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Research Article

Mechanical properties of artificially aged carbon nanotube reinforced aluminum alloy matrix composites

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ABSTRACT

The application of carbon nanotubes (CNTs) as reinforcements in aluminum (Al) matrix composites was studied enormously with different fabrication methods due to the outstanding properties of CNTs. As known, precipitation hardening is important in the mechanical property enhancement of suitable Al alloys, as well. Especially, T6 heat treatment is one of the most aforementioned treatments where solution heat treatment and artificial aging are applied, respectively. In this study, CNT-reinforced 6063 Al alloy matrix composites were fabricated by vacuum-assisted infiltration into a preform method, and T6 heat treatment of composites was realized. The aging behavior of samples was introduced by derivation of mechanical properties and results were discussed in view of microstructural characterization. It was found that decent matrix reinforcement interfacial bonding and aging capability improved by the presence of $MgAl_2O_4$ and Al_4C_3 were successfully provided through the fabrication method. After aging, composites reached a hardness of 123.6 HV where the hardness value of 59 – 65 HV can be achieved by aging of 6063 Al alloy and 79 HV by the CNT reinforcement only. Moreover, the peak tensile strength value of 254 MPa reached by the aging of CNT/6063 Al composites is 38% higher than aged 6063 Al alloy and 113% higher than solely CNT-reinforced composites. In addition, the increase in the compressive properties in composites is also remarkable. Consequently, recent findings and discussions presented in the study provided an important understanding the effects of CNT existence on the aging behavior of Al alloys.

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INTRODUCTION

The discovery of carbon nanotubes (CNTs) became a milestone in materials science and engineering areas [1]. After detecting the extraordinary properties of CNTs such as high Young's modulus and strength, there has been a great increase in the studies for their use as reinforcement material in composite materials [1-6].

CNT-reinforced Al alloy studies became visible with the publication of Kuzumaki et al. [7] and an appreciable

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increase in the tensile strength (~100%) with a high amount of (8 wt.%) carbon nanotube addition was published in their studies. Afterward, other researchers tried using CNTs as reinforcement into Al using powder metallurgy routes [8-13] mentioning fabrication, characterization, enhancement in mechanical properties including damping characteristics, friction stir processing [14] as a creative technique which is tried to disperse CNT homogenously, however, fell short, plasma spraying [15] to achieve better CNT distribution which could be a good choice for nano-coating but mechanical properties were explained limited to the hardness, microwave sintering [16] which is a limited study with wear resistance and the frictional response of the surface, and spark plasma sintering [17] which is a perfect candidate for better structure and less porosity with hot extrusion addition however results of the study didn't give the expected results.

Infiltration into a preform is often applied to fabricate composites with homogeneous reinforcement that provides good mechanical properties and was used for CNT-reinforced composites by Zhou et al. [18] using a pressureless infiltration process. In their study, molten Al alloy was infiltrated into carbon nanotubes–magnesium–aluminum preforms; however, only the wear behaviors of these composites were investigated. Vacuum-assisted infiltration of 6063 Al alloy into a CNT and Al powder/CNT preforms was performed by Kucukyildirim and Akdogan Eker [19, 20], and achieved an increase up to 330% and 250% in the compressive strength and hardness relative to the base matrix materials, respectively.

It is known that one of the important features of 6000 series Al alloys is their aging capabilities [21]. In this context, Fukuda et al. [22] investigated the mechanical behavior of aging on spark plasma sintered and extruded 6063 Al alloy composites reinforced with CNTs. They observed that the hardness of the composites decreased with the increasing CNT content, and as a result, the strengthening failure occurred due to an incomplete hardening of the Al matrix. They indicated that this failure is due to the transfer of Mg and O transfer from the matrix to the CNT sites during the sample fabrication process. In addition, Awad et al. [23] concluded that infiltration methods provide higher initial hardness values since the metallic oxidation in composites fabricated by powder metallurgy methods impacts bonding and; thus, mechanical properties.

In this study, it is aimed to enhance the mechanical properties of CNT-reinforced 6063 Al composites fabricated by vacuum-assisted preform infiltration method using T6 heat treatment by the mentioned advantages of infiltration methods stated above. In this regard, tensile and compression test results were discussed in addition to the hardness measurement results. Furthermore, fracture surfaces of composites were examined by scanning electron microscope (SEM) and X-ray diffractometer (XRD). The objective is to investigate the heat treatment behavior of infiltrated CNT/6063 Al composites and derive mechanical property developments beyond the aforementioned studies.

MATERIALS AND METHODS

Materials

CNT/6063 Al composites were fabricated by infiltrating molten Al alloy into CNT including preform by vacuum-assisted infiltration method as explained in the study of Kucukyildirim and Akdogan Eker [20]. After fabricating the composites, T6 heat treatment of composites was realized according to the ASM Handbook [24]. Fabrication, heat treatment, and testing method procedures are explained as follows.

Materials and Composite Fabrication

Industrial-type multi-walled CNTs (10-30 nm outer diameter) with a purity rate of over 85% were provided from the company named Chengdu Organic Chemicals Co. Ltd., scanning electron microscopy (SEM) and transmission electron microscopy (TEM) images of as-received CNTs are presented in Figure 1a, b. 6063 Al alloy was used as a matrix material due to its flowability and wetting characteristics in addition to the precipitation hardening and aging capability.

CNTs were functionalized with 65 wt.% HNO_3 solution by refluxing to increase the surface energy; thus the wettability of CNTs by the matrix material. The reinforcement ratio was selected as 0.5 wt.% depending on the success of CNT dispersion and mechanical enhancement achieved in the previous studies [19, 20] and mixed with. 20 vol.% Poly Vinyl Alcohol (PVA) as low-temperature binding and a pore-forming agent, 0.5 vol.% colloidal silica (10 - 20 nm grain size) as a high-temperature binding agent for preform production were used. After 48 hours of dehydration of wet preforms at room temperature, all samples were cured at 600°C for 2 hours to decompose PVA and derived a porous structure of preform, then the temperature was raised to 1100°C for 1 hour to crystallize silica, by this means a solid preform was achieved strong enough for infiltration. Subsequent to the preparation of the preforms, the melting and infiltration processes were applied the same as is explained in detail in the article of Kucukyildirim and Akdogan Eker [20]. After the successful fabrication of composites, tensile, compression, and hardness testing samples were machined according to ASTM standards [25-27].

Heat Treatment Procedure

A set of tensile, compression, and hardness test samples (5 samples for each) were subjected to solution and precipitation heat treatments according to the typical T6 heat treatment procedure given in the ASM Handbook [28]. First solution heat treatment was applied at 520°C for 3 hours and immediately water-quenched them to ambient temperature. After solution heat treatment, samples were artificially aged at 175°C for 8 hours. Temperature – time condition chart showing the T832 heat treatment procedure is given in Figure 2.

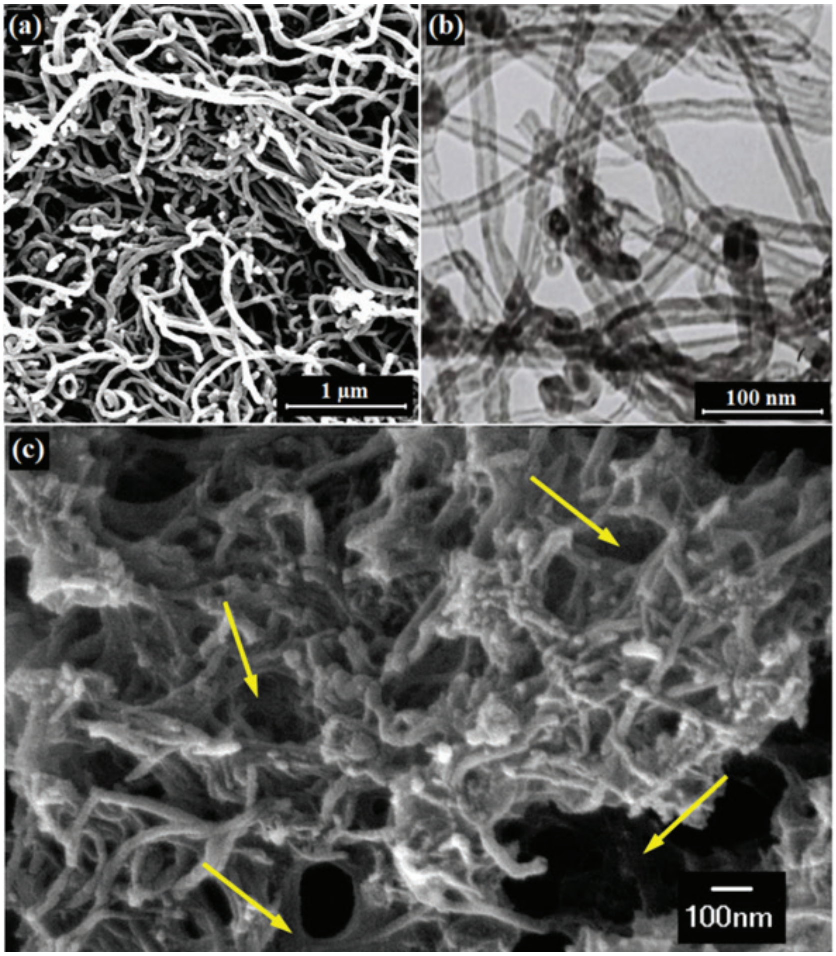

Figure 1. Electron microscope images: (a) SEM micrograph of industrial type MWCNTs; (b) TEM micrograph showing the morphology of MWCNTs; and (c) SEM micrograph of CNT containing preform before curing.

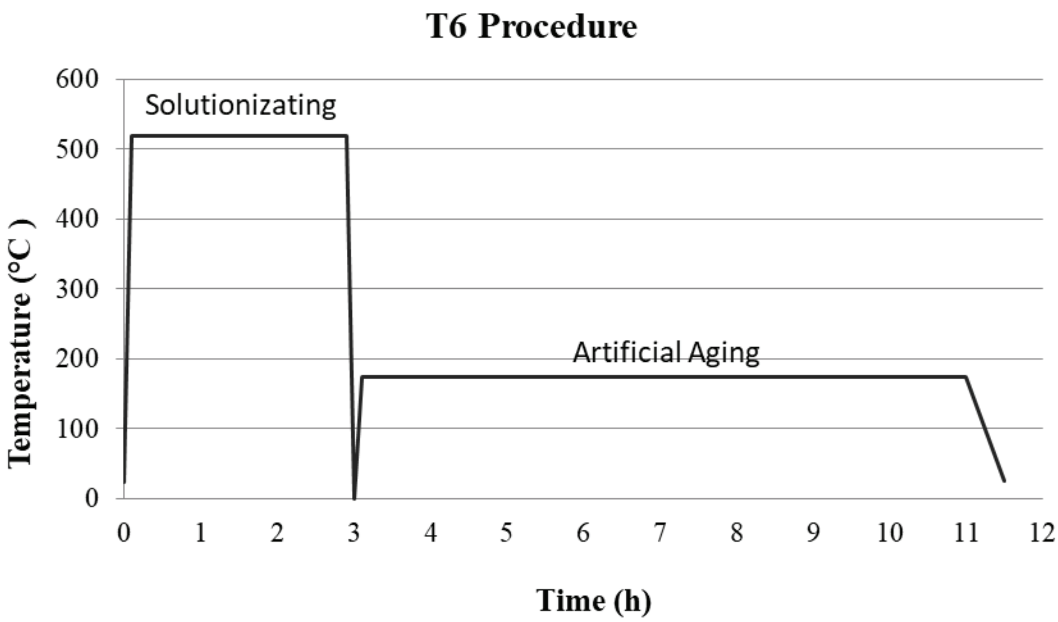

Figure 2. Temperature – Time condition chart of T6 heat treatment of 6063.

Characterization Methods

After the fabrication and heat treatment processes were completed, the densities were measured of five samples from each group using Archimedes' method to determine the porosity amount introducing the success of the infiltration process. Later, the hardness measurements were realized by micro-Vickers method using a diamond indenter with a 50 g load and 10 s dwelling time [27]. Five indentations were applied to five different samples from each group. Then, tensile and compression tests were realized according to ASTM standards and related reference documents [25, 26].

Moreover, XRD analysis of CNT/Al composites was carried out using a PANalytical X'Pert Pro X-Ray Diffractometer to determine the phases of composites. Furthermore, we investigated fracture surfaces of composites using JEOL JSM 6510-LV SEM.

RESULTS AND DISCUSSION

After the fabrication of the composites, the results of all characterization methods are given and discussed in this section. To understand the reinforcing effect of CNTs distributed in Al-CNT preform, all quantitative tests were applied to the Al powder preform containing samples as well.

Porosity

The porosity of samples was determined by the calculation of relative density from the difference of measured and theoretical densities of samples which are given in Table 1. Relative densities of the nanocomposites fabricated using powder metallurgy methods were around 95 to 99 % whereas vacuum infiltration was provided above 98 % in the literature [19, 20, 29, 30]. Relative density results achieved in the present work show us that the nanocomposite fabrication was quite successful and consistent. The first case underlying this issue is the high fluidity of selected matrix material due to the Mg content and proper casting temperature. Besides, the homogeneous distribution of CNT-Al powders in addition to the continuous and permeable pores achieved in the preform enables the capillary movement of the molten metal well enough. These pores were formed or enlarged by the evaporation of a wet binding agent in the preform shown with arrows in Figure 1c.

Table 1. Comparison of densities of as-cast and T6 heat-treated samples

Composition	Measured Density [g/cm^3]	Relative Density [%]
6063 Al	2.688 ±0.005	99.57
6063 Al-T6	2.687 ±0.005	99.52
Al/6063 Al	2.676 ±0.005	99.16
Al/6063 Al-T6	2.675 ±0.005	99.10
CNT+Al/6063 Al	2.651 ±0.009	98.33
CNT+Al/6063 Al-T6	2.650 ±0.010	98.30

Mechanical Properties

Compressive and tensile mechanical properties together with the microhardness values of samples were determined after the verification of successful fabrication of composites through porosity evaluations. Ultimate and 0.2% offset compressive strengths, ultimate tensile and 0.2% yield strengths, tensile elongation at failures, and micro hardness' average results with standard deviations are all shown in Table 2. It can be seen from the slight difference between 6063 Al matrix material and Al preform containing sample results that Al powder and colloidal silica addition has hardly any effect on the mechanical properties which was accepted to be negligible.

It can be easily noticed that the strengthening effect that CNTs lead from the compressive strength values. Approximately 150% increase in the compressive strength and 180% in the hardness was recorded by the addition of 0.5 wt.% CNT. However, we could not mention the same rate of enhancement in the tensile properties of these composites. It is known that the yield strength and hardness increase by the dispersion strengthening property of the CNTs [5, 19, 20, 31]. Especially, if the loading

Table 2. Comparison of mechanical properties of as-cast and T6 heat-treated samples

Composition	Ultimate Comp. Strength [MPa]	0.2% Offset Comp. Strength [MPa]	Ultimate Tensile Strength [MPa]	0.2% Offset Yield Strength [MPa]	Tensile Elongation at Failure [%]	Micro Hardness [HV]
6063 Al-T6	103.38 ±4.37	69.06 ±4.53	173.16 ±3.35	135.62 ±3.23	8.64 ±0.38	59.16 ±2.90
Al/6063 Al	78.52 ±3.68	55.50 ±3.67	92.76 ±3.40	50.03 ±1.99	15.30 ±1.23	28.10 ±1.05
Al/6063 Al-T6	126.34 ±5.49	88.42 ±4.02	184.34 ±3.69	146.22 ±6.10	7.50 ±0.85	65.74 ±2.13
CNT+Al/6063 Al	198.82 ±5.79	145.72 ±7.74	119.16 ±6.80	87.44 ±6.74	11.64 ±0.75	79.39 ±4.37
CNT+Al/6063 Al-T6	280.26 ±6.75	218.92 ±4.27	254.10 ±5.46	216.88 ±6.50	5.48 ±0.33	123.63 ±6.77

is compression, blocking of dislocations by CNTs and load transfer from matrix to randomly distributed reinforcement in various directions are seen as easier than the tensile load applications. Even though CNTs can be counted as nano-sized dispersions, they are also small fibers where they can carry the transferred axial load. Owing to the low ratio of nanotubes parallel to the tensile load, the amount of enhancement reduces very much. The modified Halpin-Tsai model, which is mathematically presented in Eq. 1 and 2, explains the prediction of yield strength enhancement of CNT-reinforced composites [32]:

$$\frac{\sigma_y}{\sigma_{y_m}} = \frac{1+c\eta V_f}{1-\eta V_f} \quad (1)$$

$$\eta = \frac{\alpha\frac{\sigma_{y_f}}{\sigma_{y_m}}-1}{\alpha\frac{\sigma_{y_f}}{\sigma_{y_m}}+c} \quad (2)$$

where σ_y represents the composite yield strength, V_f is the volume fraction of fiber, c is the measure of the reinforcement which depends on the boundary conditions, η is the coefficient calculated by using σ_{ym} and σ_{yf} which are the matrix and fiber yield strengths, respectively. However, there is an addition to this model, the orientation factor parameter α that is the measure of the randomness of the discontinuous fibers, which is around 0.16, for the lowest effect level. This modification numerically explains the dramatic drop in tensile strength when compared to compressive strength values. It is well-known that the major strengthening mechanisms are grain refinement (not too much in our study) and dispersion strengthening (including the Orowan mechanism) which blocks the movement of dislocations by dispersed phases and newly formed dislocations (work-hardening due to higher dislocation density) [33]. In addition to these, it is known that load transfer strengthening owing to a high CNT–matrix interface produces higher mechanical enhancement in composite materials. To provide this interface, the formation of some phases has an important effect, which is explained in the phase analysis section.

It is seen from Table 2 that the hardness results of composites are compatible with the compression test results. With 0.5 wt.% CNT addition above 200% increase was observed for the hardness of composites. The T6 heat treatment increases the hardness of our matrix from 25 HV to 59 HV, which is a fine hardening mechanism by the precipitations blocking the dislocations in the structure. The existence of CNT in the composite makes a difference and carries the hardness value to 123 HV after the heat treatment. As detailed in the phase analysis section, we cannot deny the effect of precipitation augmentation on the strengthening/hardening of composites; but at the same time, strengthening mechanisms we could address by means of CNT reinforcement carry these results to higher values.

Moreover, a significant coefficient of thermal expansion mismatch between CNTs and 6063 Al exists [31]. Therefore, prismatic punching of dislocations at the matrix-reinforcement interface would occur during the composite fabrication and the heat treatment process. For this reason, dislocation density becomes higher depending on the high surface area of CNT and this circumstance leads to work hardening of the matrix and results in strengthening.

Matrix Reinforcement Interface of Composites

It is known that the mechanical properties of composite is directly associated with the CNT-matrix interface. Transfer of the applied force from matrix to reinforcement becomes more efficient by a higher interfacial bonding. In case of a successful bonding, wetting of reinforcement would be higher and continuity of the adhesion should be saved during the loading of samples. One of the best methods to understand the effectiveness of the interface is to investigate the presence of CNT conditions at the end of the fracture. Thus, microstructural examinations of fractured surfaces were performed by SEM after the fabrication of the composites, and noteworthy images showing the strengthening or weakening mechanisms are given in Figure 3, respectively. Relative to the mechanical testing results it was assumed to observe short pull-outs and bridging of CNTs besides the uniform distributions of CNT distribution. Short pull-outs which are indicated by arrows and bridging of CNTs between fractured fractions of the matrix shown by a triangle in Figure 3(a) represents the interface between the matrix and carbon nanotubes is achieved effectively. To validate the shown morphologies are CNT a rapid point EDS analysis was carried out and a high concentration of carbon is shown in Figure 4 (a&b) for CNT1 and CNT2 indicated in Figure 3(a), respectively.

Unfortunately, a few defects are included in some sections of composites we fabricated which affects the load transfer negatively. In the composites where the homogeneous distribution of CNTs was slightly lost caused agglomeration of CNTs during the fabrication. The bundling of CNTs due to insufficient dispersion of them in preforms leads to reducing the strength of these composites. An example of these bundles is given in Figure 3(b) with an SEM image.

Phase Analysis

Figure 5 shows XRD analysis of matrix material, CNT reinforced composite before and after T6 heat treatment. Uniform precipitation of Mg_2Si occurred during the aging process due to the matrix hardening of 6063 Al alloy with both Mg and Si, and this precipitation affects the hardness of composites [34]. It is known that due to the higher oxidation tendency of Mg compared to Al, we usually encounter the reduction of Al_2O_3 and the formation of Mg-Al spinel ($MgAl_2O_4$) [35]. In our fabrication, elemental oxygen

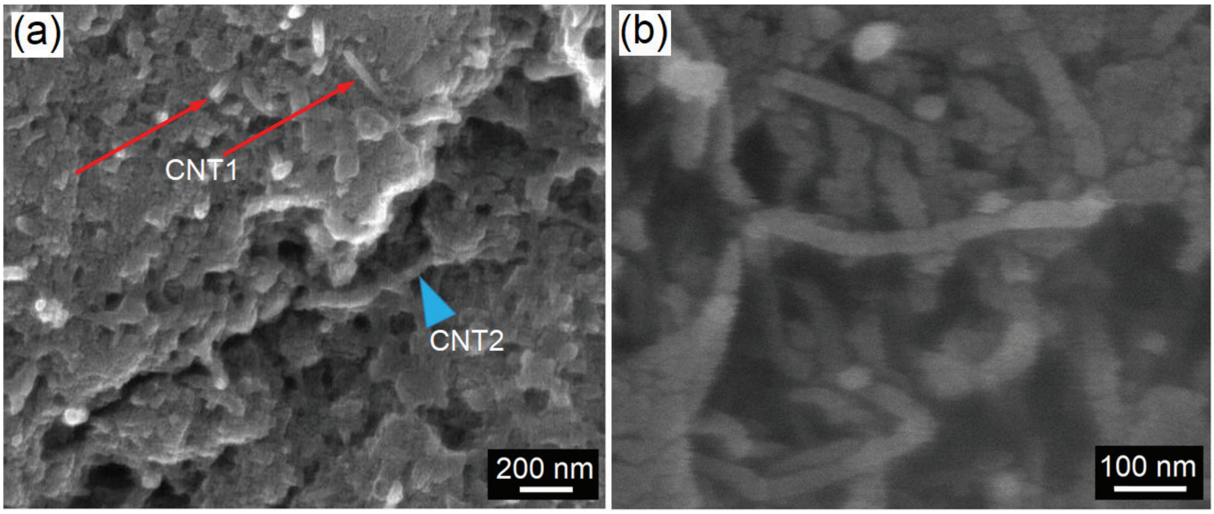

Figure 3. SEM images of CNT/Al composites showing (a) pulling-out (arrows) and bridging (triangle) of CNTs; and (b) bundles of CNTs agglomerated in the composite.

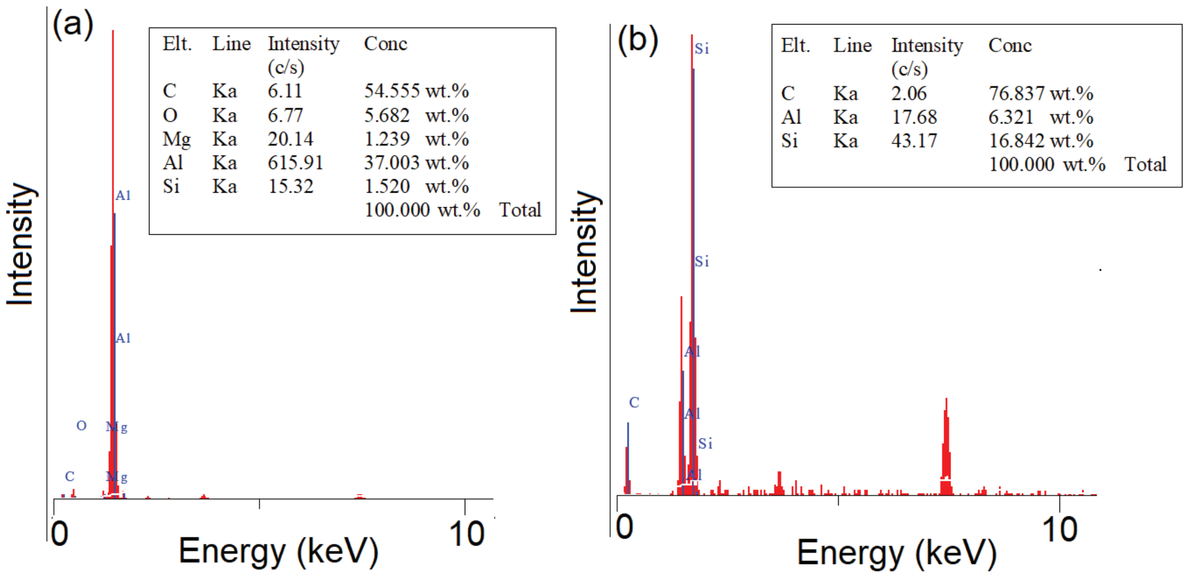

Figure 4. EDS analysis of (a) CNT1 and (b) CNT2.

coming from the beginning of the casting process and concentrated in the matrix leads to a trace quantity of $MgAl_2O_4$ formation.

In the CNT-reinforced aluminum alloy studies, one of the main problems is Mg element is trapped by oxygen rather than by CNTs; thus, the amount of Mg_2Si precipitates decreases which directly affects the aging of composites negatively [33]. Concerning the matter, Zhao et al. [36] reduced the oxygen content in CNT-reinforced Al matrix composites by a dry mixing process followed by a large deformation method. In addition, they increased the precipitation by adding an extra Mg element. In our recent study, CNT-containing preforms were prepared using Al powders, which would provide the formation of Al_2O_3 by trapping excessive oxygen and serve enhancing Mg_2Si precipitate formation. In the XRD analysis of our composites, the intensities of $MgAl_2O_4$ and Mg_2Si were gradually increased and a small amount of Al_2O_3 was observed. That means the air trapped in the preform during the casting process slowly released through the molten matrix metal and increased the formation of the mentioned phases with the help of more metal-oxygen interaction and a small quantity of alumina did not react with Mg and remained in the structure [33]. Eventually, the ascending amount of the Mg_2Si precipitates and oxides by the composite fabrication and heat treatment has a strengthening effect and this

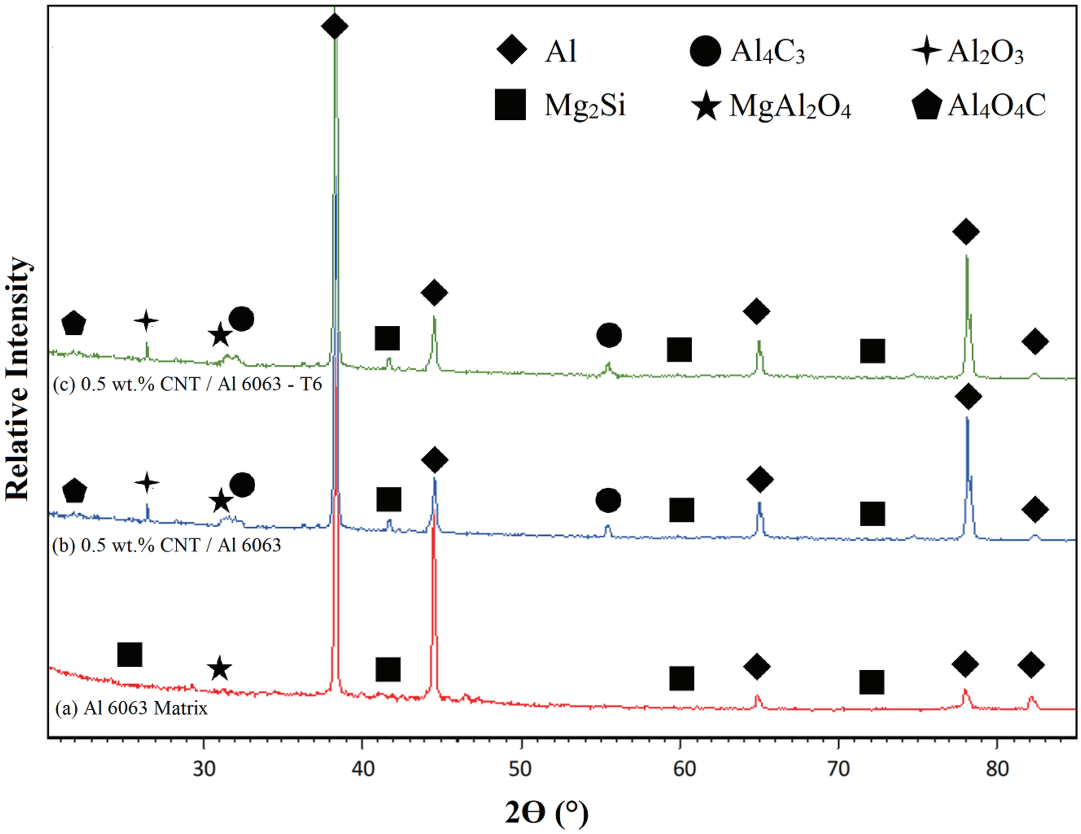

Figure 5. XRD analysis of CNT/6063 Al T6 composite material.

statement is congruent with the mechanical testing results of this study [37]. Another phase formation in the composite is Al_4C_3 and Al_4O_4C due to the CNT content of the preform. This formation is related to the reaction occurring just at the beginning of the casting process owing to the high casting temperature. Findings of some studies show that CNT – Al Matrix interfacial bonding and/or aging capability were improved by the presence of $MgAl_2O_4$ and Al_4C_3 which is consistent with our findings [38-40]. The existence of these phases causes the degradation of continuous Al_2O_3 layers around CNTs; thus, the negative effect of oxide was reduced, and interfacial bonding between the CNT and matrix increased. As a short note, Kondoh et al. [37] reported that Al_2MgC_2 has been produced by the reaction of Mg and CNTs. However, there is no detected Al_2MgC_2 peak in our XRD analysis.

CONCLUSION

CNT-reinforced 6063 Al matrix composites fabricated by vacuum-assisted preform infiltration method and were easily aged by T6 heat treatment. The effects of the CNT including preforms on the properties of composites can be listed as follows:

- The introduction of CNTs increase the mechanical properties owing to the extraordinary mechanical properties and achieved successful interface in our fabrication method. In the meantime, Al powders used in the preforms provide the formation of Al_2O_3 by trapping excessive oxygen and increased Mg_2Si precipitate formation which influences strengthening. Besides, CNT – 6063 Al Matrix interfacial bonding and aging capability were improved by the presence of $MgAl_2O_4$ and Al_4C_3 phases.
- Composites reached a hardness, tensile strength, and tensile yield strength of 123.6 HV, 254 MPa and 216 MPa, respectively where the elongation in failure slightly decreased to 5.48% where 8.64 is measured in 6063 Al – T6 matrix material. Remarkable increase in the compressive properties of composites were also achieved in this study.
- CNT reinforcement with or without aging increased the mechanical properties of composites owing to successful load transfer via high interfacial bonding despite a few amount of agglomerations found in the composite structure. Short pull-outs and bridging of CNTs observed via SEM examinations signify the efficiency of interface.

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AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE

Artificial intelligence was not used in the preparation of the article.

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