CHAPTER IV

EXPERIMENTAL WORK

The following experimental work was progressed in order to examine the physical aging effects on the mechanical properties of fiber-reinforced epoxy composites. This chapter explained the characteristics of the materials used in this research as well as all experimental techniques such as mechanical testing and sample characterization.

4.1 MATERIALS

4.1.1 Epoxy resins

Epoxy resin is widely used in many industries and could easily react with most curing agents. Reinforced with carbon or aramid fiber, laminated epoxy composite is known to impart excellent performance. The epoxy resin employed in the current work was pigmented diglycydyl ether of bisphenol A (DGEBA). It was manufactured by Master Builders Incorporation under the trade name of MBrace Saturant, Part A. The structure of DGEBA is presented in Figure 4.1. Its viscosity is about 1150 cps at 25 °C. The density is 984 kg/m^3.

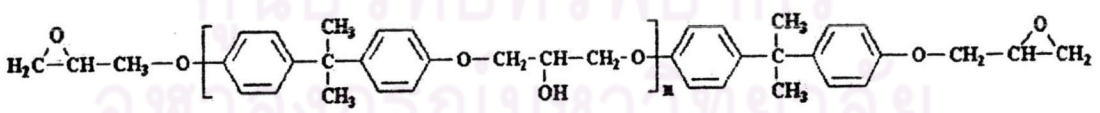

Figure 4.1: The chemical structure of diglycydyl ether of bisphenol A (DGEBA).

4.1.2 Curing agents

MBrace Saturant, Part B, also manufactured by Master Builders, Incorporation, was utilized as the curing agent in the present work. It is an aliphatic polyamine adduct in which the polyoxypropylenediamine is the largest of its part. The chemical structure of polyoxypropylenediamine is depicted in Figure 4.2. The polyoxypropylenediamine was selected to be the curing agent in concrete application due to its reaction with epoxy resin to form crosslinkages at room temperature. Polyoxypropylenediamine is a clear, colorless to slight amber liquid with low viscosity and ammoniacal odor. It has an amine content of 98% and a density of 1000 kg/m^3 approximately.

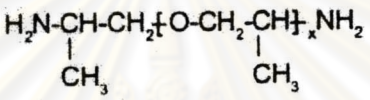

Figure 4.2: The chemical structure of polyoxypropylenediamine.

4.1.3 Carbon fiber

Carbon fiber mat-reinforced composites have been used as structural material in aerospace and manufacturing applications, especially those in which light weight, high tensile strength and non-corrosive properties are required. PAN-based carbon fiber mat from Tonen Corporation under the trade name of FORCA Tow Sheet was chosen for this present study. Figure 4.3 shows the FORCA carbon fiber in the tow mat form. It was made of carbon fiber strands bundled and oriented in unique directional arrangement. It has a density of 1.82 g/cm^3 and fiber area weight of 200 g/m^2. The thickness is 0.111 mm.

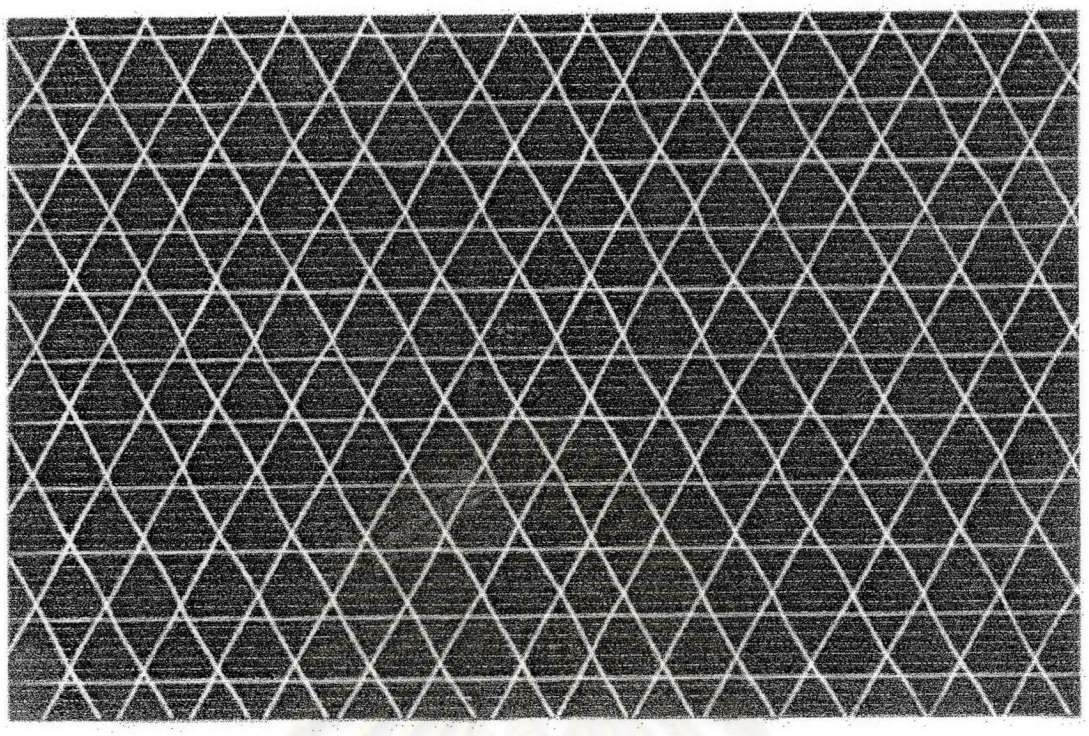

Figure 4.3: FORCA Tow Sheet.

4.1.4 Aramid fiber

Aramid fiber has been used in composites to prepare light weight, strong and stiff composites. Aramid composites are known to be resistant to fatigue, impact failure and stress rupture, and they have excellent wear resistance. In this study, the aramid fiber tow mat was obtained from Tonen Corporation. Figure 4.4 displays the structure of the aramid fiber tow mat. It has a density of 1.45 g/cm^3 and the fiber weight of 280 g/m^2. Its thickness is 0.193 mm.

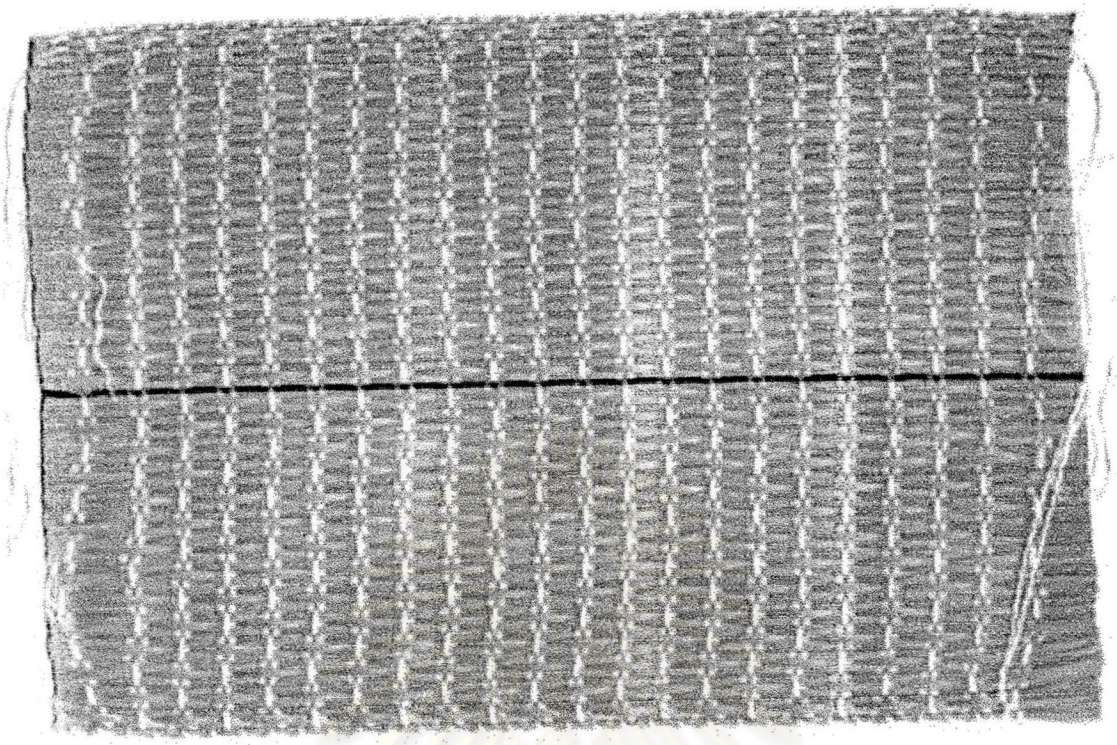

Figure 4.4: Aramid fiber tow sheet.

4.2 EXPERIMENTAL DESIGN

Many experimental studies involved the investigation of effects imparted by two or more factors as well as the joint effects of these factors. A factorial design is the most efficient approach for this type of experiment. It is widely used in research work because all possible combinations of the levels of factors could be examined in each complete trial or replication of the experiment. If there were a levels of factor A and b levels of factor B, then each replicate contained all ab treatment combinations.

In this research, there are three independent variables for the aging conditions. The quantitatively independent variable is the aging temperature, while the two qualitatively independent variables are the presence of humidity and the presence of UV light. Each variable is designed with two levels. Though qualitative, the amount of these variables were kept constant at each level. The design applied in the present study is a 2^3 factorial design. The eight possible combinations of the three variables, represented by

a, b, c, shown geometrically as a cube in Figure 4.5, would be investigated. For the aging temperature, the high level was set at 60°C, the highest temperature expected on the composite applied on strength concrete structures under outdoor service as well as the highest temperature operatable on the Accelerated Weathering tester. By the same consideration, the low level was 40°C. This is approximately the average temperature observed mostly in the concrete exposed to daily sunlight in Thailand. Since the carbon-reinforced composite and the aramid-reinforced composite in the present study are widely used in reinforcing concrete structure, the aforementioned temperature of 40°C was hence selected as the low aging temperature for the present study. The humidity of the specimen at high level was kept in wet condition, i.e. 100% humidity while the low level was operated with dry condition in vacuum oven that could be considered as close to 0% relative humidity. For UV exposure, each aging condition with UV, exposure was achieved by setting the fluorescent UV lamp to irradiate at 0.55 $W/m^2/nm$ for 168 hours. This was the high level of UV exposure. The low level was that without UV exposure at all in the aging condition.

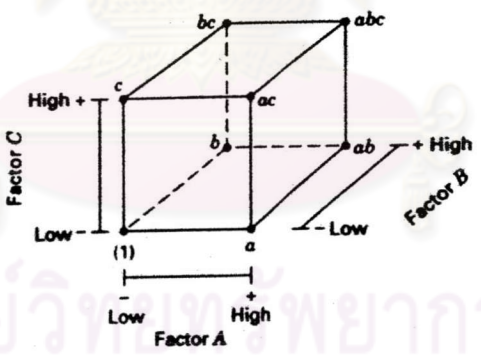

Figure 4.5: Geometric view of the eight possible effects for two-level factorial design with three variables.

From the geometric notation in the factorial design, the + and - designated notations were used to signify the high and the low factor levels respectively. A list of the eight conditions in the geometric notation for this work was exhibited in Table 4.1.

From Table 4.1, the aging conditions could be translated from + and – to real conditions for the experimental work as depicted in Table 4.2.

Aging condition	Factors		
	Aging temperature (A)	Humidity (B)	UV exposure (C)
1	-	+	+
2	-	+	-
3	-	-	+
4	-	-	-
5	+	+	+
6	+	+	-
7	+	-	+
8	+	-	-

Table 4.1: The design matrix.

Aging condition	Factors			Responses (y)
	Aging temperature (°C)	Humidity	UV exposure	
1	40	Wet	Yes	y_1
2	40	Wet	None	y_2
3	40	Dry	Yes	y_3
4	40	Dry	None	y_4
5	60	Wet	Yes	y_5
6	60	Wet	None	y_6
7	60	Dry	Yes	y_7
8	60	Dry	None	y_8

Table 4.2: Aging conditions from 2^3 the experimental design.

4.3 PROCESSING TECHNIQUE

4.3.1 Curing of the epoxy resin with curing agent

In order to cure the epoxy resin, a stoichiometric amount for both the resin and the curing agent was used. This was calculated as 100 part by weight of the epoxy resin to 33 part by weight of the curing agent. Both the epoxy resin and the curing agent were poured and mixed by stirring thoroughly. Subsequently, the mixture was transferred to an open mold that had been prepared for composite material processing.

4.3.2 Preparation of composites

Many techniques could be applied for manufacturing fiber-reinforced composites. The use of a single tool surface to yield a desired shape of composite is often referred to as an open-tool (mold) processing. They consist of the so-called 'contact molding', such as *hand lay-up* and *spray-up*, as well as *filament winding* and *centrifugal casting*.

For this study, epoxy composites were formed by hand lay-up or hand laminating process. It is the simplest and cheapest method of fabrication that involved the manual placement of the fiber reinforcement layer and the mixed resin layer on the mold surface. It is frequently essential to place several successive layers. Three-layered laminate was prepared in the present study. The metal mold was firstly coated with a mold releasing agent. Once the mold surface had dried, the top coat layer, which was the mixture of epoxy resin and curing agent, was applied to the metal mold with the help of a brush for a thickness of about 0.5 mm. The resin was left for curing at room temperature for six hours. Then, the epoxy resin mixed with the curing agent was added again to form the first ply of cured epoxy resin until it met the desired thickness of about 1 mm.

Next, the carbon fiber tow mat was laid on top of the wet resin to form the second ply. Any bubbles trapped between the resin and the carbon fiber were eliminated with the assistance of a solid-haired roller and a rubber brush. Then, the third ply, which is the epoxy resin coat, was applied. Like that of the first resin coat, the mixture of epoxy resin with curing agent was transferred to the mold with the aid of a brush till it reached the desired thickness of about 3 mm. Subsequently, the composite was left to cure for one week at ambient temperature. After the hand lay-up process was finished, the composite was cut to the desired shapes and dimensions. They were then ground and polished.

4.3.3 Physical aging

All composite specimens in this experiment were aged according to the conditions displayed in Table 4.2. They were aged in the QUV accelerated weathering tester for periods up to one week (168 hours). Figure 4.6 shows the QUV accelerated weathering tester used in this study. Figure 4.7 exhibits the top view of the specimen racks within the tester.

The QUV accelerated the aging of the composites by exposing them to alternating cycles of UV light and moisture at controlled, elevated temperature. It simulated the effect of sunlight by using fluorescent ultraviolet (UV) lamps. Simulated dew and rain was achieved by using condensed humidity and water sprays. For the aging conditions without humidity and UV exposure (or the Aging conditions 4 and 8 in Table 4.2), the carbon and the aramid-reinforced epoxy composites were aged in an oven that controlled only the temperature for identical duration. Both the vacuum oven and the air-circulating oven were used to cross check the amount of humidity remained in the air-circulating oven. For this reason, there are a total of ten aging conditions investigated in the present study as were summarized in Table 4.3.

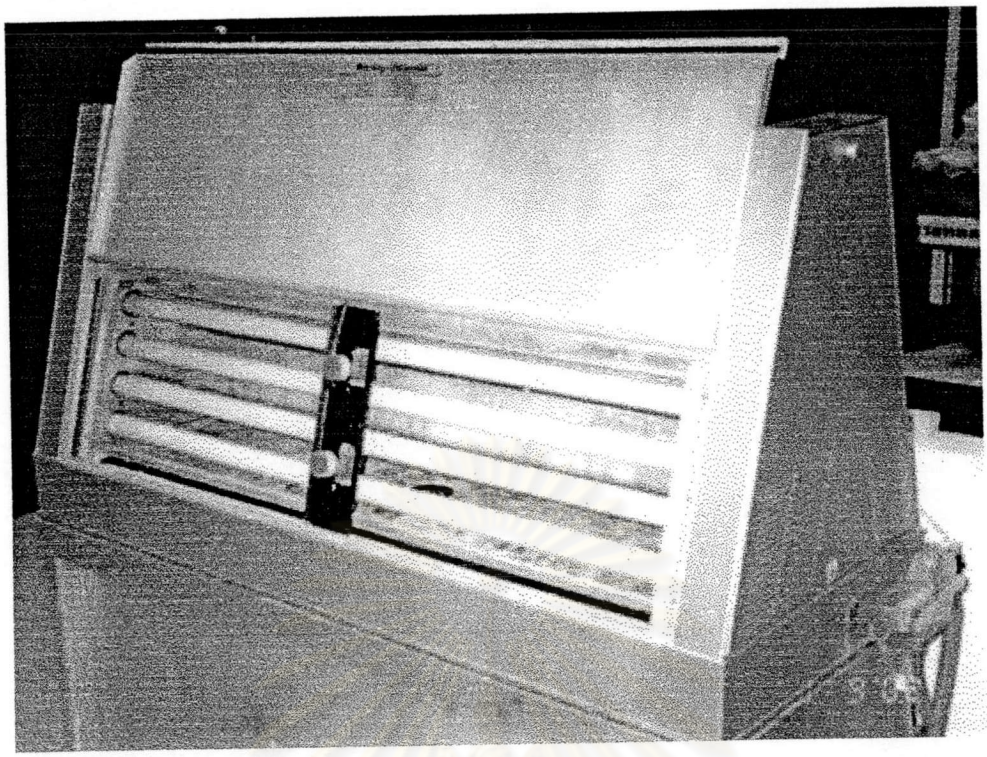

Figure 4.6: QUV accelerated weathering tester.

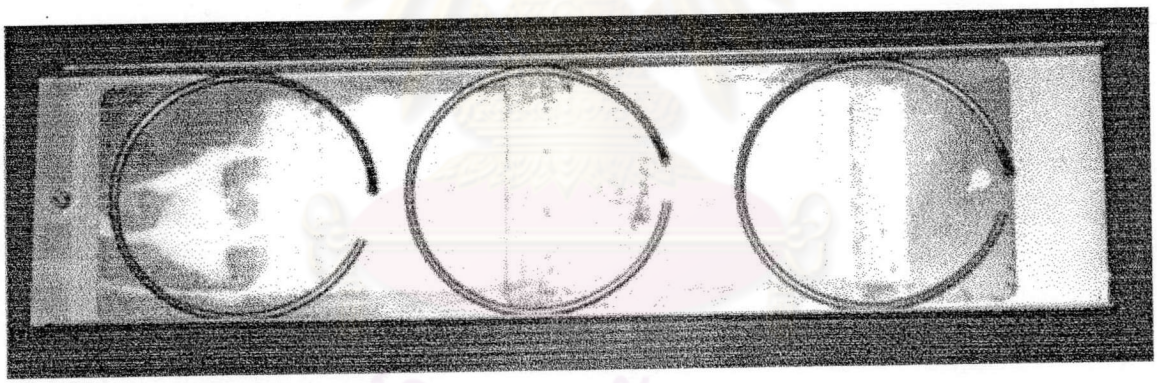

Figure 4.7: Specimen rack.

Aging condition	Factors			Responses (y)
	Aging temperature (°C)	Humidity	UV exposure	
1	40	Wet	Yes	y_1
2	40	Wet	None	y_2
3	40	Dry	Yes	y_3
4*	40	Dry	None	y_4
5**	40	Dry	None	y_5
6	60	Wet	Yes	y_6
7	60	Wet	None	y_7
8	60	Dry	Yes	y_8
9*	60	Dry	None	y_9
10**	60	Dry	None	y_{10}

* tested in air-circulating oven

** tested in vacuum oven

Table 4.3: All aging conditions from three factors, each was controlled at two levels.

4.4 MECHANICAL TESTING

4.4.1 Flexural test

Flexural testing was mostly carried out for composites under three-point bending mode as shown in Figure 4.8.

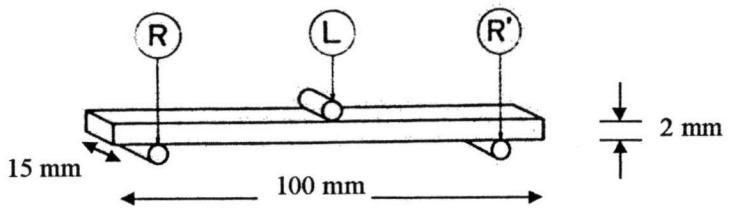

Figure 4.8: Three-point bending test where (R),(R') are supports and (L) is Loading.

Test specimens were prepared by cutting the composite material into a rectangle of 15 mm in width, 100 mm in length and 2 mm in thickness, as illustrated in Figure 4.8. In the present work, the flexural test was conducted by using a mechanical testing machine (LLOYD model 2000R). The specimens were prepared and tested according to JIS K 7074 standard test method specified for carbon fiber reinforced plastic (CFRP). It was also applied for testing aramid fiber reinforced composite for this research. For the three-point loading in this standard, the distance between the two supports, R and R' was 80 mm. The crosshead speed was set constant at 5 mm/min. Five specimens were tested for each aging condition. The testing temperature was 25 ± 2 °C and relative humidity was 50 ± 5 %. Only average values were reported.

4.4.2 Compression test

Compression test of polymeric materials was not as widely performed as the tensile or the flexural test. This was partly because it was not very simple to perform and to analyze. However, during the service life of most composites, they are often subjected to compressive load.

Compression test of polymers was performed following the standard test procedure in ASTM D695. Test specimens in the form of a square of 12.7 mm x 12.7 mm with a thickness of 3.2 mm were individually compressed between two parallel plates at a constant crosshead speed of 1.3 mm/min. The test specimen and the compression anvils were demonstrated schematically in Figure 4.9. The compression test was conducted by using a universal testing machine (Shimadzu model AG 2000G). The test was carried out at a standard condition of 25±2 °C and 50±5 % relative humidity. For anisotropic materials, ten specimens, namely five normal to and five parallel with the principal axis of anisotropy, were tested for each condition. Mean values were reported and analyzed in Section 5.2.

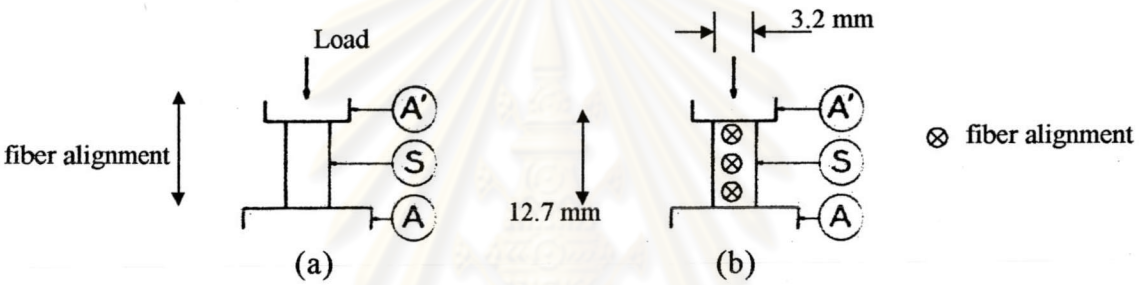

Figure 4.9: Compression testing where (A),(A') are Anvils and (S) is Test specimen; (a) is a specimen loaded parallel with the fiber alignment and (b) is a specimen loaded normal to the fiber alignment.

4.4.3 Double torsion test

The fracture toughness of a material can be measured by testing a specimen of finite size containing a machined notch. The notch itself can be extended by propagating under appropriate loading so that the tip of the notch is sharpened. Double torsion test is one type of tests designed for the determination of the critical stress concentration factor under mode I (K_{Ic}).

The double torsion specimen was a rectangular plate supported on two parallel rollers of distance 34 mm apart. Two hemispheres were placed on each side of the notch or a pre-crack at one end of the specimen. Their function was to apply compression load so that the two halves of the

specimen were in torsion. The specimen was also grooved on the lower surface along its length to ensure that the crack would propagate along the midplane. Figure 4.10 showed the shape of the double torsion specimen.

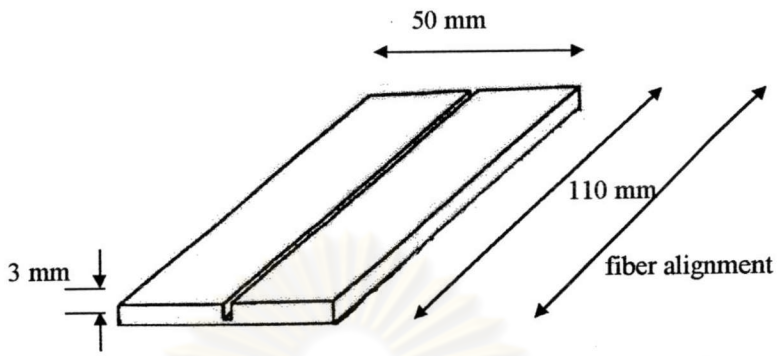

Figure 4.10: Double torsion specimen.

In this study, the dimension of the double torsion specimen was 110 mm in length, 50 mm in width and 3 mm in thickness. It was grooved along its resin face to give a U-shape groove with 2 mm in width and 1 mm in depth. One end of the groove was precracked to yield the crack initiation. The double torsion test was performed by placing the test specimen on the double torsion fixture made from stainless steel as illustrated in Figure 4.11. The fixture and the specimen were then compressed with a constant crosshead speed of 0.5 mm/min. All double torsion samples were conditioned at 23$\pm$ 2 °C and 50$\pm$5 % relative humidity for not less than 40 hours prior to the double torsion test. Three specimens were examined for each condition and average values were further analyzed in Section 5.2.

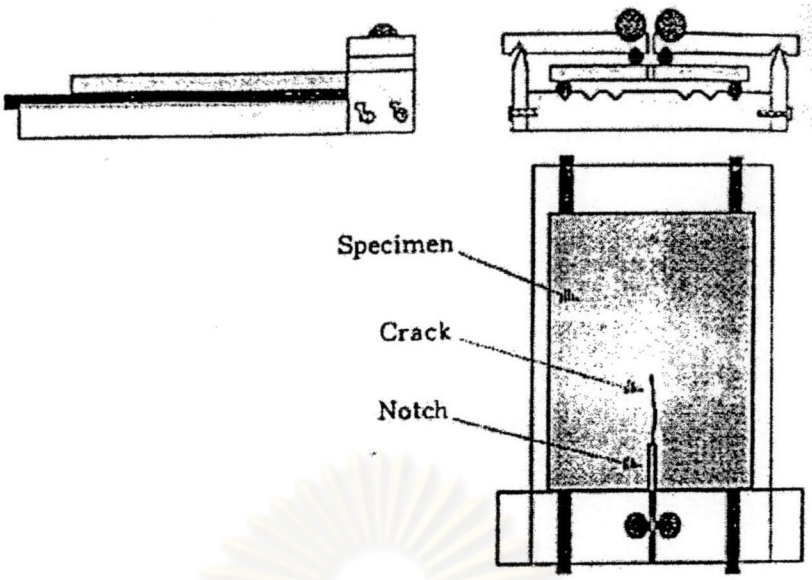

Figure 4.11: Double torsion fixture arrangement.

4.5 SAMPLE CHARACTERIZATION

4.5.1 Dynamic mechanical testing

Dynamic test is very important in the case of polymeric material due to their well-known sensitivity to the speed of loading. Such is a consequence of polymers being viscoelastic. The dynamic test is often applied when a polymeric test specimen is subjected to abrupt or repeated (cyclic) loads that are not designed to cause failure, i.e. nondestructive testing. For dynamic mechanical test, the polymer sample is deformed cyclically under small amplitude sinusoidal loads while the temperature is raised simultaneously. The deformation induced by the small amplitude sinusoidal load allows the determination of two basic quantities through the theory of linear viscoelasticity. They are the storage modulus that tends to reflect the elastic response of the material, and the loss modulus or tan δ that tended to reflects the viscous response. This technique is also used to determine the glass transition temperature (T_g), an important characteristic of amorphous polymers. It is called the "glass transition" because the structure and properties of polymer below the T_g are reminiscent of those of ordinary glass that is hard and rigid. Above the T_g, the molecules have more energy and

hence the movement of molecular segments becomes possible. In the glass transition region, polymer is soften considerably. The T_g was estimated at the peak of tan δ curve obtained from dynamic mechanical testing.

In this research, the dynamic mechanical test was performed by using a Netzsch DMA 242. The dual cantilever bending mode was selected. The specimen was 10 mm in width, 60 mm in length and 3 mm in thickness. The test was proceeded at a frequency of 1 Hz over a temperature range from 30°C to 200°C with the heating rate of 5°C/min.

4.5.2 Morphological investigation

The microscopic features on the surface of polymeric materials could be viewed by electron microscopy. Electron microscopy utilized an electron beam to examine the sample. Scanning electron microscopy (SEM) used a focused electron beam to scan the sample surface. To investigate the composites in the present study, SEM (model JOEL JSM 840A) was applied to study the fracture surfaces of the composites. An accelerating voltage of 10 kV was used. The fracture surfaces were sputtered with 300 A^0 in thickness of gold. Fiber orientation and adherence of the epoxy resin matrix phase to the fiber were observed to verify the existence of interfacial attachment and also to seek evidences of surface wetting.