The Effect of Elevated Temperature on the Mechanical and Morphological Properties of Aramid and Carbon Fiber Reinforced Epoxy/MWCNT

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Abstract. Aramid and carbon Fiber-reinforced polymer (FRP) has been widely used in automotive and aerospace industries, due to high-strength and low-density. The development of FRP composite material is needed to achieve higher mechanical properties of FRP in aerospace fabrication, especially for the performance of FRP composite exposed to elevated temperatures. This study experimentally examined the effects of elevated temperature on mechanical and morphological properties of aramid and carbon fiber-reinforced epoxy/MWCNT. The tensile test was conducted at room-temperature, 50°C, and 120°C. DGEBA and MWCNT dispersed and combined of aramid and carbon fiber by vacuum infusion method, and characterization by ultimate tensile machine (UTM) within heating by thermostatic chamber, and morphological test by scanning electron microscope, and optical microscope. The result showed incorporating MWCNT on CFRP composites improved the ultimate tensile strength, elongation at fracture, and elastic modulus of 13.88%, 11.46%, and 7.5%, respectively, at 120°C tensile test temperature. CFRP/MWCNT showed a tensile strength increase of 18.26% and 34.08% at 50°C and 120°C tensile test temperatures, respectively. Aramid fiber reinforced epoxy/MWCNT showed a tensile strength decrease of 10.71% and 12.49% at 50°C and 120°C. Morphologically, CFRP showed a great brittle failure than aramid fiber-reinforced epoxy/MWCNT. Moreover, MWCNT reduces debonding of CFRP at elevated temperatures. Therefore, this study is expected to be a reference for composite materials used and replace the metal domination in rocket components for the preparation of manufacturing rocket components at Research Organization for Aeronautics and Space, Deputy for Research, and Innovation Infrastructure.

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1 Introduction

In recent years, Fiber-reinforced polymer (FRP) has been widely used in the industrial sector, especially the automotive and aerospace industries [1]–[3]. This is due to their high strength by low density, compared to other materials, like metals and ceramics [4]. The development of FRP composite material takes more consideration to achieve higher mechanical properties in aerospace fabrication. Especially for the performance of FRP composite when exposed to elevated temperature. One of the reasons is low glass transition temperature (Tg) of the polymer. In recent studies about polymers, epoxy resin has been widely used to investigate and improve the mechanical properties of FRP composites [3], [5]–[8]. When CFRP perform above the glass transition temperature, they show a significant reduction of strength and stiffness, even to the point of composites being damaged [9]. The resistance of composite to temperature depends on the ratio and bond of epoxy and fiber. When the composite exposes to elevated temperature, they reduce the bond of epoxy and fiber [10].

Many studies have been experimentally carried out to investigate the effect of elevated temperature on the mechanical properties of FRP composites [11]–[14]. Mechanical properties of FRP are determined by compressive test, torsion test, flexural test, fatigue test, and tensile test. Among all characterization, tensile test is one of primary mechanical characterizations of the FRP composites. Besides, fiber type and orientation determine the properties of composite. Aramid fiber is widely used in aerospace components due to its high defect distribution [15]–[17]. While carbon fiber has great tensile strength and elastic modulus [18]–[20]. In other research, unidirectional fiber orientation takes more consideration because of their high-strength property [20]–[24]. Therefore, this investigation focused on tensile properties of unidirectional aramid and carbon FRP composites.

Previews study [9] investigates the tensile behavior of CFRP composite specimens at elevated temperatures ranging from 25 to 55°C. Unidirectional carbon fiber and high-tensile strength epoxy resin as an adhesive material were used. The sample was produced by 10 layers of carbon fiber and combining resin using vacuum resin infusion method. A tensile test was carried out specimen accordance with ASTM D3039. The test results showed that CFRP lost about 2.10 % of tensile strength and 17.65 % of elongation at break at highest temperature evaluated. In higher temperatures [19], the effect of elevated temperature on mechanical properties of CFRP, ranging from 25oC to 300°C was investigated. Carbon fiber and epoxy resin as an adhesive material combined using hand lay-up method and curing process in a week. The specimen was formed in accordance with ASTM D3039/D 2029-08. The result showed that CFRP lost about 45.79 % of its tensile strength and 61.24 % of elastic modulus at a critical temperature of 300°C. In an additional study [25], tensile properties of CFRP composites at elevated temperature ranging from 16 to 200°C was investigated. They used high-strength carbon fiber and epoxy resin (FR-E3P) as an adhesive material and dry carbon as a variation. The test result showed CFRP lost about 67.81 % and 48.12 % of their tensile strength at 200°C, respectively.

Lately, many studies have shown a good improvement in the mechanical properties of CFRP by adding nanofiller into the adhesive matrix [26]–[28]. In a preview study [27], the effect of incorporating multi-walled carbon nanotubes (MWCNT) to enhance the mechanical properties of CFRP was investigated. The concentration of 0.5 wt.% MWCNT was used. A tensile test was carried out to determine the transverse mechanical properties. The test result showed incorporating MWCNT increased tensile strength and elongation at break of CFRP by 11.82 % and 20.59 %, respectively.

The FRP composite is highly useful for aerospace components due to its low density and high strength, but it showed a reduced performance when exposed to elevated temperatures. Thus, a study of the effect of elevated temperature on mechanical properties of FRP is important. In this study, the effect of elevated temperature and MWCNT on aramid and carbon fiber-reinforced polymer was experimentally investigated. The object of this paper is (i) to investigate the effect of adding MWCNT to mechanical properties of CFRP composites, (ii) to investigate the effect of elevated temperature on mechanical properties of aramid and carbon fiber reinforced epoxy/MWCNT, (iii) to investigate the morphological characterization of fracture surface composites after tensile test at elevated temperature.

2 Method

2.1 Material

In this study, Unidirectional aramid (Kevlar 49) and carbon fiber (T700 12k) were used as the primary material from DuPont Co. and Toray Co. (United State). DGABA (2,2-Bis(4-glycidyloxyphenyl) propane Epoxide A) type epoxy adhesive material (Araldite LY-5052-1) and curing agent (Aradur LY-5052-1) from Huntsman Co. (United States). Multi-Walled Carbon Nanotubes (MWCNT) from XFNANO Material Tech Co. (China). Analytical acetone solvent from Merck (German). MWCNT has a diameter of 20-40 nm, length 10-30 μm, and purity about 90 %. The resin epoxy system consisted of DGBA and curing agent. They showed high potential for manufacturing the composites with wet lay-up, RTM (Resin Transfer molding), pressure molding, and filament winding methods. The ultimate tensile strength (UTS) and modulus of epoxy system are 49-71 MPa and 3350-3550 MPa, respectively [29].

2.2 Sample Preparation

Dispersion Method and Sample Preparation. Ultrasonic used to improve the dispersion of MWCNT nano-filler in an epoxy resin [30]. The MWCNT was initially added to the acetone for good dispersion on MWCNT [31]. The MWCNT/acetone in a ratio of (0.5:10) wt.% was sonicated by bath-ultrasonic for 1 hour at a frequency about 40 kHz at room temperature. After the sonication of MWCNT/acetone, the DGEBA epoxy resin was added to the MWCNT/acetone mixture and mixed by bath ultrasonic for 1 hour at a frequency of 40 kHz. Then, the MWCNT/epoxy/acetone mixture was vacuumed in the oven to degassing process for 4 hours at 60°C, to remove the bubbles and vaporize the acetone in the mixture. After degassing, the hardener was added to the MWCNT/epoxy/acetone mixture and mixed using a glass rod for 5 min at room temperature. The matrix epoxy/hardener/MWCNT/acetone mixture in the ratio of (100:38:0.5:10) wt.%, respectively.

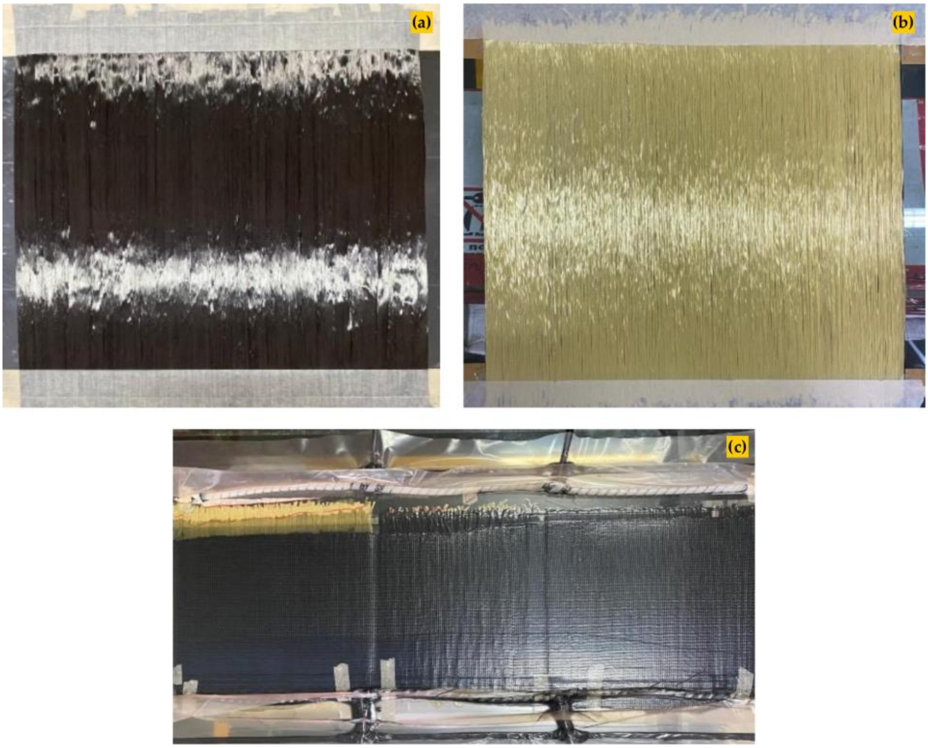

Fig. 1. The detail of sample preparation for (a) carbon laminates, (b) aramid laminates, (c) vacuum infusion method

Unidirectional carbon and aramid fiber yarn was cut about 30 cm length and arranged by hand lay-up method to dimension 30×27 cm (length × width) for a layer as presented in Fig. 1(a) and 1(b), respectively. Six aramid and carbon fiber layers fabricated the laminates of composite. The carbon and aramid laminates were infused by epoxy/MWCNT matrix by vacuum infusion method as presented in Fig. 1(c). Whereas carbon laminates infused by epoxy matrix were investigated for comparison. Before starting the vacuum infuse process, the assembling table is coated with a release agent (PMC, FMS, 700-NC, and wax) to avoid the composite adhering of the matrix to the assembling table. After vacuum infuse process, the sample was cured at RT (approximately 21.9 °C) for 48 hours.

Tensile test. The sample was cut by manual cutter according to ASTM D3039-1 for 0° unidirectional fiber orientation as presented in Fig. 2(a), which has dimensions (250×15×1) mm [32]. Fig. 2(b) shows the result of the sample.

Fig. 2. The detail of (a) ASTM D3039, and (b) sample after cut

Forty-five specimens fabricated to experimentally investigate the effect of adding MWCNT and elevated temperature of tensile properties on aramid and carbon fiber reinforced epoxy/MWCNT. According to ASTM D3039, five specimens were prepared for each test. The aramid and carbon FRP incorporating MWCNT was evaluated with elevated temperatures of room temperature (RT; approximately 21.9°C), 50°C, and 120°C. To distinguish their test specimen, the test specimen is named as shown in Table 1. The specimen was exposed to temperature in the thermostatic chamber. The thermostatic chamber has a heating capacity of up to 300 °C. The specimen was evaluated using the universal testing machine (UTM) under exposed temperature from a thermostatic chamber, which has a load capacity of 50 kN to perform the tensile test. Fig. 3(a) presents thermostatic chamber and Fig. 3(b) presents the universal testing machine used in this study.

Table 1. Variation and name of specimen

Material	Temperature test (°C)	Name
Carbon Fiber Reinforced Polymer (CFRP)	21.9 (RT)	CE-RT
	50	CE-50
	120	CE-120
Carbon Fiber Reinforced Epoxy/MWCNT	21.9 (RT)	CEM-RT
	50	CEM-50
	120	CEM-120
Aramid Fiber Reinforced Epoxy/MWCNT	21.9 (RT)	AEM-RT
	50	AEM-50
	120	AEM-120

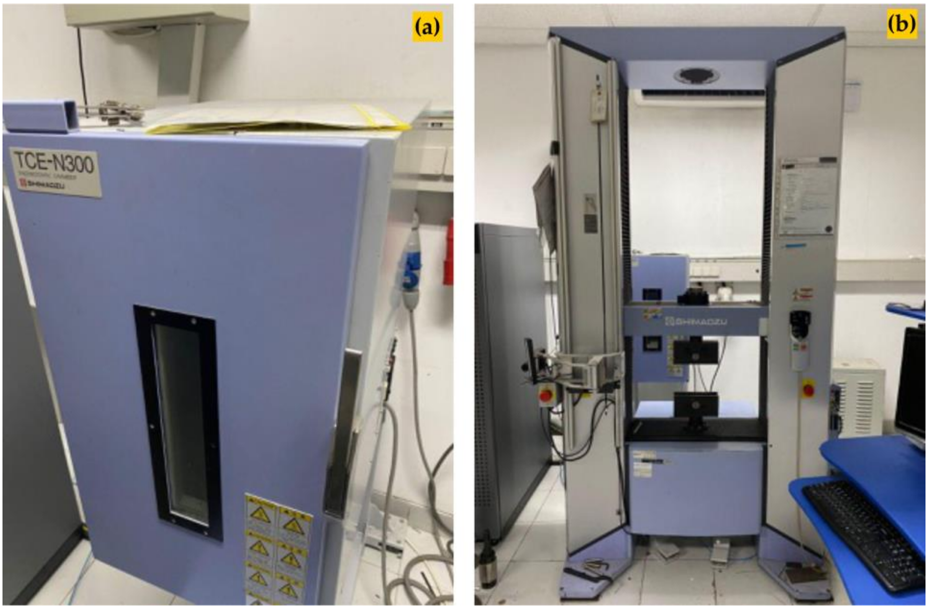

Fig. 3. Tensile machine: a) Furnace for exposing specimen by temperature; (b) universal tensile test machine

3 Result and Discussions

3.1 Tensile Properties

According to ASTM D3039, five specimens were evaluated for the variation of temperature. The result of five specimens tested at each variation showed as averages in Fig. 4. The stress-strain curve of aramid and carbon fiber reinforced epoxy/MWCNT composites and pure CFRP at elevated temperature of room temperature (RT; approximately 21.9 °C), 50 °C, and 120 °C. Fig. 4 shows aramid and carbon fiber reinforced epoxy/MWCNT exhibited brittle failure mode. Likewise, pure CFRP composites (CE) displayed a similar failure mode with a sudden drop. This means that adding MWCNT to FRP composites did not affect the failure mode [12]. Also, stress-strain curve shows a linear behavior from the starting point to the point where the specimen fractures. The stress-strain curve also shows that the carbon fiber reinforced epoxy/MWCNT composites (CEM) have the highest ultimate tensile strength and aramid fiber reinforced epoxy/MWCNT composites (AEM) show the highest strain at break than another composite. In this study, the elastic modulus is determined by dividing the stress at 0.05-0.25 of strain. Also, the other mechanical parameter of ultimate tensile strength, strain at break, and their standard deviation were computed and presented in Table 2.

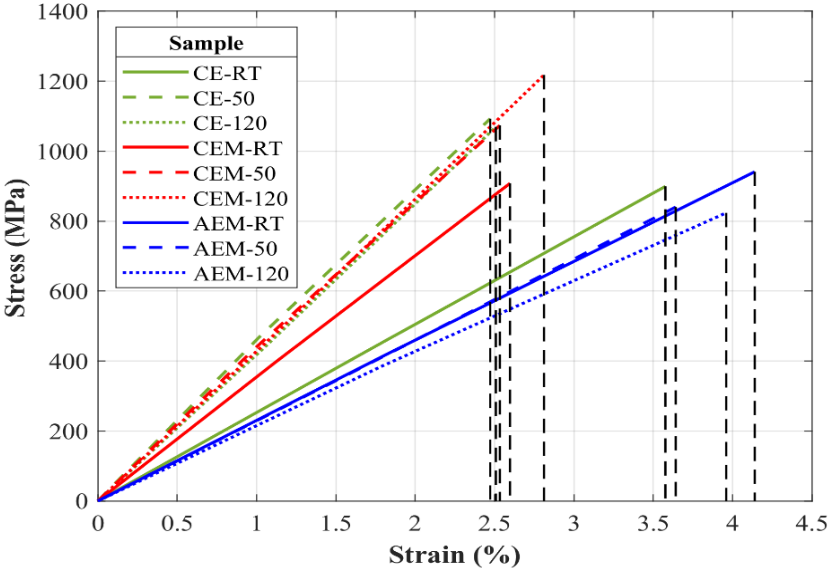

Fig. 4. Stress – strain curve of CFRP with and without MWCNT at elevated temperatures

Table 2. Tensile test result of CFRP composites with and without MWCNT and aramid FRP with MWCNT

Sample	**Elastic Modulus**	**Ultimate Tensile Strength**	**Strain at Break**
Unit	**(GPa)**	**(MPa)**	**(%)**
CE-RT	39.91 ± 6.63	899.39 ± 92.29	4.03 ± 0.72
CE-50	45.53 ± 4.91	1092.6 ± 52.42	2.91 ± 0.07
CE-120	40.17 ± 1.63	1069.2 ± 88.64	2.92 ± 0.13
CEM-RT	35.81 ± 4.33	908.10 ± 26.16	2.95 ± 0.31
CEM-50	45.13 ± 5.39	1073.9 ± 115.3	3.08 ± 0.12
CEM-120	43.43 ± 5.60	1217.6 ± 88.26	3.27 ± 0.10
AEM-RT	23.71 ± 3.64	941.10 ± 74.08	4.57 ± 0.20
AEM-50	24.21 ± 2.94	840.35 ± 126.1	4.19 ± 0.44
AEM-120	20.50 ± 1.53	823.60 ± 70.48	4.47 ± 0.36

Fig. 5(b) shown the elevated temperature affect the ultimate tensile strength of CFRP composites. Generally, the elevated temperature increasing cross-link of matrix polymer, this may be explain due to molecular mobility formed the better chain of polymer in elevated temperature [33]. At room temperature tensile test, the MWCNT existence increase the tensile strength by 0.97% of CFRP composites. The increasement of ultimate tensile strength depends on dispersion process of MWCNT in epoxy matrix. Well dispersion of MWCNT create better interlocking performance for matrix to the fiber, which very important to reduce the development of cracks in matrix and improve the load-carrying capacity of CFRP composites during tensile test [34]. Aramid fiber reinforced epoxy/MWCNT show a higher ultimate tensile strength at room temperature tensile test by 3.63% of carbon fiber reinforced epoxy MWCNT. At temperature test of 50°C, MWCNT existence shows an ultimate tensile strength that is not significantly different. Aramid reinforced epoxy/MWCNT show a reduction ultimate tensile strength by 10.71% at 50°C tensile test, due to smooth fiber surface involve poor interface interaction during mechanical load in elevated temperature [35]. Yet, the ultimate tensile strength of carbon fiber reinforced epoxy/MWCNT and pure CFRP increase by 18.26 % and 14.30 %, respectively, from temperature test of room temperature, due to the temperature of 50 °C make a great cross-link of polymer before the polymer being softening [36]. At the temperature test of 120 °C, the ultimate tensile strength of pure CFRP decreases due to glass transition temperature (T_g) of epoxy resin ranging 52-55°C [29]. Meanwhile, ultimate tensile strength of carbon fiber reinforced epoxy/MWCNT increases due to the MWCNT build better cross-link of polymer in elevated temperature [36]. Despite the softening of the epoxy resin, the mechanical properties of MWCNT did not decrease, and the MWCNT hold the cross-link of matrix increasingly [13]. Therefore, the glass transition temperature (T_g) of epoxy/MWCNT matrix was increased [37]. Basically, incorporating MWCNT on CFRP could improve the resilience to matrix deformation [20], [21], [38], [39]. Yet, aramid fiber-reinforced epoxy/MWCNT decrease at 120 °C due to a low bond matrix to the fiber, despite the polymer having a great cross-link [40].

The elevated temperature affects strain at break of composites, as shown in Fig. 5(c). Aramid fiber-reinforced epoxy/MWCNT shows the highest strain at break than carbon fiber composites. This may be explained due to aramid fiber having a great elongation than carbon fiber [41]. The MWCNT affects strain at break of carbon fiber composites, as shown at 120 °C tensile test, carbon fiber reinforced epoxy/MWCNT has a higher strain due to the elevated temperature making the MWCNT increase cross-link of polymer and bounding of fiber and matrix up to the composites being fracture [12]. Each composite shows a higher strain at break at room temperature tensile test, the one reason is the curing behavior. The curing process determines the cross-link of the matrix, and epoxy/MWCNT matrix had a low cross-link rate in low heat treatment, while faster heat rate offer less time to cross-link [29], [36]. At 50 °C of tensile temperature, each composite has the highest cross-link condition, which the strain being lower due to chain entanglement of the matrix. At 120 °C tensile temperature, strain at break for each composite showed an increase due to composites passing the glass transition temperature (T_g) of epoxy resin and the epoxy resin being softened [12], [13], [29].

Figure 5(a) shows the elastic modulus of aramid and carbon fiber reinforced epoxy/MWCNT and pure CFRP composites at elevated temperature tensile test. The elastic modulus of aramid fiber reinforced epoxy/MWCNT composites has a lowest than carbon fiber composites, due to the high strain at break of aramid fiber. Principally, the modulus is inversely proportional to the strain. At room temperature tensile test, MWCNT reduces the elastic modulus of composites due to their low cross-link between MWCNT and epoxy resin at room temperature [36]. The highest elastic modulus occurs when the temperature tensile test of 50 °C for each composite. The elastic modulus increases by 50 °C temperature tensile test, which the modulus value is 24.21 ± 2.91 GPa, 45.53 ± 4.91 GPa, and 45.13 ± 5.39 for aramid and carbon reinforced epoxy/MWCNT and CFRP composites, and higher 2.11%, 14.2% and 26.02% than temperature tensile test of room temperature, respectively. The highest elastic modulus occurs due to the elevated temperature improves the cross-link of the epoxy/MWCNT and bonding of the matrix to fiber [36], [42]. Above the glass transition temperature (T_g), the modulus of each composite decreased by 15.33%, 3.77%, and 11.77% of aramid and carbon fiber-reinforced epoxy/MWCNT and CFRP composites, respectively. The reduction of elastic modulus attributed to the softening of epoxy resin above their glass transition temperature (T_g), as in 120°C of tensile test temperature [12], [25], [43].

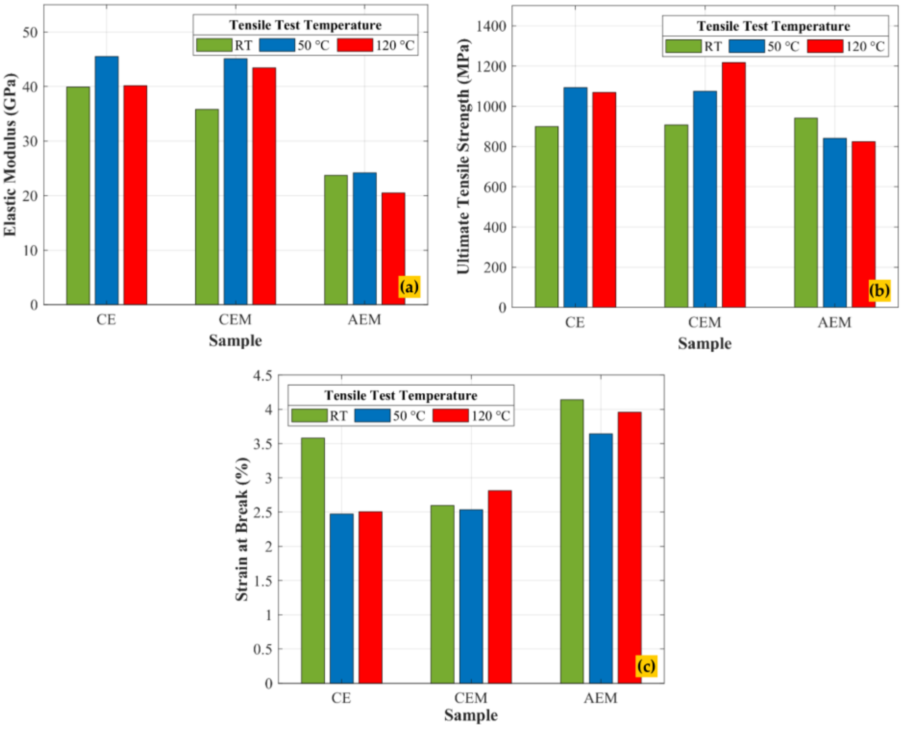

Fig. 5. Graphic tensile properties (a) elastic modulus, (b) ultimate tensile strength, (c) strain at break of CFRP with and without MWCNT

3.2 Morphological Analysis

After the specimen was evaluated by tensile strength test at elevated temperature, the fracture surface of specimen observed by scanning electron microscopy (SEM) for 120 °C temperature tensile test of specimen and optical microscopy for each variations test of the specimen. The characterization by SEM only on 120°C temperature tensile test of specimen to make sure the visualizer of fracture surface clearly in the highest temperature tensile evaluated.

Optical microscopy characterization was conducted using 200x magnification on each sample. Figure 6(a) shows the carbon fiber reinforced epoxy/MWCNT after a tensile test at room temperature, it is shown that partially the fiber that is not protected by the matrix due to low cross-links of epoxy/MWCNT matrix at room temperature tensile test, compared to Fig. 6(b) show pure CFRP that the matrix covered the fiber. Fig. 6(c) and 6(d) show the carbon fiber reinforced epoxy/MWCNT after the tensile test at 50°C, which shows a better bonding of fiber and matrix than Fig. 6(a). It proves that the elevated temperature increases the cross-link of polymer and bonding of matrix and fiber. The tensile test with a temperature of 120 °C shown in Fig. 6(e) and 6(f), shows a better bonding occurs for the carbon fiber reinforced epoxy/MWCNT than the pure CFRP composites, which attributed greater softening of the matrix make a greater debonding of the CFRP composites. Optical Microscopy characterization of aramid fiber reinforced epoxy/MWCNT fracture surface in Fig. 6(g) and 6(h) shows a lower interfacial strength than carbon fiber composites due to the higher strain of aramid fiber. Also, aramid fiber reinforced epoxy/MWCNT shows less residual epoxy/MWCNT matrix are attach at the fracture surface. This can explain due to the high surface strength of the carbon fiber imparted by the epoxy/MWCNT matrix which is responsible for the formation of strong intermolecular adhesion between the carbon fiber and the matrix resin. Conversely, aramid fiber has a low surface strength that affected fracture surface due to long organic polymer bonds with smooth structure. At elevated temperature, aramid fiber reinforced epoxy/MWCNT show a reduction of bonding between epoxy/MWCNT and fiber, that at 120 °C tensile test shows less matrix existential, which matrix left the fiber surface due to the softened epoxy resin as shown in Fig. 6(i).

Fig. 7 shows the surface characterization by scanning electron microscopy. The characterization was conducted using 1000x magnification on specimen tensile tested at temperature 120 °C. Fig. 7(a) shows the fracture surface of carbon fiber reinforced epoxy/MWCNT, which shows no experience of any fractures in the fiber after going through the tensile test. In addition, it was shown that the matrix was able to withstand the elevated temperature well as evidenced by the least debonding of the matrix to fibers. In a previous study [21], [37], the effect of adding MWCNT on CFRP composites by the fracture surface was investigated, it was shown that adding MWCNT on CFRP had less debonding than pure CFRP composites in the temperature exposed, this can be explained due to MWCNT build a bridge of matrix and fiber, then increasing the carrying load capacity. Meanwhile, Figure 7(b) shows that the pure CFRP composites experienced dominant debonding due to the elevated temperature tensile test. This occurs due to the elevated temperature softening the epoxy matrix and reducing the matrix bond to the fiber. Characterization of aramid reinforced epoxy/MWCNT shown in Fig.

7(c), that aramid composites experience an addition of length from the fracture surface until the composite is completely broken. This shows that aramid fiber has a greater strain at break than carbon fiber. Fig. 7(c) also shows the distribution of matrix flakes on the fracture surface, due to the high matrix stiffness and high strain on the fibers causes the matrix attached to the fibers to be carried away and experience cracking on the matrix.

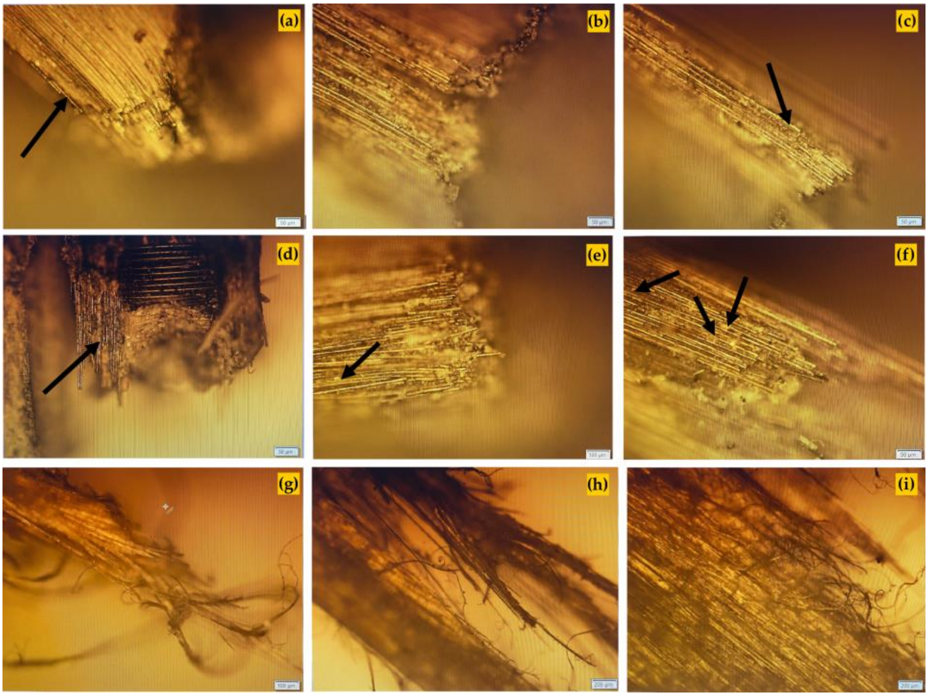

Fig. 6. Optical microscopy images of the fracture surface of (a) CEM-RT, (b) CE-RT, (c) CEM-50 °C, (d) CE-50 °C, (e) CEM-120 °C, (f) CE-120 °C, (g) AEM-RT, (h) AEM-50 °C, (i) AEM-120 °C

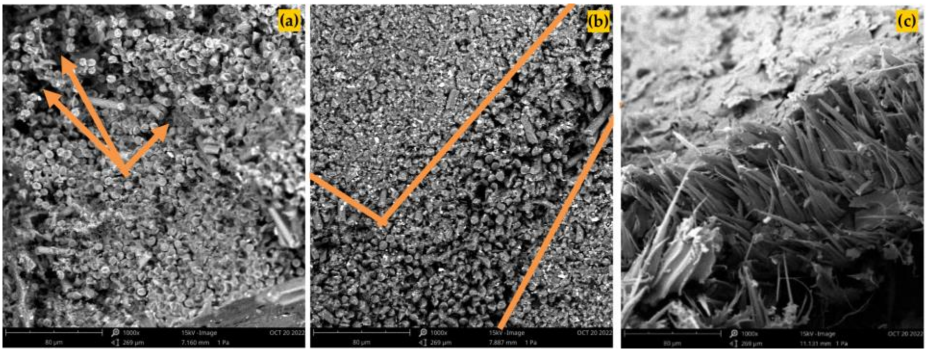

Fig. 7. SEM images of the fracture surface of (a) carbon fiber reinforced epoxy/MWCNT, (b) pure CFRP, and (c) aramid reinforced epoxy/MWCNT, at 120°C temperature tensile test

4 Conclusion

In the study of the elevated temperature effect mechanical properties of aramid and carbon fiber reinforced epoxy/MWCNT, three different temperatures of tensile test of room temperature, 50°C, and 120°C were investigated. Significant enhancements in the ultimate tensile strength and strain at break on carbon fiber reinforced epoxy/MWCNT were observed. The elevated temperature of the ultimate tensile test was increasing the ultimate tensile strength and strain at break of carbon fiber-reinforced epoxy/MWCNT by 34.08 % and 10.84 %, respectively. Yet, aramid fiber-reinforced epoxy/MWCNT decrease the ultimate tensile strength and strain at break by 12.49 % and 2.19 %, respectively. The maximum modulus of aramid and carbon fiber reinforced epoxy/MWCNT was raised by temperature tensile test of 50°C, by 24.21 GPa and 45.13 GPa, respectively.

The effect of MWCNT in CFRP composites was investigated. The MWCNT improves the glass transition temperature of CFRP composites and the modulus elastic, ultimate tensile strength, and strain at break by 21.28 %, 13.88 %, and 11.99 % respectively.

From the specimen tested at elevated temperature, the characterization fracture surface was observed. The fracture surface of specimen test at room temperature displayed a poor of cross-link between the matrix and fiber. At elevated temperature tested, the cross-link of matrix increases, which increases the ultimate tensile strength of composites. At 120°C temperature tensile test shows a matrix softened and affected the property of composites.

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