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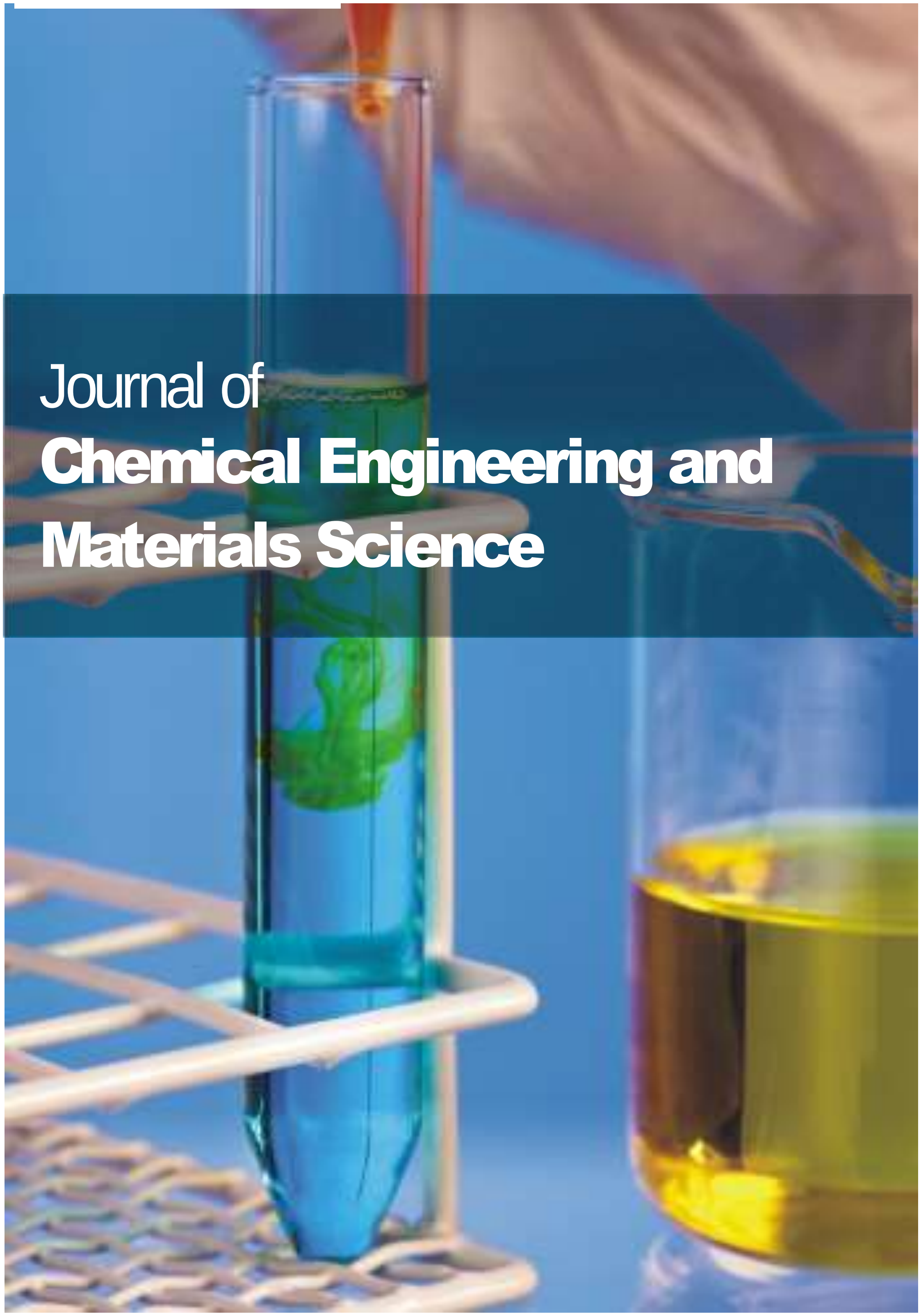

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Full Length Research Paper

Critical temperature estimation method for triple-walled carbon nanotubes (CNTs)/epoxy resin composite material

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The main objective of this research is to estimate the critical temperature for triple-walled carbon nanotubes (CNTs)/epoxy resin. Critical temperature in this study is the temperature that inter-laminar shear stress in interfaces of triple-walled CNTs and epoxy resin is the maximum value. At maximum inter-laminar shear stress, the stress concentration between triple-walled CNTs and epoxy resin is the maximum. This phenomenon may cause crack initiation, propagation and fracture in fibers/matrix interfaces. In the presented study, this temperature is derived by applying analytical method. The results of this research showed that maximum inter-laminar shear stress could occur at 85°C, which appears to be the critical temperature. The results of this research could be applied in any industry dealing with thermal stress and thermal cycles.

Key words: Critical temperature, triple-walled carbon nanotube (CNT), nanocomposites, thermal analysis, inter-laminar shear stress, coefficient of thermal expansion.

INTRODUCTION

In many industries, application of composite materials has become common such as aerospace, automobile, etc., (Geng et al., 2018). Furthermore, in recent years it is attempted to apply nanocomposites in many applications (Vilatela et al., 2012; Chae and Kumar 2006). In applying nanocomposites, instead of fibers, nanofibers are used. These nanofibers may be Single-Walled Carbon Nano-Tubes (SWCNTs), Multiple-Walled Carbon Nano-Tubes (MWCNTs), etc., (Shirasu et al., 2017). The application of nanofibers instead of fibers may increase the strength of nanocomposites. Furthermore, properties of CNTs such as electrical conductivity, mechanical and thermal, strength, and tensile and breaking mechanism have been investigated by many researchers (Ebbesen et al., 1996; Dai et al., 2008; Yu at al., 2000a; Yu et al., 2000b; Ruoff and Lorents, 1995).

"CNTs have been attracting much interest as a candidate material for nano and microscale actuators, composites and electronic devices because of their superior electrical, thermal and mechanical properties. Electronic devices may experience high temperatures during manufacture and operation processes, which lead

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to differential thermal expansion and residual stresses in devices, and affects the device reliability. Because a coefficient of thermal expansion (CTE) varies inversely with a Young's modulus, CNTs are assumed to have small (even negative) CTEs. Thus, it is expected that the combination of CNTs with polymer and metal materials which have large CTEs, makes it possible to fabricate composites with zero CTE. However, in order to make decisions on fundamental material design of composites with CNTs for zero thermal expansion, the understanding of the temperature dependence of the CTE of the CNTs is essential" (Shirasu et al., 2017).

In 2014 (Anvari, 2014), it has been estimated that the degradation of Inter-Laminar Shear Strength (ILSS) of Unidirectional Carbon Fiber/Epoxy Composite (UCFEC) is one of the most important issues that will cause failure due to thermal cycles or thermal stress.

Additionally, in 2017 (Anvari, 2017), the investigation to discover the degradation reason for UCFEC due to thermal stress, continued. The results showed that one of the main reasons that cause deterioration in UCFEC due to thermal stress could be the mismatch of CTEs between fibers and epoxy. Because the mismatch between the CTEs of carbon fiber and epoxy seems to be effective to raise Inter-ILSs in UCFEC. The reason behind is the reverse behavior between carbon fiber and epoxy in longitudinal direction because the axial CTEs for carbon fiber is negative and for epoxy is positive.

With the conclusions made from the analytical study mentioned in previous paragraph and using the previous form of the thermal stress equation (Maheswari and Prasad, 2013), an equation developed by Anvari (2018) to derive the maximum Inter-Laminar Shear stress ($ILSs_{max}$) for Unidirectional Fibers/Matrix Composites (UFMC). This relation is as follows:

$$ILSs_{max}=\Delta\alpha_{A.max}.\Delta T.G_{max}. \qquad (1)$$

Furthermore, in applying analytical method to obtain new equations in order to estimate the mechanical properties of materials, "friction coefficient variation with sliding velocity in copper with copper contact" is presented by Anvari (2016), "frictional effect on stress and displacement fields in contact region" is submitted by Adibnazari and Anvari (2017) and "cycle numbers to failure for magnesium and its alloys in human body fluid" is provided by Alijani and Anvari (2018).

Nevertheless, it appears that by increasing the application of nanocomposites in different industries such as aerospace, analysis in order to derive the mechanical properties of these materials exposed to different environments such as space environment is required. A great example of this application is satellite that rotates around the earth. As it rotates around the earth, it passes through the sun illumination and earth's shadow that are extremely hot and cold, respectively (Park et al., 2012).

Consequently, a precise thermal analysis of nanocomposites exposed to hot and cold temperatures seems necessary. Thermal analysis will provide data to calculate the $ILSs_{max}$ of the nanocomposite. It appears that as the value of $ILSs_{max}$ increases, the probability of inter-laminar de-bonding in UFMC increases. $ILSs_{max}$ is highly proportional to the mismatch of CTEs between the nanofibers and epoxy matrix. There are numbers of studies related to the research about CTEs of many fiber and epoxy materials (Yang and Qu, 2012; Deng et al., 2018). Nevertheless, it appears that there is no research regarding the thermal analysis of TWCNT/Epoxy Composite (TWCNTEC).

In the presented research, with applying a new analytical method with Equation 1 (Anvari, 2018) and using experimental results (Shirasu et al., 2017), it is attempted to derive the critical temperature for TWCNTEC that result in the highest $ILSs_{max}$. Detecting the highest $ILSs_{max}$ can be used in order to avoid the critical temperature because it might cause crack initiation, propagation, and fracture for TWCNTEC in nanofiber/matrix interfaces.

EXPERIMENTAL PROCEDURES

"Spinnable TWCNT arrays were obtained by chemical vapor deposition using C_2H_2 and $FeCl_2$ as the base material and the catalyst, respectively. The diameter of the TWCNTs was measured using a transmission electron microscope (TEM, JEOL JEM-2100F, Japan). A partially cured epoxy resin (B-stage epoxy) with a release paper was used as the starting material, where the epoxy resin comprised bisphenol-A type epoxy, novolac-type epoxy, and an aromatic diamine curing agent. The epoxy resin was then impregnated into the TWCNT monolithic sheet at 90°C for 3 min between the steel plates of a hot press (AS ONE AH-4015, Japan). After peeling off the release paper from the TWCNT sheet now impregnated with the epoxy resin (prepreg sheet), the prepreg sheet was cured at 130°C for 1.5 h at a pressure of 1 MPa using the hot press, forming a film specimen" (Shirasu et al., 2017).

"The TWCNT monolithic sheets were drawn out of the TWCNT arrays and wound onto a rotating plate. Raman scattering spectroscopy (JASCO, NRS-5100, Japan) was used to analyze the vibrational modes of TWCNTs. The measurements were carried out at room temperature under ambient conditions using an argon ion laser with an excitation wavelength of 532 nm. Aligned TWCNT/epoxy composites were prepared by a hotmelt prepreg method, wherein the MWCNT monolithic sheet was pre-impregnated with an epoxy matrix" (Shirasu et al., 2017).

"A stacked TWCNT monolithic sheet about 20 mm wide and about 45 mm in length was placed on a polytetrafluoroethylene sheet and covered with the epoxy resin film with the release paper. The weight fraction of TWCNTs was calculated from the masses of the TWCNT sheets (before impregnation of the epoxy) and the composites. Having obtained the weight fraction of the composites, TWCNT volume fractions were determined assuming that the densities of the epoxy and TWCNTs were 1.2 and 2.0 g/cm^3. The TWCNT volume fraction was adjusted by changing the number of layers of TWCNT monolithic sheets and the values for the samples prepared were found to be in the range of 9 to 31 vol.%. Because the areal density of the TWCNT monolithic sheet per layer varies according to the TWCNT diameter and each batch of MWCNT arrays (1 to 2 TWCNT arrays have been used to prepare each TWCNT monolithic sheets), it is difficult to control in advance the TWCNT volume fraction of the composites for each type of TWCNTs. Microstructural observations using a scanning electron

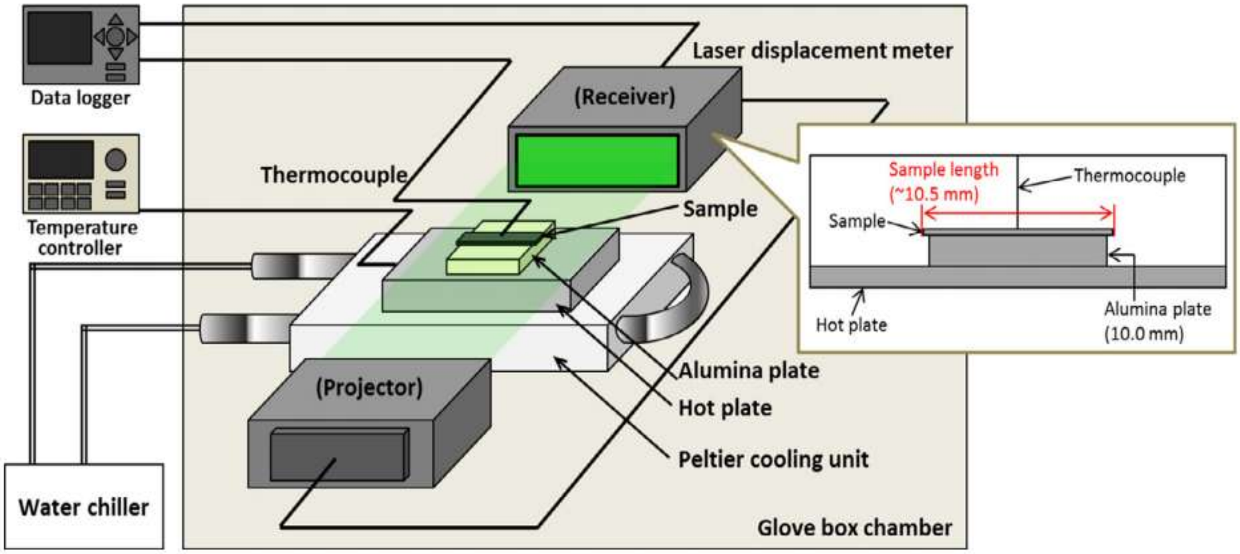

Figure 1. Schematic illustration of experimental setup for determining CTE of TWCNT/epoxy composite (Shirasu et al., 2017).

microscope (SEM , JEOL JSM6510, Japan) and TEM indicated that the epoxy resin well penetrated between TWCNTs and that densely aligned TWCNT composites were successfully fabricated using this processing method with only a limited amount of pores" (Shirasu et al., 2017).

"The CTE of the aligned TWCNT/epoxy composite films was measured using the experimental setup. The dimensions of the composite film sample were 10.5 mm × 2 mm × 0.019 - 0.030 mm (length × width × thickness), where the length is measured along the direction parallel to the MWCNT alignment. The composite film sample and an alumina plate of width 10.0 mm (used as a specimen holder) were placed on a hot plate. The assembly was further set on a Peltier cooling stage. The temperature was controlled by using the hot plate and Peltier cooling apparatus, and the sample temperature was measured by a thermocouple placed in contact with the composite film sample. The length change was then measured by a laser displacement meter (Keyence LS-7600, Japan) over the temperature range -10 to 90°C and with a heating rate of 10°C/min" (Shirasu et al., 2017). Figure 1 (Shirasu et al., 2017) shows the schematic illustration of experimental setup for determining CTE of TWCNT/epoxy composite. Furthermore, in Figure 2 (Shirasu et al., 2017) illustrates TEM images of the (a and b) 25-MWCNTs and (c and d) 41-MWCNTs used in this study.

Critical temperature derivation method

In order to derive the critical temperature for TWCNTE, it seems necessary to first derive the temperature at which the mismatch of CTEs between TWCNT and epoxy resin is maximum. In this study, the mismatch of CTEs between TWCNT and epoxy is indicated by $\Delta\alpha_A$. Thus, in the presented research, the goal is to find the temperature at which $\Delta\alpha_{A.max}$ occurs. Table 1 and Figure 3 (Shirasu et al., 2017) show CTEs for epoxy resin at different temperatures.

With the analysis of values given in Table 1 and performing calculations based on Equation 1, $\Delta\alpha_{max}$ can be derived. The temperature at which $\Delta\alpha_{A.max}$ occurs is called critical temperature because it appears that at this temperature ILSs between TWCNTs and epoxy has the highest value. According to conclusions made by the study performed in 2014 (Anvari, 2014), ILSs seems to be the main cause of fracture and delamination of UFMCs due to thermal stress and thermal cycles. Hence, deriving the critical temperature is of high significance.

RESULTS AND DISCUSSION

In performing the thermal analysis, in order to derive the critical temperature, it is important to notice that the amount of CTE for TWCNT in the temperature range -5 to 85°C is approximately constant and is equal to -2.3 e-6 (Shirasu et al., 2017). On the other hand, as it is indicated in Table 1, the CTE for epoxy changes with the variation of temperature (Shirasu et al., 2017). In Table 2 and Figure 4, the difference of axial CTEs between TWCNT and epoxy resin ($\Delta\alpha_A$) at temperature range between -5 and 85°C are indicated. It seems that at 85°C, $\Delta\alpha_A$ has the maximum value. Thus, based on Equation 1, it appears that $ILSs_{max}$ is also maximum.

$$ILSs_{max}=\Delta\alpha_{A.max}.\Delta T.G_{max} \qquad (1)$$

It is important to notice that in Equation 1, $\Delta\alpha_{A.max}$ is equal to the maximum of "axial CTE of epoxy (α_{epoxy}) minus the axial CTE for TWCNT (α_{TWCNT}). This relation is as follows.

$$\Delta\alpha_{A.max}=\alpha_{epoxy}-\alpha_{TWCNT} \qquad (2)$$

Because α_{epoxy} is a positive value and the α_{TWCNT} is equal to -2.3 e-6 (1/°C), which is a negative value, the amount of $\Delta\alpha_A$ will become a positive value and is indicated in Table 2.

In Equation 1, ΔT is the temperature difference between the crack-free temperature of TWCNTEC and

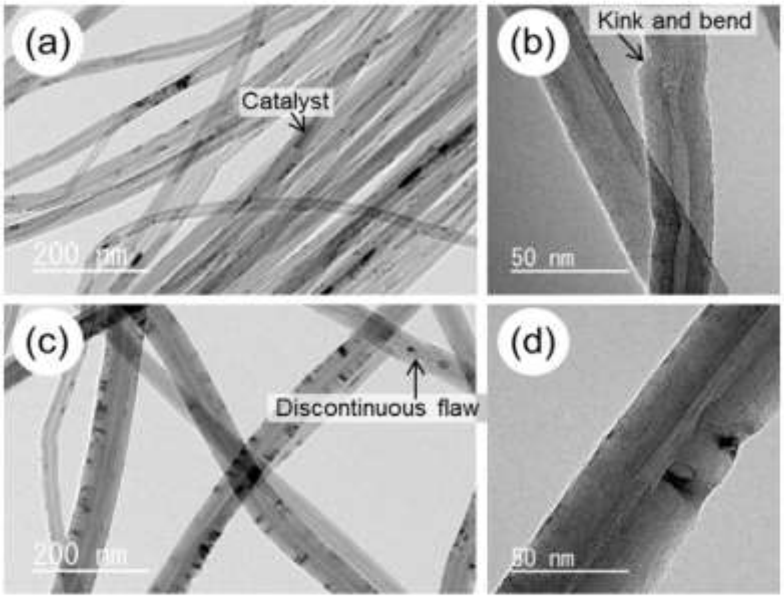

Figure 2. TEM images of the (a and b) 25-MWCNTs and (c and d) 41-MWCNTs used in this study (Shirasu et al., 2017).

Table 1. CTEs for epoxy resin at temperatures -5 to 85 °C (Shirasu et al., 2017).

S/N	Temperature (°C)	CTE (1/°C e^{-5})
1	-5	4.60
2	0	4.95
3	5	5.30
4	10	4.60
5	15	4.00
6	20	3.70
7	25	3.50
8	30	3.53
9	35	3.80
10	40	4.15
11	45	4.30
12	50	4.50
13	55	4.70
14	60	4.90
15	65	5.00
16	70	5.20
17	75	5.40
18	80	5.60
19	85	5.70

the environment temperature. G_{max} is the maximum shear modulus between TWCNT and epoxy. Because G_{max} is approximately constant at this range of temperature (-5 to 85°C), it does not affect on the variation of the ILSs in this temperature range.

In this experiment, at 85°C, the value of $\Delta\alpha_A$ is the maximum amount. As a result, it seems that ILSs has the highest value at this temperature. In the temperature range of -5 to 85°C, the axial CTE of TWCNT is about -$0.23e^{-5}$ (Shirasu et al., 2017) which is a negative value. On the other hand, the axial CTE for epoxy resin is the positive value. According to this data, as the TWCNTE is

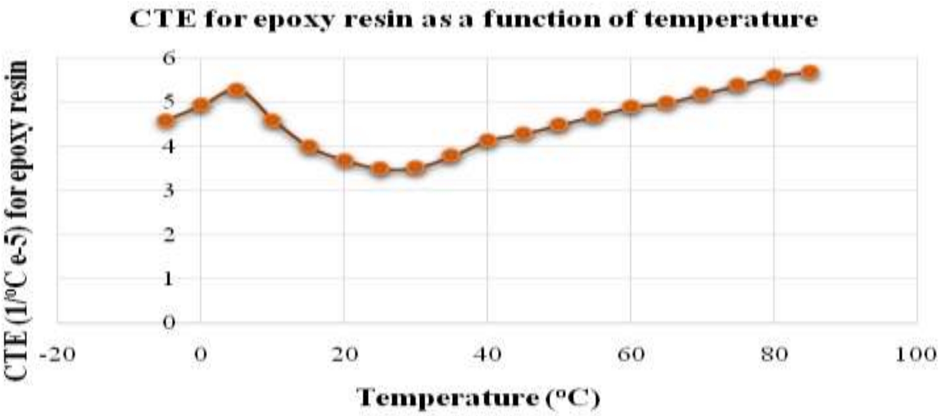

Figure 3. CTEs for epoxy resin at temperatures -5 to 85°C (Shirasu et al., 2017).

Table 2. Difference of CTEs between TWCNT and epoxy resin ($\Delta\alpha_A$) at temperature range -5 and 85°C

S/N	Temperature (°C)	Δα (1/°C e^{-5})
1	-5	4.83
2	0	5.18
3	5	5.53
4	10	4.83
5	15	4.23
6	20	3.93
7	25	3.73
8	30	3.76
9	35	4.03
10	40	4.38
11	45	4.53
12	50	4.73
13	55	4.93
14	60	5.13
15	65	5.23
16	70	5.43
17	75	5.63
18	80	5.83
19	85	5.93

heating, TWCNT is contracting in axial direction. Inversely, in this state, the epoxy resin is expanding in axial direction. Because these two materials are bonded together in TWCNTE composite, in this state and in axial direction of the composite, TWCNT is withstanding tensile stress while epoxy resin is withstanding compressive stress. In the state of cooling, the reverse occurs. It means that in axial direction, the TWCNT is withstanding compressive stress and epoxy resin is withstanding tensile stress. This stress is the ILSs in the interface of TWCNT and epoxy. The reverse behavior of the two components of the composite due to temperature variation, results in breaking bonds between them. Breaking bonds will cause crack initiation, propagation and fracture in the direction along the TWCNT axial direction.

According to the results of this study, maximum breaking bonds between TWCNT and epoxy may occurs at 85°C. It means that the probability of crack initiation, propagation and de-bonding of the TWCNTE composite is maximum at this temperature. Additionally, at temperatures around 25°C, the value for difference of CTEs between TWCNT and epoxy resin is minimum. The reason behind might be due to the reason that these temperatures are close to the ambient temperature which may be equal to stress-free temperature of the TWCNTE.

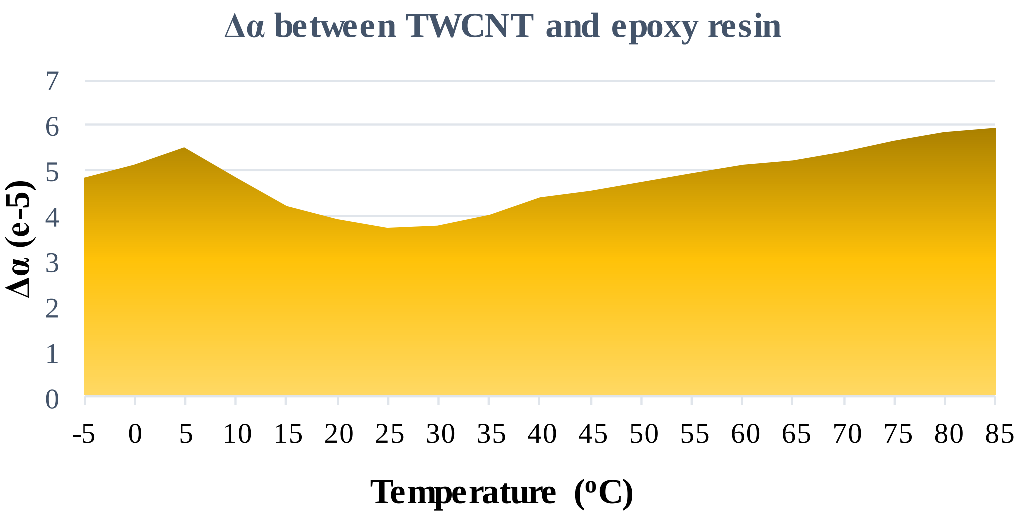

Figure 4. Difference of CTEs between TWCNT and epoxy resin ($\Delta\alpha_A$) at temperature range -5 and 85°C.

It means that around these temperatures the TWCNTE is stress-free and IlSs can be the minimum value. Moreover, as it is illustrated in Figure 4, as the temperature of the sample increases or decreases from stress-free temperature (23°C) to 85°C or -5°C, the mismatch of CTEs between TWCNT and epoxy increases. This behavior may represent that at these temperatures the reverse behavior in axial direction between TWCNT and epoxy increases.

For the future work, in order to have a satisfactory thermal analysis of TWCNTE composite material, it is recommended to perform thermal experiments to derive the CTEs for both TWCNT and epoxy in temperature range between and equal to -183 and 120°C. This temperature range is recommended because it appears that in Titan, Saturn’s moon the minimum temperature is -183°C (Lorenz and Mitton 2002) and in Low Earth Orbit, the maximum temperature is 120°C (Park et al., 2012). The recommended temperature range for thermal experiment on TWCNT and epoxy will have great results for evaluating the ILSs and thermal fatigue life of TWCNTEC for possible upcoming space mission to Titan (Regius, 2016).

Conclusions

In the presented study, by applying experimental results and using analytical method with Equation 1, the critical temperature for TWCNTE composite is estimated. TWCNTE composite is one of the nanocomposites that is currently used in many applications such as aerospace industry due to excellent mechanical properties such as high strength and lightweight. Because these nanocomposite materials in space environment are exposed to thermal stress and thermal cycles, critical temperature analysis for this nanocomposite material seems necessary. In this study, thermal analysis on TWCNT and epoxy is performed. Results of this analysis showed that the mismatch of the CTEs between TWCNT and epoxy is the maximum value in 85°C. As a result, at this temperature, the risk of de-bonding between TWCNT and epoxy is the highest value. The results also have shown that as the temperature of the TWCNTE increases or decreases from the stress-free temperature, the mismatch between the CTEs for TWCNT and epoxy increases. The reason behind might be due to the highest axial thermal stability between TWCNT and epoxy at stress-free or crack-free temperature.

CONFLICT OF INTERESTS

The author has not declared any conflict of interests.

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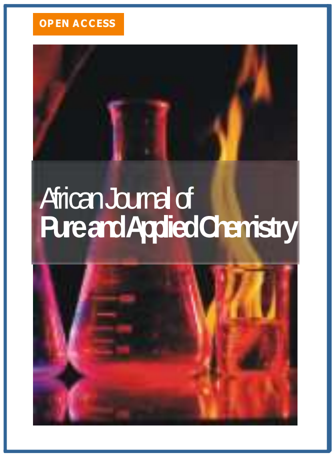

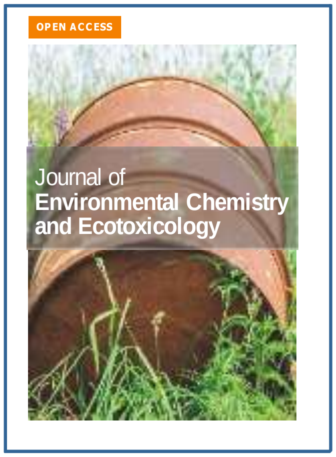

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