Effect of Thermal Treatment on the Tensile and In-Plane Shear Behavior of Carbon Fiber-Reinforced Poly(phenylene sulfide) Composite Specimens

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ABSTRACT: The effect of PPS matrix evolution occurring during thermal treatment of carbon fiber-reinforced PPS plies prior to their consolidation to laminates on the mechanical behavior of the composite material has been investigated. The thermal treatments were performed at temperatures and times, which are relevant for processing PPS composites. All thermal treatments were carried out in an oven in air to facilitate the presence of oxygen, while the subsequent consolidation was performed in an autoclave. The tensile and in-plane shear behavior of both, thermal-treated and untreated materials, was investigated. Differential scanning calorimetry and microscopy analyses were made to evaluate the effect of the performed thermal treatments on degree of crystallinity and porosity of the laminates. The mechanical tests carried out have shown an appreciable degradation of the mechanical properties investigated. The observed degradation increases with increasing thermal treatment temperature and time when thermal treatments were carried out on each single composite ply prior to the consolidation. On the other hand, when, prior to the consolidation, the whole set of plies was subjected to thermal treatment, improved mechanical properties were observed. The results were discussed under the viewpoint of PPS matrix evolution during processing of the composite plies in the presence of oxygen. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 107: 3190–3199, 2008

Key words: composites; thermoplastics; processing; mechanical properties; thermal treatment

INTRODUCTION

During the last two decades, a number of semicrystalline thermoplastic composites have emerged as attractive candidates for aircraft applications, because of their desirable properties. Higher service temperatures, processing by melting and reprocessibility as well as improved impact resistance, damage tolerance, and strength properties offered by the semicrystalline matrices represent significant technological advantages of this group of materials. Included in this group of thermoplastic matrices are polyetheretherketone (PEEK), polypropylene (PP), poly(phenylene sulfide) (PPS), etc. Among them, the semicrystalline PPS with a maximum crystallinity of 65%, T_g of 85°C, melting point of about 285°C, and processing temperatures ranging between 290 and 345°C[1,2] offer an attractive candidate. A significant technological advantage for the use of reinforced PPS in aircraft structural applications is the high service temperature of the material, which reaches 225°C; it relies on the high melting point and the good thermal stability of the material, which reflect the high bonding energy between its phenyl groups and sulfur atoms.[3] Present day aeronautical applications of PPS and its composites include seats, equipment components and assemblies, wing sections, vertical tail, rudder units, and fuselage-reinforcing elements.[4–7]

Decisive parameter for the quality of thermoplastic components is the proper selection of the processing temperature (e.g., Ref. 8). Too low processing temperatures may result in incomplete resin melting. On the other hand, it is well known that there is an evolution of semicrystalline thermoplastic matrices in the melt state, at temperatures that are relevant for processing them; it seems to concern all semicrystalline and high performance thermoplastic resins (e.g., PEEK, PEI, PEKK, PPS, PP, PEK, TPU, etc.). Heating of said resins at temperatures over the melting point is affecting their degree of crystallinity[9–15] and, hence, their mechanical properties. Mentioned effect depends on temperature,[9–12] time of holding the

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resin in the melt state,[9,10,12] crystallization time,[9] and cooling rate.[9] In Refs. 9 and 15, the effect of melt history on degree of crystallinity and transitions temperatures was investigated. It was found that the holding time of the resin over the melting temperature may appreciably change the degree of crystallinity as it influences the destruction of existing crystalline nuclei and the formation of new. Furthermore, molecular modifications take place, which cause defects along the macromolecular chain and thus reduce the ability of the macromolecule to take part in the crystalline ordered structure.[10] The crystal imperfections that are developed decrease the degree of crystallinity.[9,10] Investigations have shown that exposing said thermoplastic resins to temperatures below the melting point but over the material's T_g may also affect its degree of crystallinity (e.g., Ref. 9) and, hence, has to be accounted for when selecting the processing parameters. Reinforcing fibers influence the crystallinity growth rate and degree of crystallinity depending on type of fibers (e.g., Ref. 16) and holding temperature of the melt[17] and seem also to reduce the Avrami exponent's value.[18] The melt holding temperature has been found to influence number, location, and site of the spherulites, which influence appreciably the overall rate of crystallization; composites processed at higher melting temperatures exhibited larger spherulites far from the fibers and trans-crystalline growth morphology near the fibers.[17]

On the other hand, heating near or over the melting point may initiate a chemical reaction involving breaking of the C bond to the element characterizing the molecule (e.g., the C—S bond for the PPS matrix) and crosslinking of the chains at the amorphous regions of the resin[9,10,13,19]; it increases viscosity and makes processing of the melt difficult.

The PPS matrix under consideration can be formed as linear, branched, or slightly crosslinked resin depending on the polymerization and thermal treatment processes involved (Ref. 14 and the references therein). While the branched PPS resin is developed by polymerization in the presence of a small portion of trifunctional monomer, the slightly crosslinked resin is formed when a lower molecular weight resin is heat-treated in the presence of oxygen. Longer processing times of PPS at temperatures near or above the melting point in the presence of oxygen cause chain extension, degradation, and some crosslinking,[20] which may also reduce the degree of the resin's crystallinity. The reaction mechanism initiates by the scission of the C—S bonds; it leads to the formation of a benzyne intermediate, which reacts to other chains of the polymer and subsequently crosslinks.[9,19] Furthermore, the sulfur atoms increase the susceptibility of the PPS matrix to crosslinking when the resin is processed in the presence of oxygen, e.g., in air.[3] Experi-

ments revealed a significant increase of the viscosity of the polymer in temperatures lying in the range of 320–340°C.[2,21] The latter observation along with the melting temperature of the matrix, which lies at around 285°C, limits the temperature range for processing PPS and may also affect the mechanical properties (e.g., Refs. 14 and 22).

Evolution in crystallinity and morphology of thermoplastic composites due to thermal treatments prior to processing, during processing, or after processing affects the technological and mechanical properties of the material and also may lead to incomplete consolidation of the thermoplastic composite parts (e.g., Refs. 11–13, 22–26). Respective results for the PPS composites remain in the open literature rare. In Ref. 20, a decrease in tensile strength with increasing crystallinity was observed. This surprising result has been confirmed in Ref. 27. Flexural strength, flexural modulus, and transverse tensile strength of carbon fiber-reinforced PPS subjected to various thermal histories were measured in Ref. 26. It was found that fiber-dominated properties of the composite do not depend on degree of crystallinity and morphology of the matrix. On the other hand, increased crystallinity was found to increase the 0° flexural modulus and strength of unidirectionally reinforced C/PPS. It has also been observed that the matrix-dominated properties of the composite depend not only on the degree of crystallinity but also on the number, size, and morphology of the spherulites developed during cooling of the material, whereby increased number of large spherulites with well-defined boundaries decrease strength and make the material more brittle. Results presented in Ref. 27 related strength and ductility of the composite to the viscosity of the melt and, through it, to the thermal treatment histories involved to produce various morphologies of the matrix. The investigation revealed that thermal treatment of the composite can be decisive for the mechanical properties.

The above results underline the need for a very diligent selection of the process parameters temperature and time, particularly when the processes of concern have long duration and/or are carried out in air.

In the present work, the effects of the PPS resin evolution during melting in air, at temperatures that are relevant for processing PPS, on the mechanical properties of carbon fiber fabric-reinforced semicrystalline PPS laminates are investigated.

MANUFACTURE OF PPS LAMINATES AND EXPERIMENTAL INVESTIGATION

Manufacture of the PPS laminates

The material investigated was high performance semicrystalline thermoplastic poly(phenyl sulfide)
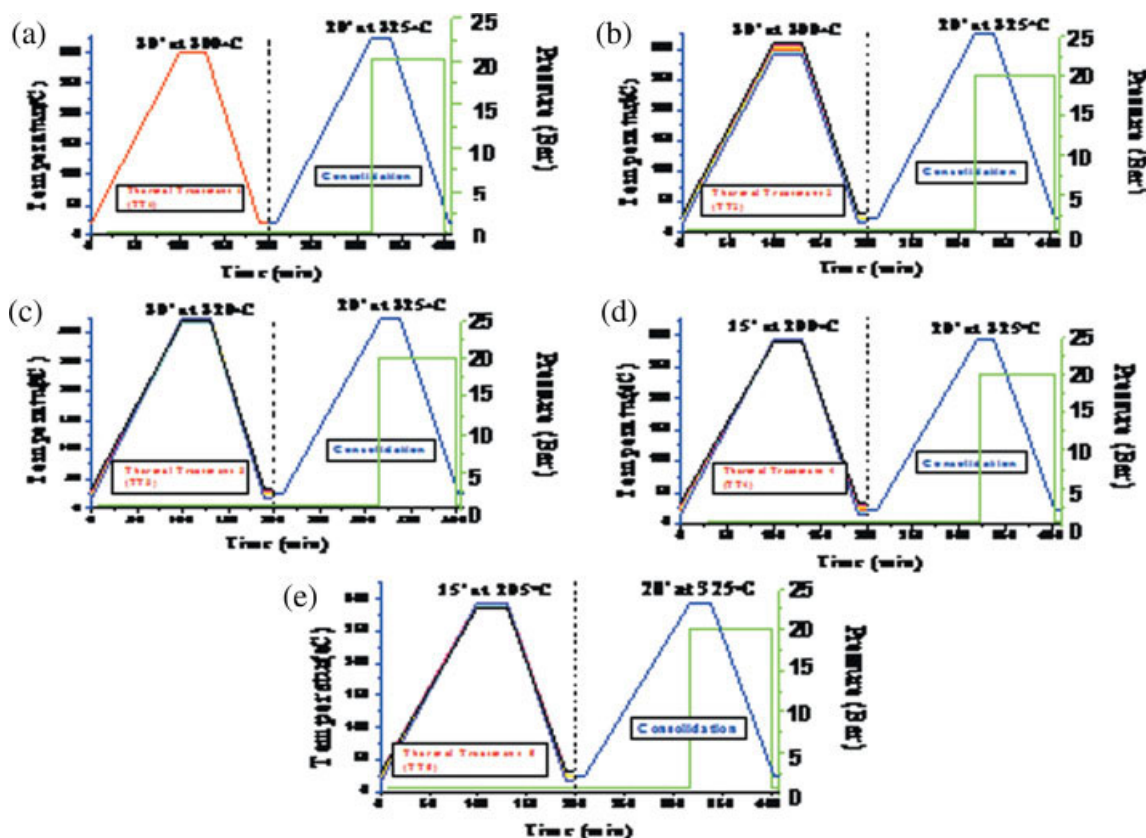
Figure 1 Process cycles for the manufacture of the C/PPS laminates. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

(PPS) reinforced with carbon fibers fabric. The material was supplied by TEN CATE (ref CD 286 050 030 8538 4300) in form of semipregs involving carbon 5 satin fabric reinforcement. The nominal thickness of the semipregs was 0.3125 mm.

Prior to the consolidation, the semipregs were subjected to different thermal treatments. All thermal treatments and consolidations of the PPS/C laminates were carried out at EADS Innovation Works. An overview of the thermal treatments history performed and the respective process cycles are displayed in Figure 1. The thermal treatment referred in Figure 1(a) as TT1 was applied on a set of plies after they were placed in the form of a sheet with the desired stacking sequence and before it has been consolidated to a laminate. The thermal treatments referred in Figure 1(b–e) as TT2, TT3, TT4 and TT5, respectively, were applied to each single ply before the plies were placed in form of a sheet in the desired stacking sequence and consolidated to a laminate. A number of laminates were consolidated without prior thermal treatment of the plies to produce reference material; in the following paragraphs, it is referred to as reference material.

The process parameters, temperature and time of the above treatments, were selected with regard to the temperature and time windows given in the literature for processing PPS.[1,2,5] Recall that at temperatures, which are lower than 290°C, the PPS matrix is not yet molten while at temperatures higher than 320°C the resin's viscosity in the melt state becomes so high[2] that it makes processing very difficult, in particular when injection processes like e.g. injection molding, etc. are concerned. Viscosity normally decreases with the temperature in the melt state (in an inert atmosphere), but oxidation of the resin in the melt state induces this drastic increase. Heating times were selected to range between 15 and 30 min. All thermal treatments were performed in an oven in air. The matrix evolution caused in the presence of oxygen accounts for the matrix evolution expected during processes carried out in air. The laminates produced included eight plies in a stacking sequence of 0/90° [0/90°]$_{2s}$. The nominal thickness of the laminates was 2.5 mm. The consolidation was performed in an autoclave at EADS Innovation Works in Suresnes. As the content of oxygen in autoclave conditions is low, the matrix evolution during consolidation is assumed to be limited as compared to the matrix evolution caused by the previous thermal treatment. The consolidation was made at 325°C (±5°C) under pressure of 20 bars. The heating speed
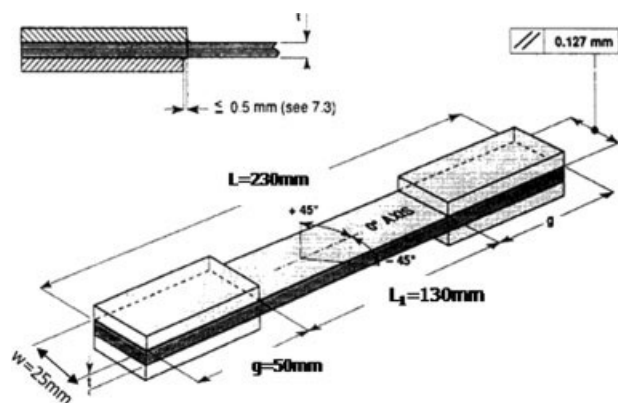
Figure 2 In-plane shear coupon according to the AITM 1-0002 specification.

to the dwell temperature was 3°C/min. The consolidation time was 20 min. Cooling occurred under pressure at a cooling rate of 5°C/min.

Experimental investigation

The experimental investigation included differential scanning calorimetry tests, micrographs of the through the thickness cross section of the laminates, in-plane shear tests and tensile tests. As the present work focuses on the investigation of the effect of thermal treatments on the mechanical behavior of C/PPS, the DSC tests and micrographs performed were limited to the extent considered as necessary to support understanding of the mechanical behavior observed.

Differential scanning calorimetry tests

To obtain the effect of the thermal treatments on the crystallinity degree of the composite under consideration, DSC tests on both reference and thermal-treated materials were carried out. For the tests, a TA Instruments Q100 differential scanning calorimeter was used. Samples (7–10 mg) were cut from both reference and thermal-treated PPS materials. For the tests, a heating rate of 10°C/min from 40 to 320°C, a 5-min dwell at 320°C, and a cooling rate of 10°C/min from 320 to 50°C were selected. From the tests, DSC data were measured and the respective DSC graphs were obtained. To determine the crystallinity degree, one run was performed.

Micrographs

To obtain the effect of the thermal treatments on the porosity of the material, micrographs of through the thickness cross sections of both reference and thermal-treated material were made by using optical microscopy. For the tests, a Moritex Scopemax MS

500A stereomicroscope with a magnification of 100 times was used. The amount of porosity was derived by involving an image analysis software.
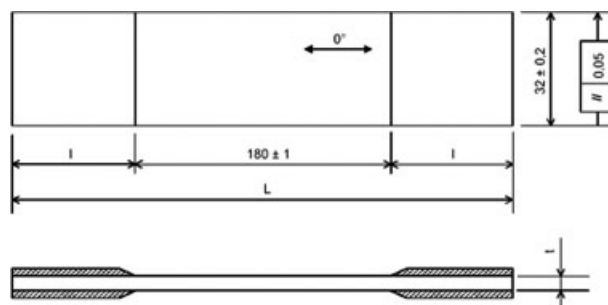
In-plane shear tests

For the in-plane shear tests, rectangular 25 mm × 230 mm coupons according to the Airbus AITM 1-0002 specification[28] were used (Fig. 2). The in-plane shear specimens were cut from the laminates in ±45° direction by means of a diamond band saw according to the ISO 2818 specification.[29] To achieve equal stress distribution over the entire coupon rectangular tabs from ±45°, C/PPS materials were bonded on the in-plane shear specimens. To measure longitudinal and transverse strain during testing, Kyowa biaxial strain gauges with a gauge length of 5 mm were attached at the centers of both faces of each coupon so as to have a mean value of two measurements. Before placing the strain gauges, the surfaces of the coupons were cleaned and the exact centers of the surfaces were located.

The tests were carried out according to the Airbus AITM 1-0002 (in-plane shear) with a constant elongation rate of 1 mm/min. The properties evaluated were in-plane shear strength τ and in-plane shear modulus G.

Tensile tests

For the tensile tests, rectangular 32 mm × 280 mm tensile coupons according to the Airbus AITM 1-0007 specification[30] were used (Fig. 3). The tensile specimens were cut from the laminates in fiber (0/90°) direction by means of a diamond band saw according to the ISO 2818 specification.[29] At the tensile specimens, rectangular tabs from ±45° C/PPS material were bonded (Fig. 3). For the strain measurements, biaxial Kyowa strain gauges with a gauge length of 5 mm were placed at the centers of both faces of the specimens. The tests were carried out according to the Airbus AITM 1-0007 specification with a constant elongation rate of 2 mm/min. The

Figure 3 Tensile coupon according to the AITM 1-0007 specification.

TABLE I
In-Plane Shear and Tensile Tests Performed for the Thermal Treatments Applied

Material code name	In-plane shear test	Tensile test
Ref	5	–
TT1	6	–
TT2	3	7
TT3	–	7
TT4	6	6
TT5	–	3

properties evaluated were tensile strength R_m and Young's modulus E.

For all the mechanical tests, a servo hydraulic MTS 100 kN machine was used. A data logger was used to store the data in a digital file. To get representative average values of the measured in-plane shear and tensile properties, all mechanical tests were repeated; numbers of test repeats are included in Table I. In total, 43 tests were performed and evaluated.

RESULTS AND DISCUSSION

Degrees of crystallinity

As expected,[14,15] thermal treatments prior to the consolidation of the laminate affect the degree of crystallinity of the C/PPS composite. An overview of the degrees of crystallinity obtained for the thermal treatments applied in the present study is given in Table II. The respective DSC curves are displayed in Figure 4. Table II and Figure 4 include also the respective data for the reference nonthermal-treated material, for comparison.

A ply-by-ply heating prior to the consolidation has been found to decrease the degree of crystallinity of the C/PPS laminate appreciably for all thermal treatments considered in the present study. The decrease of the degree of crystallinity becomes more severe with increasing thermal treatment temperature; it reaches a value of 43% as compared to the crystallinity degree of the nonthermal-treated material for the TT3 thermal treatment. The results are consistent with results available for the PEEK semicrystalline thermoplastic matrix.[9,10,15] On the contrary, a 13%

increase of the crystallinity degree, as compared to the reference material, was observed for the TT1 thermal treatment. However, as this small increase lies within the expected experimental errors, during DSC tests, the degrees of crystallinity of reference and TT1 thermal-treated material might be considered to remain unaffected by the thermal treatment. Recall that in this latter case the thermal treatment was applied after the plies were first placed in the form of a sheet with the desired stacking sequence.

Porosity evaluation

An appreciable effect on the porosity of the C/PPS laminates was observed for all thermal treatments applied. An overview of the porosities observed for both reference and thermal-treated materials is given in Table II. The respective micrographs are shown in Figure 5. It should be noticed that the amount of porosity of laminates and consequently all properties dominated by the interphases between the plies depend on the technology of stacking. As it can be seen, the ply-by-ply thermal treatment of the material increases the amount of voids, which results in increased intraply porosity and local delamination. As expected, it facilitates excessive or lower resin infusion and leads to resin-rich or resin-poor areas; resin-rich or resin-poor areas have been made responsible for increased porosity.

Starting from a low level of porosity (around 1%) for the reference material, the amount of porosity increases with increasing thermal treatment temperature and reaches 3.8% for TT3 thermal treatment; it represents an increase on porosity of 280%, which is expected to degrade matrix-dominated mechanical properties of the composite. The results are consistent with results available for the PEEK semicrystalline thermoplastic matrix.[9,15] On the contrary, thermal treatment referred to in Figure 1 as TT1 has nearly the same level of porosity as compared to the reference material with a value of around 1%.

In-plane shear properties

The results of the in-plane shear tests performed are displayed in Figures 6 and 7, where R stands for the

TABLE II
Crystallinity Degree and Amount of Porosity of Reference and Thermal-Treated Materials

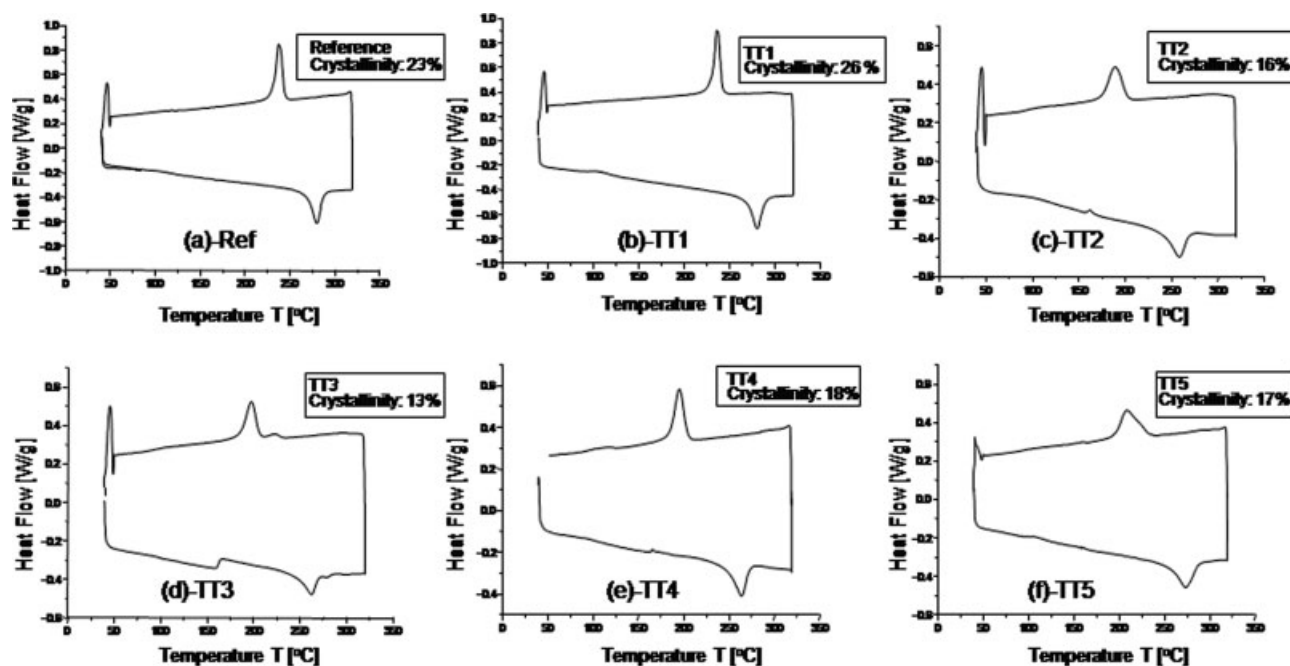| Material	Crystallinity degree (%)	Difference to reference value (%)	Amount of porosity (%)	Difference to reference value (%)
Ref	23	–	1	–
TT4	18	22	1.8	80
TT5	17	26	2.5	150
TT1	26	(+)13	0.8	(−)20
TT2	16	30	3.2	220
TT3	13	43	3.8	280

Figure 4 Differential scanning calorimetry graphs of reference (a) and thermal-treated materials (b–f).

reference material and TT for the thermal-treated materials. All values in these figures are average values. The values of in-plane shear strength and modulus derived for the reference material fit well to the respective values of the same material given in Ref. 14; in Ref. 14, the values given for the above properties are 110 MPa and 4.17 GPa, respectively.

The in-plane shear stress–strain curves for materials subjected to thermal treatments referred to as TT1 and TT2 conditions in Figure 1 are shown in
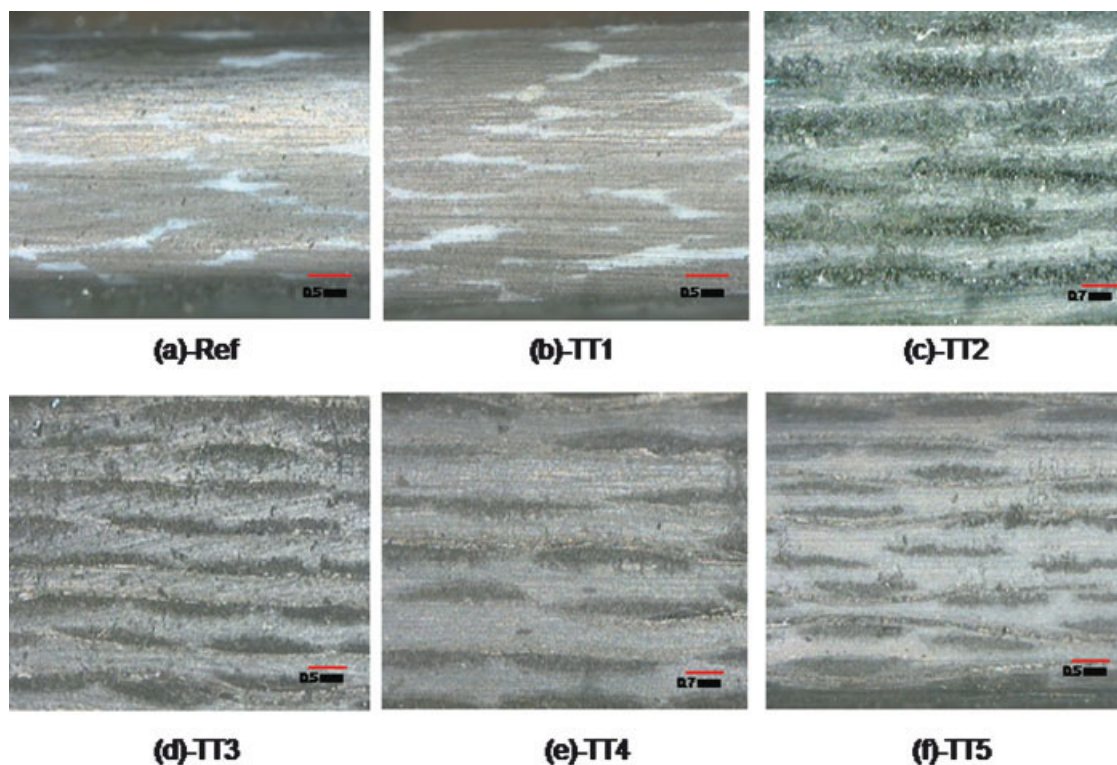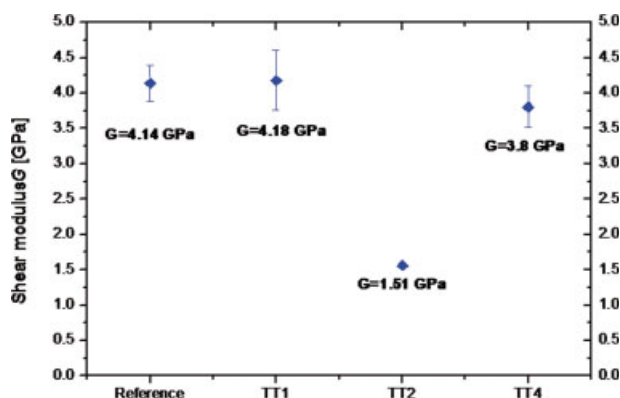

Figure 5 Micrographs of through the thickness of reference (a) and thermal-treated coupons (b–f) showing the porosities. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
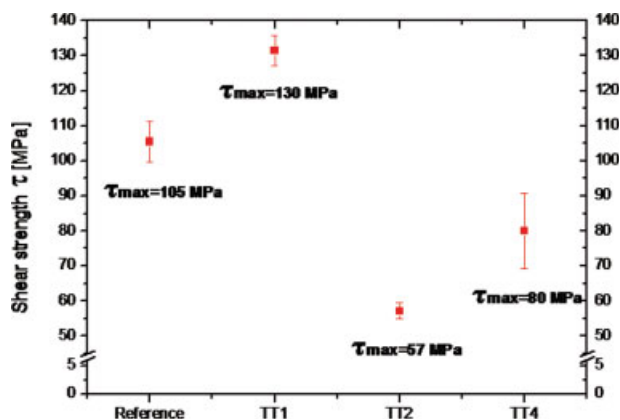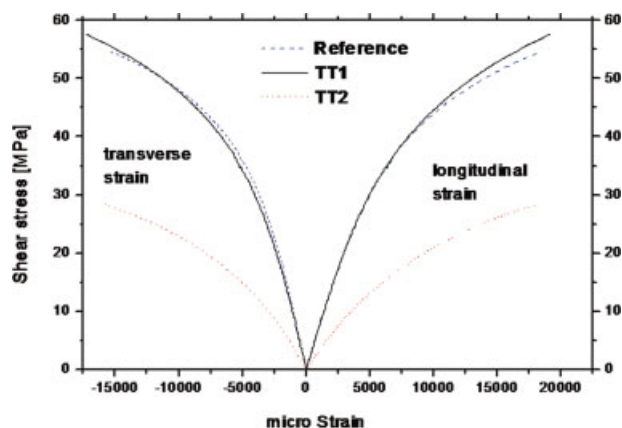
Figure 6 In-plane shear strength of reference and thermal-treated materials. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figure 8 In-plane shear stress–strain curves of reference and thermal-treated specimens for various treatment durations and temperatures. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figure 8. For comparison, stress–strain curves for the reference material are displayed in the same figure as well. The derived in-plane shear strength values for both the reference and the thermal-treated materials are displayed over the respective thermal treatment procedures involved in Figure 6. The standard deviation in the derived properties has also been given in the figure. As it can be seen, there is a decrease of the in-plane shear strength after TT2 and TT4 heat treatment procedures of 46 and 24%, respectively. On the contrary, TT1 thermal treatment leads to 24% improvement in in-plane shear strength values.

The average values of in-plane shear moduli of reference and thermal-treated specimens are displayed in Figure 7. A significant 64% in-plane shear modulus decrease was observed after TT2 thermal treatment, and a 8% in-plane shear modulus decrease was observed after TT4 thermal treatment. TT1 treatment seems to do not affect the in-plane shear modulus value; it gives a marginal increase of 1%.

The above results are summarized in three-axis plots (Fig. 9), relating treatment temperature, treatment duration, and in-plane shear property.

As it can be seen, thermal treatment on the composite sheet prior to the consolidation improves the in-plane shear properties of the material. This result is attributed not only to interdiffusion of long macromolecular thermoplastic chains through the ply surfaces and some entanglements at the interface but also to some real 3D polymer networks building, which finally reinforce the interply behavior. Recall that in-plane shear strength properties are matrix-dominated properties. On the contrary, in-plane shear strength properties of materials subjected to ply-by-ply thermal treatment decrease (Fig. 6). The decrease is higher when thermal treatment temperature and/or duration increase. It is reasonable to expect that a ply-by-ply treatment allows for matrix degrading phenomena (e.g., decrease of crystallinity, increase of porosity, crosslinking effects) to better occur. On the other hand, when the whole set of plies is subjected to thermal treatment for relatively short times, one may expect that matrix degrading processes will take place rather at the outer plies than in plies lying in the interior of the laminate. Hence, the absence of oxygen-driven detrimental reactions between the plies could be considered as a factor contributing to the high shear property values obtained. An additional hypothesis could be that heating of the whole set of plies at 300°C for 30 min prior to the consolidation might cause some crosslinking, which is likely to reinforce the ply-to-ply cohesion thus improving consolidation and hence the in-plane shear properties. Although there is no clear evidence for this latter hypothesis, it is supported by the reduced porosity observed for this material.
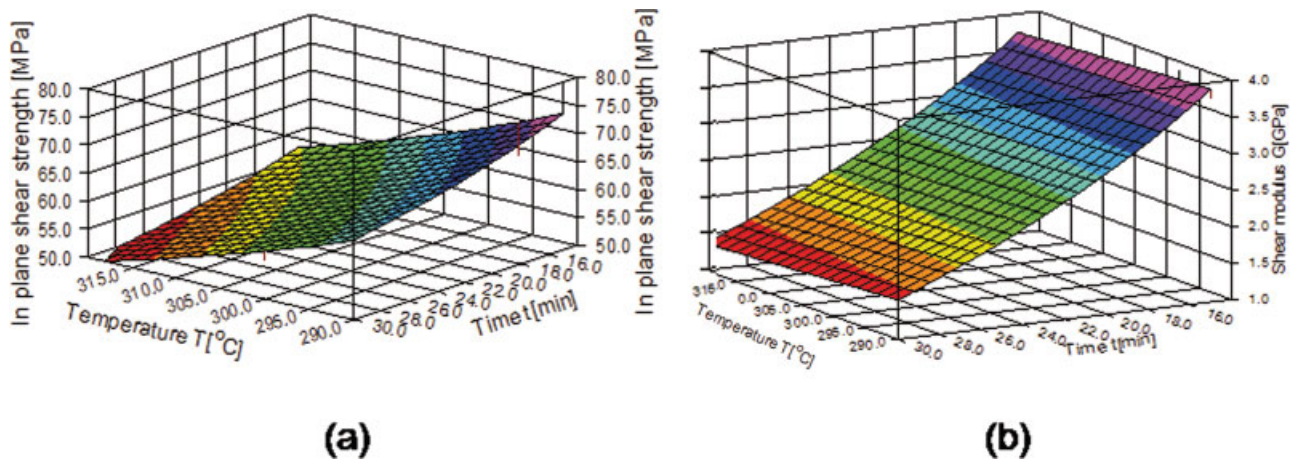

Figure 7 In-plane shear moduli of reference and thermal-treated materials. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
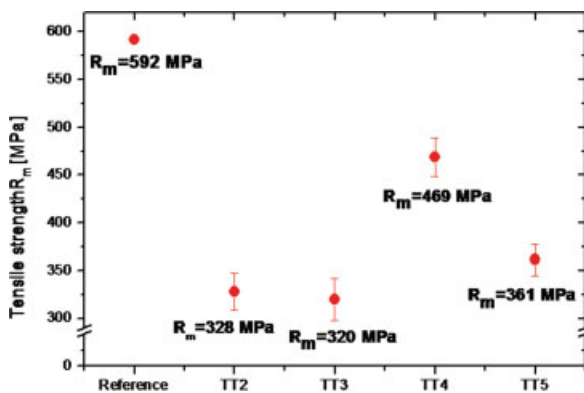
Figure 9 Three-axis map combining heating temperature and heating time with in-plane shear strength (a) and in-plane shear modulus (b). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Tensile properties

The average tensile properties of the thermal-treated materials along with the respective properties of the reference material for comparison are displayed in Figures 10 and 11. Typical stress–strain curves for the investigated thermal treatment conditions are displayed in Figure 12. It is worth noting that all thermal treatment procedures for this part of investigation refer to a ply-by-ply treatment of the material.
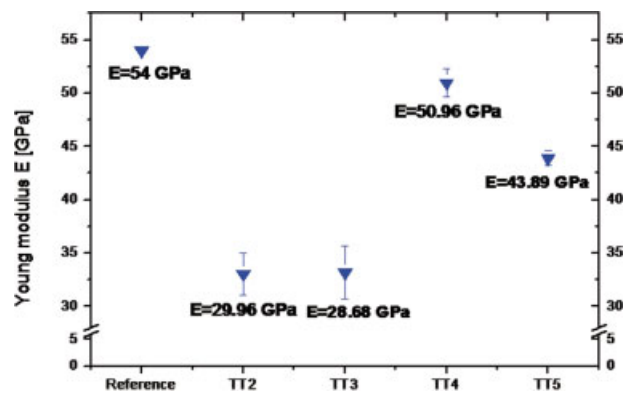
After the thermal treatments, significant decrease in the tensile strength is observed as compared to the reference value (Fig. 10). The higher decrease of tensile properties was observed for the TT2 and TT3 conditions (45 and 46%, respectively), which refer to the higher temperatures of 300 and 320°C, respectively. The TT4 and TT5 treatments that refer to lower temperatures (290 and 295°C, respectively) also cause a lower decrease in tensile properties (39 and 21%, respectively). Notice that TT4 and TT5 conditions also involve shorter thermal treatment duration as compared to the TT2 and TT3 conditions.

Tensile moduli of the reference and the thermal-treated specimens are displayed in Figure 11. Similar to the tensile strength values, thermal treatments prior to consolidation decrease the tensile modulus. For the thermal treatments TT2 and TT3, the tensile moduli are dramatically decreased by 45 and 47%, respectively. A decrease was observed also after the TT3 and TT4 treatments; yet it was lower and did not exceed 6 and 19%, respectively. Tensile strength and modulus results are summarized in three-axis plots (Fig. 13), relating treatment temperature, treatment duration, and tensile property.

Although tensile strength is a fiber-dominated property, for a $0/90°$ $[0/90°]_{2s}$ stacking sequence, as it is the case for the specimens used, the contribution of the matrices tensile strength, through the $90°$ plies, to the strength of the composite cannot be ignored. By involving the lamination theory to calculate the tensile properties of a composite C/PPS specimen with same stacking sequence and volume fraction of fiber and matrix as the specimens investi-

Figure 10 Tensile strength of reference and thermal-treated materials. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

Figure 11 Tensile moduli of reference and thermal-treated materials. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]
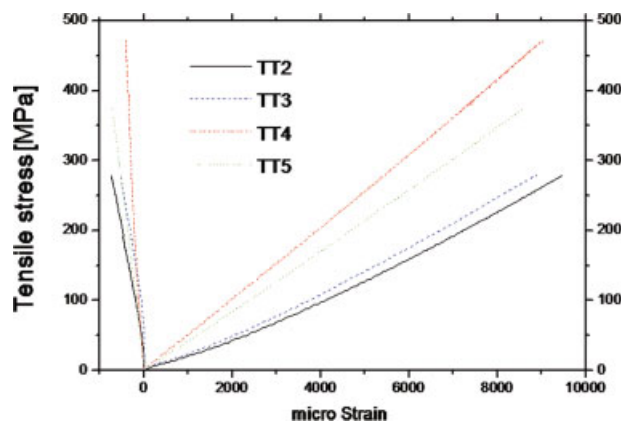
Figure 12 Tensile stress–strain curves of thermal-treated specimens for various treatment durations and temperatures. [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

posite specimens subjected to tensile tests parallel to the fibers have shown that the strength of the interfaces between fibers and matrix, which is significantly influenced by the amount of porosity, may change the failure mode and lead to appreciable changes of the tensile strength values. As shown in the same investigation, the effect of the interface's strength on the tensile strength of specimens loaded transverse to fiber can become dramatic. In any case, further systematic investigation is needed to understand the underlying physical processes resulting to the degradation observed for the tensile properties.

gated in the present study and by using a tensile strength value σ_f for the fiber fabric of 840 MPa[31] and a tensile strength value σ_m for the matrix of 90 MPa,[5] it gives a composite tensile strength value of 540 MPa. As discussed in the previous chapters, thermal treatments may cause appreciable decrease on the degree of crystallinity and increase of porosity; both effects are decreasing the tensile strength of the matrix significantly. By taking as an example a matrix tensile strength value reduced to 45 MPa, the composite's tensile strength value calculated by the lamination theory decreases to 495 MPa. A further reduction of the composite's tensile strength for the thermal-treated specimens could be attributed to the increased porosity at the interfaces between the plies and in particular between fibers and matrix, which is also influencing the strength of the plies in 0° direction. Investigation in Ref. 22 performed on unidirectional AT-400/PPS and AT-400/Nylon6 com-

CONCLUSIONS

- The effect of matrix evolution caused by thermal treatments of carbon fiber-reinforced PPS composite specimens, at temperature and time conditions that are relevant for processing PPS composites, on in-plane shear and tensile properties of the composite has been investigated. Thermal treatments were made in air in an oven to account for matrix evolution in the presence of oxygen, which refers to processes carried out in air.
- The performed mechanical tests have shown an appreciable decrease of both tensile and in-plane shear strength properties when each single ply of the material was subjected to thermal treatment prior to the consolidation of the plies to a laminate. On the contrary, a property increase was observed when, prior to the consolidation the whole set of plies placed in form of a sheet was subjected to thermal treatment.
- Changes on the degree of crystallinity and porosity caused by the thermal treatments may explain the observed changes of the in-plane shear properties. On the other hand, mentioned changes may only partially explain the observed
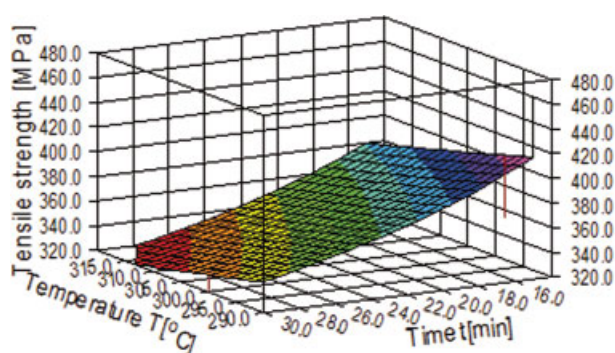
(a)
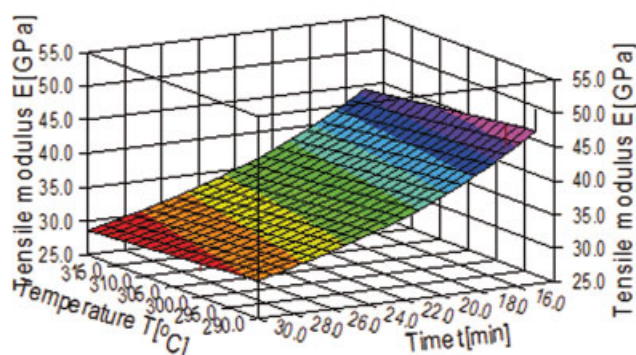
(b)

Figure 13 Three-axis map combining heating temperature and heating time with tensile strength (a) and tensile modulus (b). [Color figure can be viewed in the online issue, which is available at www.interscience.wiley.com.]

degradation of the fiber-dominated tensile strength properties. Further, systematic investigation is needed to understand the underlying physical processes causing the observed tensile strength reduction.

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