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Review Article

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Homogenous dispersion and interfacial bonding of carbon nanotube reinforced with aluminum matrix composite: A review

Abstract: Recent developments in the field of carbon nanotube (CNT) have attracted attentions on a new development of aluminum matrix composite (AMC). Homogenous dispersion and interfacial bonding of CNT/metal matrix are the two main problems yet to have sound solutions. In general, to ensure uniform dispersion, the overall process can be divided into three steps: preprocessing, mixing and post processing. This paper summarizes previous works on solid and liquid processing techniques which some are more successful than others by looking at the improvement of the composite tensile strengths. On the other hand the interfacial bonding depends on the existance of Al_4C_3 phase and physical conditions of CNT. Both elements must be controlled for the optimized results. The review presented here would be used as references in the future works to fabricate higher strength CNT/aluminum composites.

Keywords: multiwalled carbon nanotube; metal matrix composite; composite processing; interfacial bonding

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1 Introduction

Aluminum alloy is one of the most widely used materials in automotive and aerospace industries owing to its good mechanical and light weight properties [1–3]. In recent years, there has been a growing interest in incorporating reinforced materials into the alloy matrix such as alumina (Al_2O_3), silicon carbide (SiC), boron carbide (B_4C), graphene nanoplatelets (GNPs), and carbon nanotube (CNT) to become metal matrix composites (MMC) [4–7]. MMC possesses higher specific strength/stiffness, higher wear resistances, and low density compared to the original alloys [8, 9]. In addition, since the discovery of CNT by Ijima in 1991, there has been a large volume of published studies describing the roles of CNT as a reinforcement material in the matrix. CNT has exceptional mechanical properties, Young's modulus of 1 Tpa, tensile strength of 30-150 Gpa, and low thermal expansion, which led to greater interest in exploring a CNT-aluminium matrix composite (CNT-AMC) [10, 11]. Therefore, the purpose of this paper is to review recent research focusing on the CNT-AMC exclusively.

The presence of extremely small particles with homogenous dispersion and effective interfacial bonding of the CNT in the metal matrix helps to ensure that there are absolute specific qualities [12–14]. The CNT nano particles tend to agglomerate due to strong Van der Waals forces between the atoms, which make it difficult to dampen and overcome the surface tension of the matrix [15]. Moreover, the interfacial bonding is formed by interdiffusion between carbon and aluminium at certain temperatures, as seen in aluminium carbide (Al_4C_3), which provides adhesion strength [16–20]. To date, several studies have investigated the mechanical characteristices of CNT, such as length, size, waviness and structural defects, which contribute to the strength and formation of Al_4C_3 [9, 21–23]. Both homogenous dispersion and interfacial bonding provide the mechanisms of mechanical strengthening of CNT-AMC, as in load transfer, Orowan strengthening, thermal expansion mismatch, and grain refinement of matrix and

CNT [21, 24, 25]. Despite the positive impacts of the reinforced particles, previouly it was observed that the performance of a new monolithic composite is limited by the reduction in ductility [14, 26, 27].

Basically, the production of CNT-AMC can be grouped into five categories: (1) Solid state (powder metallurgy), (2) Liquid state (stir casting), (3) Thermal spray, (4) Electrochemical disposition, and (5) other novel techniques. Powder metallurgy has been the most preferred CNT-AMC production method mainly due to agglomeration and floating because of density issues. However, liquid state production is cheaper for bulk production and intricate parts compared to others [18, 19, 28]. To date, the problems of homogenous dispersion, agglomeration, and interfacial bonding especially for liquid state processing have still not been explored comprehensively. Therefore, this paper provides an overview of recent research of CNT-AMC in the solid and liquid state processing routes, the impact on improving the mechanical properties as well as some of the critical approaches taken to overcome the problems that have been discussed.

2 CNTs as reinforced materials

CNT can be produced via several techniques, for example, electrolysis, laser ablation, chemical vapour deposition (CVD), arc discharge, and sonochemicals [20]. The most common type of CNT used in real applications is multi-walled carbon nanotube (MWCNT) compared to single-walled carbon nanotube (SWCNT) [29]. The process to produce single-walled is more expensive and difficult to purify compared to multi-walled [4]. Each concentric layer of graphitic carbon rolled is held together by Van der Waals bonding [30] and can be up to hundreds of walls [31]. Furthermore, the simplicity of SWCNT structure results in a higher Young's modulus with less defect compared to the MWCNT. Stein *et al.* [32] compared the Young's modulus, tensile strength, and ultimate tensile strength between SWCNT and MWCNT with AA5083 wrought alloy and found that single-walled composite had slightly lower values of these properties compared to multi-walled. Therefore, they concluded that the single-walled was more prone to damage in structures. However, in other observations, the amount of CNT in the matrix will determine the mechanical properties rather than types of CNT. Moreover, Guiderdoni *et al.* [33], conducted a series of trials on a different number of CNT-walls (2,3,8 and 20) with copper (Cu) matrix and their effects to hardness, friction, and wear properties. It was discovered that the microhardness of

these multiple-walled composites were higher compared to those based with Cu, nevertheless the 2MCNT produced good friction and wear properties compared to 8MCNT and 20MCNT. Therefore, too many layers give a negative impact due to damaged carbon layers and porosity. In addition, Varshney [34] also pointed out some issues of CNT, namely the lack of solubility and the difficulty to maintain high quality with less impurities [34]. Figure 1 shows a high-resolution transmission electron microscopy (HRTEM) image showing the morphology and structure of the MWCNT.
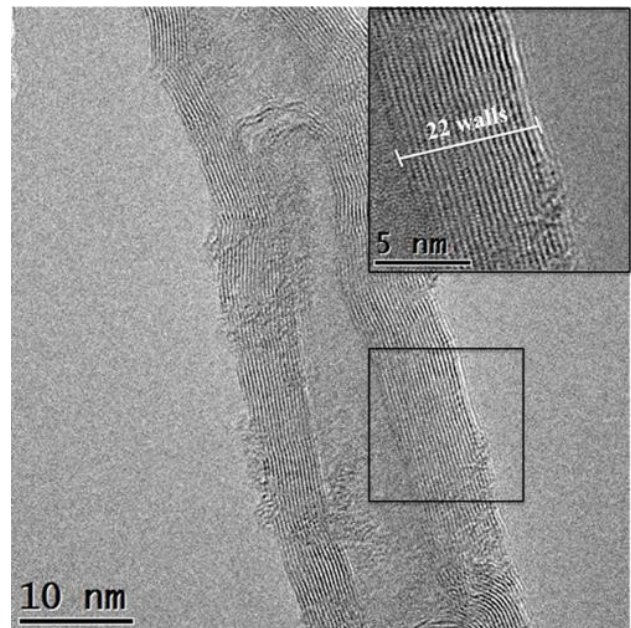

Figure 1: HRTEM image of MWCNT showing 22 number of walls [35].

The data also showed that only a small percentage of CNT, as low as 0.5% by weight percentage, was required in the matrix to improve the tensile strength and hardness [3]. Yang *et al.* [36] summarised the optimised CNT content by weight percentage (wt%) used by others which varied up to 5.0 wt%. In addition, the tensile and hardness properties of the composites increased relative to the amount of CNT. However, the properties started to deteriorate beyond these values due to agglomeration [37].

In a sound composite structure, CNT acts as shear stress absorber and load transfer to increase fracture strength. Figure 2 shows that the FESEM image of MWCNT in the fracture acts as the load transfer mechanism and similar figures have been reported by Bradbury *et al.* [38]. Hence, in order to achieve optimal performance for the composite, an exceptional and homogenous matrix of interfacial bonding plus good reinforcement are very crucial.
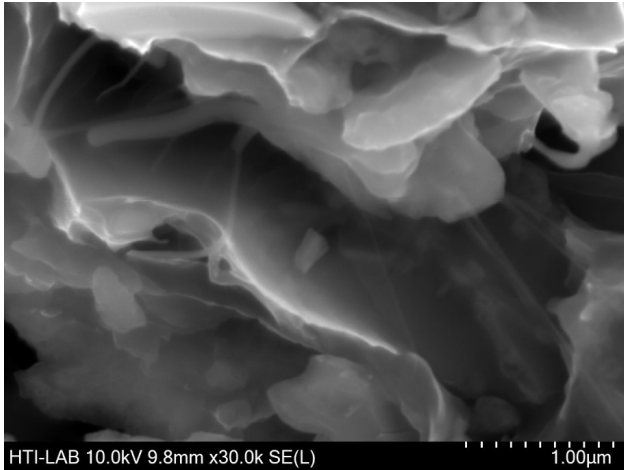
Figure 2: FESEM image shows the MWCNT acts as bridges between the grains from the fractured surface after the tensile test.

Figure 3: Homogenous Dispersion Process Strategy (HDPS)

Park *et al.* [39] studied the formation of Al4C3 subelements with good covalent bonding between Al and CNT which act as load transfer. Moreover, Xiang *et al.* [40] examined the sizes, conditions, and dislocations of CNT in the matrix which improved the composite strength.

3 Homogenous dispersion of CNT

Several factors which influenced the homogenous dispersion of CNT in metal matrix were explored. Firstly, the density of CNT range from 0.03 g/cm^3 to 0.22 g/cm^3 [29] was lower compared to aluminum liquid density 2.37 g/cm^3 and most metals [41]. Hence, the huge difference in densities caused CNT to float while being mixed with molten metal [42]. Consequently, the research to date is focusing more on powder or solid state rather than liquid state processings. Secondly, the strong Van der Waals forces that exist between the carbon atoms caused them to stick and agglomerate to form clusters [22]. Besides, according to the Percolation theory, the threshold amount of CNT in the matrix is very critical in optimising the dispersion throughout in the matrix. In addition, incorrect amount of CNT will either cause minimum improvement or weaken the composite, resulting in a higher agglomeration tendency [43–45].

Several researchers whom have investigated CNT-AMC composites, have utilised the process strategies as shown in Figure 3. The homogenous dispersion process strategy (HDPS) can be divided into a three-step process.

In the first step, the preprocessing stage ensures that CNT is properly cleaned from contamination, purified and detangled from the bundles. Chemicals like isoproplyl alcohol (IPA) and ethanol are commonly used in the clean-
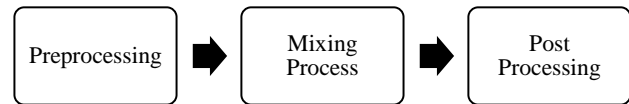
ing process [46, 47]. In some cases, CNT is milled to shape it into a flake form which helps to improve the surface area and energy [48, 49]. In addition, similar threatments made to aluminium have been taken, especially when dealing with powder processing. Major advantages of preprocessing are to minimise porosity, inclusion and fatigue defects which might result from a non-uniform dispersion of CNT and unwanted gaseous dissipation during the mixing process. Step two involves the mixing process where different methods have been applied depending on the routes. In this stage, there is a need for proper selection of hardwares such as ball milling, crucible, stirrer; as well as different types of chemicals with the necessary setup parameters such as temperatures and time and material ratio. Step three includes the post processing stage where heat treatments such as T5 or T6, hot or cold extrusions and rollings are often carried out to further enhance the mechanical properties of the composites. The following chapters present the findings of the previous research, focusing on the HDPS steps based on solid and liquid processing route.

3.1 Solid state processing route

Solid or powder state processing route had been established as one of the most preferable routes for producing the CNT-AMC. One of the major benefits of this approach is that the density is different between the reinforced materials, and that the matrix materials can be ignored when not dealing with the liquid state. However, a great deal of previous research into CNT-AMC has focused on pure aluminum instead of an alloy as the metal matrix. So far, there has been little discussion on other types of aluminium alloys. This might be due to an inadequate understanding of the CNT particles behaviour and interaction with the alloy elements. For the past 10 years, several studies have investigated the various CNT-AMC processing steps and some are discussed as follows:

Rikhtegar *et al.* [50] evaluated the microstructural and mechanical properties of MWCNT in pure aluminium matrix composite. The amorphous MWCNT was preprocessed to become functionalised in nitric acid, followed by being rinsed and dried in an oven. The mixing steps were separated into two different powder metallurgical techniques:

semi-wet (SW) and slurry based (SB). For SW, the mixtures underwent utrasonic vibration and planetary ball milling. Ethanol was added to unbundle the tangled MWCNT. The mixture was electromagnetically stirred and later ultrasonically vibrated. On the other hand, for SB, the powder was ball milled for 2 hrs until flaky powders were produced. The powder was then mixed with MWCNT-COOH and sodium dodecyl solfate into deionised water. Both specimens were compacted, sintered, and extruded at a high temperature of 400°C. The tensile strengths of both SW and SB improved by 25% and 49%, respectively.

Yang *et al.* [36] studied the effects of chemical vapour deposition (CVD) in-situ fabrication of CNT and pure aluminium (CNT/Al) using cobalt/aluminium catalyst. This preprocessing step helped to disperse and coat CNT around the Al powder matrix which prevented contamination. Next, the CNT/Al was further put through a process of ball milling using planetary agitation and then compacted for mixing process. Finally, for post processing, the samples were hot extruded at 500°C. The tensile strength and hardness of the monolithic composite increased by 2.4 and 2.3 times higher, respectively.

Bunakov *et al.* [51] performed a functionalisation process of attaching polar molecules such as – OH or – COOH to the MWCNT by dipping in the sulphuric and nitric acids to enhance the dispersion. Next, for the mixing process, both pure aluminium powder and MWCNT were mixed in ethanol. The solution was stirred intensively and put through an ultrasonic treatment before it was dried off for 3 hrs. Then, the specimens were compacted and ran through spark plasma sintering. There were 30% and 16% improvements of microhardness and tensile strength, respectively, for 0.1%wt of MWCNT. Further additions of MWCNT in the matrix resulted in lower values due to agglomeration.

A comprehensive preprocessing and mixing method were taken by Chen *et al.* [49] which involved multiple steps. A solution of MWCNT/IPA was mixed and planetary ball milled to produce flaky aluminium powder coated with MWCNT. After 15 minutes, the solution was then removed out from the container and left over coated flaky aluminium powder which sanked at the bottom and then dried off. Then, the samples were sintered by sparking plasma followed by hot extrusions for post processing. There was a noticeable improvement in tensile strength from 149 MPa of pure aluminum to 192 MPa of the composite.

Esawi *et al.* [52] conducted a series of trials in whereby pure aluminum was mixed with different quantities of MWCNT. In the preprocessing step, high purity >95% and large aspect ration between CNT and the matrix had been choosen. Hence, assuming no futher process of cleaning or

pretreatment was placed on both the matrix and reinforcement materials. However, methanol was dded into the mix during the ball milling process to prevent cold welding and powder sticking before it was compacted at 475 MPa. Lastly, the samples were hot extruded and annealed for some time during post processing. The maximum tensile strength and stiffness were improved by 50% and 23%, respectively, with the addition of 2%wt MWCNT.

Furthermore, in the preprocessing step, Choi *et al.* [53] purified the MWCNT from amorphous carbon using nitric and hydrochloric acids. Then, in the mixing step, stearic acid was added into the ball milling process to prevent agglomeration. Finally, the post processing steps involved hot rolling and heating the compact. Additionally, hot rolling took place 27 times, which helpd to reduce it by more than 300% compared to the original thickness. As a result, 4.5vol% of MWCNT/Al composite improved significantly in terms of wear and mechanical properties.

Overall, these studies exhibited the need for a three-step composite process to ensure that uniform and homogeneous dispersion is obtained. The evidence presented in this section suggests that higher tensile strength and hardness can be achieved when the processes are carried out properly. The next section provides some of the latest studies on CNT-AMMC conducted on the liquid state processing route.

3.2 Liquid state processing route

Despite the difficulties in overcoming issues with floating, agglomeration, and wettability in the liquid state processing route, it is still a cheaper process as compared to the solid state. But, high potentital for bulk production makes the liquid processing route more interesting and worthy of trying [54]. The difficulties or issues which take place during the mixing process was due to the density gaps between CNT and the metal matrix which causes the CNT to float. As a result, the strong Van der Waals bonding between carbon atoms causes them to stick to one another and the surface tensions of CNT 45.3 mN/m was lowered compared to aluminium 955 mN/m during the melting phase, due to poor wettability [55]. Some of the latest studies on the issues involving the three-stage liquid state processing are discussed as follows:

Abbasipour *et al.* [8] prepared the preprocessing step by depositing the MWCNT particles onto aluminum using the Ni-P electroless plating technique. The MWCNT and aluminium particles were first purified, pretreated and degreased, subsequently treated with acid and ultrasonically cleaned before coating, respectively. While mixing, the P-

Ni-CNT-Al was injected directly into molten aluminium alloy A356 at 700°C above melting temperature. 1% of magnesium (Mg) was also injected together into the molten product. By precoating aluminium particles with CNT gave way to slow dispersion into the matrix and Mg acted as a wettability agent. Finally, in the post processing, some specimens were poured into the steel die at different temperatures corresponding to their solid fractions. The hardness of the composite improved by 23% compared to the original A356 alloy.

Mansoor and Shahid [54] treated aluminum AA1199 with sodium hydroxide solution to wash any oil and oxide layer from the surface for the preprocessing step. Next, during the mixing process, grinded MWCNT was mixed with flux and injected into molten aluminium. Induction stirring was applied and added with another cleaning flux to remove unwanted materials inside the induction furnace. The molten composite was than poured into a mould and later put through cold rolling as the post processing step. The tensile strength and hardness increased as much as 52% and 45%, respectively.

Elshalakany *et al.* [28] fabricated a block of MWCNT, aluminum powder, and stearic acid to increase the density which submerged into the molten form in the preprocessing step. The mixing step was carried out in a resistance furnace and 0.75%wt of Mg was added. The blocks were then introduced into the molten and stirred mechanically for 1 min. The molten mixture was then poured into a mould and squeezed instantenously. The optimum amount of MWCNT was 1.5%wt in the composite to enable the ultimate tensile strength to improve by 50% and to significantly increased in hardness.

A recent study by Park *et al.* [39] involved intensive preprocessing steps, the MWCNT was first grinded and dispersed into dichloroethane solution. Then, the mixtures were dried, de-oiled, sintered, and finally pressed into blocks. Next, the mixing process was carried out by inserting CNT-Al blocks into completely melted pure aluminium ingot using an induction furnace. The molten composite was stirred by a graphite impeller for 20 mins. Finally, the post processing hot extrusion at 550°C was applied to the composite. Based on these process steps, CNT-AMMC improved by 60% and 23% of the yield strength and tensile strength, respectively.

Hayan and Niroumand [56] prepared a multilayer MWCNT-Al composite strips via a rolling process called accumulative roll bonding (ARB) at the preprocessing step. The MWCNT powder was spread onto an aluminium stripe alternately and rolled at 50% thickness reduction each time for 4 cycles. The mixing process was carried out by melting a A356 alloy into a crucible in an electrically resistant furnace. The strips were then put into the crucible and stirred when the alloy was completely melted. The total ratio of 0.5%wt MWCNT specimen was produced for the post processing step. The slurry was poured into a die and squeezed immediately at different pressures. There was a significant improvement of hardness and shear strength of 38% and 20%, respectively. Figure 4 shows homogeneous dispersion of MWCNT throughout A356 alloy.
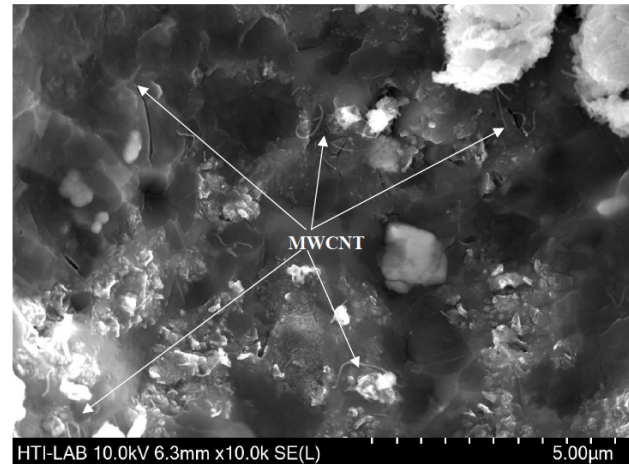

Figure 4: FESEM images of MWCNT uniform distribution in the A356 alloy matrix

This section has conferred the different methods of the preprocessing, mixing, and post processing steps were applied to ensure maximum homogeneous dispersion of CNT throughout the aluminium matrix. Normal melting and casting routes may not be suitable in the fabrication of CNT-AMMC. The next section discusses another two mechanisms that contributed to composite strengthening.

4 Interfacial bonding

Despite the importance of uniform and homogenous dispersion of CNT, without strong interfacial bonding at the interface between CNT and metal matrix, load transfer can be ineffective [38, 57]. Furthermore, wettability is also affected by the reaction at this interface where chemical bonds are formed. In addition, Kumar *et al.* [58] reported that corrosion resistance was also dependent on the intergrity of interfacial bonding. In CNT-AMC fabrication, CNT reacted with the aluminium matrix and formed needle-like structure of aluminum carbide (Al_4C_3) compound with a 20-40 nm average diameter. The Al_4C_3 acts like a pin or binder of the CNT to the matrix [59]. Deng *et*

al. [60] identified the formation of Al_4C_3 was likely to take place when molten aluminium is involved and the temperature is above 656°C. The formation of these phases help to minimise shear stress and increase the effective load transfer between the matrix and reinforced material [61, 62]. On the other hand, the presence of excess Al_2O_3 will have a negative impact to the bonding strength due to agglomeration and poor wettability [63, 64]. Therefore in order to get positive results, the presence of sub-products in the region must be controlled for positive results.

A significant analysis and discussion on the subject were presented by Bakshi and Agawal [65], showing that in their list, about 20% of the CNT-AMC studies utilised metallurgical powder or solid process route had formed the Al_4C_3 phase. Others reported no formation of Al_4C_3 phase at the interface. These observations might be due to an insufficient temperature level to form the phases thermodynamically or undetected AL_4C_3 amount during characterisation process. In addition, the chemical reaction of $4Al + 3C \longrightarrow Al_4C_3$ required negative standard free energy, which explains the high tendency of phase formation. In conclusion, the chemical interaction between matrix and CNT is necessary to enable large stress load transfer and stronger composite.

Bakshi *et al.* [65] studied the formation of Al_4C_3 phase between MWCNT and aluminium silicon (Al-Si) composites and discovered that as the Al_4C_3 fraction reduces the Si fraction increases. In this case, Al-16%wt Si showed clear formation of Al_4C_3 needle phases, which helped to enhance the mechanical properties of the composite alloy.

Moreover, Laha *et al.* [66] investigated the type of interface phases formed between hypereutectic Al-23%wt Si alloy matrix and MWCNT. Based on the findings, due to lower free energy of β-SiC phase formation ($-280\,kJ\,mol^{-1}$), it is thermodynamically more attainable as compared to Al_4C_3 ($-64\,kJ\,mol^{-1}$). The formation of these carbides is confined by the existence of prism plane (100 and 110) as in a graphitic structure. Therefore, the phases are more likely to form due to physical damages occuring at the outer surface of the MWCNT be it intentional or not.

Hence, the next section discusses the two effects of the physical conditions of MWCNT and process temperatures for the formation of interfacial bonding.

5 Processing temperatures

This subsection deliberates the importance of precise processing temperatures that help to optimise the formation of interfacial phases in the regions. Kwon *et al.* [67] exam-

ined the effect of sintering at 600°C and heat treatment at slightly above pure aluminium melting point 670°C and at 800°C for the formation of Al_4C_3. Based on this study, small amount of carbide in Al_4C_3 was already present between the CNT and matrix. However, the needles of Al_4C_3 were finer and coarse lumps were distributed at every boundary. These conditions might be caused by the ability of the disordered CNT to flow into the boundary matrix at slightly above melting temperature. Furthermore, at 800°C, the average size of the carbide got smaller which displayed a degree of overlapping with γ-alumina. Hence, this complex phase led to the degradation of the chemical bond of the composite. Moreover, Housaer *et al.* [63] reported another important intefacial phase of the amorphous aluminum oxide Al_2O_3 at a temperature of 580°C. This phase was formed naturally due to the oxidation of aluminium powder which was not desirable to the strengthening process. The XRD detected significant peaks of Al_4C_3 appearing at temperatures of 620°C and 645°C compared to the normal peak at 600°C. These simple findings concluded the influence and importance of process termperature for carbide formation.

The studies presented thus far, provide evidence for the importance of controlling the process temperature in the formation of the Al_4C_3 phase.

6 Physical conditions of CNT

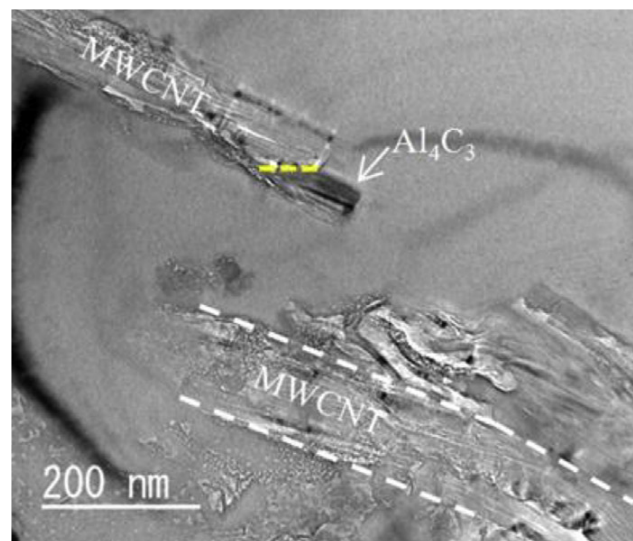The adhesion and shear strength properties of the interfacial phase are determined by structural damage, mor-

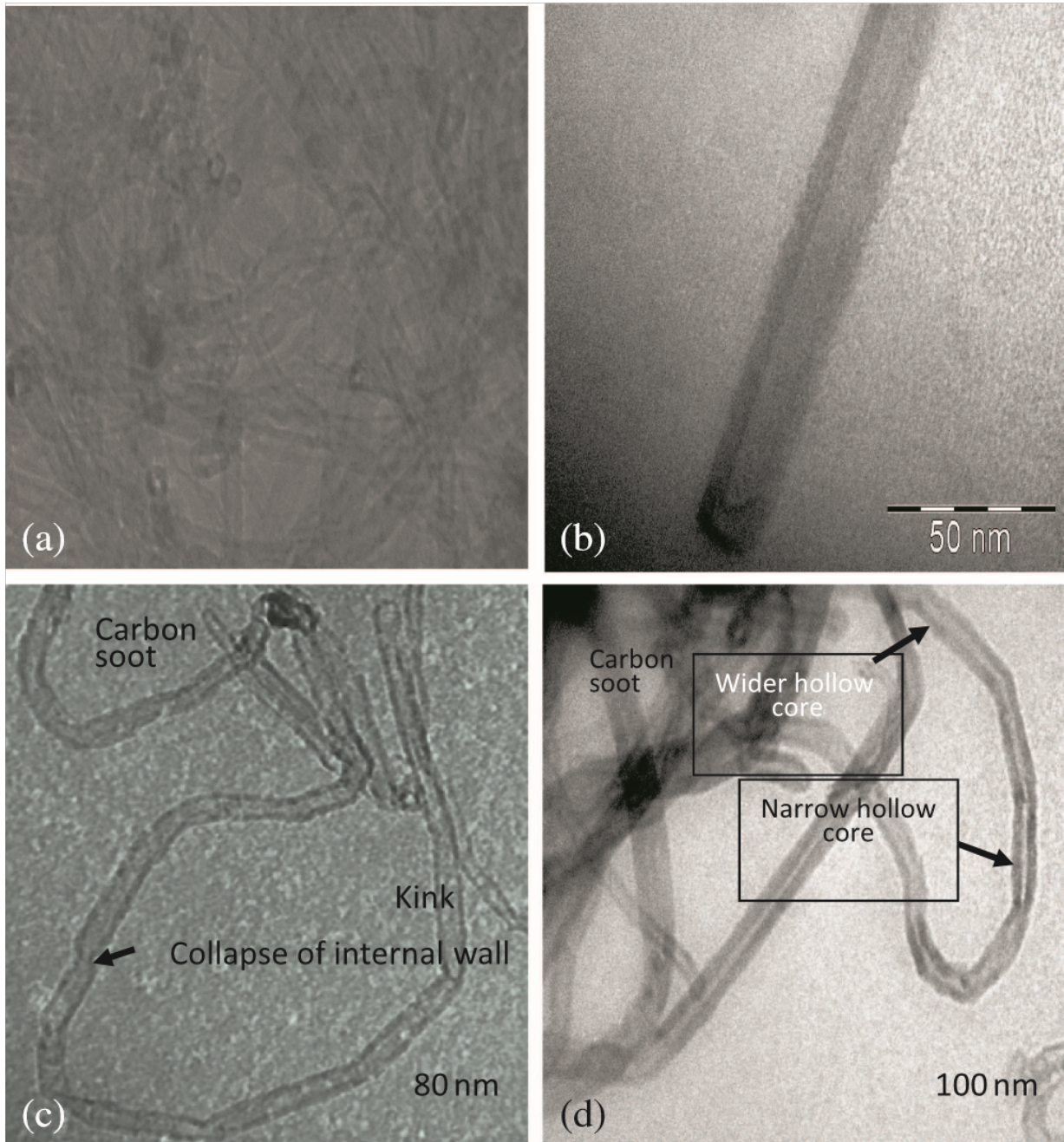**Figure 5:** TEM images of Al_4C_3 formation at the end of MWCNT [69].

Figure 6: TEM images of MWCNTs: (a) raw, (b) 60 min heat-treated, (c) 120 min heat-treated, (d) 120 min heat-treated [**?**]

phology, and diameter of CNT [15]. These properties determine he load transfer efficiency between CNT and the matrix [68]. In a study which investigated interface phases, Zhou *et al*. [69] reported that a significant fraction of Al_4C_3 formed at the nanodefects and open ends of MWCNT structures as shown in Figure 5. The evidence presented in the studies suggests that the nanodefect promotes simultaneous carbide growth in <100> and <110> directions and open end in <100> direction of MWCNT only. The functions

of the carbide phases are as follows: The covalent bond acts as a bridge to block peeling of the outmost wall from the inner wall under load-bearing. Next, improvement in interfacial shear resistance and prevention of pullout of MWCNT and finally, the formation of strong anchors for MWCNT in the Al matrix.

Hassan *et al*. [12] demonstrated that the levels of CNT structural damage affected the tensile strength of the composite. Comparisons were made based on received, mildly,

and severely damaged CNTs which were prepared by ball milling and confirmed by FESEM analysis. For the additional 2%wt CNT in the matrix, there was an improvement on the 95.9% as-received to 97.5% mildly damaged compared to pure Al. However, for the severely damaged CNT, there were some reduction in the value. These results showed that with disorders or open structure of CNT, there was an increased promotion of interfacial growth which helped to strengthen the composite. Yet, with the observation by FESEM which exhibited further damage or destroyed turbular structure, agglomeration started to occur which weakened the bonding.

On the other hand, Peng and Chang [3] suggested that damaged structures of CNT prevented the full translation of CNT effectiveness in the matrix. Based on the studies, the fully intact structures can be achieved by using wet powder in a shake-mixing process. However, the enhancing effects of the undamaged MWCNT to the mechanical strength has not yet reported as in the studies.

Srikanth *et al.* [70] reported the high temperature and duration effect on MWCNT structure through TEM observation. Using thermogravimetric analysis (TGA), the studies also showed that as received raw MWCNT starts to loose its weight at $550°C$ and stablize at $650°C$ due to 36 wt% of metallic impurities. On the other hand, heat treated MWCNT cleared the impurities and stablized up to $700°C$. Further heat treated at $2600°C$ for 60 min had shown some structures degradation and further damaged can be seen at longer duration of 120 min. Figure 6 shows the structural started to kink and eventually collaped of the internal walls.

When considering all of the evidences, it seems that the control of exposed physical conditions of the CNT structure plays important roles in ensuring the development of effective interfacial phase growth and strengthening of the composite. Severely damaged CNT structure prompts the formation of Al_4C_3 nanorods especially at higher sintering temperatures which weakens the strenghtening effect.

7 Conclusion

The insights gained from this study may be of assistance in the understanding of CNT homogenous dispersion strategies and contribution factors to maximise interfacial phase formation in the aluminum matrix.

For solid or liquid state processing routes, a three-step strategy must be implemented for homogenous dispersion; preprocessing, mixing, and post processing. Eventhough, solid state route is more preferable, a more robust liquid state route requires more attention in the future due to its suitability for high volume and cheaper production method.

Furthermore, the shear stress strength or effective load transfer is largely dependent on the presence of Al_4C_3 phase. The chemical bonding and adhesion of this phase was determined by process temperatures and physical conditions of CNT structures. The right amount of Al_4C_3 in the region is also critical in ensuring the strengthening effect of the reinforced material.

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