Doctoral Dissertation

Fatigue Fracture Properties of Nanoclay and Wood Plastic Composites

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# Table of Contents

Nomenclature ........................................................................................................ iii
Symbols and Units ................................................................................................ iv

## Chapter 1 General Introduction

1.1 Background .......................................................................................................... 1
   1.1.1 Polymer nanocomposite ............................................................................. 7
   1.1.2 Wood-Plastic Composites (WPCs) .......................................................... 9
1.2 Objectives and outlines .................................................................................... 12


2.1 Introduction ........................................................................................................ 14
2.2 Experimental methods .................................................................................... 17
   2.2.1 Materials .................................................................................................. 17
   2.2.2 Specimen fabrication ............................................................................... 18
   2.2.3 Tensile and fatigue tests ......................................................................... 23
   2.2.4 Elemental observation .............................................................................. 25
2.3 Experimental results ....................................................................................... 27
   2.3.1 Tensile and fatigue properties ................................................................. 27
   2.3.2 EPMA observation .................................................................................... 29
2.4 Discussion ......................................................................................................... 37
   2.4.1 Agglomerate evaluation by image analysis .............................................. 37
   2.4.2 Evaluation using the nearest neighbor functions .................................... 43
2.5 Conclusions ...................................................................................................... 46

## Chapter 3 Study on Fatigue Properties of Wood Filled Polypropylene Composites: Fabrication and Interfacial Adhesion

3.1 Introduction ....................................................................................................... 47
3.2 Experimental methods ..................................................................................... 50
   3.2.1 Materials ................................................................................................. 50
Chapter 4 Evaluation of Fatigue Fracture Surface of Wood Filled Polypropylene Composites

4.1 Introduction ................................................................. 68
4.2 Experimental methods ................................................. 70
  4.2.1 Materials ................................................................. 70
  4.2.2 Specimen fabrication ............................................... 70
  4.2.3 Tensile and fatigue tests ........................................... 70
  4.2.4 Fracture surface observation ..................................... 74
4.3 Experimental results ..................................................... 77
  4.3.1 Optimal fabrication conditions .................................... 77
  4.3.2 Tensile properties .................................................... 79
  4.3.3 Fatigue properties .................................................... 80
4.4 Discussion ................................................................. 82
  4.4.1 Cross-sectional and fracture surface observation .............. 82
  4.4.2 Fatigue damage of WPC and failure mechanism ............... 85
4.5 Conclusions ............................................................... 89

Chapter 5 Summary ............................................................. 90

References ........................................................................ 93
Acknowledgements ............................................................ 97
### Nomenclature

<table>
<thead>
<tr>
<th>Abbreviation</th>
<th>Full Form</th>
</tr>
</thead>
<tbody>
<tr>
<td>CMCs</td>
<td>Ceramic matrix composites</td>
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<tr>
<td>MMCs</td>
<td>Metal matrix composites</td>
</tr>
<tr>
<td>PMCs</td>
<td>Polymer matrix composites</td>
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<tr>
<td>FRP</td>
<td>Fiber reinforced plastic</td>
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<tr>
<td>UD</td>
<td>Unidirectional</td>
</tr>
<tr>
<td>XRD</td>
<td>X-ray diffraction</td>
</tr>
<tr>
<td>TEM</td>
<td>Transmission electron micrographic</td>
</tr>
<tr>
<td>WAXD</td>
<td>Wide angle X-ray diffraction</td>
</tr>
<tr>
<td>SEM</td>
<td>Scanning electron microscopy</td>
</tr>
<tr>
<td>EPMA</td>
<td>Electron probe micro-analyzer</td>
</tr>
<tr>
<td>WPCs</td>
<td>Wood-plastic composites</td>
</tr>
<tr>
<td>PP</td>
<td>Polypropylene</td>
</tr>
<tr>
<td>NE-</td>
<td>Nanoclay-epoxy</td>
</tr>
<tr>
<td>MAPP</td>
<td>Maleic anhydride polypropylene</td>
</tr>
<tr>
<td>S-N</td>
<td>Stress to the number of cycles to fracture</td>
</tr>
<tr>
<td>MDF</td>
<td>Medium density fiberboard</td>
</tr>
<tr>
<td>MA</td>
<td>Maleic anhydride</td>
</tr>
<tr>
<td>AA</td>
<td>Acrylic acid</td>
</tr>
<tr>
<td>MFI</td>
<td>Melt flow index</td>
</tr>
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</table>
Symbols and Units

$F_{ci}$  
Fatigue fracture surface at crack initiation area

$F_{pi}$  
Fatigue fracture surface at peak intensity area

$G(R)$  
nearest neighbor function

$R$  
radius from nanoclay agglomerate's centroid

$d_i$  
nearest neighbor distance

rpm  
rotation per minute

phr  
part(s) per hundred parts of resin

wt.%  
percentage of weight (mass) fraction

°C  
degree Celsius

h  
hour(s)
Chapter I  General Introduction

1.1  Background

Development of new advanced materials due the needs from the industries has placed a number of conditions such as light in weight, high durability and low production cost as a minimum prerequisite that are necessary to be full filled. The production of composite materials for example has put these elements as a main goal that is by combining two or more materials with different properties in order to give a performance in service which is superior to the properties of the individual materials. The composite materials have been used over centuries as construction materials - ancient Egyptians mixed the wheat straws with the Nil's mud to make bricks for their house, where the straws functioned as reinforcement to the mud to make the bricks strong, besides keep the house cool when it is hot in summer and vice versa. The implementation of the 'combining materials' idea bring interest to many people from various fields over the time until recent, which is known as a composite materials in the present-day. Fig. 1.1 illustrates the schematic of reinforcing and binding elements of composite materials. It is commonly use in many applications such as aerospace industry, where the high thermal shock resistance and excellent fracture toughness of ceramic matrix composites (CMCs) material is applied for heat shield of the space vehicles, and also widely applied in automobiles and as a material for sports equipments due to its light weight, tremendous elasticity and strength. Metal matrix composites (MMCs) are practically used for vehicle parts such as engine, rotors and disc brakes; because of relatively high thermal properties of the composite materials, and also commonly applied as a structure frames for many sports applications such as bicycles, rackets and as well as golf shafts.
Composite materials are designed to suit its properties to a certain application in order to optimize the consumption of the raw materials while giving a maximum performance. Similar to the other types of composites stated above, polymer matrix composites (PMCs) is applied to a certain parts of products in which appropriate with its range of properties and performances such as the interior and exterior parts of the vehicles, containers and also as a building panels. Over decades, numerous studies on PMCs properties as well as the development of new type of PMCs had been carried out. Fiber reinforced plastic (FRP) composites, for instance, gain interest from both manufacturers and researcher on improving the bulk properties of the materials to maximize the overall performances regarding to the applied field. Consequently, the FRP materials show
higher modulus per unit weight, *specific modulus* and strength per unit weight, *specific strength* when compared to other metal or monolithic materials, i.e., imply the weight savings over the whole structure, thus resulted in greater efficiency and energy savings to the outcome of the composites products. Plastic is defined as a polymeric material that formed by large molecules that are composed of many (poly-) repeats of simpler structural unit, the *mer*, connected by covalent chemical bonds. There are two types of polymeric plastic regarding to the engineering materials definition; thermosets and thermoplastics [1]. Thermosets are low-molecular-weight polymer that formed a cross-linked structure from the chemical reactions driven by the heating that is generated either by the reactions or from the external supplied heat. In opposites, thermoplastics are high-molecular-weight polymer that held together by weak secondary bonding. The cross-linking reaction does not formed in the thermoplastics as thermosets does, however, they can be reprocessed. The weak secondary bonds break down when high temperature is applied and reform during the cooling process. The differences between thermosets and thermoplastics were summarized as shown in Table 1.1. It is known that the virgin plastic or the pure polymeric materials have low in strength and stiffness properties; moreover, most are less resistance towards high temperature, which make it difficult to be applied individually in many engineering applications. Making the polymeric plastic as the binding constituent of composite materials, thus, by incorporating the fillers like fibers or particulates into the matrix, the mechanical and thermal properties of the plastic materials indicated an excellent improvements [2-5,20] despites several difficulties such as poor distribution and lack in interfacial adhesion between the fillers and the matrix, that need to be encountered. In this study, the PMCs were chosen as the subject material that would be varied by the difference of reinforcement fillers,
where the performances of these different PMCs material were measured and evaluated through their reliability from the mechanical behavior.

The reinforcement agent or fillers in composite materials plays an important role in order to hold the matrix that is by transferring the load and act as a *crack-bridge* at a crack tip upon failure. Fig. 1.2 indicates several examples of reinforcements that are commonly used for composite materials. The compatibility of fillers with the matrices has put extra advantageous for composites to uphold the local area towards given loads and therefore enhanced the performance of the overall composite materials. There are several types of fillers depends on the (i) structure or geometrical shapes, (ii) size and (iii) origin of the fillers.

i. **Structure or geometrical shapes** - Unidirectional (UD) fiber is commonly used as reinforcement agent in many composite materials. It may be includes under the continuous fiber type that provides strong strengthening result to the matrix, and may stacked together of different layers and directions to form a laminae structure. The laminae structure also may consist of woven roving of continuous fiber or a
mix of aligned unidirectional layer with random short fibers including chopped fibers or whiskers. High stiffness of particulates fillers of various geometrical shapes such as spheres, plates, ellipsoids or hollow, are often used as fillers to obtain a micro- or nano-size effect which offers an excellent improvements of composites properties.

ii. **Size** - The order of the size of fillers that is from nano- to micron-size, gives variety in results on the performance of the composite materials. The improvement of the composite materials properties can be achieved by the addition of low amounts of well dispersed small size fillers, in which a higher interfacial surface area results from an increased aspect ratio or surface–volume ratio of the fillers. Therefore, for example, the required amount of nano-size fillers is much lower than that of conventional micro-order fillers, such as ceramic particles and short fibers, in order to maximize the properties of the composites which also bring along the economic benefits.

iii. **Origin** - Generally, fillers can be differentiate from its material such as the glass, carbon, boron fibers or the fibers that originated from the plants or animal, which is namely as the natural-based fibers. The use of natural-based fibers had been practically used in PMCs type composites over years and increasingly due to the awareness of the environmental problems. Commonly, the waste from the forestry or agriculture were processed as a fillers of PMCs which bring lessen to the used of petroleum-based matrices while optimizing the natural resources.
Fig. 1.2 Different types of reinforcements applicable to fabricate composite materials.
1.1.1 Polymer nanocomposites

Polymer nanocomposite is a combination of nano-size stiff fillers with the polymer plastic matrix. The nano-size fillers can be defined as the particle that has at least one dimension in nanometer scale. The incorporation of nano-size fillers into plastic polymer or rubber creates a new class of material with an outstanding performance in functional properties such as gas barrier, as well as mechanical and thermal ones [3-5]. From the end of the last century, nanocomposite materials have been attracting a great attention to industrial sectors and academic researchers, who focus on the broad possibility of nano-size fillers compared to micro-size ones. As mentioned above, there is an economical point such that the required amount of nano-size fillers is lower than the conventional micro-order fillers, i.e. ceramic particles, short fibers, and many more. Moreover, great attentions to the nano-size fillers emerged for several other reasons including [6, 7]:

i. The fillers usually have different properties from bulk properties of the same materials;

ii. The fillers are small defect compared to micro-size fillers which are similar in size to the critical crack size, and this will cause a premature crack;

iii. Due to larger surface area of fillers, nanocomposites have a large volume of interfacial matrix material with properties.

However, insufficient dispersion of nano-size fillers in polymeric matrices brings less mechanical properties [4, 8] because of the fillers often form micron-size agglomerates. The portion that is containing more agglomerated fillers tends to impart higher stress concentration on polymeric matrix than the other parts. This reaction leads to a crack formation and thus reduces the potential of the polymer nanocomposite materials. Consequently, there are many
factors that contribute to the insufficient dispersion that affect the properties of the nanocomposites, such as the synthesis methods during the fabrication process, the types of nano-particles that been used and also the characteristics of polymer matrix, where it is compatible or not with the compounded fillers.

The dispersion of fillers in polymer commonly tends to become partially exfoliated and intercalated clusters, where the fillers are homogeneously distributed. The distribution states have been verified through an X-ray Diffraction (XRD) method analysis or with high resolution observation by using transmission electron micrographic (TEM) [5, 9]. As concern by many in this field, however, it is almost difficult to achieve a fully exfoliated state only by a single physical process. We need to take into account the other factors such as chemical modification of nano-size particles that might influence appropriate formation for the internal microstructure of the polymer nanocomposites. Evaluation on dispersion order of nanoclay particles commonly has been done through a qualitative analysis of which the nanocomposites’ structure can simply be done using a wide angle X-ray diffraction (WAXD) analysis, scanning electron microscopy (SEM) or TEM observation.
1.1.2 Wood-Plastic Composites (WPCs)

Current issues on composite materials have been discussed and published in many literatures, including some virtuous inventions on improvement of mechanical properties and hybridizing conventional composites with bio-based fillers. This work includes the development of woods as reinforcing fillers for polymeric matrix to form an environmentally friendly composite material. Wood materials have been used in many products such as household articles, furniture and other interior items. The combination of wood flour with polymeric matrices, so-called as wood-polymer composites (WPCs), brings a new sight in wood-based application, i.e. high durability and excellent mechanical properties such as high tensile strength and stiffness [10-12] and low density. As fillers, these natural particles play a role of transferring the load and act as a crack-bridge at a crack tip upon failure. However due to the incompatibility of wood surface structure with the polymer matrix, the maximum performances of compounded wood particles in the composite materials are difficult to achieve. Fig. 1.3 illustrates the role of

![Diagram of coupling agent between hydrophilic fiber and hydrophobic matrix polymer.](source)

Fig. 1.3 Schematic figure of the mechanism of coupling agent between hydrophilic fiber and hydrophobic matrix polymer. Source [13].
coupling agent / compatibilizer in WPCs material. Moreover, the characteristics of wood flour are different with respect to the type of trees and harvesting time and it is easily influenced by the presence of moisture.

Another factor that contributes to the performance of WPCs material is the distribution of wood flour, i.e. the optimal amount of wood flour creates an appropriate condition for the fillers to transfer the loads and avoids the formation of wood cluster due to the lack of mixing process during the fabrication of WPCs. A study by Leu et al. [14] offers an idea of the optimization of wood fillers and other material composition in WPCs that helps to achieve ideal properties of the material. It is stated that by compounding up to 50 wt.% with the maximum value of coupling agent, maleic anhydride polypropylene (MAPP) at 3 wt.% able to enhanced the mechanical properties of WPCs. However, less data can be achieved regarding to the fatigue properties of the WPCs materials, which is highly recommended to be clarified. This is because the fatigue behaviors of WPCs material are important to estimate the life span or durability of the WPCs since the material itself has large potential to be applied in many applications. For an example, according to the fatigue behavior of natural fiber, Dijan et al. [15] suggested that high loading frequency reduced fatigue life of coir fiber reinforced polypropylene composites due to higher heat generation and less time available to dissipate the heat. This study imply the effect of heat that generated from loading and unloading actions, which is considerably close to the real applications, i.e. different loading frequency take place will results in heating and softening effects of the polymeric resin. Here, by understanding the fatigue behavior of WPCs material helps to contribute to a safer and reliable structure design for various applications in the future.

The failure behavior of green composites has recently gains a great attention, especially for certain research area that relates with the composition and characteristic of the natural fibers,
which is relatively difficult to control, and thus, need a particular methods and solution to overcome the difficulties. Also, the environmental factors such as the vibrations and the weathering effects may become an important point that causes a catastrophic failure of the bio-composite materials. These factors are more likely to be related with improper analysis of dynamic loading frequency or inappropriate fiber composition and its compounding behavior. Therefore, the fatigue test - a method for determining the behavior of materials under fluctuating loads - revealed a significant indication on the durability of materials, which is needed to estimate the life span of the material used. The fatigue process of a material is thought to begin at an internal or surface flaw, and the failure effect accumulates after several repetitive loads actions that may caused stress concentration at a point of cumulated damage, that is called as an initial crack point. Then, it generates a shear flow along the slip planes that resembles a crack, and propagates along the weak regions before caused a catastrophic failure. The fatigue study on new materials has become a must in order to design a strong and safety structure with high durability characteristic. A study by Shao Xiong et al. [16] on the properties of flax-epoxy layered composites under fatigue loading underlined the importance of interfacial adhesion between the natural based fillers and hydrophobic resin for the enhancement of the performance of the composites material. Larger number of fiber pulls out and fiber prints indicate the poor adhesion between the flax fibers and epoxy matrix. The interfacial adhesion of green composites had been studied literally; since the combination of these two constituents without the use of an appropriate compitibilizer or coupling agents is commonly lessen the properties of the materials [17]. Indeed, the enlargement of natural based fibers as a potential reinforcement agent in composite material leads to interesting findings, nevertheless, undiscovered factors and behaviors that result in the failure, which are essential to be predicted and analyzed.
1.2 Objectives and outlines

This study aims to understand the fatigue failure mechanism of particulates filled polymer-based composite materials and to clarify the fatigue fracture behavior from the fracture surface observation. For these purposes, two types of composite materials; nanoclay nanocomposites and wood-polymer composites (WPCs) were fabricated and tested. The tensile and fatigue tests were carried out for each type specimen - significant means to clarify the reliability of the materials and to understand the failure mechanism. As for the matrices, epoxy and polypropylene (PP) were used as a binder constituent for nanoclay nanocomposites and WPCs, respectively. The compounding of wood fibers and nanoclay fillers into the polymer matrix brings a structural change that caused by the presence of the micro-structure of the fillers itself or as a result from the clustering of the fillers that is incompatible with the polymer. Here, the distribution of the fillers and the compatibility of natural-based fillers with the hydrophobic polymer matrix were taken as the major subject in this study, i.e. by clarifying the relation between the mechanical properties with these factors, it is expected to fulfill the aims of this study.

In Chapter II, the neat epoxy matrix and nanoclay-epoxy (NE-) composites specimens were prepared with different nanoclay amount to investigate the influence of the nano-size fillers amount to the tensile and fatigue properties. The distribution of nanoclay particles was discussed and evaluated through an analysis conducted at the fatigue fracture surface and at the arbitrary cross-sectional area. It is assumed that the cross-sectional area contains a moderate distribution level that result in the lower stress concentration at as compared with at the fracture surface. The electron probe microanalyzer (EPMA) machine was used to conduct an elemental study on both fracture and cross-sectional surface, and a qualitative analysis was performed from the observation images results. From this chapter, the distribution states of nanoclay particles, i.e. the
agglomeration and clustering state, play an important part in determining the failure factors of this material despite lessen the properties of NE composite materials.

In Chapter III, different coupling agent MAPP amount of WPCs specimens was fabricated in order to investigate the effect of coupling agent on fatigue properties of WPCs. The tensile and fatigue test were carried out for each type of specimen. The obtained mechanical properties were discussed regarding to the different effects of coupling agent amount in the composites that expected to improve interfacial adhesion between wood fibers and PP matrix. The cross-sectional area and the fracture surface were characterized by the SEM and 3D laser microscope, respectively.

In Chapter IV, WPCs specimens with different weight fraction of wood fibers were prepared and the tensile and fatigue tests were carried out for each type specimen. The influence of wood fibers on fatigue properties was clarified and the fatigue fracture behavior was characterized from the image analysis. Moreover, further discussion on the fracture damage mechanism of WPCs will be carried out in this chapter.

Finally, Chapter V summarizes all conclusions obtained from the present studies.
Chapter II  Electron Probe-Microanalyzer Evaluation of Fatigue Fracture Surface of Nanoclay–Epoxy Composite Materials

2.1 Introduction

Among the wide possibilities presented by various materials, polymer-based nanocomposites have been used in many applications, particularly in the automotive and packaging industries, because of their rigidity, light weight, and high endurance properties. These materials are anticipated for wider use for other applications. Meanwhile, the incorporation of nano-size fillers such as clay particles into polymer matrix materials of fiber reinforced plastic (FRP) composites has been studied [9, 18]. This study assesses the importance of toughening the matrix material for additional applications of composite materials. To date, many studies have examined the preparation, testing, modeling, and analyses of nanoclay-filled polymer-based composites [19].

Nanoclay is known as an ideal nano filler to improve virgin polymer because of its high aspect ratio, low cost, and ease of availability [20]. Nanoclays are classified into three types based on the condensation ratio of silica to an alumina sheet, such as two-sheet minerals (dimorphic), three-sheet minerals (trimorphic), and four-sheet minerals (tetramorphic) [20]. This filler has 1 nm thickness with up to hundreds of nanometers of length. It consists of several elements such as silicon, aluminum or magnesium, and oxygen.

Montmorillonite is typical nanoclay, which is often used as a reinforcement of nanocomposites. This material is a trimorphic clay, i.e. an alumina sheet is sandwiched by silica sheets, from the smectite family, as shown in Fig. 2.1. The surface of each layer is negatively
charged, which attracts inorganic metal cations such as Fe$^{2+}$, Ca$^{2+}$, and Na$^+$, and which forms a positively charged layer that is commonly designated as an interlayer (intergallery) between the clay layers. An organic modification occurring with an exchange of inorganic cations with the organic modifiers, results in an expansion of the interlayer space, which allows the polymer to permeate easily into the galleries, thereby forming intercalated and exfoliated clays within the polymer-based matrix. However, nanoclay platelets are likely to be stacked together to form a clustered phase or combined as agglomerate. They might be unable to function completely as a nano-order reinforcing agent. Poor dispersion methods and the usage of non-modified clay particles or low viscous polymer can contribute to this phenomenon. In most cases, poor dispersion of clay particles during preparation often causes nanoclay agglomeration, and induces a decrease in the mechanical properties of the material.

Fig. 2.1 Schematic of chemical structure of montmorillonite mineral.
To realize the importance of morphological analysis of nanoclay cluster or agglomerates, an electron probe micro-analyzer (EPMA) was used in this study. Indeed, this approach is important to ascertain the relation between the microstructure at the fracture surface and mechanical properties. Fatigue failure commonly originates from a local point within the material, at which high stress concentration occurs, resulting in crack initiation. Consequently, this study is of great importance in elucidating how the crack initiation is related with the morphology of the nanoclay cluster or agglomerates in the polymer matrix because the fatigue life of materials often depends on the number of cycles causing the crack initiation.

In this chapter, a study was conducted to introduce quantitative analysis of nanoclay agglomerates for use with observation of fatigue-fractured nanoclay-filled epoxy resin matrix composites, and to clarify nanoclay agglomerates’ size and dispersion states. For this purpose, different amounts of nanoclays were incorporated into the resin through the conventional mechanical and ultrasonic processes. Analyses were conducted using EPMA at arbitrary and crack initiation areas on the fatigue-fractured surface. Finally, we examined the affinity between the results obtained from quantitative analysis and fatigue tests.
2.2 Experimental methods

2.2.1 Materials

In this study, bisphenol-F type epoxy resin (EP-4901, Density: 1.19 Mg/m$^3$) was used as a matrix material, and polyetheramine hardener (Jeffamine T-403, Density: 0.978 Mg/m$^3$; Huntsman Co., Ltd.) was used as a curing agent as shown in Fig. 2.2. Both EP-4901 and Jeffamine T-403 were mixed in the weight ratio of 100:46 to produce a thermosets resin. Regarding reinforcement, nanoclay (Nanomer®I.28E, Bulk density: 0.25-0.30 Mg/m$^3$; Nanocor Inc.) was used as shown in Fig. 2.3. As described in earlier reports, clay particles can be dispersed properly throughout the polymer matrix despite small weight percent values [9, 18, 21–23]. Consequently, it is expected that the clay particles can create much higher surface area for polymer/filler interaction than conventional micro-order clays can.

![Overview of (a) bisphenol-F type epoxy resin and (b) polyetheramine hardener.](image)

Fig. 2.2 Overview of (a) bisphenol-F type epoxy resin and (b) polyetheramine hardener.
2.2.2 Specimen fabrication

First, 1 phr, 3 phr, or 5 phr of nanoclay amounts were measured. They were mixed with EP-4901 epoxy resin in a beaker with a mechanical stirring device (PM-201; As-One Corp.) at 2500 rpm for about 3 min. Then, the mixture was mixed further using an ultrasonic homogenizer device (Branson Sonifier 450; CSC Co.) for another 10 min. An overview of the mechanical stirring device and the ultrasonic homogenizer are shown in Fig. 2.4. During the sonication process, the beaker was placed in cold water to avoid increasing the solution’s temperature [9]. The solution’s temperature was measured using a thermometer every one minute. Subsequently, the Jeffamine T-403 hardener was added to the solution and was stirred manually for about three more minutes at room temperature.

Then, nanoclay–epoxy resin solution was poured into a metallic mold as shown in Fig. 2.5. A mold lubricant (QZ13; Huntsman Co., Ltd.) was applied beforehand to prevent adhesion to the metallic mold of the epoxy resin. The mold was then placed in a vacuum pump for 10 min to remove air bubbles from the resin. The specimens were cured completely after placing them in a homo-isothermal drier (MOV-112F; Sanyo Co.) for 24 h at 30 °C; then for 6.5 h at 60 °C. The
nanoclay volume fractions of 1 phr, 3 phr, and 5 phr specimens were 0.007, 0.019, and 0.032 respectively. The overview of vacuum pump and the homo-isothermal drier are shown in Fig. 2.6.

The specimen dimensions of tensile and fatigue tests are presented in Fig. 2.7. The thickness and width of each specimen were measured respectively at three points: both ends and the center of the gauge part. These average values were recognized respectively as the specimen thickness and width. Then, the aluminum tabs were attached to both ends of the specimens using an adhesive.