

# **CARBON FIBER FABRICS REINFORCED PPS LAMINATES : INFLUENCE OF TEMPERATURE ON MECHANICAL PROPERTIES AND BEHAVIOR**

J. Aucher, B. Vieille, L. Taleb  
Groupe de Physique des Matériaux, UMR 6634 CNRS, INSA de Rouen  
76801 Saint Etienne du Rouvray – France  
Jeremie.Aucher@insa-rouen.fr

## **SUMMARY**

Depending on the applied thermomechanical loading, a temperature increase up to the glass transition temperature ( $T_g=120^\circ\text{C}$ ) of fiber fabrics reinforced PPS laminates degrades significantly the quality of the adhesion at the fiber/matrix interface. However, it does not seem to be detrimental for the tensile and the double lap joint properties.

*Keywords: carbon fabrics - PPS resin – interlaminar shear - bolted joints - temperature*

## **INTRODUCTION**

Thermosetting resin based composite materials have been extensively used over the past 30 years. Even if they do present interesting mechanical properties, they are also characterized by undeniable drawbacks (low temperature storing - difficult-to-control reticulation process - long curing process - handmade draping that generates most of the non reversible defects of the manufacturing process). High performance thermoplastic resins and more particularly polyphenylenesulfide (PPS) represent a promising alternative to thermosetting resins problems because it offers a number of advantages over epoxy resins (chemical and impact resistance over a wide range of temperatures). Thus, a wide range of TP is available but in the area of high performances TP, PEEK and PPS are probably the most widely reported TP semi-crystalline resins [1]. At elevated temperatures, the nonlinear behavior of fiber-reinforced composites becomes significant, especially under off-axis loading conditions [2][3]. This nonlinear response, associated to the shear deformation of the polymer matrix along reinforcing fibers, is enhanced at high temperatures due to the viscoplastic nature of the TP resins [4][5][6]. The load bearing performances of pin connected UD carbon/PPS composites have been evaluated at  $21^\circ\text{C}$  [7]. There is no study currently available in the literature which investigates the joint strength at elevated temperature for carbon fabrics reinforced PPS. Thus, very few works highlight the behavior of carbon fabrics PPS laminates [8][9] and a few authors investigated the influence of high temperatures on the behavior of UD reinforced PPS resins [10][11][12][13].

## **ABOUT THE INFLUENCE OF TEMPERATURE ON POLYMERS**

Crystallinity in high-performances polymers is important because it has a strong influence on chemical and mechanical properties: the crystalline phase tends to increase

the stiffness and tensile strength while the amorphous phase is more effective in absorbing impact energy [2]. The high temperature behavior of polymeric based composites is then narrowly associated to the degree of crystallinity of the matrix material.

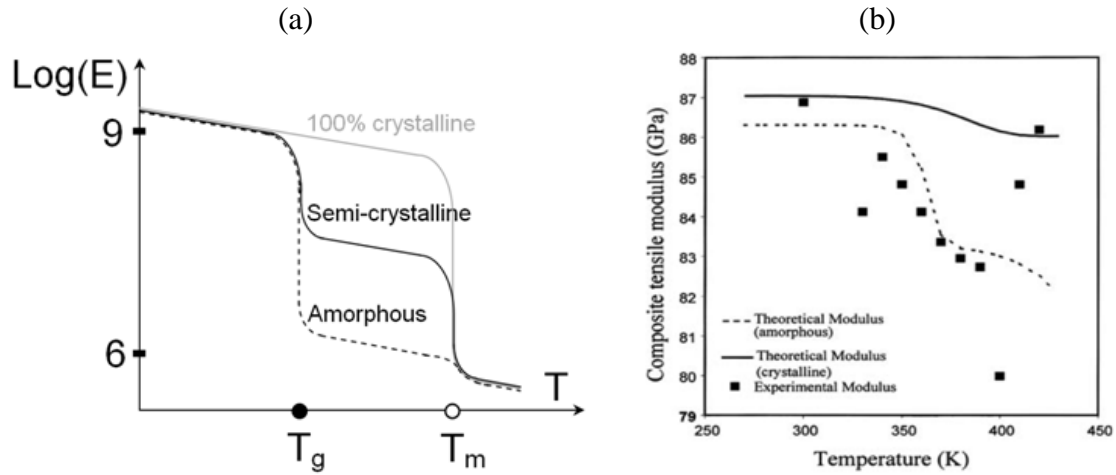


Fig. 1 - Evolution of the stiffness with temperature:  
 (a) Amorphous and semi-crystalline polymers - (b) C/PPS UD laminate [10]

The degree of crystallinity is determined by many factors including the type of polymer and the processing conditions. Thermoplastic polymers can be amorphous or semi-crystalline contrary to thermosetting polymers that are only amorphous (Fig. 1a). Semi-crystalline polymers like PPS resin have a degree of crystallinity ranging from 10 to 60% (26.3% in this work, determined by DSC) and they are characterized by a glass transition temperature  $T_g=90^\circ\text{C}$  and a melt temperature  $T_m=280^\circ\text{C}$ . Amorphous polymers like epoxy resin only have glass transition temperature  $T_g$ . As reported in [10], the tensile strength of the UD ply carbon/PPS laminate decreased by 17 percent from  $30^\circ\text{C}$  to  $140^\circ\text{C}$ . Between  $30^\circ\text{C}$  and  $130^\circ\text{C}$ , the tensile modulus decreased by 7.7 percent. These changes appear to occur near two distinct points, around the glass transition temperature of the neat PPS resin and the PPS based composite ( $T_g=126^\circ\text{C}$ ) as pointed out by Fig. 1b. For the carbon fiber fabrics reinforced PPS laminates studied in this work, a Dynamic Mechanical Thermal Analysis (DMTA) performed by the CRISMAT laboratory gives  $T_g=120^\circ\text{C}$ .

## EXPERIMENTAL PROCEDURE

The composite materials studied in this work are carbon fabrics reinforced PPS prepreg laminates plates. The woven ply prepreg laminate consists of 5-harness satin weave carbon fiber fabrics and the volume fraction of carbon fibers is 50%. The specimens tested in this work have the same stacking sequence: [0/45/0/45/0/45/0] and its lay-up can be considered as being quasi-isotropic. All the quasi-static tests were performed using a 100 kN capacity load cell of a MTS 810 servo-hydraulic testing machine at room moisture. The operating temperature ranges from room temperature (R.T.) to  $120^\circ\text{C}$ . Different tests were carried out on the carbon fiber fabrics reinforced PPS laminates specimens: tensile test, open hole tensile test, inter laminar shear test, single-bolt double lap joint and single-bolt single lap joint tests.

## RESULTS AND ANALYSIS

### Tensile tests on notched and unnotched laminates

These tests were carried out to investigate the role of the resin ductility, potentially enhanced by temperature, on the overstress accommodation around the hole and the notch sensitivity of both laminates. The hole factor  $C_h$  represents the hole sensitivity of the laminate ultimate strength. It is defined by  $C_h = \frac{\sigma_{notched}^u}{\sigma_{unnotched}^u}$  where  $\sigma_{notched}^u = F_{notched}^u/t.w$  and  $\sigma_{unnotched}^u = F_{unnotched}^u/t.w$  are respectively the notched and unnotched ultimate stresses,  $t$  and  $w$  being the thickness and width of the specimens. From these definitions, the stress-strain curves of notched and unnotched laminate can be compared for both laminates at both temperatures (Fig. 2).

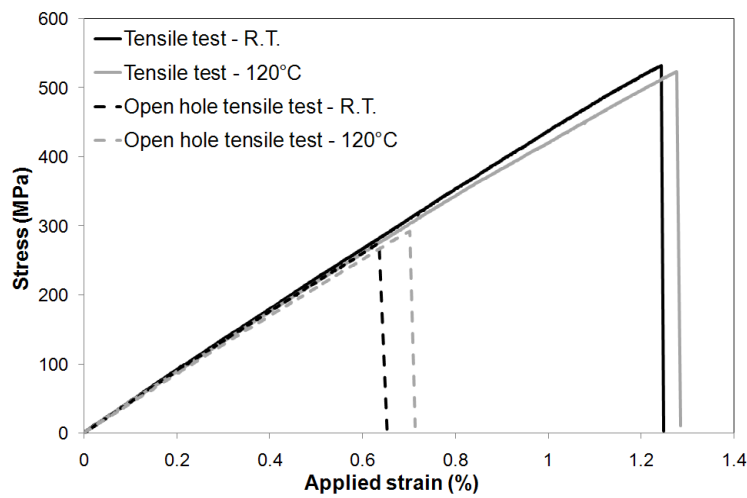


Fig. 2 - Tensile vs open hole tensile tests at both temperatures for C/PPS laminates

For the studied stacking sequence, the  $0^\circ$  oriented fibers (57% of the plies) carry the load and the laminates responses are dominated by the behavior of these  $0^\circ$  fibers resulting in a behavior independent from temperature. This is the reason why the laminate behavior is almost elastic-brittle in spite of the ductile nature of the resin. Then this slightly non-linear behavior is not associated to a plastic behavior of the PPS resin but to the progressive failure of  $0^\circ$  oriented fibers during loading. It is consistent with the literature results because the maximum test temperature is equal to the glass transition temperature ( $T_g=120^\circ\text{C}$ ) of C/PPS composites.

Tab. 2 - Tensile vs open hole tensile tests at both temperatures for C/PPS laminates:  
Mechanical properties

| $\sigma_{unnotched}^u$ (MPa) |       |          | $\sigma_{notched}^u$ (MPa) |       |          | $E_L$ (GPa) |       |          | $C_h$ |       |          |
|------------------------------|-------|----------|----------------------------|-------|----------|-------------|-------|----------|-------|-------|----------|
| R.T.                         | 120°C | R.C. (%) | R.T.                       | 120°C | R.C. (%) | R.T.        | 120°C | R.C. (%) | R.T.  | 120°C | R.C. (%) |
| 506                          | 469   | -7       | 255                        | 247   | -3       | 43          | 41    | -4       | 0.5   | 0.53  | +4       |

Considering the mechanical properties of notched and unnotched laminates (Tab. 2), temperature seems to influence very little both strength and stiffness with relative changes (R.C.) ranging from -3 to -7%. Thus, a service temperature equal to 120°C is not significantly detrimental for the mechanical behavior of PPS based composites submitted to such loadings. With increasing temperature, the hole sensitivity decreases a little what indicates temperature slightly influences the redistribution of stresses around the hole through a localized but little more extended plasticization of the PPS resin [14].

### Inter laminar shear test

The short-beam shear test is a commonly used method for characterizing the interlaminar failure resistance of fiber-reinforced composites. This test method involves loading a beam under three-point bending with dimensions (a short-beam) such that an interlaminar shear failure is induced (Fig. 3). This method measures the "apparent" interlaminar shear strength of composite materials. Thus, the short-beam shear method is not appropriate for generating design allowables [15] but it provides information about the quality of the adhesion at the fiber/matrix interface. The interlaminar shear strength  $\tau$  is defined by  $\tau = 3F^m/4wt$  where  $F^m$  corresponds to the maximum load applied to the specimen,  $w$  and  $t$  being respectively the width and thickness of the specimen. This strength is significantly affected by a temperature increase up to 120°C since it changes from  $\tau=54\text{MPa}$  to  $\tau=40\text{MPa}$  (-26%).

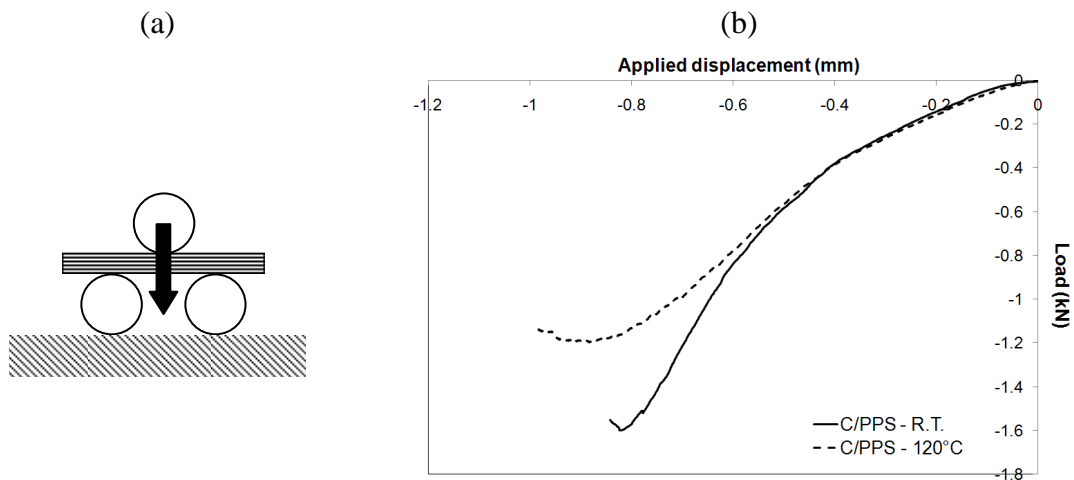


Fig. 3 - Inter laminar shear test: (a) Three-point bending of a short beam - (b) Responses at both temperatures for C/PPS laminates

The load-displacement curves show that the mechanical response of the laminate is the same from the elastic behavior point of view but the interlaminar shear of the PPS resin between plies is associated to a ductile behavior which is enhanced by a temperature increase. Thus, the elongation at maximum load increases by 7%. From the edge macroscopic views (Fig. 4), the residual plastic deformation of samples at both temperatures, and more particularly at 120°C, indicates that the interlaminar shear strength of the laminate does not exactly corresponds to an ultimate strength due to a

pure interlaminar shear. However, the influence of temperature on the load transfer between plies can be investigated through these values. The SEM observations of the failed samples edge clearly show multiple interlaminar shear cracks mostly around the neutral axis and these cracks propagate transversely to load direction. Moreover, the PPS resin being less ductile at R.T., it results in more cracks (Fig. 4). However, the geometry of the fabrics reinforcement opposes some resistance to this propagation at the plies interface. Since the fibers are broken, the upper and lower plies of the specimens seem to fail respectively in tension and compression.

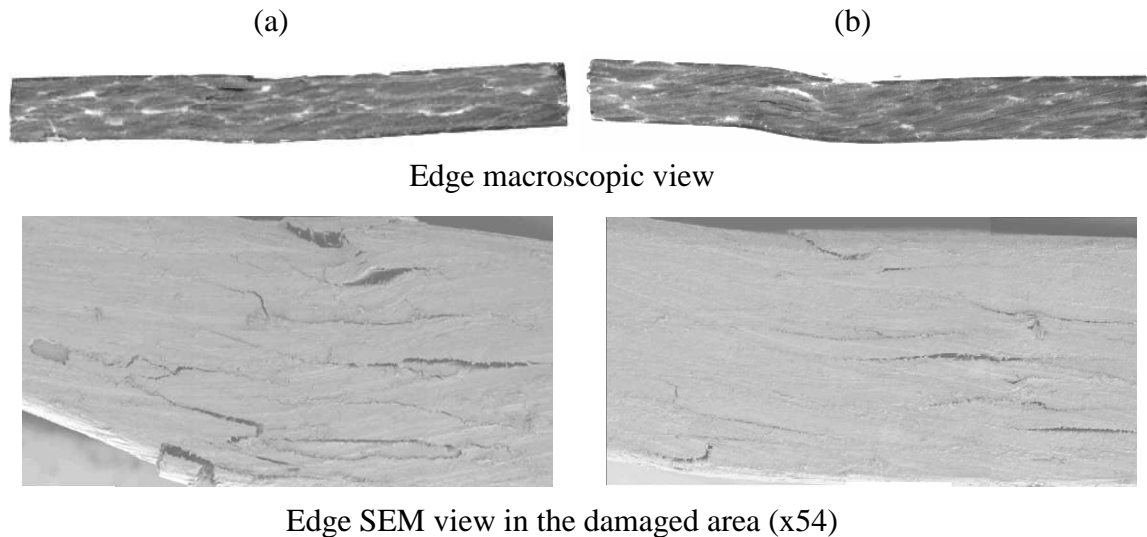


Fig. 4 - Observations of the edge of failed specimens after inter laminar shear tests:  
(a) R.T. - (b) 120°C

Finally, it is possible to conclude that a temperature increase around the glass transition temperature degrades significantly the properties of the fiber/matrix interface. The viscoplastic behavior of the resin enhanced by elevated temperatures seems to be detrimental for the load transfer by shear between plies.

### Bolted joint tests

In advanced technology fields, the joining of composites to other structures (metals or composites) with mechanical fasteners is often required. Structures are usually joined with bolts or rivets according to two geometrical configurations: single-bolt single lap joint and single-bolt double lap joint tests (Fig. 5).



Fig. 5 - Schematic representation of single-bolt single lap joint and single-bolt double lap joint tests [16]

For practical purposes, the ultimate bearing strength  $\sigma_b$  is used for dimensioning bolted joints and corresponds to the ultimate force  $F^u$  carried by the specimen. It is defined by  $\sigma_b = F^u/d.t$  where  $d$  and  $t$  are respectively the hole diameter and the specimen thickness. Similarly, the damage stress  $\sigma_d$  is defined by  $\sigma_d = F/d.t$  where  $F$  is the force carried by the specimen when a clear first sign of damage appears. From the following curves (Fig. 6), it is therefore possible to calculate these values reported in the following table (Tab. 3).

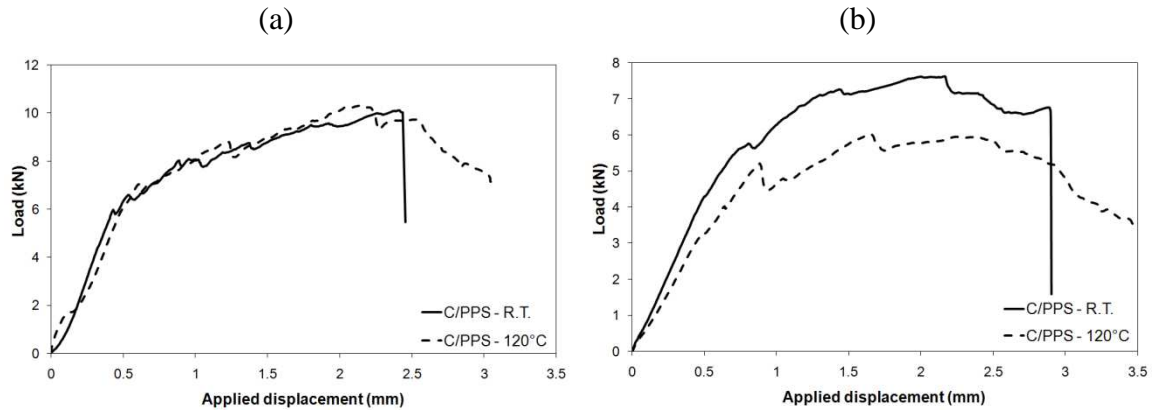


Fig. 6 – Bolted joint tests on C/PPS laminates: (a) Double lap - (b) Single lap

Tab. 3 – Single lap and double lap joint tests: comparison of damage and bearing stresses for C/PPS laminates

| Single lap       |       |          |                  |       |          | Double lap       |       |          |                  |       |          |
|------------------|-------|----------|------------------|-------|----------|------------------|-------|----------|------------------|-------|----------|
| $\sigma_d$ (MPa) |       |          | $\sigma_b$ (MPa) |       |          | $\sigma_d$ (MPa) |       |          | $\sigma_b$ (MPa) |       |          |
| R.T.             | 120°C | R.C. (%) | R.T.             | 120°C | R.C. (%) | R.T.             | 120°C | R.C. (%) | R.T.             | 120°C | R.C. (%) |
| 540              | 492   | -9       | 720              | 568   | -21      | 415              | 504   | +21      | 723              | 740   | +2       |

From the strength standpoint, it seems that that temperature influence depends on the geometrical configuration of the joint (single or double lap). The single lap joint is a lot more detrimental for both damage and bearing stresses with relative changes (R.C.) of respectively -9% and -21% (Tab. 3). However, the bearing strength of the double joint seems to be unaffected by a temperature increase up to 120°C but the outset of damage is clearly higher (+21%) at elevated temperature. Even though they are not studied in this work, the dimensions of both kinds of joints must be underlined because they have a strong influence on the failure mode of the joints:  $w/d = 7.9$  and  $t/d = 2.4$  for double lap joints and  $w/d = 5$  and  $t/d = 2.4$  for single lap joints.

### Single-bolt double lap joint tests

For C/PPS laminates, temperature seems to shift the failure mode from cleavage to bearing what is more interesting from the service behavior standpoint of structures because it is a progressive failure mode (Fig. 7a).

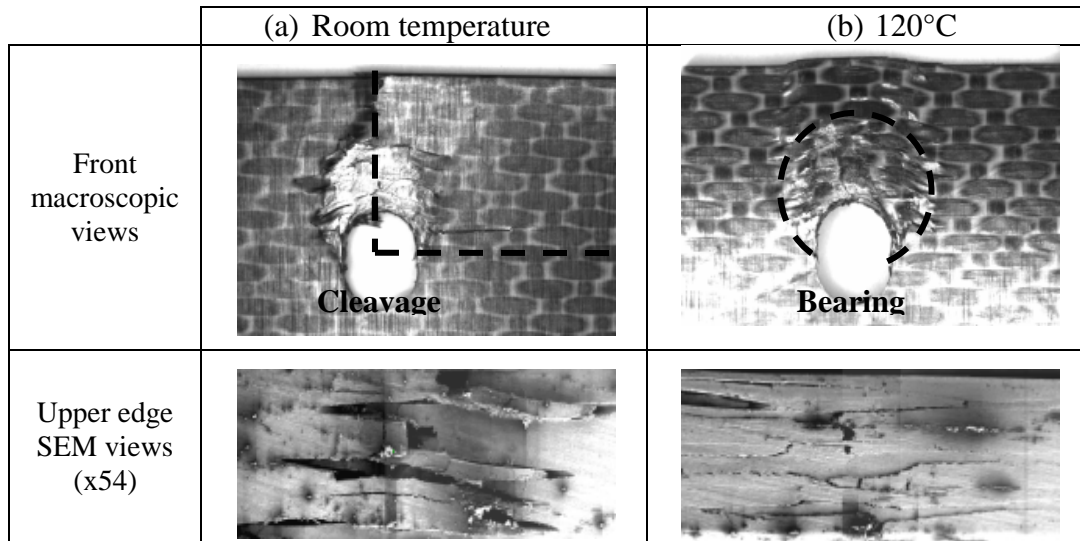


Fig. 7 – Double-lap joint test - Observation of failure surfaces at both temperatures for C/PPS laminates

From the upper edge SEM view of C/PPS specimens, the cleavage failure mode is related to the debonding of the fibers/matrix interface and the transverse fibers breakage at room temperature (Fig. 7b). At 120°C, there is no debonding and the outset of damage observed on the upper edge of the specimen (mainly cracks at the fiber/matrix interfaces) is associated to the extension of the bearing area to the upper edge of the specimen (Fig. 7b). It confirms the mechanism of plasticization of the resin during the vertical displacement of the bolt. In the meantime, the longitudinal fibers above the bolt/hole area are submitted to compression and experience buckling at some level of the load whereas the transverse fibers wind around the bolt. It results in an inflection of the load-displacement curve (Fig. 6a). One possible explanation is that the ductile nature of the PPS resin at elevated temperature promotes a localized and cumulated plastic deformation (bearing) above the bolt/hole contact area and a bearing failure of the bolted joint. The increase of the damage stress at 120°C is probably associated to an extensive plasticization of the resin (indicated by the observation of an extended bearing area in Fig. 7a) what delays the outset of damage.

### Single-bolt single lap joint tests

Contrary to the double-lap joint, the geometry of the single-lap joint is non-symmetric. When a tensile load is applied to the joint, this asymmetry and the bolt-hole clearance cause the fastener to tilt in the hole, which implies that the bolt/hole contact pressure becomes non-uniform through the plate thickness. A stress concentration appears at the hole edge close to the shear surface of the joint. The non-uniform stress distribution may cause the plate to bend out-of-plane which is referred to as secondary bending when the clearance is too low [16]. Like in the case of double lap joints, temperature seems to shift the failure mode from cleavage to bearing for C/PPS laminates. From the upper edge macroscopic views, the important out-of-plane deformation (white-dotted line in Fig. 8) observed at both temperatures is directly associated to the secondary bending. This deformation seems to be enhanced at elevated temperature as well as the

debonding of the fiber/matrix interface. It results in a significant decrease in the joint strength (-21%) when temperature increases.

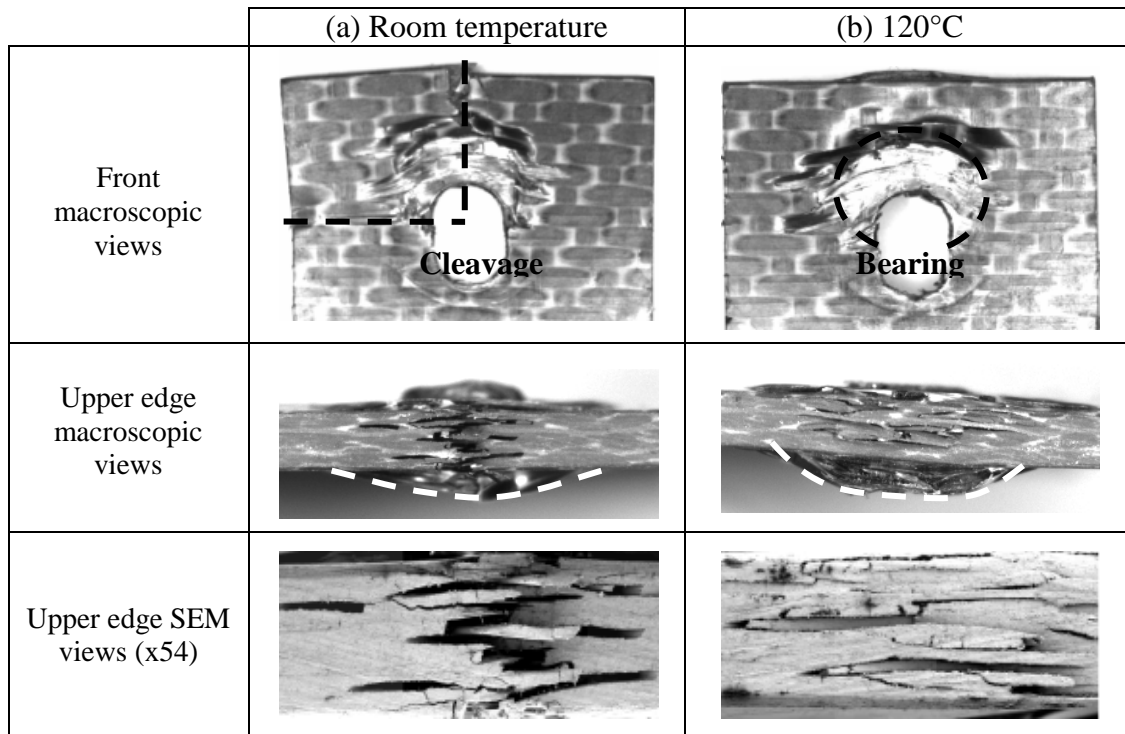


Fig. 8 – Single-lap joint test - Observation of failure surfaces at both temperatures for C/PPS laminates

From the upper edge SEM view of C/PPS specimens (Fig. 8), the cleavage failure mode is related to the debonding of the fibers/matrix interface and the transverse fibers breakage at room temperature. At 120°C, an important debonding of the fiber/matrix interfaces can be observed. It is associated to the extension of the bearing area to the upper edge of the specimen. During the vertical displacement of the bolt, two mechanisms contribute to damage the joint: plasticization of the resin (bearing) on the one hand and out-of-plane deformation on the other hand.

### CONCLUSION

This work aimed at investigating the influence of temperature on the mechanical behavior of carbon fiber fabrics reinforced polyphenylenesulfide (PPS) laminates submitted to different stress states at different temperatures (23°C and 120°C): tensile and open hole tensile tests, inter laminar shear test, single-bolt double lap or single lap joint tests. About the mechanical properties of notched and unnotched laminates, a service temperature equal to the glass transition temperature of C/PPS composites (i.e.  $T_g=120^\circ\text{C}$ ) seems to influence very little both strength and stiffness. In addition, the hole sensitivity decreases a little what indicates temperature slightly influences the redistribution of stresses around the hole through a localized but little more extended



plasticization of the PPS resin. Considering the interlaminar shear tests results, a temperature increase around the glass transition temperature of C/PPS composite materials degrades significantly the quality of the adhesion at the fiber/matrix interface. The viscoplastic behavior of the resin enhanced by elevated temperatures seems to be detrimental for the interlaminar shear strength (-26%) hence for the load transfer between plies. For C/PPS laminates joints (double and single lap), temperature seems to shift the failure mode from cleavage to bearing what is more interesting from the service behavior standpoint of structures because it is a progressive failure mode. The bearing strength of the double joint seems to be unaffected by a temperature increase up to 120°C but the outset of damage is clearly higher (+21%) what is probably associated to an extensive plasticization of the resin delaying the outset of damage. For single lap joints, the out-of-plane deformation associated to the secondary bending seems to be enhanced at elevated temperature as well as the debonding of the fiber/matrix interface. It results in a significant decrease in the joint strength (-21%) when temperature increases up to 120°C.

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