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Effect of Copper Coated Multiwalled Carbon Nanotubes on Dispersion and Wettability in Fine Aluminum Matrix

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Abstract - *Dispersion and wettability problems are the main obstacles to obtain good metal-CNTs composites due to the attraction of Van der Waals forces in carbon nanotubes. Attempts to produce Al-CNTs composites by melt stirring method (stir casting) were not satisfactory due to poor wettability of the Al-CNTs system, due to high Al viscosity, too low CNTs density and strong CNTs agglomeration.*

Efforts to increase the wettability of metal matrix composites have been carried out using various methods, namely; coating the matrix or its reinforcement, adding alloying elements to the matrix alloy, and treating the matrix. However, in principle, the main concept to increase wettability is to increase the surface energy of the solid, decrease the surface tension of the liquid alloy or decrease the interfacial energy of the solid-liquid at the particle-matrix interface.

This study aims to address the problem of dispersion and wettability of multiwalled carbon nanotubes in aluminum matrix processed by the liquid state processing (stir casting) method. The research procedure was carried out in several stages, namely the first stage of coating on multiwalled carbon nanotubes, the second stage of carrying out a wettability test, and the third stage of manufacturing Cu/MWCNTs-Al composites with induction heating.

The results showed that by coating the multiwalled carbon nanotubes using copper, it increased the dispersion of the multiwalled carbon nanotubes in the aluminum fine matrix. Likewise, the wettability between multiwalled carbon nanotubes and aluminum fines increases, due to the occurrence of greater adhesion forces when multiwalled carbon nanotubes are coated with copper.

Key words: *multiwalled carbon nanotubes, coating, dispersion, wettability, aluminum fine*

1. INTRODUCTION

In the last few decades, several methods have been used in the fabrication of CNTs-Al composites, including; high-energy ball milling (HEBM) [1], friction stir-processing [2], flake metallurgy [3], powder metallurgy, stir casting and nanoscale dispersion [4]. However, most of the research has focused on the problem of CNTs dispersion, which sometimes causes damage to the CNTs or residual contamination in the matrix. Little research has been done to study the CNTs/Al interface, which is an essential key to understanding the behavior of CNTs in CNTs-Al matrix composites [5].

The bonding and interface properties that exist between the reinforcement and the aluminum matrix play an important role in the properties of AMCs [6]. The structural strength of composite materials largely depends on the chemical properties, atomic structure, and bonding at the interface, where the transfer of mechanical loads from the matrix to the reinforcement occurs through the interface [7]. The metal matrix should have good wettability with the reinforcement material to avoid the formation of micro-scale cavities and ensure good adhesion at the interface to avoid delamination at the interface [8].

In the fabrication of CNTs/Al composites, CNTs is not easily mixed with Al due to the large difference in surface tension between the two materials. The surface tension of Al is 955 $mN.m^{-1}$ [9], while the surface tension of CNTs is 45.3 $mN.m^{-1}$ [10]. A technical obstacle to forming CNTs/Al composites with high mechanical strength is the high oxidizing ability of Al, which causes Al particles to easily oxidize and lose their metallic characteristics. Due to these difficulties, the wettability of Al on the CNTs surface is extremely difficult to achieve.

A simple method in fabricating CNTs-Al composites to obtain high mechanical strength is to overcome large surface tension differences and increase the wettability of Al on the CNT surface. Coating the CNTs surface with a metallic layer is a good choice to form a strong and ductile CNTs/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, SiC, copper, Ni-P, and aluminum materials [11]. All these studies showed better wettability and stronger interfacial bonding between aluminum matrix and coated-CNTs. However, not all of these studies observed the contact angle formed between liquid aluminum and coated-CNT.

In this study, the CNTs surface was coated with copper using the electroless plating method, which aims to increase the wettability between the aluminum matrix and the CNTs. Copper is a transition metal that has a strong bond with carbon [12]. The metal matrix tends to form a more stable bond with the metal phase [13]. The process of electroless plating copper on the CNT surface is a good choice, because it is efficient and effective.

2. MATERIALS AND METHODS

2.1 Materials

The multiwalled carbon nanotubes were supplied from Chengdu Organic Chemicals Co. Ltd., China (OD: 10 - 20 nm, length: 10 - 30 ⍰m and purity > 98%) was used in this study. Colloidal palladium-tin catalyst was prepared with the composition of 0.5 g of palladium chloride ($PdCl_2$), 50 ml of 37% hydrochloric acid (HCl), 200 ml of deionized water, 25 g of stannous chloride. Cupric Sulphate Pentahydrate (98.5% Assay) and Sodium Carbonate Anhydrous (99.5% Assay) were supplied from Bofa Laboratotium. Sodium Hydroxide (99% Assay) was supplied from Bofa Laboratotium. Potassium Sodium Tartrate Tetrahydrate otherwise known as Rochelle salt (99% Assay) is supplied from Bofa Laboratotium. Cobalt (II) Chloride Hexahydrate (99% Assay) was supplied from Bofa Laboratotium. Formaldehyde 37% was supplied from Bofa Laboratotium.

2.2 Methods

The process of coating the surface of MWCNTs with copper is carried out in three steps. Starting with the surface activation process of MWCNTs using Pd-Sn colloidal particles. The next step is the acceleration process to remove stanno hydroxide deposits on the surface of the activated MWCNTs. The last process, electroplating of Cu-Co on the surface of the catalyzed MWCNTs. The above procedure globally is shown in Figure 1.

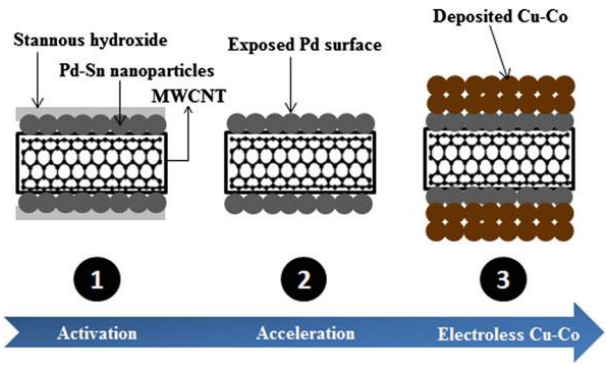


Figure -1: General schematic of Cu-Co electroless plating process on MWCNTs

2.2.1 Activation of MWCNTs in Pd-Sn colloid solution

The number of MWCNTs used in this process is 0.1 grams of MWCNTs. MWCNTs were activated using a Pd-Sn colloidal solution adapted from the Plating on Plastics (POP) industry. The activation process was carried out in a mixture of 37.5 ml of Pd-Sn colloid solution, 37.5 ml of HCl (37%), and 175 ml of DI water. When using, mix 15% palladium-tin colloidal catalyst solution and 15% hydrochloric acid (37%) together, and balance with deionized water, then heated to 50 - 60 °C to get a better catalytic effect.

For activation, MWCNTs were dispersed with a magnetic stirrer in colloidal solution for 30 minutes. After the stirring was completed, the treated MWCNTs were filtered using a 0.22 ⍰m PTFE filter membrane on the microfiltration kit. The filtered MWCNTs were re-dispersed in DI water and filtered again to remove excess colloidal particles and residual colloid solution from the activated MWCNTs. After filtration, MWCNTs were collected from the membrane using tweezers.

2.2.2 MWCNTs acceleration in acid mixture

The activated MWCNTs are then introduced into a mixed acid solution known as an accelerator. The accelerator serves to remove excess tin hydroxide on the surface of the catalytic particles in the MWCNTs allowing the palladium surface to be exposed. The acceleration process will not remove lead from the core of colloidal particles [14]. The acceleration process uses 55% (50 mL) HF acid in 500 mL of DI water.

After acceleration, MWCNTs were redispersed in water and filtered again to remove the acid film. Following the previous step, the surface of the MWCNTs became catalytic.

2.2.3 Electroless Plating Cu-Co on MWCNTs

The catalyzed MWCNTs were put into a 1 liter solution of Cu-Co electrolyte with concentrations as shown in Table 1.

Table -1: Composition of Cu-Co electrolyte solution [15]

Copper-Cobalt electrolyte	Concentrations
$CuSO_4.6H_2O$	6.99 g/L
Na_2CO_3	2 g/L
$CoCl_2.6H_2O$	1.09 g/L
$KNaC_4H_4O_6{\cdot}4H_2O$ (Rochelle Salt)	22.57 g/L
NaOH	4.5 g/L
Formaldehyde 37%	6 ml/L

All precursor powders were dissolved in DI water and stirred using a magnetic stirrer for 5 minutes. After making sure all the powder is dissolved in the solution, formaldehyde is added to the solution. Subsequently, the activated MWCNTs were placed in an electroless bath under a magnetic stirrer for 10 min and the reaction started on the catalytic surface of the Pd-coated MWCNTs. Air bubbles began to emerge from the solution after the MWCNTs were added. This occurs due to the dissolution of hydrogen from the palladium surface and the oxidation of formaldehyde which produces hydrogen. Then the solution in a glass beaker was stirred using a magnetic stirrer for 30 minutes. When the air bubbles stop, it gives a good indication that the copper has completely covered the entire surface of the catalyst. In this case the copper surface became auto-catalyzed and the solution turned

dark brown indicating the coverage of MWCNTs by copper. After stirring is complete, the copper-coated MWCNTs begin to accumulate on the bottom of the glass due to their increased density. Then the solution was filtered using a 0.22 m PTFE filter membrane. The color of the filtered solution appears to be a light pink color indicating the consumption of all copper ions in the solution prior to filtration. The color of the copper-coated MWCNTs powder obtained, is shown in Figure 2.

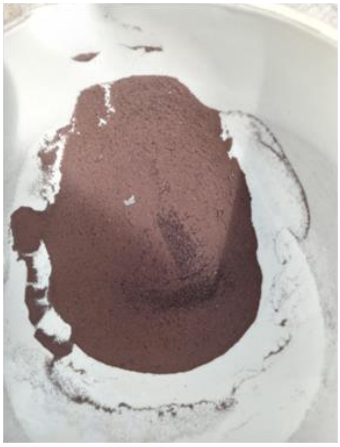


Figure-2: Copper coated MWCNT's brown

The characterization of copper-coated MWCNTs was carried out at the Mechanical Engineering Materials Laboratory of Udayana University using scanning electron microscopy (SEM) analysis using (JEOL-JSM 6510 A).

2.2.4 Wettability testing

As the first step in the wettability test, the manufacture of Cu/MWCNTs pellets and Al specimens. Cu/MWCNTs pellets are made with sizes ; diameter of 10 mm and thickness of 3 mm using a metal mold and press, with a press pressure of 500 MPa at room temperature. Meanwhile, cylindrical Aluminum pellets with a size of ∅2 mm x 2 mm are made by a casting process. The surface of the aluminum pellet is polished prior to testing to reduce surface oxidation. Polishing was carried out using a diamond abrasive (6 μm) followed by cleaning with isopropanol [16].

Wettability test using the sessile drop method was carried out in the Lab. Mechanical Engineering Mechanical Engineering Bali State Polytechnic, with the procedure;

- A cylindrical fine aluminum pellet with a size of ∅ 2 mm x 2 mm was placed on a Cu/MWCNTs pellet, and placed in an induction coil and heated to a temperature of 700 ^{o}C for 10 minutes.
- The heater is turned off until the working temperature reaches room temperature.
- Taking photos of the test samples with a digital camera at room temperature.

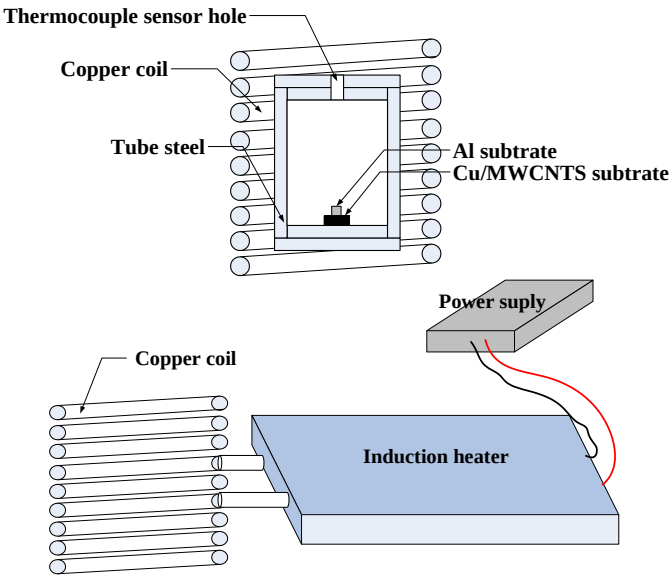


Figure-3: Schematic of wettability test equipment

The results of the wettability test (contact angle) were analyzed using an image processing program and the interface formed between Al and MWCNTs was analyzed using SEM-EDX.

2.2.5 Manufacturing Process of Al-MWCNTs Composite

Materials for composites, aluminum and multiwalled carbon nanotubes of varying composition were included in the smelting kowi. Heated to a temperature of 700 ^{o}C with a time of 10 minutes and a stirrer speed of 200 rpm. The composite melt is poured into a cylindrical metal (steel) mold at room temperature. The equipment used in this stage is shown in Figure 4.

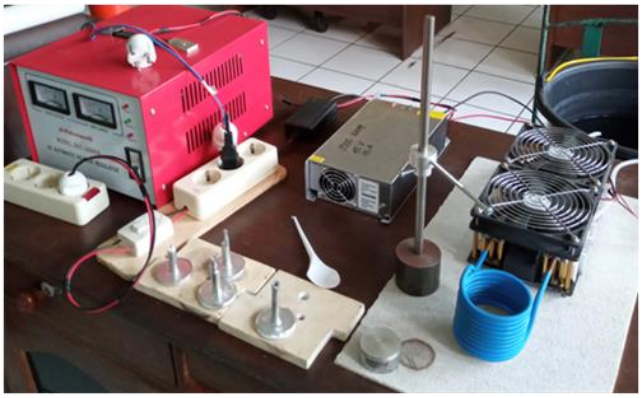


Figure-4: Equipment system design for composite fabrication

3. RESULTS AND DISCUSSION

3.1 Characteristics of copper coated MWCNTs

The addition of cobalt(II) chloride to the electroless copper solution assists in the autocatalytic reduction of copper ions in the electroless solution increasing the deposition rate remarkably. The use of colloidal Pd-Sn nanoparticle catalytic system limits the catalyst optimization process to two factors (colloidal particle concentration and solution volume relative to the number of MWCNTs) rather than four factors in the predecessor system. In addition, the new system provides a fixed average size of the colloidal nanoparticles for a better fit of the layer over the MWCNTs. The optimized catalyst concentration and volume required to cover the surface area of a fixed number of MWCNTs helps in controlling the catalyst concentration-dependent initial copper deposition rate.

SEM test results in Fig. 5 shows MWCNTs after being coated with copper. The increase in the thickness of the MWCNTs indicates the presence of copper on the surface of the MWCNTs. The mean diameter of the copper-clad MWCNTs was found to be 85 nm compared to the diameter of the initial MWCNTs, averaging 10–20 nm. The SEM images show a uniform layer that completely covers all the surfaces of the MWCNTs.

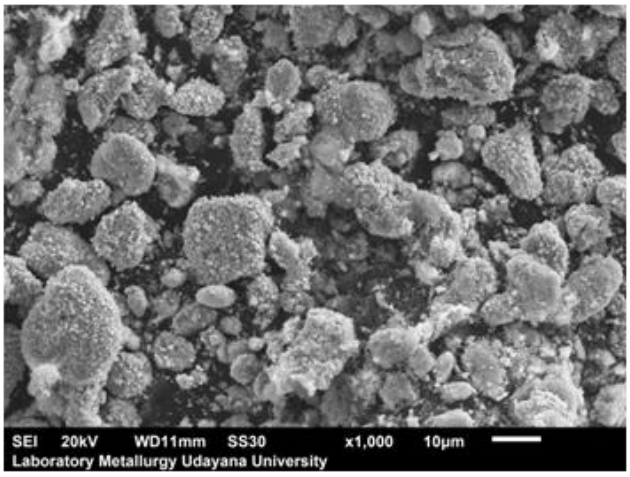


Figure-5: Copper coated MWCNTs from SEM test results

To determine the percentage of the elements present, chemical analysis was carried out using energy dispersive X-ray (EDX). The spectrum obtained is shown in Figure 6.

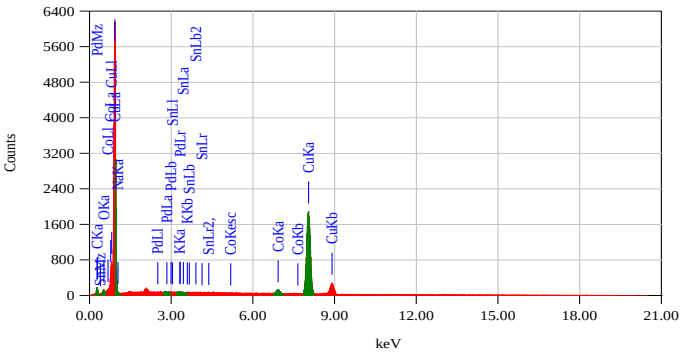


Figure-6: EDX Spectrum of copper coated MWCNTs

The results of the analysis showed that the weight of copper was 84.10 % and 12.08% C . Other elements such as Na, K, Co and Sn were found in minimal percentages as listed in Table 2.

Table -2: EDX Analysis of copper coated MWCNTs ZAF Method Standardless Quantitative Analysis(Oxide) Fitting Coefficient : 0.0326 Total Oxide : 24.0

Element	(keV)	Mass %	Sigma	Mol%	Compound	Mass %	Cation	K
C K	0.277	12.08	0.24	47.47	C	12.08	0.00	2.9938
O		17.81						
Na K	1.041	1.30	0.12	1.34	Na_2O	1.76	1.22	0.6077
K K	3.312	0.05	0.03	0.03	K_2O	0.07	0.03	0.0754
Co K	6.924	1.53	0.09	1.23	CoO	1.95	0.56	2.7533
Cu K	8.04	67.19	0.71	49.92	CuO	84.10	22.8	93.52

	0						0	4
Pd L								
Sn L	3.442	0.04	0.07	0.01	SnO_2	0.05	0.01	0.0458
Total		100.00		100.00		100.00	24.62	

The process of coating MWCNTs with copper produces different amounts of elements, compared to the results of previous studies [16]. This is influenced by the type of catalyst, the type of accelerator, and the concentration of the solution used when immersing the MWCNTs. In this study, the SEM-EDX results showed that there was no Pd in the copper coated MWCNTs powder. The use of a commercial Pd-Sn Colloidal Solution catalyst from Dupont gave a better effect, obtained elemental content of Cu 98.56% and Pd 0.43% [17]

3.2 Wettability test results of Al-Cu/MWCNTs system

Figure 7 shows an optical image after the sessile drop, showing the contact angle between the Al drop and the copper-coated MWCNTs substrate. To increase the level of confidence, images were taken at various positions, to characterize the contact angle, shown in table 3.

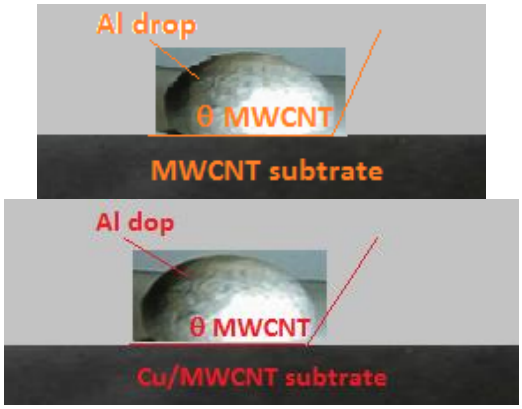


Figure-7: Wettabilty test results with the sessile drop method

From table 3, the case of wetting Al on the MWCNTs substrate, the average contact angle, θ_{MWCNT} = 152 °. However, the average contact angle of Al on the copper-coated MWCNTs substrate was reduced to 120 °. The work of adhesion between the liquid and the substrate, expressed in terms of the Young-Dupre equation, is as follows:

$$W_A = \gamma_{LV} (1+ \cos \theta)$$

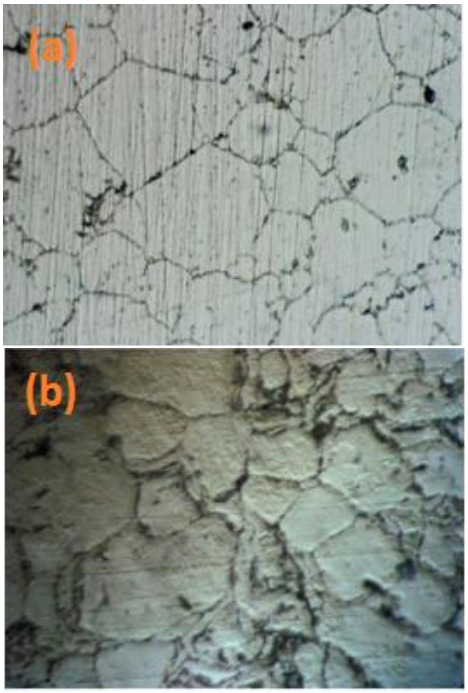
Where, is the contact angle and γ_{LV} = 850 mJ/m² is the liquid-pavor surface energy. The adhesion action of Al-MWCNTs and Al-Cu/MWCNTs based on calculations was 99 and 421 mJ/m². Therefore, it is expected that a strong interfacial bond will occur with the formation of an intermetallic $CuAl_2$ phase which results in increased load transfer from the Al matrix to MWCNTs.

Table-3: Contact angles measured at various positions after sessile drop aluminum at 700 °C

Direction of view	Front	Rear	Right	Left	Average	Adhesion work (mJ/m²)
θ_{MWCNT} (°)	152,9	149,2	154,8	151,5	152,1	99
$\theta_{Cu/MWCNT}$ (°)	123,4	118,3	122,7	116,8	120,3	421

3.3 Microstructure of Al-Cu/MWCNTs Composite

In the sample casting Al - Cu/MWCNTs composite, the grain morphology of Al did not change much compared to Al fine, as shown in Fig. 8. This is because gravity casting is applied during the formation of the composite, which is beneficial to the plasticizing of the powder to achieve full density. Grain boundaries are seen more clearly after repeated etching. In the pure Al samples, small grain growth (Figs. 8a) and equiaxed grains were observed in all composites (Figs. 8b and 8c).



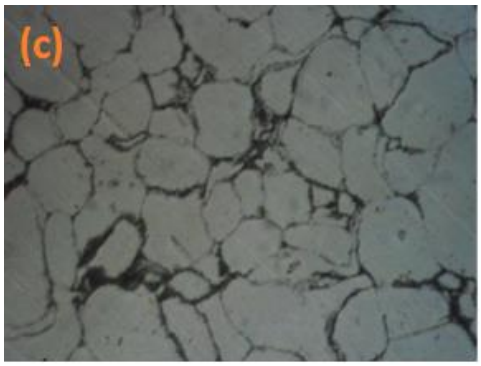


Figure-8: Microstructure image etched (a) Al fine, 1.0 wt.% (b) MWCNTs/Al uncoated composite and (c) Cu-coated MWCNTs/Al composite

In the composite sample, the MWCNTs were homogeneously dispersed at the grain boundaries and within the Al particles. By comparing the grain size of the sample composites (Figs. 8b and 8c) with the Al powder particles, it can be seen that the particle growth is very small. This is due to the embedding effect of MWCNTs which inhibits particle growth. It is important to note that the effect of Cu-coated MWCNTs enhances more of the Al matrix binding interface. Therefore, small grain sizes were obtained in the Cu-coated MWCNTs/Al composites as compared to the uncoated MWCNTs/Al composites, where little grain growth resulting in relatively larger grains was observed. Due to poor wettability in the uncoated MWCNTs, the embedding of MWCNTs in Al particles was less during casting compared to Cu coated MWCNTs/Al composites.

3.4 Mechanical Properties of Al-Cu/MWCNTs Composites

As previously mentioned, copper coated MWCNTs are added in varying percentages to the pure aluminum melt using casting techniques. The aim was to improve the wettability and dispersion between aluminum and MWCNTs which was reported to be very poor in a previous review [18]. The hardness of the sample was tested using a Vickers hardness tester. About 10 penetrations were carried out on a different area of each sample. The small variability of the Vickers hardness between the different indentations is indicative of the homogeneous distribution of Cu coated MWCNTs in the aluminum matrix. The results of the Vickers hardness number for various percentages of copper coated MWCNTs are presented in Table 4.

Table-4: Vickers hardness numbers of cast aluminum with variations in the percentage of copper coated MWCNTs

Cu-MWCNTs (% wt)	Vickers Hardness Numbers (HV) on 1000 g load
Pure Al	37.4
Al + 0.5% Cu-MWCNTs	42.8
Al + 1% Cu-MWCNTs	48.2
Al + 2% Cu-MWCNTs	54.6

It was found that adding 0.5, 1 and 2% copper coated MWCNTs to pure aluminum resulted in significant increases in Vickers hardness of 14.3, 28.9, and 45.9 % especially that, for example, 2% copper coated MWCNTs had less than 0,1 % of MWCNTs. This confirms the potential of the process used in producing good quality cast composites from Al-Cu/MWCNTs. Efforts are underway to optimize the casting process and to fully investigate the mechanical behavior of the composite.

4. CONCLUSIONS

Based on the results of the analysis and discussion, in the MWCNTs electroless plating process with copper on dispersion and wettability properties in fine aluminum matrix, it can be concluded that:

a. Factors that affect the morphology of the Cu coated MWCNTs, namely; the composition of the colloidal palladium–tin catalyst, the type of catalyst, the activation temperature, the volume of the HF acid solution during the acceleration process, and the volume of the electrolyte solution bath.

b. The volume of the electrolyte solution bath is more in electroless plating, resulting in a better sample.

c. The hardness of the Al-Cu/MWCNTs composite increased with the increase in the Cu/MWCNTs content in the Al matrix.

d. Homogeneous hardness test showed that the Cu coated MWCNTs were evenly distributed in the Al matrix.

e. In the Al - Cu/MWCNTs composite samples from the casting results, the morphology of the Al grains did not change much compared to the fine Al.

f. The results of the wettability test using the sessile drop method at a temperature of 700 ^{o}C, showed that the wettability between the Al drop and the Cu coated MWCNTs substrate increased.

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BIOGRAPHIES

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