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IN-SITU HEATING EXPERIMENT OF ALUMINIUM AND CNT MIXTURE USING HIGH RESOLUTION TEM

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ABSTRACT

As lightweight and high strength material, carbon nanotube (CNT) has a high potential used in wide range of application to increase the strength of material like Aluminum, which is already used in many area. To understand interaction of CNT-Al, we conduct insitu-heating experiment up to 800°C; during heating the process was observed in Transmission Electron Microscopy. The results shows that deformation was occur in Al.

Keywords: carbon nanotube (CNT), electron microscopy, mechanical properties.

INTRODUCTION

CNT is known to have good mechanical and electrical properties [1] so that it has a high potential to be used in a wide range of applications. One of its purposes is to act as filler on a mixture of metals to increase their mechanical strength. Aluminum is widely used in many products, so that it is desirable to improve the mechanical properties of aluminum. The addition of CNT into aluminum during the manufacturing process could help to increase the aluminum mechanical properties [2]. To understand mechanism of interaction 2 material based on insitu heating experiment was very interesting, for example a chemical reaction between Si and graphite at around 1400C in high-resolution electron microscopy [3], insitu experiment in Al melting and freezing at high temperature was reported [4]. Therefore, it is interesting to evaluate the interaction between the CNT and the metals when they undergo a heating procedure during the manufacturing process.

In this experiment, the effect of heating on the mixture of CNT and aluminum is observed and reported.

Experimental procedure

In order to reduce the agglomeration of the multi wall CNT (MWCNT) powder is mixed with ethanol and then sonicated for 15 minutes and then followed by centrifugal spinning for 10 minutes and then vacuumed dry. The aluminum powder was mechanically grinded using mortar and pestle, the MWCNT is added to form an aluminum and MWCNT mixture, the mixture sample was cold pressed to got a bulk of Al-MWCNT. The TEM sample is fabricated from the cold-pressed Al-MWCNT by ion beam on Hitachi FB2200 with an operated voltage of 40 kV. Thin TEM sample is mounted on a designated heating filament, which control thetemperature based on Kamino holder [3]; as can be seen in Figure-1. The sample was observed and heatedin-situusing the TEM Hitachi H9500 at operating voltage of 200kV to find out the effect of heating to this Al-MWCNT sample. All experiments were conducted in Research Center of Nanoscience and Nanotechnology, InstitutTeknologi Bandung.

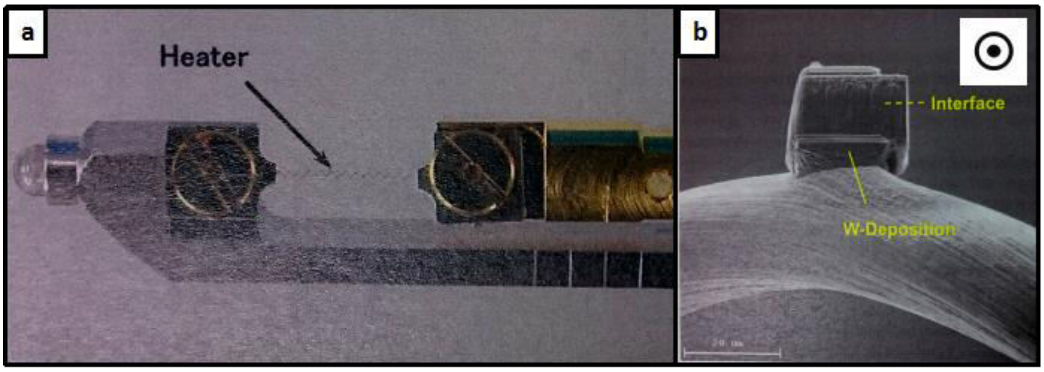

Figure-1. Al-MWCNT on heating element at TEM heating holder.

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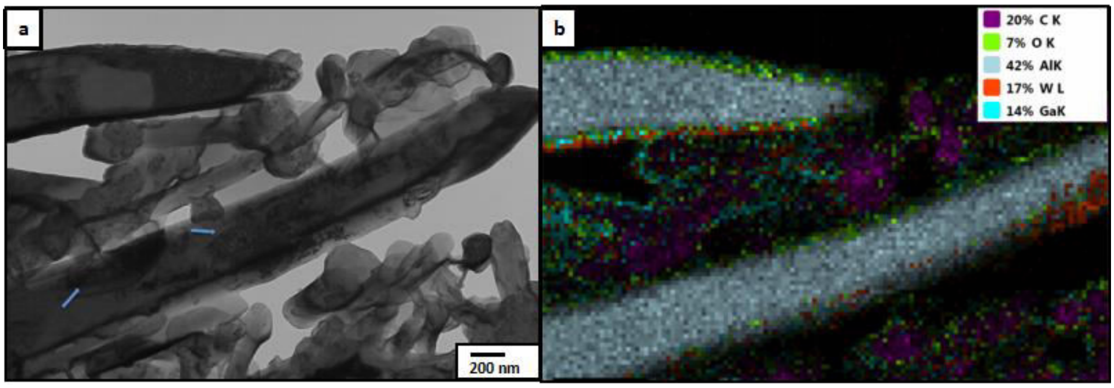

Figure-2. TEM EDX mapping of Al-MWCNT.

RESULTS AND DISCUSSIONS

Figure-2a shows TEM image of Al-MWCNT before heating, showing that the aluminum and MWCNT layers are a little bit loose where there are some space that can be filled by air between the layers. To make sure the element composition of the area, the EDX analysis was applied. Based on the TEM mapping result as can be seen in Figure-2b, oxygen was detected on the gap between each layer, indicated that aluminum oxide layers were created on the gapduring TEM sample fabrication. The area inside the red box (in Figure-1) showing a quite large gap between aluminum that contains a large portion of empty space and a small amount of MWCNT. This area seems to have more portion of MWCNT before, but the MWCNT was severely damaged by the FIB microsampling procedure so that it is crumpled and shrunkas it is shown in Figure-3. The part inside the yellow box is magnified further to see the structure of MWCNT after FIB microsampling procedure.

The TEM mapping shown in Figure-2 also shows that there is some tungsten and gallium also deposited on the surface of aluminum and CNT. The presence of Gallium and Tungsten is due to the micro sampling process on the FIB. Gallium is the ion used to bombard the specimen during the milling and tungsten is the ion deposited on top of the sample surface for protection during the milling. The tungsten and gallium is in the ionized state so that they are easily moving around during the heating. Oxygen also detected mainly in the surface of aluminum. It is probably due to the addition of water during the cold pressing treatment so that the aluminum surface was oxidized.

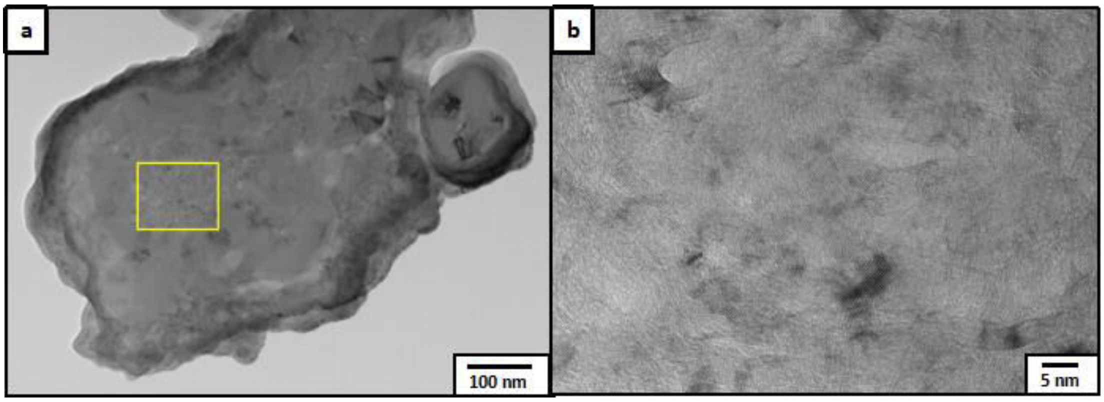

Figure-3. Crumpled MWCNT due to the side effect of FIB microsampling process.

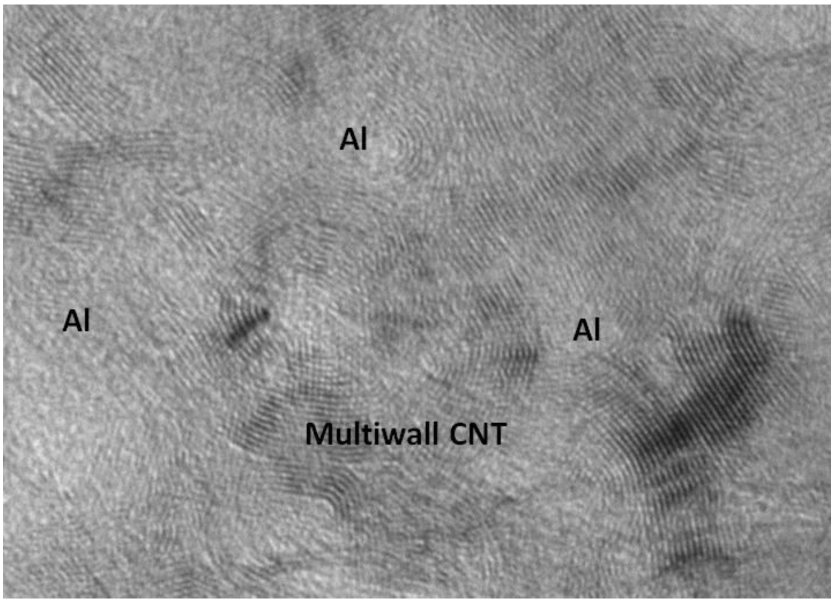

Figure-4. The high magnification of MWCNT.

High magnification TEM image of Al-MWCNT shown at Figure-4 shows that CNT layers have a very clear circular pattern. We found that MWCNT are encloses Al in that irregular pattern.

The heating experiment was performed with gradual increase of heating temperature. The result can be seen in Figure-5. The in-situ heating experiment that was performed in this area showed that the dislocations observed on the aluminum chunks start to move around towards the free surface at (T = 200˚C) as shown at Figure -5a. The increase of heating temperature leads to a significant shape change of the sample and the dislocations to suddenly disappear completely from the aluminum chunk when the temperature almost reaching 300˚C, which is around aluminium recrystallization temperature. The aluminum then start to disintegrate and the aluminum particles start to move around at (T = 300˚C).

When the temperature reaches 600˚C, which isnear the melting temperature of aluminum at T = 660˚C, a significant deformation of the sample occurs, indicating that the flow of aluminum is increased significantly around that temperature so that the sample deforms to accommodate that flow. The deformation indicated by the formation of empty spaces and some lumps at the aluminum surface as shown by red arrow on Figure-5b. On the other hand, some of the aluminum seems flowing into the carbon nanotube and fill the space inside and on the MWCNT; furthermore, some of the aluminum also sticks at the surface of MWCNT as is shown by the yellow box on Figure-5b.

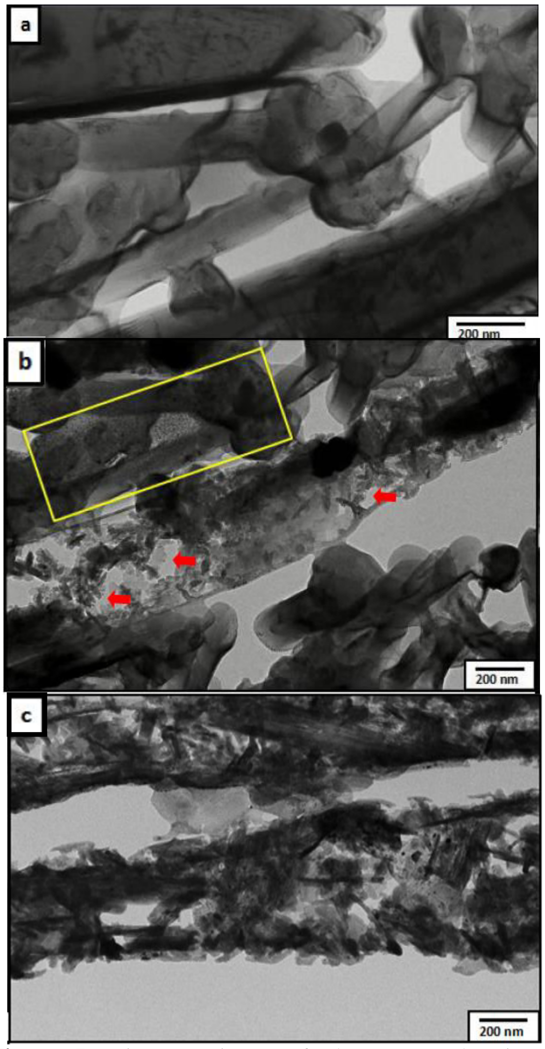

Figure-5. Insitu experiment of Al-MWCN Theating. (a) 200˚C, (b) 600˚C and (c) 700˚C.

When the temperature reaches near the melting temperature of aluminum at T = 660˚C, a significant deformation of the sample occurs, indicating that the flow of aluminum is increased significantly around that temperature so that the sample deforms to accommodate that flow. When the temperature reaches 700˚C,the aluminum looks flowing freely all over the sample, so it is already distributed all over the sample as is shown on Figure-5c. When the temperature reaches 800˚C, the flow and the deformation of the sample is no longer significant.

This is because the melting temperature of aluminum is lower than that of MWCNT so that when the temperature is increased near the melting temperature of aluminum, the bonding on aluminum starts to break and the aluminum particles starts to move freely. Some of the aluminum is flowing through the spacing between CNT layers and some others are evaporated and deposited in the surface of CNT.

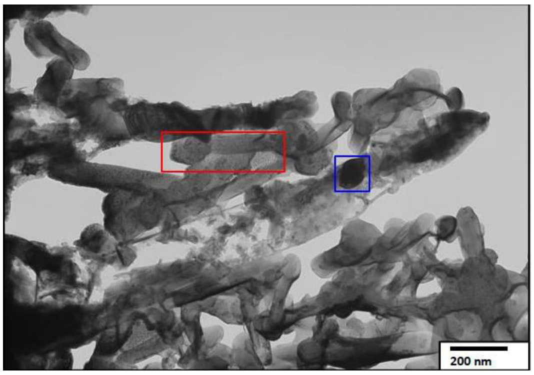

Figure-6. Al-MWCNT after heating up to 800˚C.

Figure-6 shows that the aluminum is heavily deformed and reduced after heating experiment. Gallium and tungsten also observed to be spread all over the sample surface. Gallium melting temperature is around 30˚C that makes the gallium is already saturated so that it spread all over the sample surface. On the other hand, the tungsten melting temperature is around 3400˚C so that it is quite strange that tungsten particle is able to move around at the heating temperature of about 500˚C.

It is most probably due to the fact that the tungsten in this sample comes from the tungsten deposition layer that created by the induction of the adsorbed tungsten gas on top of the sample surface by the FIB beam [5]. Because of that, the bonding between tungsten and aluminum or MWCNT is not too strong so that low temperature heating could already break the bond so that the tungsten can move freely around the sample. The tungsten is moving freely and sometimes creates some lumps as shown by the area inside the blue square on Figure-6. This is possible because there is quite a lot of tungsten available in the sample because tungsten was used as a protective layer during FIB microsampling process.

SUMMARY

As summary we can conclude that aluminum has a lower melting temperature than CNT so that during heating process, Al could flow into the CNT or stick on the CNT surface. Effect of heating the aluminum on its crystallization temperature was observed, that is the dislocations are suddenly disappear completely from the aluminum. From this observation also can be seen that there is a big effect from FIB preparation process such as CNT crumpling, as well as movement of gallium and tungsten particles.

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