT coated Cu with 30 minutes deposition time

Table-2 : Composition of elements in MWCNT coated Cu with 30 minutes deposition time

Element	Weight %	Weight % σ	Atomic %
Carbon	96.773	1.860	99.374
Copper	3.227	1.860	0.626

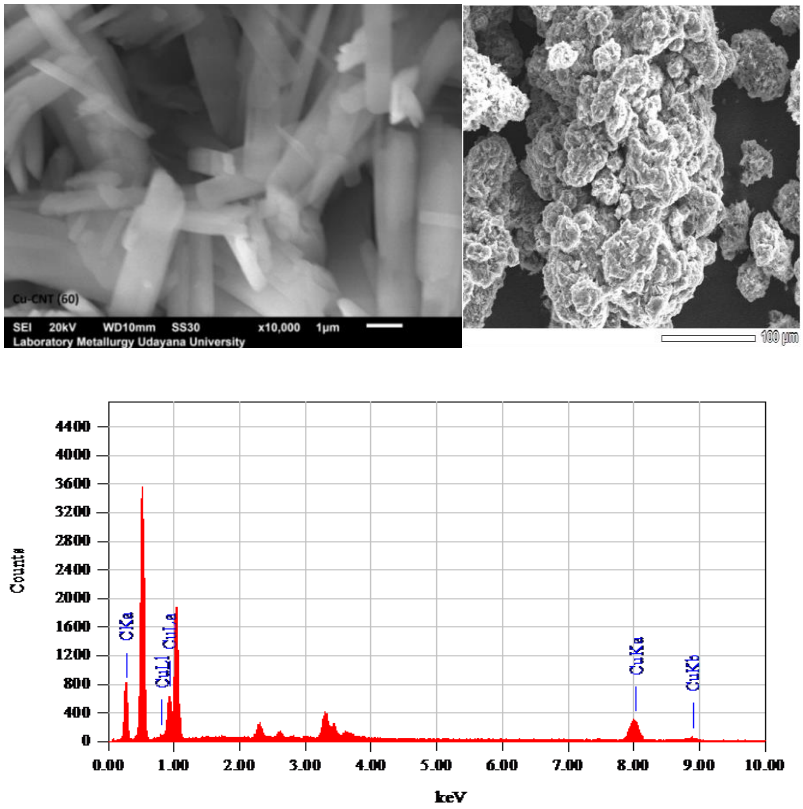


Fig - 6 : SEM / EDX test results of MWCNT coated Cu with 60 minutes deposition time

Table-3 : Composition of elements in MWCNT coated Cu with 60 minutes deposition time

Element	(keV)	Mass%	Sigma	Atom%	K
C K	0.277	66.44	0.54	91.28	39.5256
Cu K	8.040	33.56	0.77	8.72	60.4744
Total		100.00		100.00	

C. XRD analysis

Based on data analysis using X'Pert HighScore software, the following data is obtained:

1). The pure MWCNT

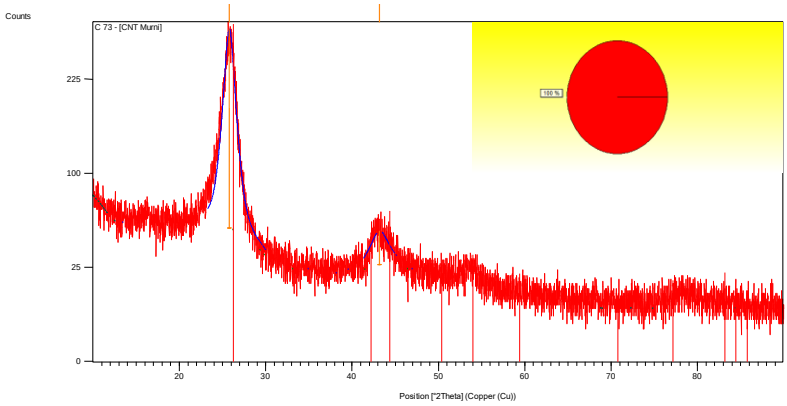


Fig - 7: Diffraction patterns of pure MWCNT from X'Pert HighScore analysis

HighScore X'Pert analysis results show that there is 100% content of C (Reference code: 01-075-1621). Identification of the plotted phase at start position [° 2Th.] = 10,0100 and end position [° 2Th.] = 89,9900, shown in Fig. 8.

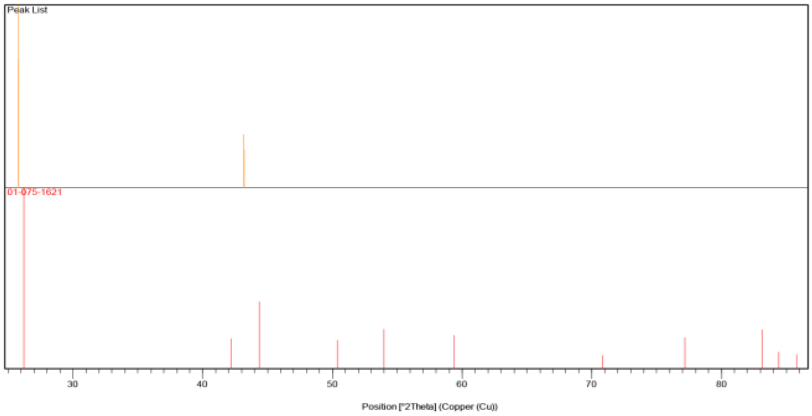


Fig - 8 : Phase identification of pure MWCNT results from X'Pert HighScore analysis

Table-4 : Diffraction peaks of pure MWCNT

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip width [°2Th.]	Matched by
25.7737	260.96	1.6371	3.45385	100.00	1.9645	01-075-1621
25.8389	130.48	1.6371	3.45385	50.00	1.9645	
43.1387	22.33	2.4951	2.09533	8.56	2.9941	01-075-1621
43.2513	11.17	2.4951	2.09533	4.28	2.9941	

2). The MWCNT coated Cu with 15 minutes deposition time

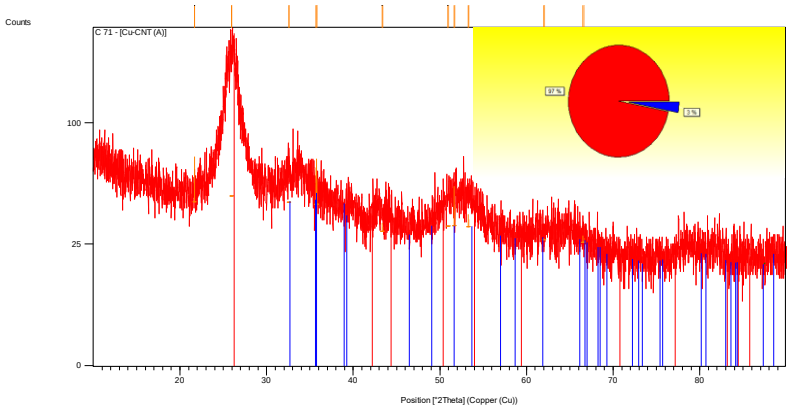


Fig - 9 : Diffraction pattern of MWCNT coated Cu with 15 minutes deposition time results from X'Pert HighScore analysis

HighScore X'Pert analysis results show that there are 97% C (Reference code: 01-075-1621) and 3% CuO / Copper Oxide content (Reference code: 01-074-1021). Identification of the phase is plotted at start position [° 2Th.] = 10,0100 and end position [° 2Th.] = 89,9900, shown in Fig. 10

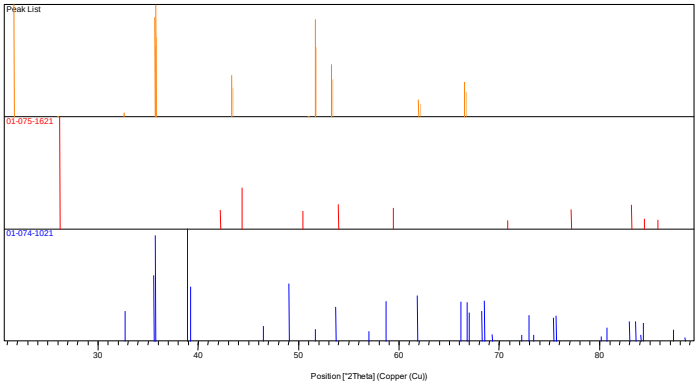


Fig - 10 : Phase identification of MWCNT coated Cu with 15 minutes deposition time results from X'Pert HighScore analysis

Table-5 : Diffraction peaks of MWCNT coated Cu with 15 minutes deposition time

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip width [°2Th.]	Matched by
21.6857	28.53	0.1055	4.09821	100.00	0.1265	
25.9700	0.00	4.0000	3.42818	0.00	4.8000	01-075-1621
26.0357	0.00	4.0000	3.42818	0.00	4.8000	
32.5700	0.04	0.0010	2.74699	0.15	0.0012	01-074-1021
32.6532	0.02	0.0010	2.74699	0.07	0.0012	
35.6744	22.44	0.4299	2.51474	78.66	0.5159	01-074-1021
35.7660	11.22	0.4299	2.51474	39.33	0.5159	
35.7781	28.44	0.0593	2.50769	99.70	0.0711	01-074-1021
35.8700	14.22	0.0593	2.50769	49.85	0.0711	
43.3300	3.90	0.0900	2.08652	13.67	0.1080	
43.4431	1.95	0.0900	2.08652	6.83	0.1080	
50.9300	0.00	0.0010	1.79156	0.00	0.0012	
51.0656	0.00	0.0010	1.79156	0.00	0.0012	
51.6280	21.53	0.0687	1.76897	75.46	0.0825	01-074-1021
51.7657	10.76	0.0687	1.76897	37.73	0.0825	
53.2500	6.23	0.0900	1.71885	21.85	0.1080	01-074-1021
53.3928	3.12	0.0900	1.71885	10.92	0.1080	
61.9700	0.66	0.0900	1.49627	2.32	0.1080	01-074-1021
62.1411	0.33	0.0900	1.49627	1.16	0.1080	
66.5300	2.72	0.0900	1.40434	9.55	0.1080	01-074-1021
66.7169	1.36	0.0900	1.40434	4.78	0.1080	

3). The MWCNT coated Cu with 30 minutes deposition time

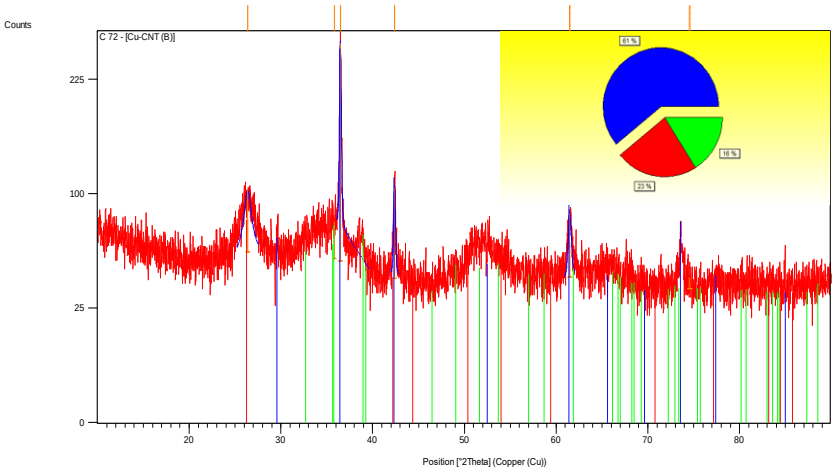


Fig - 11 : Diffraction pattern of MWCNT coated Cu with 30 minute deposition time results from X'Pert HighScore analysis

HighScore X'Pert analysis results show that there is a content of C (Reference code: 01-075-1621) of 21%, CuO / Copper Oxide (Reference code: 01-074-1021) of 16% and Cu2O / Cuprite (Reference code: 01 -078-2076) 61%. Identification of the plotted phase at start position [° 2Th.] = 10,0100 and end position [° 2Th.] = 89,9900, shown in Fig. 11.

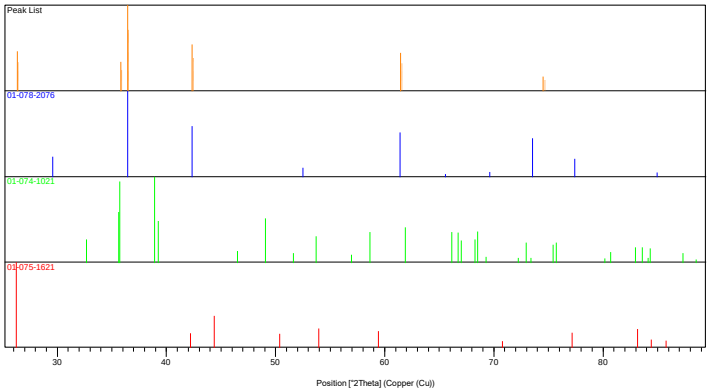


Fig - 12 : Phase identification of MWCNT coated Cu with 30 minutes deposition time results from X'Pert HighScore analysis

Table-6 : Diffraction peaks of MWCNT coated Cu with 30 minutes deposition time

Pos. [°2Th.]	Height [cts]	FWHM [°2Th.]	d-spacing [Å]	Rel. Int. [%]	Tip width [°2Th.]	Matched by
26.3528	47.32	0.9567	3.37925	21.11	1.1480	01-075-1621
26.4195	23.66	0.9567	3.37925	10.55	1.1480	
35.8118	25.87	4.0000	2.50540	11.54	4.8000	01-078-2076; 01-074-1021
35.9039	12.93	4.0000	2.50540	5.77	4.8000	
36.4662	224.19	0.1695	2.46194	100.00	0.2034	01-078-2076; 01-074-1021
36.5600	112.10	0.1695	2.46194	50.00	0.2034	
42.3570	66.02	0.2010	2.13217	29.45	0.2412	01-078-2076; 01-075-1621
42.4674	33.01	0.2010	2.13217	14.72	0.2412	
61.4336	43.32	0.3798	1.50804	19.32	0.4557	01-078-2076; 01-074-1021
61.6029	21.66	0.3798	1.50804	9.66	0.4557	
74.4965	6.13	4.0000	1.27265	2.74	4.8000	01-078-2076; 01-074-1021
74.7131	3.07	4.0000	1.27265	1.37	4.8000	

4. CONCLUSIONS

In the MWCNT coating process with Cu using electroless plating method, with varying deposition times, it shows that increasing deposition time causes the Cu content in MWCNT to increase. From the experimental results, it was found that copper content was 1.141%, 3.227% and 33.56% by weight in MWMWCNT for 15, 30, and 60 minutes deposition time.

The XRD test results showed that with 15 minutes deposition time, there was 97 % MWCNT and 3 % copper oxide compound. While cuprite compounds formed 61%, 16 % copper oxide compounds and 23% MWCNT at 30 minutes deposition time.

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