

# Comparison Between Microwave and Thermal Curing of Glass Fiber–Epoxy Composites: Effect of Microwave-Heating Cycle on Mechanical Properties

Varaporn Tanrattanakul, Dumrong Jaroendee

*Polymer Science Program, Faculty of Science, Prince of Songkla University, Hadyai, Songkla 90112, Thailand*

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**ABSTRACT:** The objective of this study was to compare the mechanical properties between epoxy composites cured by thermal heating and microwave heating. Epoxy-anhydride resins reinforced with glass fiber were cured in a domestic microwave oven and in a thermal oven. Hardening agents included methyl tetrahydrophthalic anhydride and methyl hexahydrophthalic anhydride. Microwave curing was carried out at various conditions, including 1-, 2-, and 3-step heating cycle, whereby each cycle employed different power level and time. Mechanical properties were tested according to ASTM standards. It is found that the microwave-cured composites produced mechanical properties as good as the thermally cured composites. The 2- and 3-step heating cycle used in the microwave curing process pro-

duced better mechanical properties higher than those obtained from the microwaved 1-step and thermally curing process. This is attributed to the slow increase in temperature during the beginning of the microwave curing process whereby the very low power level was applied in the first cycle of the multistep heating process. This affected the slower rate of viscosity increment, resulting in better wettability of the glass fiber with enhanced interfacial adhesion between the fibers and the resins. The viscosity of resins affected the homogeneity of the crosslinked structure. © 2006 Wiley Periodicals, Inc. *J Appl Polym Sci* 102: 1059–1070, 2006

**Key words:** composites; crosslinking; curing of polymers; resins; glass fiber; epoxy

## INTRODUCTION

Since the mid-1980s, there has been a great deal of interest in microwave-cured processing of polymers. There have been a lot of researchers in the area of microwave-cured processing for epoxy resins as reviewed by Tanrattanakul and SaeTiaw.<sup>1</sup> Microwave processing for polymer applications include crosslinking of polymer networks, polymerization of thermoplastics, curing of laminates, and joining and repairing of composites. The use of microwave-cured processing is expected to greatly reduce curing time and therefore the operating cost would be lower. A reduction in residual stress in the processed materials and an improvement in the final properties of the material are to be expected. Although most published articles of microwave-cured processing in polymer have been reported on crosslinking and polymerization of resins, however some researchers studied the microwave-

cured composites, for examples, glass fiber/epoxy composites,<sup>2–8</sup> glass fiber/epoxy laminates,<sup>9</sup> carbon fiber/epoxy composites,<sup>10,11</sup> glass-graphite/polyimide composites,<sup>12</sup> carbon fiber/polyimide composites,<sup>13</sup> glass fiber/PMMA composites,<sup>14,15</sup> graphite/epoxy laminates,<sup>16</sup> and thermoplastic composites.<sup>17</sup> Boey et al.<sup>2–4</sup> showed that the microwave-cured epoxy-amine system reinforced with glass fiber had strength and stiffness as high as or better than that cured in a thermal curing process. They suggested that the two main process parameters affecting the final mechanical properties of the thermoset composites are the cure cycle and the reduction in void content. To affect the former, a conventional thermal process is used normally, with the cycle duration lasting a matter of hours. It is commonly known that in the case of polymer matrix composites, the load is taken mainly by the fiber and the fiber–matrix interfacial strength is very important because of load transfer. Interfacial properties of the microwave-cured composites have been reported.<sup>6,7</sup> In general, the fiber–matrix interface is considered to be an intensive chemico-physical linking, which depends on the local curing process, or more precisely, local thermal transfer across the interface.<sup>6</sup> For the E-glass fiber/epoxy system, the E-glass fiber cannot be heated by microwaves because of the

Correspondence to: V. Tanrattanakul (varaporn.t@psu.ac.th)

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transparency, the epoxy is heated and it then heats the E-glass fibers by thermal conduction. So heat passes through the interface from the epoxy to the glass fiber. In the case of thermal curing, the E-glass fibers are heated first and then the epoxy. The thermal gradient across the interface decreases from the fiber to the epoxy. Bai and Djafari<sup>6</sup> reported that although the microwave-cured samples provided more voids but their mechanical properties were similar to those of the thermally cured samples, and the fiber-matrix interface of the microwave-cured samples was stronger than that of the thermally cured samples. They proposed that if the curing is too fast and the pressure is too low, the voids are susceptible to being trapped in the materials after curing, which results in a degradation of properties. Yue and Looi<sup>7</sup> also found that the interfacial shear strength of the microwave-cured composites was higher, but the strength from the pull-out test became lower. Liu et al.<sup>12</sup> employed a domestic microwave oven for fabricating polyimide composites by using multistep heating cycle because of the reaction path of resin used. They mentioned that the electrically conducting fibers, such as graphite and carbon fibers, absorb the microwave energy more efficiently than the nonconducting materials, and microwave process may enhance the bonding between resin and fiber matrix. Fang and Scola<sup>13</sup> reported that microwave-cured carbon fiber/polyimide composites showed better interfacial adhesion than that of thermally cured composites.

The aim of this study was to study the processability of microwave curing process for epoxy-anhydride system reinforced with glass fiber by using a domestic microwave oven. Mechanical properties of the microwave-cured composites have been compared with those of the conventional thermal cured composite. The improvement of mechanical properties of the microwave-cured composites and the origin of this improvement will be discussed. The direct comparisons between the kinetics of curing activated by thermal and microwave heating are beyond the scope of this study, and the kinetic mechanisms during the polymerization of the reactive system are not the aim of this article.

## EXPERIMENTAL

### Materials

The epoxy resin was a general-purpose-grade of diglycidylether of bisphenol A (DGEBA) with  $n = 0.15$  and epoxy equivalent weight (EEW) = 184–189 g/equiv. Two hardeners were employed: methyl tetrahydrophthalic anhydride (MTHPA) and methyl hexahydrophthalic anhydride (MHHPA). The accelerator included tris-2,4,6-dimethyl aminomethyl phenol (DMP-30). MTHPA contained an unknown accelera-

tor, whereas MHHPA contained no accelerator. The glass fiber was chopped strand mat with specific weight = 300 g/m<sup>2</sup>. All chemicals were commercial-grade and were used as received.

### Sample preparation

The main suppliers of the anhydride hardeners suggested using approximately 80–90 wt % of the anhydride/epoxy stoichiometric ratio to obtain better properties. For that reason, a ratio of 80 : 100 anhydride/epoxy was selected. The concentration of the accelerator (DMP-30) was 4 parts per 100 parts of epoxy resin. The sample designation and compositions are listed in Table I. After good mixing, air bubbles were released from the resin before it was poured into a mold. A Teflon mold (20 cm in diameter and 5 cm deep) was used for both thermal and microwave curing. The resin and the glass fiber were weighed to maintain a constant resin-to-fiber ratio (15 wt % fiber). After pouring the resin for 1/4 of mold volume, a glass fiber mat soaked with the resin was placed into the mold, and the rest of the resin was filled. A Memmert U500 oven (Schwabach, Germany) was employed for thermal curing. Microwave curing was performed in a Sanyo EM-X412 commercial microwave oven (Sanyo Thailand Co, Thailand) at a frequency of 2.45 GHz. The microwave oven was equipped with a turntable to prevent formation of hot spots because of nonuniform heating. This microwave oven was fitted with a voltage controller to facilitate the processing power (to be specified). The maximum output is 800 W, and the power output can be manually adjusted between 10 and 100%, represented by power levels (L1–L10). Each power level consisted of a time period when the power was on followed by a period when the power was off, and the timings are given in Table II. The curing samples were relatively large to avoid the nonuniformity of microwave field in the microwave oven. The microwave oven calibration was reported earlier.<sup>1</sup> In this work, the applied power was based on physical performance and mechanical properties of cured samples. No air bubbles and no burning were criteria for good performance specimens. The heating conditions for microwave and thermal curing are described in Table III. Microwave curing processes were classified into three types: 1-step heating (1S), 2-step heating (2S), and 3-step heating (3S). The single-step heating (1S) was done by using

TABLE I  
Sample Designation and Composition

Composite	Hardener	Accelerator
I	MTHPA	Unknown
II	MHHPA	4% DMP-30

**TABLE II**  
Duration of Microwave Heating at Each Power Level

Power setting (level)	Duration power on (s)	Duration power off (s)
2	3	14
3	5	12
4	7	10
5	9	8
6	11	6

only one power level. The multistep heating was done by using two or three power levels successively. Based on our product design, thermal curing was operated at 150°C and cure time should be between 15 and 30 min. The composite I needed 25 min to get a solid specimen, whereas the composite II could be solidified within 15 min. Initially, curing condition in the microwave oven was produced by trial and error, and the aim was to use the lowest power level and the shortest time. Curing conditions for both composites were not necessary to be similar.

### Mechanical property testing

The tensile properties, flexural properties (3-point bending), and notched Izod impact resistance were tested according to ASTM D638, ASTM D790, and ASTM D256, respectively. The Shimadzu 100kNG universal testing machine (Japan) was employed for the tensile and flexural properties testing, and the Zwick 5102B impact tester (Germany) was employed for impact testing. The tensile testing was conducted at a tension speed of 5 mm/min and at a gauge length of 50 mm. The three-point-bending test was executed at a speed of 5.3 mm/min with a span width of 100 mm; the specimen dimensions were 25 mm by 120 mm. The

impact resistance was tested with a 2J-pendulum. The specimen thickness was [ $<]3577\epsilon 3.5$  mm. Five specimens or more were tested for every sample for all the testing.

### Sample characterization

The extent of conversion of the cured samples was determined by using a PerkinElmer DSC7 (Norwalk, CT) at a heating rate of 10°C/min from 30 to 300°C. The Rheometric Scientific DMTA V (Piscataway, NJ) was employed to investigate  $\alpha$  transitions. The testing was performed in a three-point-bending mode at a heating rate of 5°C/min with a strain control of 0.02%. The applied frequency was 1.8 Hz within the temperature range of -120 to 250°C. The thermal gravimetric analysis was performed by using a PerkinElmer TGA7 (Norwalk, CT) at a heating rate of 10°C/min from 30 to 800°C under nitrogen environment. Fractured surfaces of impact-tested specimens were coated with gold prior to observation with a scanning electron microscope (Jeol JSM5800LV, Japan). An increase in temperature of curing resin was investigated. After curing at a certain time, the resin was taken away from the heating source and the resin temperature at the center of the mold was measured immediately by using an infrared thermometer. This process took time [ $<]3577\epsilon 5$  s. The temperature profile was plotted as a function of cure time. Viscosity of resin cured in a certain time was determined by using Brookfield RV-DII viscometer. Those cured resins were kept in the freezer (-10°C) prior to measurement.

## RESULTS AND DISCUSSION

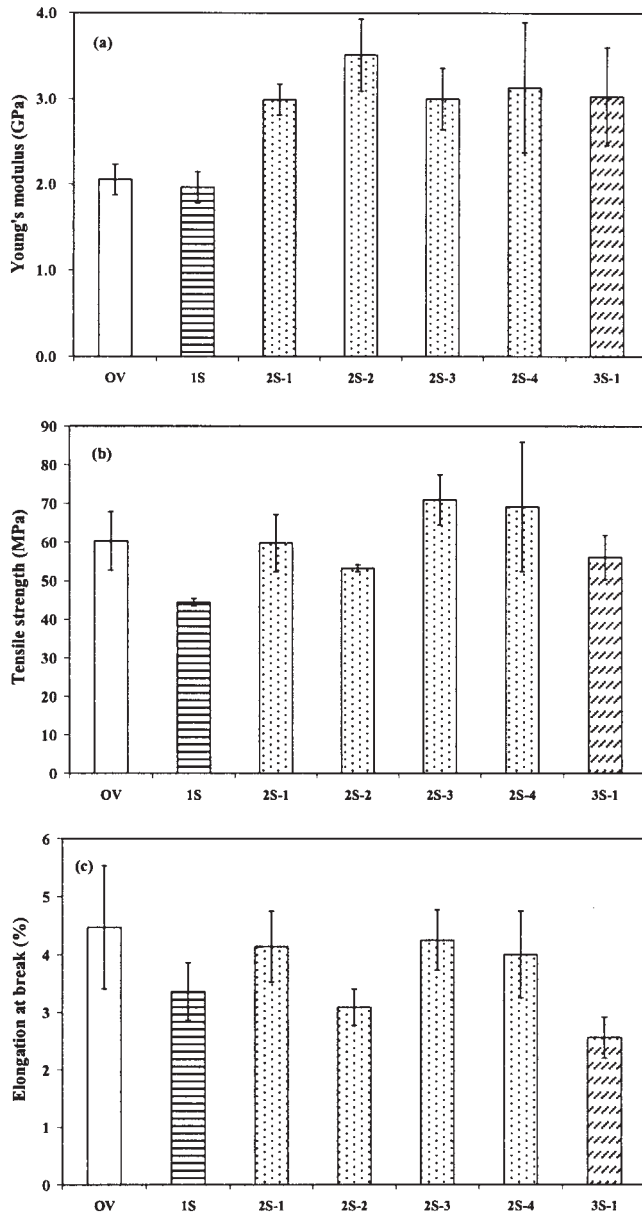
### Mechanical properties of composites

Figures 1–3 represent tensile properties, flexural properties, and impact strength of the composite I, respec-

**TABLE III**  
Heating Condition

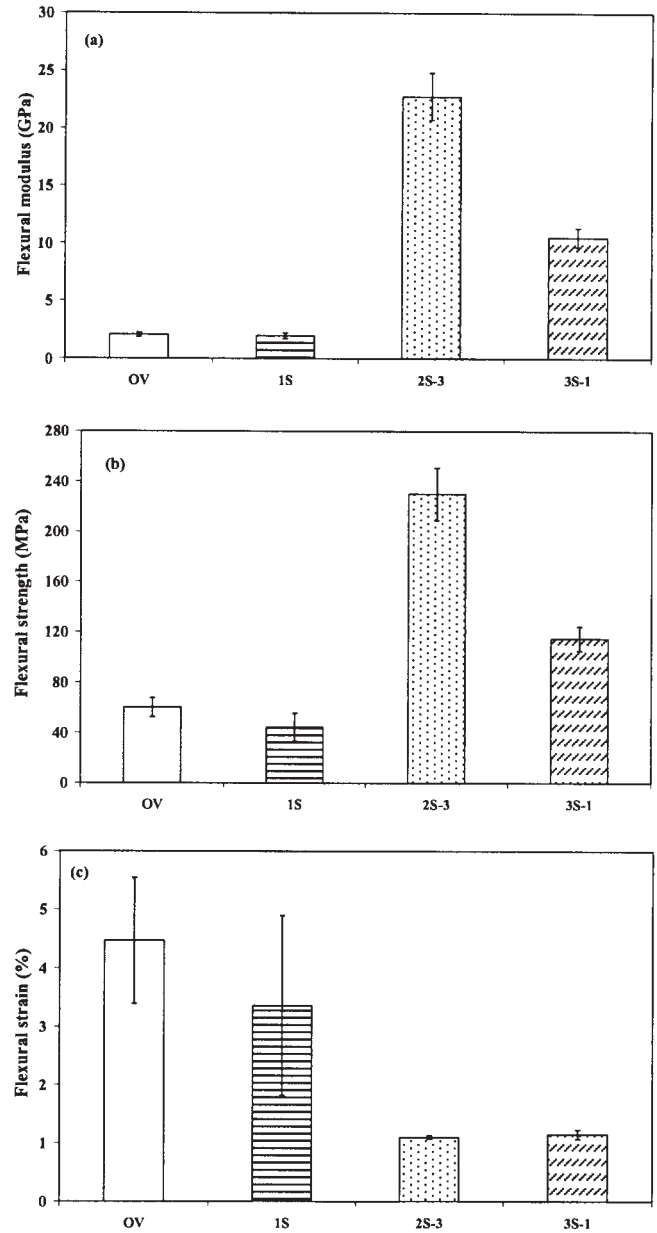
Code	Source of heat	Composites I		Composites II	
		Condition	$T^*$ (sec)	Condition	$T^*$ (sec)
OV	Thermal oven	150°C, 25 min	1500	150°C, 15 min	900
1S	Microwave oven	L3/10	180	L3/7	125
2S-1		L2/20 + L4/5	339	L2/10 + L4/5	234
2S-2		L2/25 + L3/5	357	L3/5 + L4/3	167
2S-3		L2/25 + L6/5	465	L2/15 + L4/5	285
2S-4		L2/20 + L3/10	393	L2/10 + L4/7	285
2S-5		–	–	L3/5 + L4/5	216
3S-1		L2/20 + L3/10 + L5/5	555	L2/10 + L3/5 + L4/3	275
3S-2		–	–	L2/10 + L3/5 + L4/5	324
3S-3		–	–	L2/10 + L3/7 + L4/3	310
3S-4		–	–	L2/10 + L3/3 + L4/7	338
3S-5		–	–	L2/15 + L3/5 + L4/5	375

Note: Lx/y = curing at microwave power level "x" for "y" min.  $T^*$  = actual heating time.

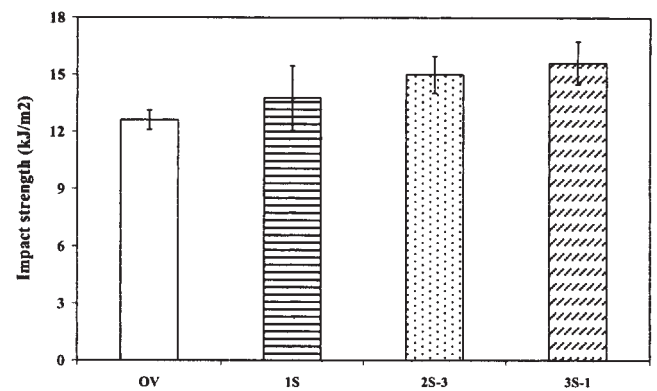


**Figure 1** Tensile properties of composites I: (a) Young's modulus; (b) tensile strength; (c) elongation at break. OV = oven curing, 1S = 1-step curing, 2S = 2-step curing, and 3-S = 3-step curing.

tively. The "OV" (thermal curing) and "1S" (1-step microwave curing) samples are similar in terms of single-step process. The thermally cured samples showed higher tensile and flexural properties than the microwaved samples, except strain at break. In contrast, impact strength of the microwaved samples was higher. Mechanical properties of the composite II are shown in Figures 4–6. Comparing the "OV" with "1S" samples, the microwaved sample exhibited higher flexural modulus and strength, whereas the rest of their mechanical properties were lower than those of thermally cured samples. The uniformity of the microwave field was determined. The extent of cure was



**Figure 2** Flexural properties of Composites I: (a) flexural modulus; (b) flexural strength; (c) flexural strain.



**Figure 3** Impact strength of composites I.

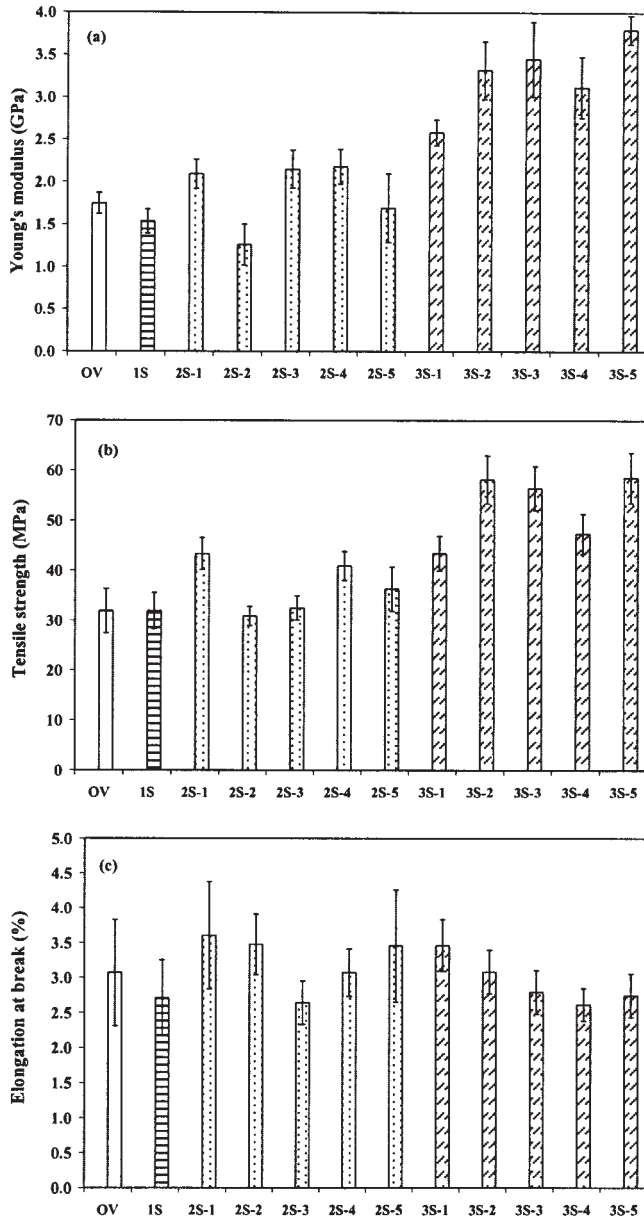


Figure 4 Tensile properties of composites II: (a) Young's modulus; (b) tensile strength; (c) elongation at break.

investigated by using DSC technique. The specimens were cut from the center and the edge of the 20-cm diameter cured samples. No significant difference in the extent of cure between both positions was observed. This indicates the uniform microwave field. Considering the curing time of both composites, the microwaved samples were cured under shorter time period. The composite I took 25 and 10 min in the thermal oven and microwave oven, respectively. The composite II took 15 and 7 min in the thermal oven and microwave oven, respectively. As mentioned earlier, the requirement for microwave curing condition was using the minimum power level and time. For single-step curing of the composite I, the power Level

3 (L3) was the minimum applicable level; however, the cure time cannot be longer than 10 min because if more than 10 min used, the specimens will be burnt. Likewise, curing at L3 for 7 min was the mildest condition for 1-step heating of the composite II. The higher power level or the longer time caused the samples to be burnt or very brittle. For a domestic microwave oven, the setting time is not equal to the actual heating time.<sup>1</sup> The actual heating time for L3 at 10 and 7 min was 180 and 125 s, respectively. This means the microwave oven spent only 30% of the setting time for irradiation. Although good mechanical properties from the single-step process with shorter time in microwave oven could be derived, it is possible to obtain the better mechanical properties of the microwaved samples if the proper heating condition was selected.

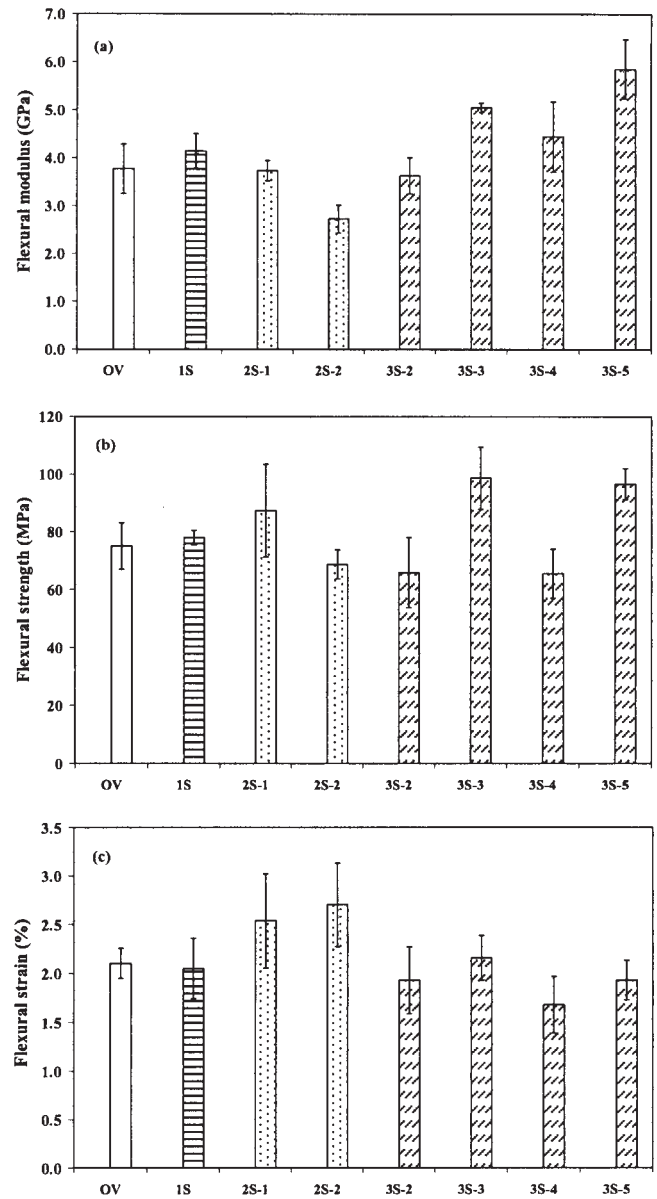


Figure 5 Flexural properties of Composites II: (a) flexural modulus; (b) flexural strength; (c) flexural strain.

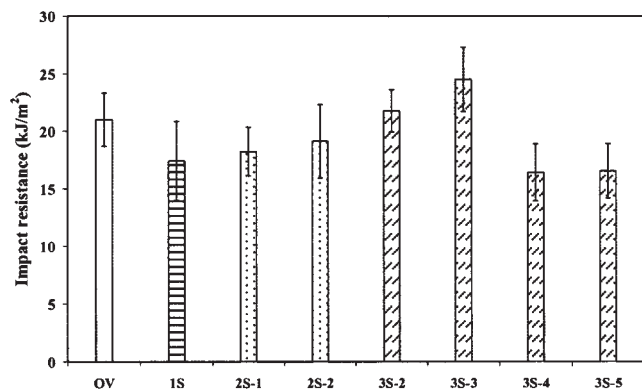


Figure 6 Impact strength of composites II.

It is well known that microwave heating is different from thermal heating. The microwave energy is absorbed by the molecules through the polarization or dipole reorientation of functional groups, which is consequently converted into thermal energy.<sup>12</sup> As a result, heat is generated within the molecule, and, thereby, a homogeneous heating pattern is created in the materials. This process is attributed to inside-out solidification of curing resin. On the other hands, in conventional thermal heating, heat energy is transferred from the surfaces (outside) to inside of the material by the conduction of the heating medium, resulting in outside-in solidification. Different states of heating between microwave and thermal heat may cause differences in viscosity and temperature profile of curing resin, which should affect crosslinking, fiber-matrix interfacial adhesion, and homogeneity of resin-microstructure. It is believed that to obtain the better mechanical properties, the cure time in the microwave oven must be increased. To do so, it is necessary to preheat the resin. Therefore, the present strategy was using cycled heating by preheating in the beginning and followed by regular heating, as called "multistep heating".

As expected, the multistep process (2S and 3S) increased the mechanical properties as shown in Figures 1–6. Remarkably, modulus and strength of both composites cured by multistep heating were higher than those of thermally cured samples and single-step microwaved samples. Because the proper condition for multistep heating was difficult to predict, therefore, the initial experiment was by trial and error. However, several conditions for both composites have been tested, and conditions that offered good mechanical properties are shown in Table III. The composite I needed longer cure time than the composite II, this may be due to different accelerator type and content and different hardener type. On the basis of the preliminary study, the 3-step heating of the composite I took too much cure time, hence, only one condition (3S-1) has been selected. The 2S-3 heating condition

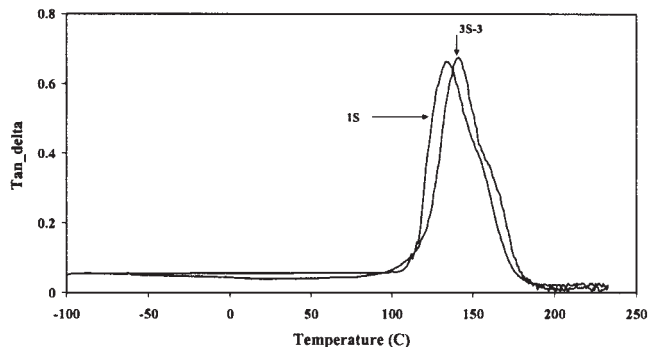


Figure 7 The  $\alpha$  transition temperature of composites II. The 1S sample obtained from the 1-step heating process shows lower transition temperature than that obtained from the 3-step heating process.

seemed to be the best condition of the composite I. It showed extremely high flexural properties and higher tensile strength than the 3S-1 and OV sample. Conclusively, the 2-step heating is appropriate for the composite I in this study. There was no effort to obtain the very good mechanical properties from the 3-step heating of the composite. In contrast, the 3S-3 sample,

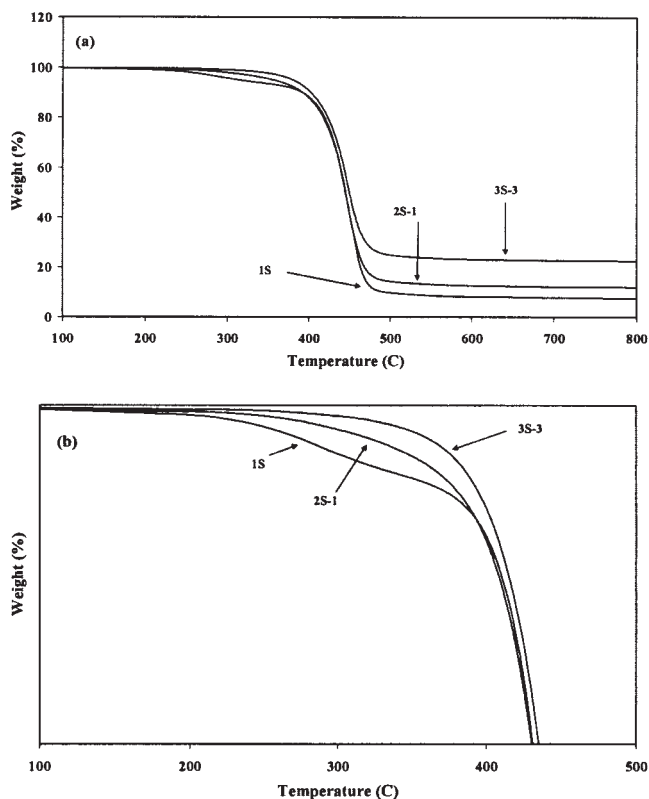
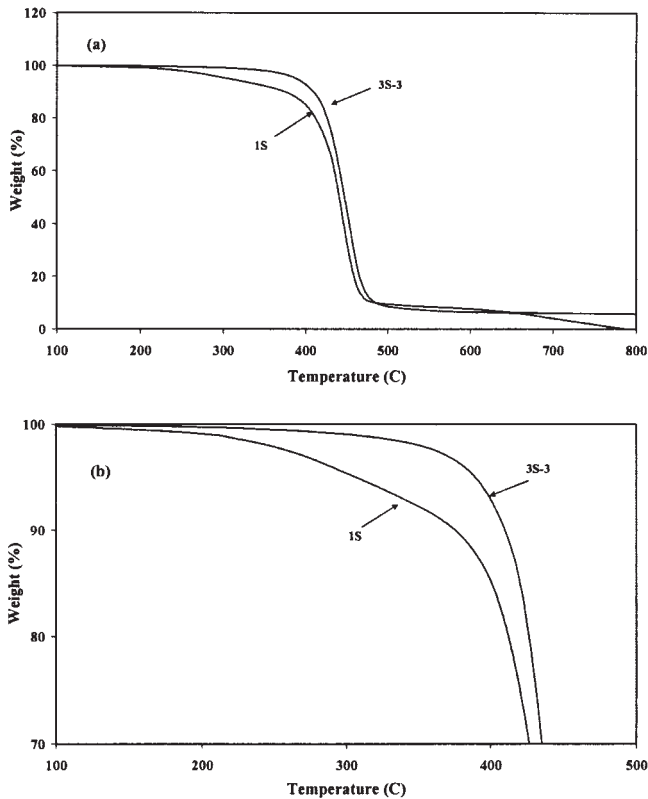


Figure 8 TGA thermograms of composites II. The 1S and 2S-1 samples (obtained from 1- and 2-step heating processes) show 2-step degradation process, indicating the entrapment of uncrosslinked epoxy in the network. The 3-step heating process offered highest crosslink density. (b) is the enlarged scale of (a).



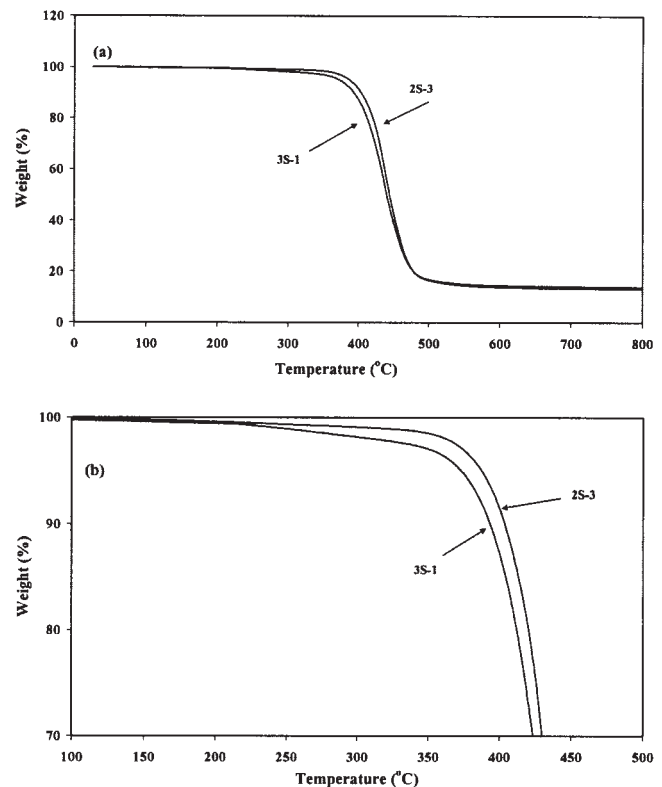
**Figure 9** TGA thermograms of Resins II. The 1S samples (1-step heating process) show 2-step degradation process, indicating the entrapment of uncrosslinked epoxy in the network. The figure (b) is the enlarged scale of figure (a).

3-step heating, provided the best mechanical properties among the composite II. All its properties were higher than those of OV-, 1S-, and 2S-samples. It is known that the volume fraction of fibers and the void content are critical parameters. To confirm the results obtained from the multistep heating, mechanical properties of some composites were retested and reproducible data were received. As a result, it is believed that the differences in the moduli of the composites in this study come from the heating cycle because the experiment was a small scale and well controlled. In these studies, it is necessary to employ the multistep heating in the microwave oven to obtain higher mechanical properties, and this procedure also yielded higher mechanical properties than the thermal curing. It is believed that the interfacial adhesion will increase when the right multistep process is used, further proved by the increase in flexural strength.

### Curing characteristics

It was successful to obtain the high modulus and strength from the multistep heating process in the microwave oven, 2S-3 and 3S-3 for the composite I and II, respectively. It was interesting to study why the

multistep heating was better than the single-step heating and why the composite I preferred the 2-step heating whereas the composite II preferred the 3-step heating. On the basis of the DSC results, no exothermic peak was observed. Normally, one expected that higher glass-transition temperature of the composite showed higher modulus and strength. The  $\alpha$  transition temperature of the 1S and 3S-3 samples of the composite II were 138 and 143°C, respectively (Fig. 7). Unfortunately, the  $\beta$  transition temperature could not be observed. The storage modulus at the rubbery plateau region (at 200°C) of the 3S-3 sample was  $2 \times 10^7$  Pa and that of the 1S sample was  $1.2 \times 10^7$  Pa, reflecting the higher crosslink density in the 3S-3 sample. Although the difference in the transition temperature was not to a great extent, DSC results showed significant differences.  $T_g$  of the 1S, 2S-1, and 3S-3 samples became 78, 106, and 115°C, respectively. As a result, the relationship between mechanical properties and molecular architecture (or morphology) of the composites in this study is corresponded to the above assumption. However, the differences in  $T_g$  may not always relate to mechanical properties. For example, the thermally cured carbon fiber/polyimide compos-



**Figure 10** TGA thermograms of composites I. The 3S-1 sample degraded prior to the 2S-3 sample, indicating the higher crosslinking reaction in the 2S-3 sample. The entrapment of uncrosslinked epoxy in the network may occur in the 3S-1 sample. The figure (b) is the enlarged scale of figure (a).

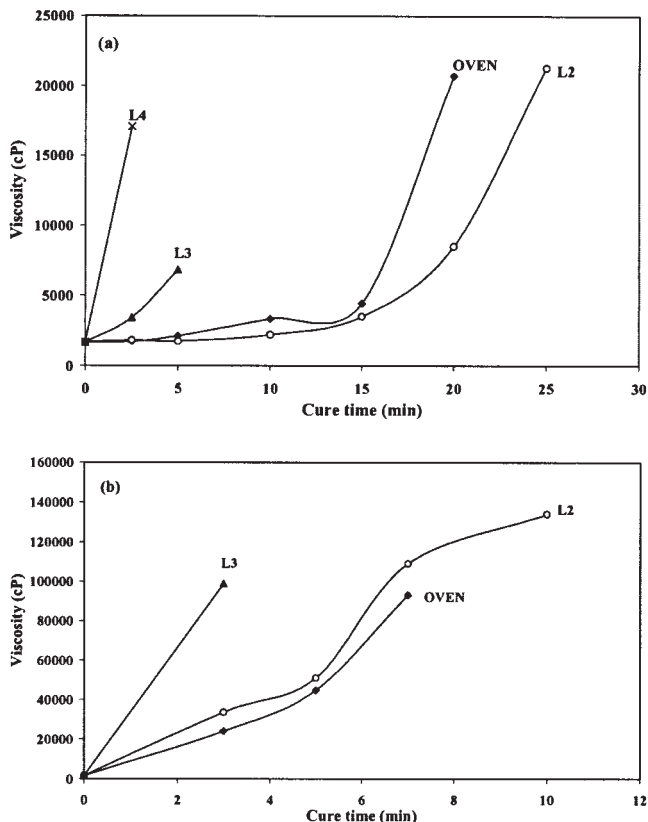


Figure 11 Viscosity of resin during curing in the thermal oven (OV) at 150°C and microwave oven at various power levels (L2–L4): (a) composite I; (b) composite II.

ite<sup>13</sup> showed lower  $T_g$  but higher flexural strength than the microwave-cured composite. This is because the interfacial adhesion between fiber and matrix also plays an important role on the mechanical properties.

TGA technique exhibited very useful information. The TGA thermograms of 1S, 2S-1, and 3S-3 samples of the composite II are displayed in Figure 8. The 1S and 2S-1 samples showed 2-step degradation process and started degradation around 200°C, whereas the 3S-3 sample showed 1-step degradation process and started degradation after 240°C. An extent of the first degradation decreased from 6.6% in the 1S sample to 4.2% in the 2S-1 sample. On the basis of the TGA results, it is believed that the higher crosslink density provides higher thermal degradation. It seemed that the crosslink density ranked in the following order: 3S-3 > 2S-1 > 1S. These phenomena were verified by investigating the resin II, containing no glass fiber. The experimental results were similar, the 1S and 3S-3 samples showed 2- and 1-step degradation processes, respectively (Fig. 9). There are differences in the 1- and 2-step degradation process because of the differences in microstructure or structure homogeneity of the resins. For some reasons, there were some defects in the crosslinked structure, some prepolymer or some uncrosslinked epoxy in the 1S and 2S-1 samples and this

imperfection was attributed to the first degradation and relatively low mechanical properties. Therefore, the 3S-3 sample of the composite II was the best sample. This assumption was supported by TGA thermograms of the composite I as illustrated in Figure 10. The 3S-1 sample showed the small degradation before 200°C, whereas this degradation was not observed in the 2S-3 sample. As stated earlier, the 2-step heating process was the preferential process for the composite I. This is because the 3S-1 sample may contain imperfection as stated earlier, represented by the first degradation before 200°C. It appeared that TGA data coincided with its mechanical properties such as the higher mechanical properties and the higher degradation temperature. It is established that the lower degree of cure was observed in microwave samples and explained by the entrapment of reactive function within the network.<sup>18</sup> This is not in this study because DSC results showed no further crosslinking reaction, therefore, it is plausible that the imperfection of the samples derived from the entrapment of uncrosslinked resin in the network.

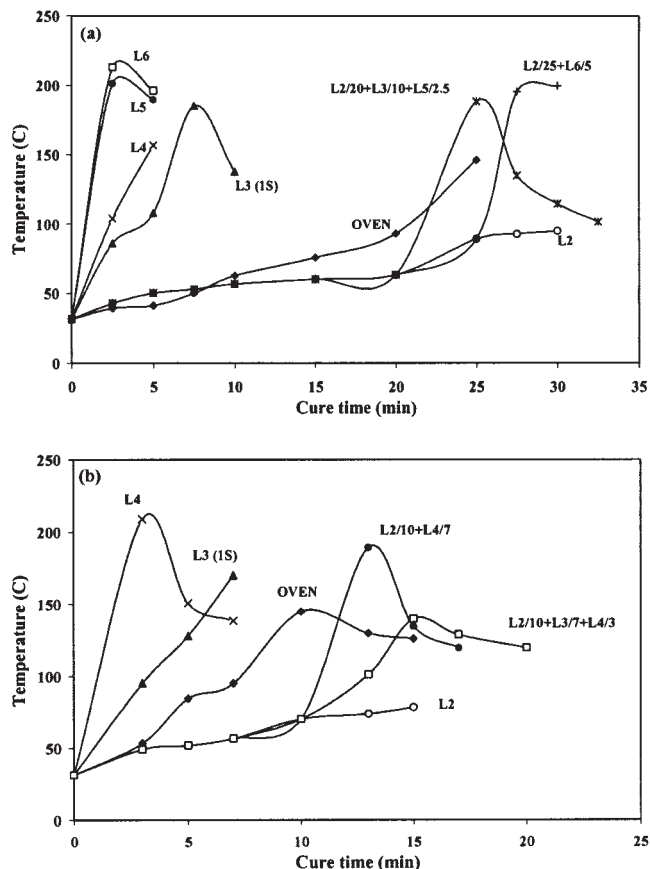
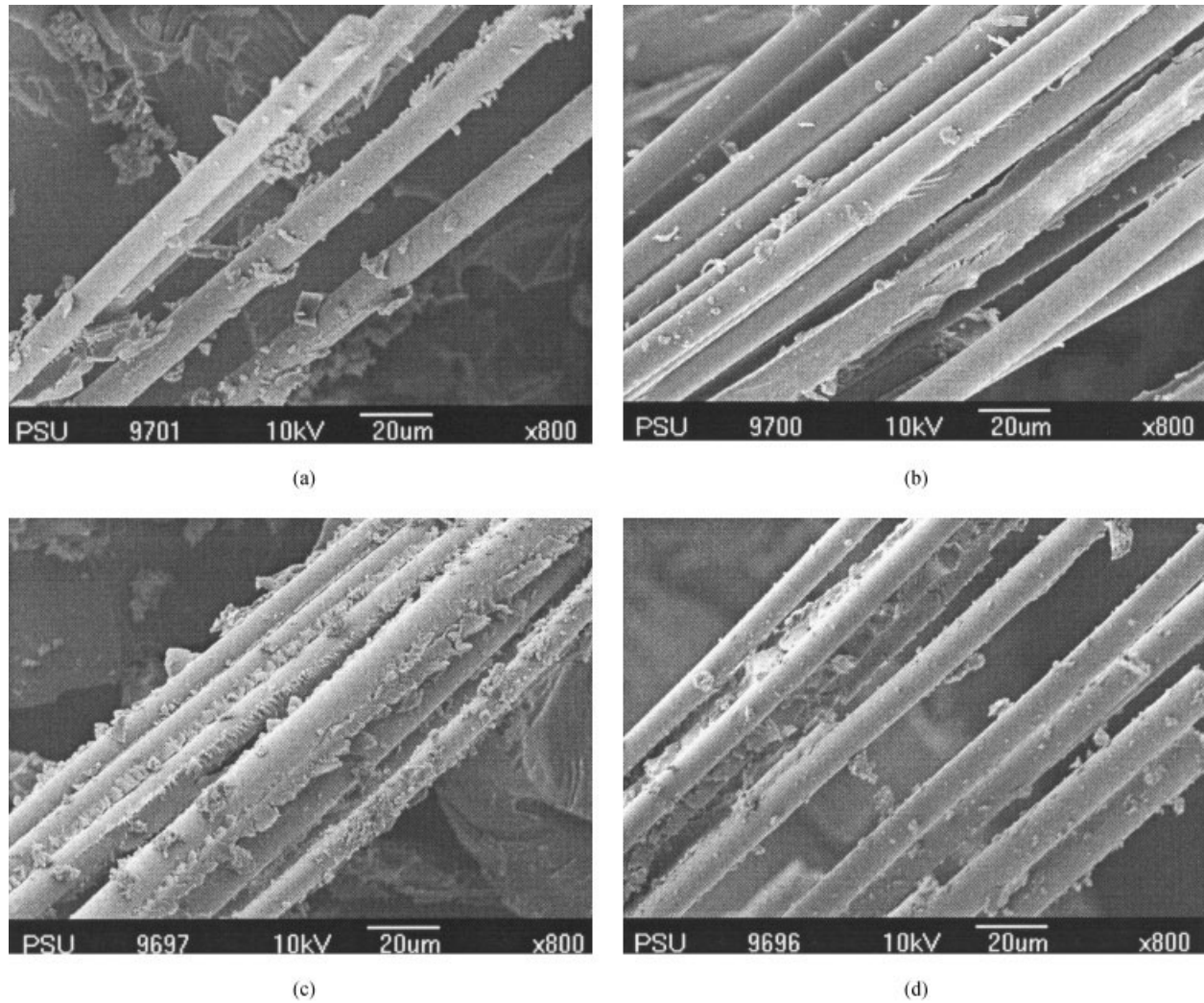


Figure 12 Temperature of resin during curing in the thermal oven (OV) at 150°C and microwave oven at various power levels: (a) composite I; (b) composite II.





**Figure 13** SEM micrographs of fractured surfaces of composites I: (a) the OV sample; (b) the 1S sample; (c) the 2S-3 sample; (d) the 3S-1 sample. The 2S-3 sample (c) showed most resin adhered on the fiber than other samples, indicating most interfacial adhesion in this sample.

### Viscosity and temperature profile effect

It is assumed that factors affecting structure homogeneity of epoxy resin and fiber-matrix interfacial adhesion were resin viscosity and temperature profile during curing process. Rapid increase in viscosity may cause imperfection such as defects in crosslinking because of the entrapment of uncrosslinked resin.<sup>19</sup> When the resin became too viscous in the early state of crosslinking process, flowability or diffusion of epoxy and hardener were lowered, which caused locally inhomogeneous structure. The viscosity effect, associated with the progress of the reaction, hindered more and more the Brownian motion and lowered the kinetics of curing.<sup>20</sup> Viscosity of resins cured in the thermal oven and microwave oven at a certain period were measured. The setting temperature of the thermal oven was 150°C. Changes in viscosity during curing are shown in Figure 11. Viscosity increased

with curing time. Some parts of the composite I were slightly solidified or became jelly-like at the following conditions: L3 for 7.5 min and L5 for 2.5 min, whereas the following conditions provided solid fraction: L4–L6 for 5 min. These specimens were unable to measure viscosity. In the same way, the composite II containing jelly-like or solid-like fraction was disregarded, including L2 for 15 min, L3 for 5 min, and L4 for 3 min. It appeared that the increase in viscosity of the resins obtained from the thermal oven and the microwave oven at L2 was similar, and the higher power level, the higher rate of changes in viscosity. Remarkably, viscosity at L2 for 25 min and 10 min of the composite I and II, respectively, were very high when compared with viscosity at L3 for 5 min and 3 min of the composite I and II, respectively. Concerning to heating condition of the composite I, the 1S sample was converted into a partial solid around 7.5 min and

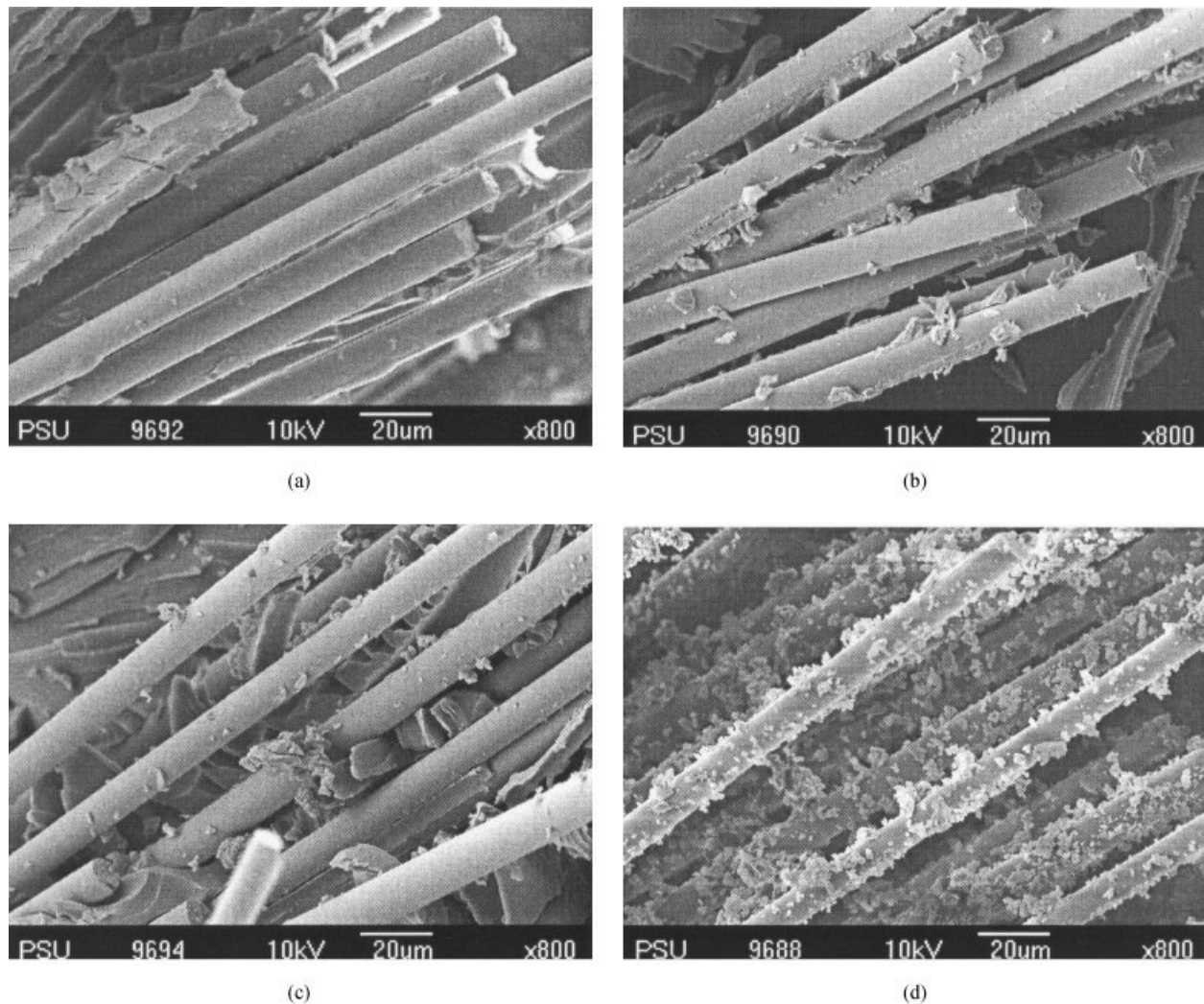
became a complete solid at 10 min at L3. Although the 1S sample solidified prior to the 2S-3 sample (curing condition: L2/25min + L6/25min), its viscosity before gelation (3420 cP at L3 for 5 min) was lower than that of the resin cured at L2 for 25 min (21,300 cP). This indicated that the rate of change in viscosity seemed to be more important than the viscosity before gelation and the slower rate was more favorable. Changes in viscosity of the multistep composite II, the 2S-1 and 3S-3 samples were different. Their curing conditions were L2/10 min + L4/5 min and L2/10 min + L3/5 min + L4/3 min. Both samples became a partial solid at the second heating of cure cycle, L4/3 min and L3/5 min for the 2S-1 and 3S-3 samples, respectively. The viscosity before gelation of the 1S sample (98,900 cP at L3/3 min) lowered than that of the 2S-1 and 3S-3 samples (134,000 cP at L2/10 min). The system preferred relatively slow rate of increase in viscosity.

The temperature profile of the curing resins elucidated the effect of curing cycle. Figure 12 illustrates the temperature profile of both composites. Fresh resin was employed for each cure time, and sample size was the same as that prepared for mechanical properties testing. The thermal oven was set at 150°C, the microwave oven was set at L2–L6 and also set for the multistep condition. Temperature was measured immediately after turning off the microwave power or taking away from the thermal oven. Therefore, these temperatures reflect the true sample temperature. Although this measure is less accurate than the *in situ* measurement, some researchers<sup>21</sup> also employed this technique. The measurement was done within 5 s, which was longer than the response time of the IR thermometer. If some error occurred, all measurement should obtain same error. Therefore, comparison of the temperature is acceptable. Temperature increased with increasing cure time, and some conditions showed a peak of the maximum temperature. These peaks represented the solidification temperature. For the 1-step heating process, the higher power level provided the higher rate of temperature increment, similarly to the viscosity behavior. The lowest rate of temperature increment was obtained from L2, and the maximum temperature at L2 was lower than 100°C for both composites. Undoubtedly, the resins could not be cured at L2, no matter how long the curing duration. The slow increase in temperature of L2 lowered the viscosity increment when compared with using other power levels. The faster rate of temperature increment at L3–L6 brought about the higher viscosity, which may accelerate the local crosslinking process or entrap the uncrosslinked epoxy in the network resulting in an inhomogeneous microstructure. For the multistep heating of the composite I, the 3-step heating condition may be inappropriate, compared with the 2-step heating process. Obviously, the 3-step sample solidified faster than the 2-step one, as represented by the

maximum temperature. Similar behavior was observed in the composite II, the 2-step cured sample became a solid prior to the 3-step cured sample (the better sample). This study showed that the slower solidification produced better mechanical properties. Similarly to the viscosity results, the slow rate of increase in temperature is preferable. It should be noted that the 2-step heating process could be better or worse than the 3-step, depending on the cure cycle. To obtain the optimum results, the best condition allowing the slow rates of temperature and viscosity increment must be available.

### Fiber–matrix interfacial adhesion

The higher power level caused the higher temperature in the resin because the higher power level provided longer time for irradiation, thus more heat generation occurred inside the resins. This was attributed to higher viscosity. By using the very low power level (L2) to act as preheating and to gradually increase viscosity, this enhanced the wettability of glass fiber and promoted interfacial adhesion between glass fiber and epoxy resin. The effect of microwave resin preheating on RTM laminates was reported.<sup>22</sup> It was proposed that preheating altered the viscosity and thermal “age” of the thermosetting resins. It was anticipated that a lower resin viscosity would improve fiber wet-out, increasing the mechanical properties of the laminate. SEM micrographs of the composite I and II are demonstrated in Figures 13 and 14, respectively. No void in the matrix was observed in every sample. The interfacial adhesion could be observed from the fractured surface. The cleaner, smoother surface of the glass fiber indicated the lower interfacial adhesion because lesser matrix adhered on the fiber. The entire fractured surface was observed. More resin adhered to the fiber surface of the microwave-cured (multistep) composite than to the fiber surface of the thermally cured composites. This suggests that adhesion of the resin to the fiber surface for the microwave-cured (multistep) composite was better than in the thermally cured composite. The 2S-3 and 3S-3 sample of the composite I and II, respectively, showed a considerable amount of resin adhering to the fiber surface, whereas the failure surface of the OV and 1S samples were essentially free of resin. This was a strong indication that better wetting of the fiber by the resin occurred in the multistep microwave-cured system than in the thermally cured system. The interfacial adhesion of the present composites may be ranked in the following order: 2S-3 > 3S-1 > OV, 1S (for the composite I); 3S-3 > 2S-1 > 1S > OV (for the composite II). This result is similar to that reported by Bai and Djafari<sup>6</sup> and Fang and Scola,<sup>13</sup> the fiber–matrix interface of the microwave-cured composites was stronger than that of the thermally cured composites. However,



**Figure 14** SEM micrographs of fractured surfaces of composites II: (a) the OV sample; (b) the 1S sample; (c) the 2S-1 sample; (d) the 3S-3 sample. The 3S-3 sample (d) showed most resin adhered on the fiber than other samples, indicating most interfacial adhesion in this sample.

in the case of microwave curing, the cure cycle is one of the most important parameter that should be of concern.

### CONCLUSIONS

This work has shown that the microwave oven was able to cure the glass-reinforced epoxy composites as good as or better than the thermal oven, and the multistep heating in the microwave oven was necessary. Appropriate heating cycle in the microwave oven was critical to provide good mechanical properties. By means of preheating, the increase in temperature was not too high and this gave rise to the slow rate of increase in viscosity, which improved fiber wet-out and reduced entrapment of uncrosslinked resin in the network. The rate of change in viscosity seemed to be more important than the viscosity before

gelation, and the slow rate was preferable. The improvement in mechanical properties of the appropriate multistep heating in the microwave oven was derived from better interfacial adhesion.

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