Effect of Quasi-Carbonization Processing Parameters on the Mechanical Properties of Quasi-Carbon/Phenolic Composites

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ABSTRACT: In this work, quasi-carbon fabrics were produced by quasi-carbonization processes conducted at and below 1200°C. Stabilized polyacrylonitrile (PAN) fabrics and quasi-carbon fabrics were used as reinforcements of phenolic composites with a 50 wt %/50 wt % ratio of the fabric to the phenolic resin. The effect of the quasi-carbonization process on the flexural properties, interfacial strength, and dynamic mechanical properties of quasi-carbon/phenolic composites was investigated in terms of the flexural strength and modulus, interlaminar shear strength, and storage modulus. The results were also compared with those of a stabilized PAN fabric/phenolic composite. The flexural, interlaminar, and dynamic mechanical results were quite consistent with one another. On the basis of all the results, the quasi-static and dynamic mechanical properties of quasi-carbon/phenolic composites increased with the applied external tension and heat-treatment temperature increasing and with the heating rate decreasing for the quasi-carbonization process. This study shows that control of the processing parameters strongly influences not only the mechanical properties of quasi-carbon/phenolic composites but also the interlaminar shear strength between the fibers and the matrix resin. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 3350–3357, 2008

Key words: composites; fibers; mechanical properties; high temperature materials; processing

INTRODUCTION

In general, carbon fibers have many advantages for diverse applications, such as excellent thermal and electrical conductivity, thermal stability, tensile properties, thermal-shock resistance, chemical inertness, and low thermal expansion.[1] However, they also have some disadvantages such as an extremely high heat-treatment temperature (HTT), low processibility, and high production costs. In particular, if one needs polyacrylonitrile (PAN)-based carbon fibers for carbon-fiber-reinforced phenolic composites with acceptable thermal insulation as well as heat resistance, commercial carbon fibers of high thermal conductivity may not be desirable.

Commercial PAN-based carbon fibers of general-purpose grade are, in general, proprietarily manufactured through a series of heat-treatment processes above 1400°C in an inert atmosphere.[2] When stabilized PAN fibers are treated at high temperatures, the chemical and physical characteristics of the carbon fibers obtained strongly depend on a variety of processing parameters.[3–5] Among the parameters, final HTT is most critical. It has been reported that HTT is very important for controlling the mechanical, thermal, and electrical properties of PAN-based fibers partially carbonized in the range of 400–950°C.[6–8] This implies that the properties of carbon fibers may be controlled and designed by the variation of the carbonization processing conditions according to their uses.

Stabilized PAN fibers can be successfully transformed into quasi-carbon fibers or partially carbonized fibers when they are heat-treated at carbonization temperatures lower than those for general-purpose carbon fibers.[7–11] Such a low-temperature carbonization process producing partially carbonized fibers is called *quasi-carbonization*. It has been reported that quasi-carbon fibers generally have lower mechanical properties, lower thermal conductivity, and higher electrical resistivity than general-purpose carbon fibers, depending on the quasi-carbonization process.[10,12] Therefore, it would be desirable if quasi-carbon fibers at a relatively low cost could, to some extent, replace conventional carbon fibers for thermal insulation applications such as heat-sink, heat-shielding, and ablation-resistant materials.[13] It is, of course, important to minimize the reduction of the properties and performances of
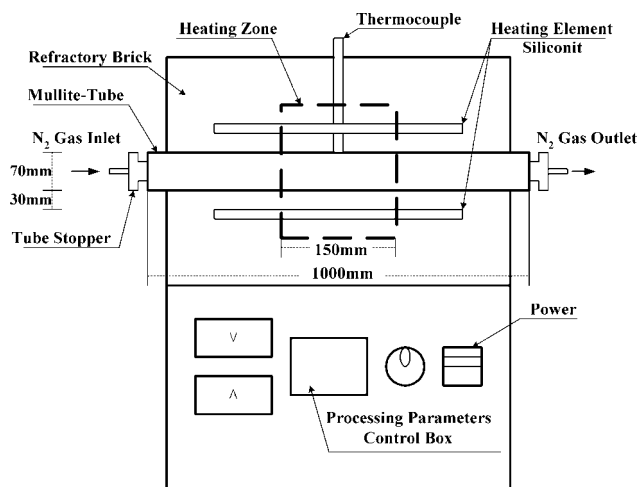
Figure 1 Schematic layout of the tube-type carbonization furnace used for the quasi-carbonization processes of stabilized PAN fabrics in this study.

quasi-carbon-fiber-reinforced composites. One practical approach for that is to optimize the processing parameters for quasi-carbonization, such as the HTT, heating rate, tension, and heating step.[3,5]

A thermosetting phenolic resin has good thermal insulation, thermal stability, heat-resistant mechanical properties, and interfacial adhesion with carbon fibers. Hence, phenolic resins have been widely used for a long time not only as frequently used materials in commodity and industrial applications but also as advanced materials in aerospace and military applications. One may combine their individual benefits with quasi-carbon-fiber-reinforced phenolic composites. However, no study has been reported on how the processing parameters for producing quasi-carbon fibers influence the properties of quasi-carbon fiber/phenolic composites.

Consequently, the objective of this study is to investigate the effect of quasi-carbonization processes conducted at and below 1200°C on the flexural properties, interlaminar shear strength (ILSS), and

dynamic mechanical properties of quasi-carbon/phenolic composites reinforced with the quasi-carbon fabrics produced from spun-type, stabilized PAN-based woven fabrics.

EXPERIMENTAL

Materials

Stabilized PAN-based woven fabrics composed of staple-spun yarns with an eight harness satin texture, supplied by Zoltek Co. (Bridgeton, MO), were used as starting materials for preparing quasi-carbon fabrics. The length of a staple-spun yarn was about 85 mm. The average diameter of a single filament of the stabilized PAN fiber was about 13.8 μm. The as-received stabilized PAN-based fabrics were heat-treated without any further modification according to the scheduled processing conditions. A resole-type phenolic resin used as a matrix was supplied by Kang Nam Chemical Co. (Ansan, Korea). Poly(vinyl alcohol) (PVA), which was purchased from Aldrich Chemical Co. (St. Louis, MO), was used as a sizing material for the quasi-carbon fabrics.

Quasi-carbonization processes

Quasi-carbonization processes were performed in a Siliconit mullite, tube-type carbonization furnace (Ajeon Heating Industrial Co., Nam Yang Ju, Korea) under an inert atmosphere, with the processing parameters, such as the final HTT, heating rate, and tension, being varied. The cylindrical heating zone in the furnace was 150 mm long and 70 mm high. Figure 1 illustrates the layout of the carbonization furnace used. Before the quasi-carbonization process, the stabilized PAN-based woven fabrics were cut to 300 mm in the warp direction and 50 mm in the fill direction. The thickness of a single ply of each fabric was 0.64 mm on average. Eight plies of the fabrics were tightly gripped with a tailor-made device for applying tension.
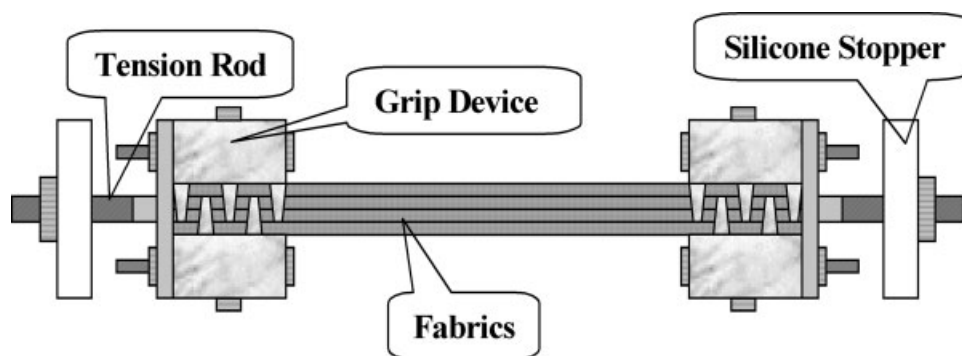

Figure 2 Schematic illustration of the stabilized PAN fabrics gripped with the tension device designed for the quasi-carbonization process.

Figure 2 displays an assembly of the fabrics gripped with the tension devices. The tension was applied by rotation of the stainless steel threaded rod directly attached to the grips in two opposite directions. The level of tension was controlled from the outside of the furnace before heat treatment, and it was kept constant throughout the quasi-carbonization process. The plied fabrics with grips were placed in the heating zone during the quasi-carbonization process. After the quasi-carbonization, the fabrics in the heating zone were cut to 130 mm in length and used for composite fabrication. The furnace was designed to manipulate all the processing parameters by programming temperature and time variables on purpose.

Fabrication of the quasi-carbon/phenolic composites

It was found in our earlier study[5] that the heating rate, HTT, and tension are the most critical processing parameters for controlling the mechanical properties of PAN-based quasi-carbon fabrics. Accordingly, two-directional quasi-carbon/phenolic composites were prepared with quasi-carbon fabrics produced with the three processing parameters previously mentioned.

Before composite fabrication, as-received stabilized PAN fabrics and quasi-carbon fabrics were sized with a 1.0 wt % PVA aqueous solution by a dip-coating method. The sized fabrics were fully dried at 110°C for 30 min. Quasi-carbon fabric/phenolic prepregs were prepared at 80°C for 5 min in an air-circulating oven. The ratio of the fabric to the phenolic resin was 1/1 (50% : 50%) by weight. The prepregs were cut to 50 mm × 50 mm and then placed in a stainless steel mold for compression molding. A stack of the prepregs was compacted by a debulking process to remove entrapped air.

The molding was performed with a hot press according to a cure profile as follows: at 70°C for 1 h, at 110°C for 2 h, and then at 150°C for 3 h. A pressure of 1200 psi was applied after a period of 20 min at 110°C. After the cure was complete, the molded composite was cooled to the ambient temperature, the applied pressure being held constant.

Flexural testing

Three-point flexural tests were conducted with an Instron (Norwood, MA) 4467 universal testing machine according to ASTM D 790. A load cell of 30 kN was used. The crosshead speed was 0.85 mm/min. The span-to-depth ratio was 16. Ten specimens were tested to obtain the average values of the flexural strength and modulus of each composite.

Short-beam shear testing

Short-beam shear tests were conducted with an Instron 4467 universal testing machine according to ASTM D 2344. A load cell of 30 kN was used. The crosshead speed was 1.3 mm/min, and the span-to-depth ratio was 4. To measure the average value of ILSS of each composite, 10 specimens were tested.

Dynamic mechanical analysis (DMA)

DMA was performed with a dynamic mechanical analyzer (DMA 983, TA Instruments, New Castle, DE). A single cantilever bending mode was used with a fixed frequency of 0.1 Hz. The oscillation amplitude was 0.2 mm. The heating rate of 2°C/min was used to avoid possible thermal lag between the program temperature and the sample temperature during the measurement. The DMA measurements were carried out from the ambient temperature to 400°C. Dry nitrogen gas was purged at a rate of 30 cm^3/min.

Scanning electron microscopy

A Hitachi S-2400 scanning electron microscope (Pleasanton, CA) was used to observe the texture pattern of quasi-carbon fabrics before and after the application of different tensions to the fabrics.

RESULTS AND DISCUSSION

To investigate the effect of quasi-carbonization processing parameters on quasi-static (flexural and short-beam shear tests) and dynamic (DMA) mechanical properties of quasi-carbon/phenolic composites, all the composites used were made under the same processing conditions and characterized with the same testing parameters, with the exception of quasi-carbonization processes of stabilized PAN-based woven fabrics.

Flexural properties

Among the three processing parameters for quasi-carbonization previously mentioned, the effect of the applied tension was first examined to determine the presence or absence of tension. Then, the heating rate and HTT were examined. Figure 3 shows the variation of the flexural strength and modulus of quasi-carbon/phenolic composites reinforced with quasi-carbon fabrics. The composites were processed at 1200°C at a heating rate of 60°C/h in the absence (0 kgf) and presence (33 kgf) of applied external tension. During the quasi-carbonization process, the tension was continuously applied from the outside of the furnace with the device illustrated in Figure 2. The applied external tension was quantitatively
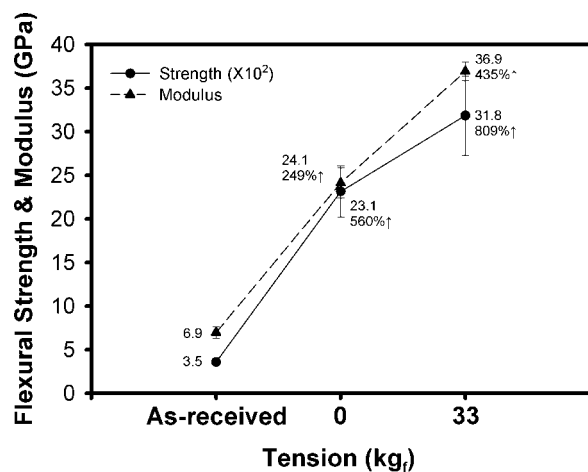
Figure 3 Flexural strength and modulus of the stabilized PAN/phenolic and quasi-carbon/phenolic composites in the absence and presence of applied external tension.

evaluated by the conversion of the numerical values of the longitudinal extension of the gripped fabrics to the corresponding force measured with the attached tension device including the fabrics.

The word *stabilized* in the figures indicates the as-received stabilized PAN-based woven fabrics without quasi-carbonization. Here, *tension 0 kgf* designates that the stabilized PAN fabrics were firmly gripped with the tension device but no external tension from the outside of the furnace was applied during the quasi-carbonization process. *Tension 33 kgf* designates the external tension continuously applied by rotation of the stainless steel threaded rod from the outside of the furnace during the quasi-carbonization process, as described earlier. The reason for applying the tension of 33 kgf in this work was that the breaking strength and modulus of quasi-carbon fabrics were increased with the applied tension increasing from 0 to 33 kgf, and they were most greatly enhanced with the tension of 33 kgf, as found in our earlier study.[5] As can be seen, the flexural strength and modulus of quasi-carbon/phenolic composites were greatest in the presence of the tension of 33 kgf. The flexural strength and modulus of the stabilized PAN/phenolic composite were greatly increased by 560 and 249%, respectively, compared with those of the quasi-carbon/phenolic composite in the presence of 0 kgf during the quasi-carbon fabric preparation. The flexural strength and modulus of the quasi-carbon/phenolic composite without tension were further enhanced about 38 and 53%, respectively, compared with those of the phenolic composite reinforced with quasi-carbon fabrics prepared in the presence of 33 kgf.

This can be explained by the fact that the alignment and preferred orientation of crystalline fibers consisting of the stabilized PAN fabrics were

induced by the externally applied tension. The formation of a well-aligned fiber structure with the assistance of applied tension may significantly contribute to enhancing the strength and modulus of the quasi-carbon/phenolic composite. Such behavior was similarly found from the breaking strength and modulus of quasi-carbon fabric studied earlier.[5] No fiber breakage or damage due to the tension applied in this work was observed in the quasi-carbon fabrics and the composites.

Figure 4 shows the effect of the heating rate on the flexural strength and modulus of quasi-carbon/phenolic composites. The quasi-carbon fabrics used here for composite fabrication were processed at 1100°C with different heating rates of 30, 60, and 100°C/h in the presence of the applied tension of 33 kgf. The flexural strength and modulus increased with the heating rate decreasing. The greatest values of the strength and modulus with a heating rate of 30°C/h were obtained. The flexural strength and modulus of the quasi-carbon/phenolic composite with a heating rate of 30°C/h were about 34 and 58% greater than those of the composite with a heating rate of 100°C/h, respectively. The result also indicates that the heating rate influenced the flexural modulus more than the strength of the composite.

It is obvious that the stabilized PAN fabrics were exposed to a heat-treatment condition for a longer period of time at a lower heating rate than at a higher heating rate during the quasi-carbonization process. The former processing condition may provide a more thermally stable structure to the resulting fabrics. In addition, the quasi-carbon fabrics processed at a slower heating rate may have developed to a greater degree an aromatic structure with a preferred orientation of the fibers and a greater areal density of the fabric than that with a faster rate.
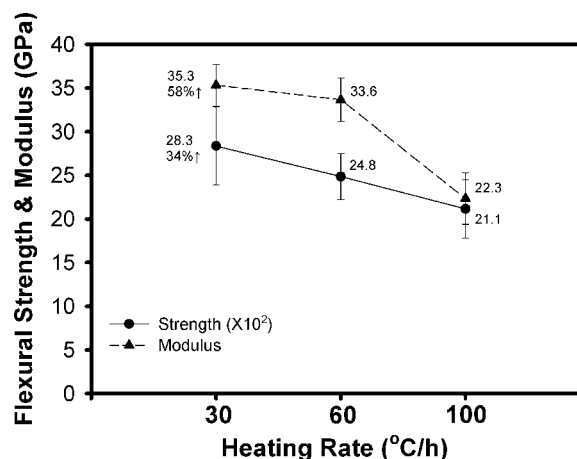

Figure 4 Variation of the flexural properties of the quasi-carbon/phenolic composites as a function of the heating rate.
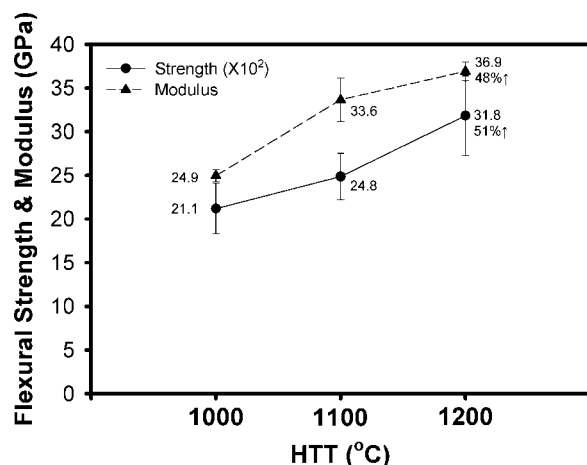
Figure 5 Variation of the flexural properties of the quasi-carbon/phenolic composites as a function of HTT.

As a result, it is concluded that the use of quasi-carbon fabrics significantly contributes to enhancing the flexural properties of stabilized PAN/phenolic composites. It can be suggested that a slow heating rate may be more effective than a fast heating rate for obtaining better flexural properties for quasi-carbon/phenolic composites. However, an appropriate heating rate should be used because of the balance between the processing time and cost.

Figure 5 presents the effect of HTT on the flexural properties of quasi-carbon/phenolic composites. The composites were fabricated with quasi-carbon fabrics prepared with a heating rate of 60°C/h in the presence of tension of 33 kgf. The flexural strength and modulus of the composite increased with increasing HTT. The composite fabricated with quasi-carbon fabrics heat-treated at 1200°C exhibited a flexural strength and modulus 51 and 48% higher than those of the one heat-treated at 1000°C, respectively. In our earlier study,[5] it was reported that the higher the final HTT was, the greater the breaking strength and modulus of the quasi-carbon fabrics were. The breaking strength and modulus became greater with increasing applied tension. The applied tension more significantly contributed to the final HTT than to the heating rate.

The increase in the flexural strength and modulus with increasing HTT may be explained by the fact that the aromatic structure of quasi-carbon fabric can be developed to a greater extent at higher HTT and that graphene layers with a turbostratic structure in the more ordered pattern can also be formed. Such quasi-carbon fabrics can more effectively distribute the flexural load applied to the surrounding phenolic matrix. Accordingly, the flexural properties of the composite can be improved. Therefore, it is concluded that the final HTT as well as the heating rate and applied tension importantly contributes to

improving the flexural properties of quasi-carbon/phenolic composites.

ILSS

The flexural and dynamic mechanical properties of a fiber-reinforced polymer composite strongly depend on the interfacial strength between the reinforcing fibers and the matrix in the composite.[14] This is because the reinforcing fibers and the polymer matrix may be chemically and/or physically bonded to each other at their interfaces. In general, the interfacial strength plays an important role in the mechanical resistance of the composite to the external load. A short-beam shear test is a useful method for exploring the interfacial strength between the woven fabric and the phenolic matrix,[15,16] especially in this two-directional quasi-carbon/phenolic composite system.

ILSS can be measured with the short-beam shear test. This test is normally carried out by the removal of the applied load as soon as the initial delamination between the woven fabric ply and the matrix takes place through the thickness in the midplane of a composite right after the yield point in the stress–strain behavior. The average ILSS values were obtained from the maximum load of a load–displacement curve for each specimen with the following equation:

$$\tau_{max} = 3P_{max}/4b \times t \qquad (1)$$

where τ_{max} is the interlaminar shear strength, P_{max} is the maximum load, b is the width of the composite specimen, and t is the thickness of the specimen.

Figure 6(A) shows the effect of applied tension on ILSS of quasi-carbon/phenolic composites. The composites were fabricated with the quasi-carbon fabrics processed at 1200°C with a heating rate of 60°C/h. ILSS of the stabilized PAN fabric/phenolic composite was also measured for comparison. ILSS of the stabilized PAN fabric/phenolic composite was greatly enhanced by about 150–420% by the incorporation of quasi-carbon fabrics as reinforcements into the phenolic matrix. The result depends on the absence (0 kgf) and presence (33 kgf) of applied external tension. Figure 6(B) shows the effect of the heating rate on ILSS of the composites. The composites were fabricated with the quasi-carbon fabrics processed at 1100°C in the presence of tension of 33 kgf. The ILSS value was increased by about 28% with the heating rate decreasing from 100 to 30°C/h. Compared with that of the stabilized PAN fabric/phenolic composite, ILSS of the quasi-carbon/phenolic composite at 30°C/h was remarkably increased by 428%. Figure 6(C) depicts the effect of HTT on ILSS of the composite. The applied tension and the heat-
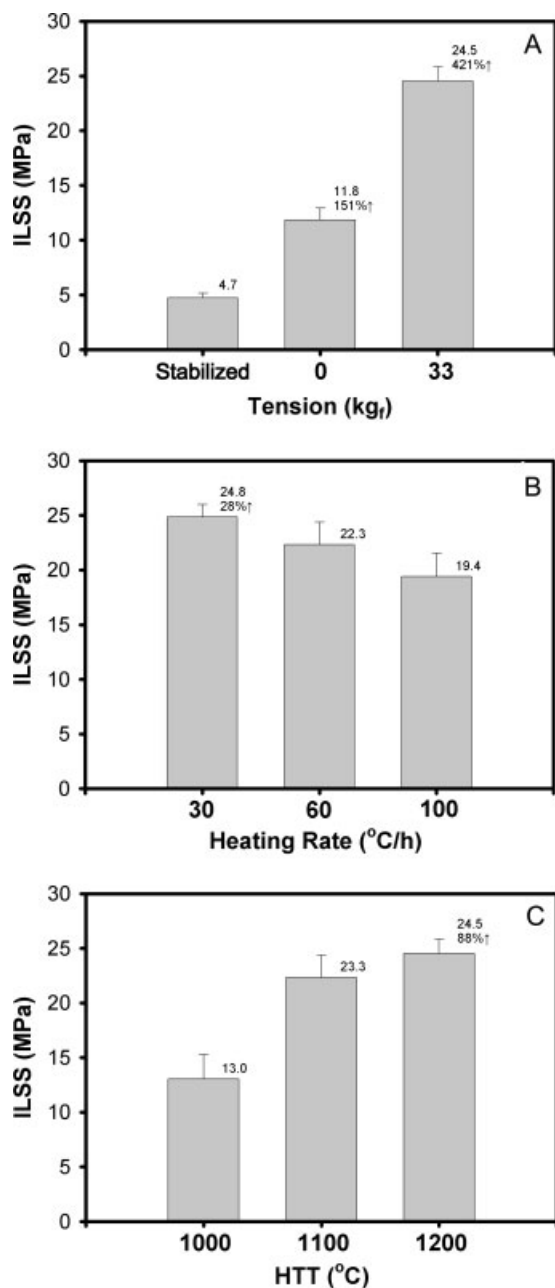
Figure 6 Variations of ILSS of the quasi-carbon/phenolic composites showing the effect of (A) the tension, (B) the heating rate, and (C) HTT.

ing rate were fixed at 33 kgf and 60°C/h, respectively, during the quasi-carbonization process of the stabilized fabrics. The ILSS value was significantly increased by about 88% with HTT increasing to 1200°C. ILSS of the quasi-carbon/phenolic composite at 1200°C was 421% greater than that of the stabilized PAN fabric/phenolic composite.

The fabric texture patterns in Figure 7 support the effect of applied tension on ILSS. As can be seen, the number of tows and crimps per unit area of the fabric was increased in the presence of applied tension.

The longitudinal width of each tow became narrow along the applied tension direction. It resulted in an increase in the surface area of the quasi-carbon fabric at the same magnification (×20). This may cause an increase in the interfacial adhesion between the quasi-carbon fabrics and the phenolic matrix in the composite, leading to a further increase in the presence of applied tension. In addition, the increased mechanical properties of the fabric obtained by the
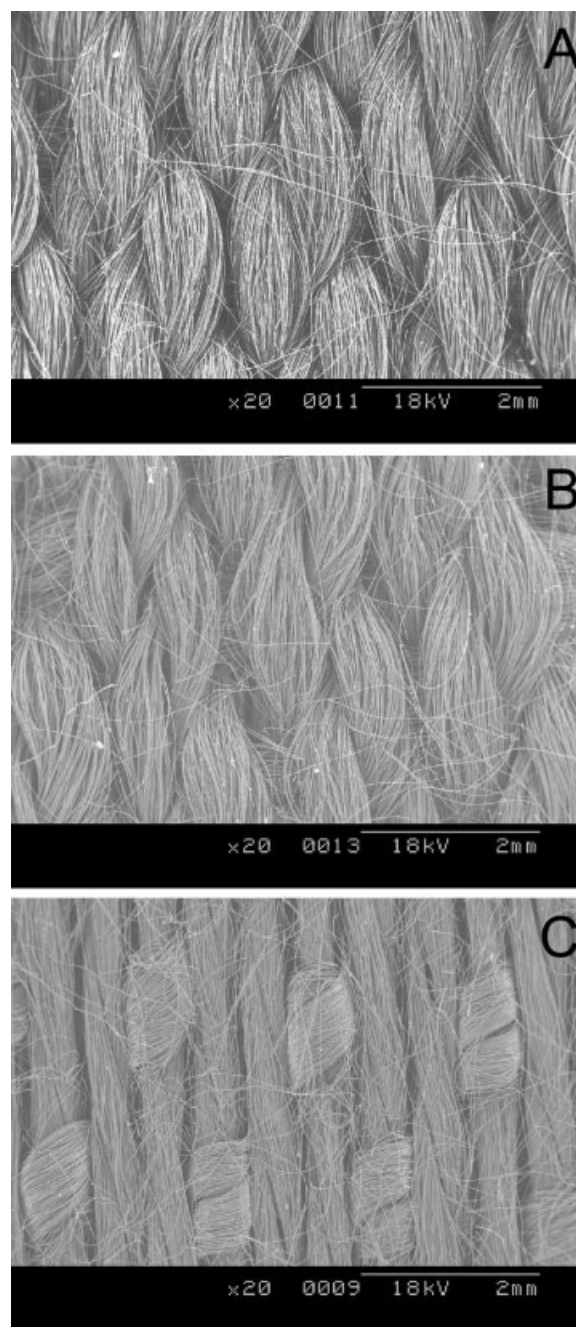

Figure 7 Scanning electron micrographs showing the texture pattern of (A) the stabilized PAN fabric, (B) the quasi-carbon fabric (0 kgf), and (C) the quasi-carbon fabric (33 kgf).
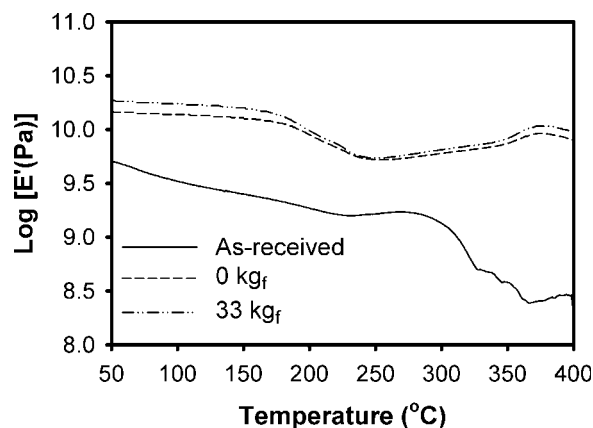
Figure 8 Effect of the applied external tension on the storage modulus (E') of the stabilized PAN/phenolic and quasi-carbon/phenolic composites.

Figure 9 Effect of the heating rate on the storage modulus (E') of the quasi-carbon/phenolic composites.

quasi-carbonization process may contribute to distributing more or less of the external load to the matrix of the composite. As a result, ILSS in the two-directional quasi-carbon/phenolic composite was significantly improved.

Dynamic mechanical properties

DMA is a useful method for studying the thermal mechanical properties of composite materials as well as viscoelastic polymer materials because of the temperature dependence of the dynamic storage modulus. It is also frequently used to understand the relation of the dynamic mechanical behavior to the interfacial behavior between the fiber and the polymer matrix in a composite system.[17–19]

Figure 8 presents the effect of the absence (0 kgf) and presence (33 kgf) of applied tension on the storage modulus of quasi-carbon/phenolic composites. The composites were fabricated with the quasi-carbon fabrics processed at 1200°C with a heating rate of 60°C/h. The storage modulus of the quasi-carbon/phenolic composite was much greater than that of the stabilized PAN fabric/phenolic composite. The storage modulus of the stabilized PAN fabric/phenolic composite gradually decreased with the measuring temperature increasing. This was because the stabilized fabrics therein were chemically and physically changed with temperature, and this accompanied some primary weight loss of the stabilized PAN fabrics. This may have led to some deterioration of the original fiber properties. The slight increase in the storage modulus above 240°C was ascribed to the postcure effect of the phenolic resin matrix. The storage modulus further decreased at about 280°C because of the secondary weight loss from the stabilized PAN fabrics in the composite.
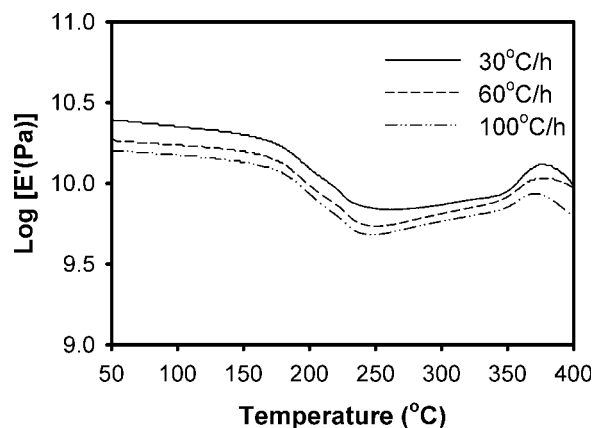
On the other hand, the initial storage modulus of the two quasi-carbon/phenolic composites was retained with increasing temperature below about 150°C. The decrease of the storage modulus above 150°C occurred because the molding temperature for processing quasi-carbon/phenolic composites was 150°C. It also occurred because the small amount of the phenolic resin remaining unreacted in the matrix could further react during the DMA measurement in the given temperature range. The postcure effect of the phenolic resin matrix led to an increase in the storage modulus above 250°C again. A small decrease in the value of the logarithm of the storage modulus near 380°C was attributed to the degradation of the phenolic matrix in the composite. It seems that the applied tension of 33 kgf slightly increased the storage modulus of the quasi-carbon/phenolic composite in comparison with the case without the external tension applied.

Figures 9 and 10 compare the variation of the storage modulus as a function of temperature. The results show the effects of the heating rate and final
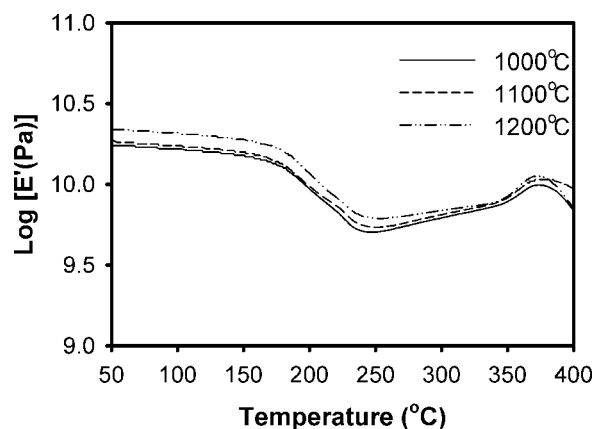

Figure 10 Effect of HTT on the storage modulus (E') of the quasi-carbon/phenolic composites.

HTT used for the quasi-carbonization process in the presence of 33 kgf, respectively. The overall variation of each DMA thermogram was similar to that observed for the quasi-carbon/phenolic composite in Figure 8. The result indicates that the slower heating rate and higher HTT were more effective in improving the storage modulus of the quasi-carbon/phenolic composite, as demonstrated by the flexural and short-beam shear test results. It has been concluded that the improvement of ILSS of quasi-carbon/phenolic composites plays a significant role in increasing not only the flexural strength and modulus but also the dynamic storage modulus. The improvement depends on the processing parameters used for quasi-carbonization of stabilized PAN-based fabrics.

CONCLUSIONS

The flexural strength and modulus of the stabilized PAN/phenolic composite were greatly improved by the reinforcement of the phenolic resin with quasi-carbon fabrics. The flexural properties of the quasi-carbon/phenolic composites were increased with the heating rate decreasing and also with HTT increasing.

ILSS of the stabilized PAN fabric/phenolic composite was markedly improved by the incorporation of quasi-carbon fabrics into the phenolic matrix. ILSS was increased with the heating rate decreasing and with HTT increasing, as similarly found from the flexural results. On the basis of the percent improvement of the ILSS values, the applied tension exhibited the greatest effect, and the heating rate showed the lowest.

The dynamic storage modulus of the stabilized PAN fabric/phenolic composites was significantly improved in the quasi-carbon/phenolic composites, strongly depending on the quasi-carbonization process parameters used in this work.

On the basis of all the results, the quasi-static and dynamic mechanical properties of quasi-carbon/phenolic composites increased with the applied external tension and HTT increasing and with the heating rate decreasing for the quasi-carbonization process. This study demonstrates that control of the processing parameters directly influences the mechanical properties of quasi-carbon/phenolic composites as well as the interlaminar strength between the fibers and the matrix resin.

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