Investigation on Mechanical Properties and Fracture Mechanism of Carbon Fiber and Glass Fiber Composite at Different Curing Temperature

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Abstract
Weight reduction is one of the big challenges in the auto industry. In the aerospace industry, the high modulus of composite material makes weight savings to replace alloys such as aluminium and titanium. It is one way to reduce fuel consumption and reducing emissions in vehicles by using composite. Among them, carbon fiber and glass fiber are the most useful materials in composite to reduce the weight in vehicles not only in the automotive field but also in aircraft, marine, medicine, sport, etc. In this study, hand lay-up (wet lay-up) method is used to fabricate carbon fiber and glass fiber composites with different curing temperature 30°C (the equivalent of room temperature) until it is dry and 80°C for curing 6 hours. The strength of carbon fiber and glass fiber composites were increased over 10% and the hardness of carbon fiber composite was also increased two times at 80°C curing temperature. Furthermore, the fracture mechanism of carbon fiber and glass fiber composites were also observed by using Scanning Electron Microscopy (SEM) image processing method.

Keywords: Composites, Curing temperature, Mechanical Properties, Fracture Mechanism.

1. Introduction
Carbon fibers are fibers about 5–10 micrometers in one filament diameter and consist of at least 92 wt%, mostly more than 99 wt% of the carbon in the non-graphitic state [1]. Recently fiber composites are rapidly developing in the auto industry due to their unique characters and several advantages including low weight, high stiffness, high tensile strength, high chemical resistance, high-temperature tolerance, corrosion resistance, and rigidity [2, 3]. Their properties could be used not only in automotive parts, aerospace, marine, but also, they are used in civil engineering, medicine, military,
motorsports, and others [4]. There are several types of carbon fiber such as pitch, aramid, PAN and mesopitch carbon fiber. Among them, precursor polyacrylonitrile (PAN) is used as the most useful carbon fiber production because of its high strength and young modulus compared to other materials that contained PAN formula so-called C₃H₃N. And this formula is made from propylene and ammonia [5]. Nevertheless, they are proportionately expensive when compared with similar fibers like glass fibers or plastic fibers. Glass fiber is one of the most flexible materials that can be used in various fields. Most of them are based on silica (SiO₂) and combined with other chemical substances, such as oxides of calcium, boron, sodium, aluminum, iron. Glass comes from sand and it needs good care to get free of impurities material of process [6]. A composite material is a combination of two or more materials with significantly different in terms of physical or chemical properties. The two main functions in the composite are fiber and matrix [7, 8]. In composite, carbon fiber is the main substance and matrix is the one material, which can produce bonding strength between the fibers [9]. The polymer matrix resin can be divided into thermoset and thermoplastic resins. The overall influencing resin for composite structural is a thermoset, giving lower cost processability and high performance. For a thermoset, an irreversible cross-linking of the polymer chain consolidates the resin into an amorphous polymer forming the matrix. The main requirements for the resin are high ductility, high toughness, low viscosity, excellent processability, low shrinkage, and compatibility with fibers, sizing, and adhesives [10]. N. Ozsoy [11] observed 0, 90˚ and 0/90˚ orientation of both carbon fiber and glass fiber by using lay-up process and the composite was investigated in the tensile and flexural tests.

In an analysis of nano-indentation technique, Hardiman (2015) [12] found that the technique of a nano-indentation can identify the hardness and modulus of carbon fiber polymer matrix composite with small pyramid shape spindle diamond knife. Singh (2018) [13] studied the effect of curing temperature ranging from 80°C to 130°C on natural jute fiber reinforced epoxy polymer matrix-based composites for various mechanical properties. While Cui et al. (2018) [14] observed carbon fibers coated with graphene reinforced TiAl alloys composite bulks sintered at 620°C for 10 minutes using the field emission scanning electron microscopy (FESEM S-4800, Hitachi, Japan). This study revealed that this scanning was equipped with Energy-Dispersive Spectrometry (henceforth EDS), and it leads to a characterization of the microstructure of the specimens. The objective of the study was to investigate the mechanical properties and microstructure fracture of composites when increasing the curing temperature. This study seeks to explain the fracture mechanism of carbon fiber and glass fiber composites that are the microstructures weakness using electron microscopy analysis with different curing temperature.

2. Material Properties and Experimental Setup

2.1 Material Properties

In this experimentation, two types of fibers have been used with epoxy resin YD 582 and hardener TH 7253 as reinforcement in this composite; carbon fiber plain weave based on poly-acrylonitrile (PAN) and E-glass fiber fabrics were obtained from a local market. The properties of the two fibers and matrix material are mentioned in Table 1.

| Material                  | Based     | Tensile Strength (MPa) | Elongation (%) | Tensile Modulus (GPa) | Chemical Composition | Product name |
|---------------------------|-----------|------------------------|----------------|-----------------------|----------------------|--------------|
| Carbon Fiber Plain        | PAN       | 152.70                 | 1.83           | 17.253                | C (95–99%)           | PC6951500    |
| Glass Fiber Fabrics       | E-Glass   | 114.40                 | 2.97           | 8.230                 | Si (32–37%)          | Glass Cloth EW200 |
| Epoxy/Hardener            | Thermoset | 60                     | 4-6            | 2.850                 | Hydrocarbon          | YD 582/TH 7253 |
2.1.1 SEM with Energy-Dispersive Spectrometry (EDS) analysis on raw material

The carbon fiber plain (CFP) and glass fiber fabric (GFF) was checked in SEM/EDS to determine the elemental composition of fibers found, as shown in Figure 1. The cross-section of carbon fiber and glass fiber were cut by fiber cutter scissor. The surface of the carbon fiber plain consists of roughness, pitting sharp but there is no porous in the cross-section area of the fiber. The chemical composition of carbon fiber plain is carbon-containing 96 wt% and oxygen-containing 4 wt%, respectively.

Regarding glass fiber fabric, there are several defects on the fiber surface along the fiber axis, cracking area and porous and are found in the cross-section area. The chemical composition of glass fiber fabric contained Zn element in the range of 0.9-1.4% based on three filament measurement, as shown in Figure 5 (b). The chemical percentage of Al was not found in the fiber, some were from the holder of the sample while SEM analysis is performed. Table 2 provides a summary of the EDS chemical composite in each carbon fiber and glass fiber.

![Electron Image 1B](image1.png)
(a) Elemental composition of carbon fiber plain

![Electron Image 2B](image2.png)
(b) Elemental composition of glass fiber fabric

**Figure 1.** Energy-Dispersive Spectrometry (EDS) analysis of fibers
2.2 Fabrication of composites
All the reinforced polymer composites were fabricated using hand lay-up process and adjusted bidirectional 0/90 degree. Weave fibers were used with reinforcement and epoxy resin as matrix material. There are two parts of the epoxy resin mixing ratio such as epoxy and hardener mixing ratio (3:1) and they are calculated by equations (1-2). The weight of the composites was controlled based on the fabric weight of each layer. All the composites were cut by computer numerical control (CNC) machine Mazak FJV-20 with the parameter of spindle speed 150 mm/min and feed speed 50-100 mm/min according to ASTM specimens shape. Additionally, after cutting dried composites at 30 °C until it was dry, other specimens were cured at 80ºC for 6 hours. Among them, the three specimens were tested twice in each mechanical testing and all the laid-up conditions (see Table 3).

Table 2. EDS result of CFP and GFF filaments

| Fiber type | Element (Weight %) |
|------------|-------------------|
|            | Carbon | Oxygen | Silicone | Ca | Al | Zn | Mg |
| CFP        | Spect:34 | 95.9    | 4.1      | -  | -  | -  | -  |
| GFF        | Spect:42 | 13.7    | 18.8     | 34.4 | 21.6 | 9.7 | 1.4 | 0.3 |

2.3. Mechanical testing

2.3.1. Tensile Test
The tensile test based on the ASTM D638 was used with the three samples of carbon fiber composite and glass fiber composite. After that, all the samples were tested INSTRON 8801 with the condition of 50 mm/min, 95 kN load cell.

2.3.2. Flexural Test
The flexural test was performed on 51% RH with INSTRON 55R4502 machine, 24°C temperature by three-point bending test. ASTM D790 method was used on 5.2 mm/min testing rate speed with cell load 10 kN for all the tests.

2.3.3. Indentation Test
Indentation test was used to observe the mechanical properties of the materials especially young modulus and hardness of composite that is cured at room temperature until it was dry and cured composite at 80ºC for 6 hours. In this research, Elionix (ENT -1100a) indentation tester was used in
the operating condition at room temperature with max load 200 mN. The time set up was 10,000 sec and 500 steps (200 sec/step).

2.3.4. Morphological analysis
Morphological analysis was investigated at the room temperature (RT) 30˚C and RT + 80˚C at a curing temperature to understand the fiber-matrix interface and mechanism of failure of the composites using SEM image analysis.

3. Result and Discussion

3.1. Tensile test results on carbon fiber and glass fiber composites with different curing temperature

In this research, tensile test for three specimens of carbon fiber plain and glass fiber fabrics epoxy-based composites were investigated with two different curing temperature such as (1) at room temperature until it was dry; (2) at room temperature + 80˚C for 6 hours. As can be seen in Figure 2, the result shows the effect of curing temperature on tensile properties of carbon fiber and glass fiber composites. The ultimate tensile strength of CF80 was recorded as around 18% higher than CF30 while GF30 increased to 12% compared to GF80. The tensile strain of carbon fiber is two times shorter than glass fiber elongation. GF30 and GF80 are nearly similar in tensile strain around 2.9% elongation. The results from the CF80 slightly increased by about 13% than the CF30. Similarly, the results show a similarity of the effect of curing temperature 30°C and 80°C on the young modulus of the composites. The value of carbon fiber composite is just about two times higher than glass fiber composite of the young modulus. Higher tensile strain represents the higher ductility of composite that was found in glass fiber composite. It can be explained that glass fiber composite has ductile behaviour compared to carbon fiber. The two composites did not have the same thickness, but their thickness does not affect the young modulus of materials because the material properties are not influenced. In this experiment, the strain gauge was not used so that the elongation of composites is calculated from sample crosshead displacement. The effect of temperature on tensile strength, tensile strain, and tensile modulus of composites are shown in Figure 2 (c), Figure (d) and Figure (e).
3.2 Flexural test results on carbon fiber and glass fiber with different curing temperature

Regarding the flexural test condition, three specimens are used for flexural strength and flexural strain. They were calculated with the machine automatically, while the flexural modulus was calculated by the ratio of stress and strain. The mechanical properties of carbon fiber and glass fiber composites were observed at 30°C (room temperature) and RT+80°C for 6 hours. All the tests were performed with the strain rate 5.2 mm/min and all the stress-strain results of each specimen are shown in Figure 3 (a) and Figure (b). Besides, the two different curing temperature of carbon fiber composite, the high flexural strength of CF80 is higher than CF30 (almost 17%). The elongation of CF80 composite is slightly shorter than CF30 and it can be considered the heating at the oven for 6 hours with 80°C released all the moisture and evaporate. In contrast, the young modulus of CF80 is 28% higher than CF30. For glass fiber composite, its the flexural strength was 240 MPa for GF30 and 326 MPa for GF80 whereas the elongation rate also increases by 24% as shown in Figure 3 (c) and Figure (d). Young Modulus of both GF30 and GF80 did not show a significant change but it slightly increased later, as shown in Figure 3 (e).

(a) Three specimens of carbon fiber composite  
(b) Three specimens of glass fiber composite
3.3 Indentation test results on carbon fiber and glass fiber with different curing temperature

In the indentation test, 10mm$^2$ of composites were prepared by band saw (electric saw) and test 5 times in different location with 20 μm of the triangle shape spindle diameter. It was found that the hardness of composite and modulus of composites were investigated with two different curing temperature such as room temperature and 80°C for 6 hours. At 30°C cured result, glass fiber composite has the lowest depth at 4.5 μm where carbon fiber is the deepest depth at 6.5 μm. Glass fiber fabric contains a plain weave one by one bundle. The filament bundle thickness is much smaller, but the composite was built up by 16 layers of fabric. It will lead to difficulties for a spindle to penetrate through the composite. Concerning carbon fiber, the woven in plain weave and the layers were 12 layers fiber in composite and the filament bundle, and its amount is smaller than the glass fiber. Therefore, it will be easier to penetrate through composite.

Figure 4 (c), Figure (d) and Figure (e) show the hardness, max depth and modulus of all type of fibers composite. The results from the composite show that it is cured at 80°C for 6 hours, the depth of CF80 was less than 1.7 μm compared to CF30. While the depth of GF80 slightly increased around 0.4 μm because the glass fiber composites increased in ductility based on the increase of curing temperature. In addition, the results of the higher curing temperature showed higher hardness. The results of the modulus in carbon fiber composite contrasts with the glass fiber composite result. The results remained unchanged when the curing temperature was increased. The main point of higher hardness and modulus might be higher adhesive cross-linking between fiber and epoxy. The spindle dent area image of both composites is shown in Figure 4 (f) with different curing temperature.
3.4. Field-Emission Scanning Electron Microscope (FE-SEM) Analysis

The fracture mechanism of carbon fiber and glass fiber composite was observed by using SEM image analysis at 30°C and 80°C cured fracture mechanism especially on tensile test failure, as shown in Figure 5. The fracture mechanism of carbon fiber composite was from vertical direction laid up fibers, it may start cracking when the load was applied the vertical axis of tensile force. Regarding the fracture mechanism of CF30, it indicated that several voids and less resin were attached between fibers and matrix. Whilst the CF80 is more compact than the CF30 because high temperature can remove the moisture and make brittle on composite, as shown in Figure 5 (b) and Figure (c). As can be seen in Figure 5 (a), carbon fiber was found to be a narrow channel around the fiber surface. A possible explanation for this might be that the mechanical bonding with a matrix can be a composite. Also, it was cured at a higher temperature for a long time so that it could be a brittle composite.

For glass fiber composite, their fracture area was quite large compared to carbon fiber composite due to the main extracted fibers from the matrix. Figure 5 (e) shows several defects and cracks on the cross-section of GF30 surface because the voids from the composites are one of the factors that allowed the cracking of the composite. In contrast, the results of the GF80 revealed a different failure condition compared to GF30 because the fiber and matrix are bonding tightly than the curing room temperature. And, the results were found to be less pull out fiber in this fracture in Figure 5 (f). The matrix holds the fiber tightly for not being pulled off from external force at high temperature. However, the results indicated that the fibers kept pulling out in GF80 due to the surface of the smooth condition of the glass fiber along the filament axis, as shown in Figure 5 (d). Therefore, the pull-off fibers are become breakage easily due to a very weak in the chemical reaction between the fiber and the matrix.
4. Conclusion
The present study was designed to determine the effect of carbon fiber and glass fiber reinforced epoxy polymer matrix-based composites at different curing temperatures ranging from 30°C (RT) and 80°C. The SEM method was selected to identify the mechanical properties such as tensile, flexural, indentation, and morphology of the fracture mechanisms of the composites. The element composition of fibers was also observed using SEM-EDS.

During the tensile testing, carbon fiber and glass fiber composites are improved in tensile strength 18% and 12% with the increase of curing temperature. For the flexural test, both fiber composites are significantly increased in flexural strength 17% and 36% but it does not affect the modulus of flexural strength of glass fiber composite. In the indentation test, the hardness of CF80 was two times higher than CF30 while the hardness of GF80 was slightly 19% lower than CF30.

The fracture mechanism of composites and fiber filaments were observed by using SEM image analysis. The results from the carbon fiber composite at 30°C cured show that several voids could start cracking easily when the load is applied. The main failure mode is pulling out of the fiber due to the chemical bonding fiber and matrix were not well interlocked. At 80°C temperature cured, the composite becomes brittle due to the increase of cross-linking in the matrix resulting in higher strength. However, it can create holes and cavities since fibers are shrunk at high temperature. For glass fiber composite, the fracture mechanism was quite different because glass fiber composite is broken gradually at load applied to point and break at the outer surface of the composite. This study set out to represent the ductile behavior of glass fiber composite with a large fracture area.

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