Investigation of processing parameters on static properties of glass/polyamide laminated thermoplastic composites

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Abstract

Thermoplastic composite materials such as E-glass/polyamide have gained much interest in industry due to their recyclability, high toughness and chemical resistance characteristics. However, their use in high volume production applications have been limited to date as a result of drawbacks with thermoplastics, including susceptibility to oxidation and challenges in controlling fiber alignment during processing. In this study, multi-layered composite laminates were produced by hot press moulding E-glass/polyamide 6 prepregs. The effects of peak temperature and holding time at peak temperature were investigated and optimized, while processing pressure and mean cooling rate were held constant. Optical microscopy was performed on the fabricated laminates to investigate the consolidation and quality. Tensile quasi-static mechanical tests were performed on laminates with $[0]_8$, $[90]_8$ and $[0_2/90_2]_s$ stacking sequences that were fabricated using two processing cycles. In addition, scanning electron microscopy (SEM) was used to study the modes of failure. The laminates processed with a 240°C maximum temperature and no holding at maximum temperature exhibited lower oxidation levels and improved fiber matrix adhesion, and therefore superior mechanical properties which was confirmed by SEM. As was expected, $[0]_8$ specimens exhibited a linear deformational behavior until failure, and the longitudinal strength was approximately 885 MPa which is comparable to similar E-glass/epoxy laminates. The $[90]_8$ specimens exhibited a maximum strength of 20 MPa, which is lower than E-glass/epoxy and can be attributed to the weaker bonding between the fiber and matrix when compared to glass/epoxy. Also, the E-glass/polyamide cross-ply laminates had higher strength compared to E-glass/epoxy while having the same modulus. These findings support the feasibility of producing high quality continuous glass/polyamide laminates that may be suitable for use in high performance applications, which an attractive alternative to more conventional glass/epoxy laminates due to their recyclability.

1 Introduction

Thermoplastic composite materials are a major interest within industry due to their many advantages when compared to thermosetting based composites, including high ductility, high toughness, good chemical resistance characteristics, recyclability, as well as the ability to weld thermoplastic components together. However, their use in high volume production applications is limited to date due to some key challenges. For example, some thermoplastics, including polyamide, readily oxidize

when heated above their melting temperature. Also, the ability to control fiber alignment during processing is challenging due to high shear forces imparted by the matrix. Glass and carbon fibers are commonly used to reinforce thermoplastic polymers such as polyamide, polypropylene and PEEK. These materials can be categorized according to the length of the reinforcing fibers: short and long fiber reinforced composites (SFC, LFC) and continuous fiber reinforced composites (CFC). Many studies focused on the processing and mechanical characterization of short and long fiber thermoplastic composites have been reported [1-4]. These studies show that increasing fiber length from short fiber to long fiber improves the tensile strength and tensile modulus of these composites [5].

CFCs are known to have superior mechanical properties and also retention of strength and modulus in different environmental conditions when compared to their SFC and LFC counterparts [6]. However, the volume of published work on processing and characterization of continuous fiber thermoplastic composites is limited to high performance composites such as carbon/PEEK, which are frequently processed by compression moulding.

Polyamides are known to have excellent mechanical properties, including high stiffness and strength, and when they are reinforced with glass fibers good mechanical properties are anticipated when compared to high performance thermoplastic composites. However, prior to implementing continuous glass fiber/polyamide composites into structural components such as wind turbine blades and automotive parts [7], their characteristics and mechanical performance under different loading conditions must be better understood and studied. Currently glass/epoxy is widely used for manufacturing wind turbine blades, thus in order to substitute glass/epoxy with glass/polyamide, the mechanical properties of the two material systems should be compared.

Some reported studies have focused on the mechanical behavior of continuous glass fiber/polyamide composites, particularly in shear and flexural loading. Cinquin et al. [8] studied the effect of different additives on static and dynamic shear and the flexural fatigue behavior of unidirectional glass/polyamide composites. They identified the significant effect of thermal stabilizers in improving the dynamic shear modulus and flexural fatigue strength of glass/polyamide composites. Yan et al. [9] investigated the flexural and shear strength of vacuum infusion moulded glass/polyamide composites. They investigated the content of catalyst and processing temperature on the degree of crystallinity and strength of the produced composite. Cartledge and Baillie [10] studied the effect of cooling rate on the Iosipescu shear strength and transverse flexural strength of glass/polyamide composites, and concluded that lowering cooling rate enhanced the interfacial tensile and shear strength through improving the crystallinity of the polyamide and also the transecrystalinity between glass fibers and polyamide. Von Rijswijk [11, 12] studied the processing of woven glass fiber reinforced polyamide via resin transfer moulding of anionic polyamide-6 (APA-6) to produce wind turbine blades. He optimized the mould temperature for maximizing the interfacial shear strength and fatigue strength of this material and compared these properties with those of glass/epoxy. Probhakaran et al. [13] examined the compression, bending and short beam shear behavior of glass fiber reinforced polyamides with three different commercial prepregs and comingles. Generally, the laminates produced from comingles showed better mechanical properties due to the proper alignment of the fibers, even distribution and improved wetting of the fibers with the matrix. Haspel et al. [14] characterized the shear strength of glass/polyamide and glass/epoxy composites produced by resin transfer moulding. The assessment was done by using a push out test. They concluded that glass/epoxy has a very high interfacial shear strength compared to glass/polyamide.

A review of the literature indicates that there is limited research assessing the performance of unidirectional-ply glass/polyamide laminates, which would otherwise be valuable for the design and manufacturing of corresponding composite structures. The goal of this study was to investigate the influence of processing parameters on the performance of unidirectional and cross-ply glass/polyamide laminates produced by compression moulding. The quality of impregnation of the laminates was investigated by optical microscopy. Furthermore, the performance of these laminates under quasi-static tensile loading was studied and compared to that of glass/epoxy and

glass/polypropylene laminates with similar fiber volume fractions. Fracture surfaces were also analyzed by scanning electron microscopy.

2 Material and processing

2.1 Materials

Unidirectional E-glass/polyamide 6 tape supplied by Jonam Composites Ltd was used for producing composite laminates. Prior to processing, the nominal volume fraction and the total areal weight of the prepregs were 45 % and 370 g/m^2, respectively, while the width and thickness of the tape were 110 mm and 0.2 mm, respectively.

2.2 Processing

Two processes were used for fabricating the composite laminate plates as will be described in this section. Picture frame moulds were prepared from 6061 aluminum alloy, and used for both processes. The prepregs were stacked in the mould cavity according to the specified lamination.

The first group of laminates were processed using a manual hot press machine. The platens were pre-heated to 255 °C, then the mould was placed between the platens and the pressure-temperature cycle shown in Fig. 1 was applied. Cooling of the platens was accomplished by water such that the mean cooling rate was 10 °C/min. This will hereafter be denoted as process A.

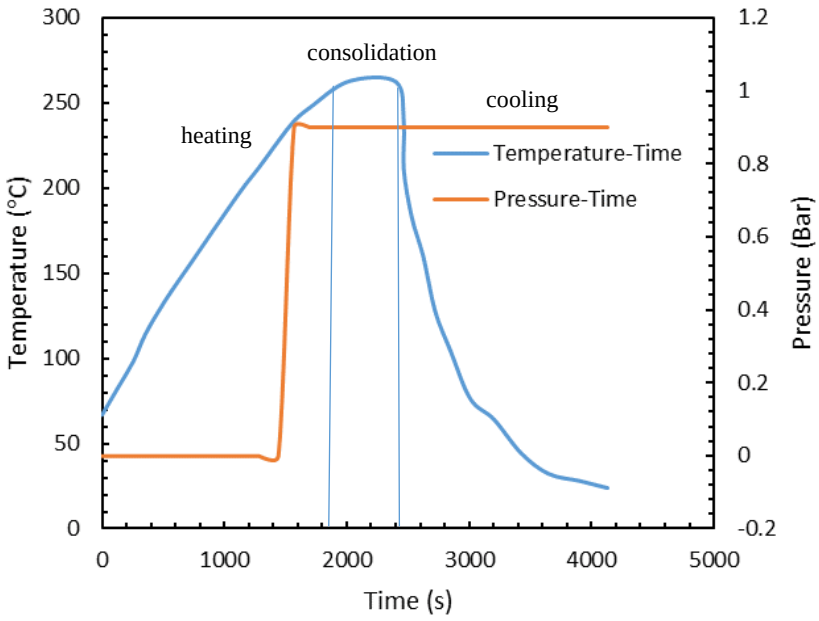

Figure 1. Temperature-time and pressure-time process diagram for fabrication of glass/polyamide used in process A.

To limit the observed oxidation produced along the edges and that permeated through to the middle of the panels with the manual hot press, two key modifications to the procedure were made. First, an automatic 30-ton Carver hot press with a notably higher heating capacity was used, allowing the mould to reach the maximum temperature four times faster than the manual hot press. Secondly, the dwell time was removed from the process.

To further decrease the onset of oxidation, the maximum temperature was also slightly reduced while the mean cooling rate was kept constant to 10 °C/min. In the new temperature-time cycle, the time at which samples were exposed to temperatures higher than the prepreg melting temperature was reduced to 2-3 minutes to ensure a reduction in oxidation reaction time. Temperature-time and pressure-time cycle of process B is shown in Fig. 2.

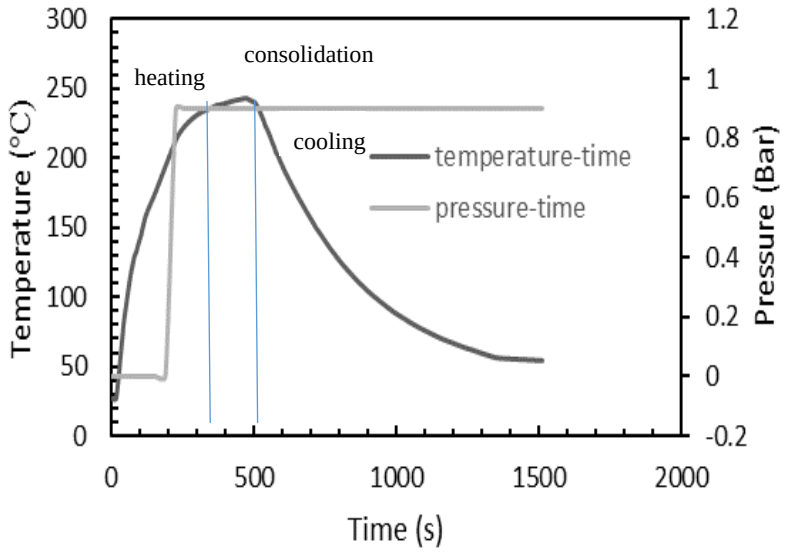

Figure 2. Temperature-time and pressure-time process diagram for fabrication of glass/polyamide used in process B.

2.3 Optical Microscopy

Optical microscopy was used on the laminates produced using both process A and process B to investigate the impregnation of the glass fibers. As seen in Fig. 3, the quality of the impregnation is noticeably high. It should be noted that some voids and resin rich areas have been observed in both types of specimens as is shown in the figure. Qualitative observations have confirmed that the void content is similar for laminates produces using both process A and B.

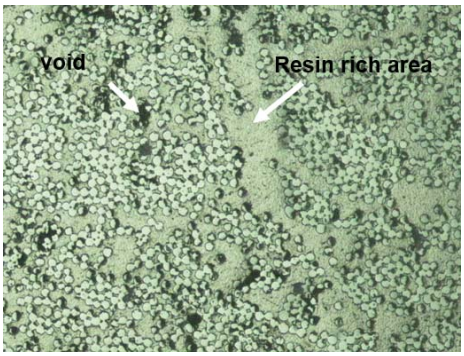

Figure 3. Optical microscopy of unidirectional glass/polyamide composite cross-section.

The optical images were also used in ImageJ software to compute the volume fraction of the specimens. The obtained volume fraction for all specimens was approximately 58 %.

3 Mechanical testing results and discussion

Tensile static tests were performed on $[0]_8$, $[90]_8$, $[0_2/90_2]_s$ and $[0_4/90_4]_s$ specimens in accordance with ASTM D3039. For this purpose, an MTS 810 hydraulic test frame was used for the $[0]_8$ and $[90]_8$ laminations, and for the remaining laminations an Instron 8874 hydraulic test frame was used. The displacement rate was set at 2 mm/min for all tests. Strain was measured by a 10 mm gauge extensometer for some specimens, while digital image correlation (DIC) was also used for a number of the test specimens. At least five specimens were tested for each lamination, and the mean value was reported for both strength and Young Modulus. Aluminum end tabs of 2 mm by 2 mm size were bonded to both ends of all specimens. Fig. 4 and Table 1 summarize the dimensions of the different test specimens used in this study.

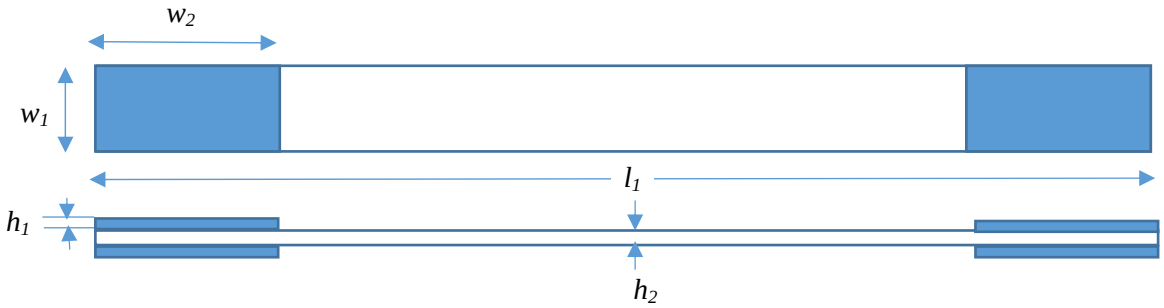

Figure 4. Geometry of the specimens used for static tensile tests.

Lamination	l_1 (mm)	w_1 (mm)	w_2 (mm)	h_2 (mm)	h_1 (mm)
$[0]_8$	110	15	20	1.4±0.1	1
$[90]_8$	110	20, 15	20	1.4±0.1	1
$[0_2/90_2]_s$	110	15	20	1.4±0.1	1
$[0_4/90_4]_s$	110	15	20	2.9±0.1	1.5

Table 1: Dimensions of the specimens used for static tensile tests.

After completion of the tensile static tests, some of the fractured specimens were gold coated using Polaron Instruments E5100 SEM Coating System, and LEO (Zeiss) 1550 and Philips XL30 microscopes were used to observe the morphology of fractured surfaces.

3.1 $[0]_8$ laminates

The representative stress-strain curves for glass/polyamide $[0]_8$ laminates fabricated using process A and B are presented in Fig. 5, which exhibit a linear profile as shown. The mean strength of the material for the process A samples is 904±3.5 MPa and the Young modulus is 43.2 GPa. The mean strength for process B specimens is 885±78.5 MPa and the mean Young modulus is 41.6±3.7 GPa, which is comparable to the process A specimens. Stress-strain curves of the process A samples exhibited a step like behavior (see Fig. 5). This behavior may be due to lower mechanical properties of polyamide caused by oxidation using process A, and thus lower interface strength between the glass and polyamide in the samples. This is confirmed through SEM images in the following section. It should be noted that the failure strain for each glass/polyamide specimen was approximately 2.5%.

The experimental stress-strain curves for the E-glass/polyamide $[0]_8$ specimens were also compared with those of glass/epoxy and glass/polypropylene with maleic anhydride (MA) having the same fiber content. As shown in the Fig. 5, the longitudinal Young modulus of both glass/polyamide specimens are approximately the same as glass/epoxy, however, the strength of the glass/polyamide specimens is slightly lower than glass/epoxy. This demonstrates that in fiber-dominated loading conditions, the performance of the glass/polyamide specimens is comparable to that of glass/epoxy. The longitudinal Young modulus of glass/polyamide is comparable to that of glass/polypropylene (with and without MA) which is expected since polypropylene and polyamide have comparable Young's modulus. Also, the strength of glass/polyamide is similar to glass/polypropylene with 10 % wt. MA, which is due to the improved fiber and matrix bonding in this percentage of MA.

According to the rule of mixtures, with a fiber volume fraction of 0.58 the theoretical Young's modulus for glass/polyamide is 43.39 GPa. This value is very close to the measured experimental value for the specimens fabricated using processes A and B, which suggests that the resulting void content is considered to be low.

For obtaining the Poisson's ratio of glass/polyamide, DIC was used for some of the tested samples. After analyzing the strain fields and according to the definition $\nu_{12} = -\varepsilon_2 / \varepsilon_1$, the measured Poisson's ratio was equal to 0.28. The Poisson ratio value obtained using rule of mixtures with a fiber volume fraction of 0.58 was 0.29, which is also comparable.

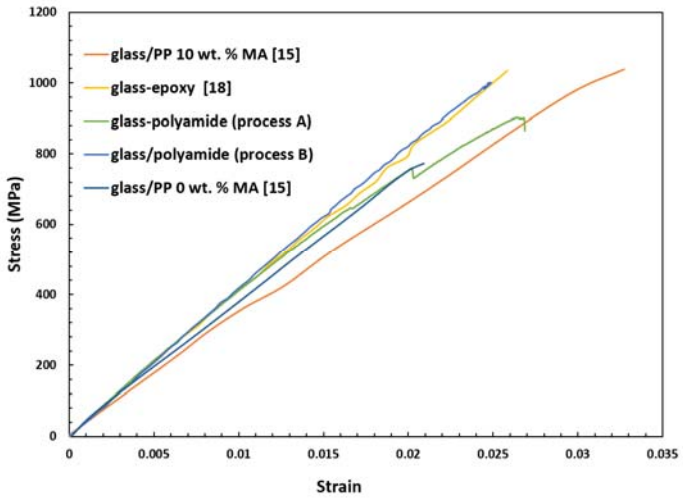

Figure 5. Tensile stress–strain curves for various $[0]_8$ laminates of glass/polyamide, glass/epoxy and glass polypropylene composites.

3.1.1 Scanning Electron Microscopy (SEM) of $[0]_8$ laminates

The SEM images perpendicular to the fractured surfaces for the two $[0]_8$ laminates which were processed with process A and process B are shown in Fig. 6. As shown in the SEM images, for both processing conditions cracks initiated in the matrix and also at the interface between fiber and matrix, and propagated along the fiber direction until they formed into long splitting cracks. The sequences of splitting type failures occurred in different sites of the specimen, which was followed by fiber fracture and finally resulting in failure of the specimen. It should be mentioned that fiber fracture was very localized in a small zone, which has been also observed in Ref. [16].

As illustrated in Fig. 6, process A specimens had a significant number of bare fibers at the fracture surface in comparison to the process B specimens, confirming that these specimens have a lower fiber/matrix interface strength. This was likely caused by the degradation of matrix resulting from oxidation, which promoted poor adhesion between the fibers and matrix.

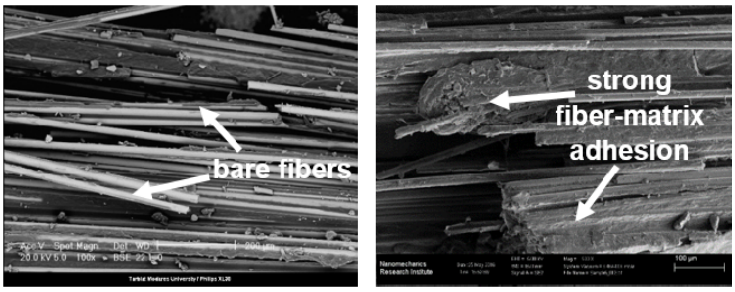

Figure 6. SEM micrographs of fracture surfaces for $[0]_8$ glass/polyamide specimens loaded in the longitudinal direction: a) fabricated with process A, and b) fabricated with process B.

3.2 $[90]_8$ laminates

The representative stress-strain curves for $[90]_8$ laminates of glass/polyamide processed with process A and process B, glass/epoxy (50% fiber content) with different sizings, and glass/polypropylene with 10 wt. % of MA and without MA are shown in Fig. 7. The mean transverse strength increased by nearly 35% and the mean Young modulus increased by 15% from process A to process B, which again suggests that the optimized process results in superior fiber/matrix interfacial strength. The failure strains for both glass/polyamide specimens are between 0.35% and 0.4%, which corresponds

to the matrix failure strain of 0.3%. The transverse modulus evaluated with the rule of mixtures results in a value of E_2 = 5.69 GPa, which is comparable to the measured value of the specimens fabricated with process B. This further supports the improved quality of the process B specimens.

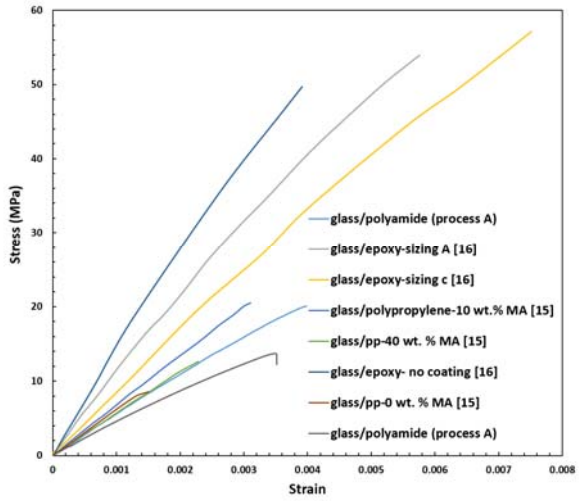

Figure 7. Tensile stress-strain curves for various $[90]_8$ glass/polyamide, glass/epoxy and glass/polypropylene specimens.

From Fig. 7, the strength of $[90]_8$ glass/epoxy laminates is at least 2.5 times more than that of $[90]_8$ glass/polyamide, with a notably greater modulus. The decreased performance of the glass/polyamide specimens in the transverse direction when compared to glass/epoxy may be a result of weaker bonding between the glass fibers and polyamide [17]. Furthermore, the strength of the $[90]_8$ glass/polypropylene specimens is lower than that of the glass/polyamide samples tested here, however, by adding 10 % wt. MA the glass/polypropylene has the same transverse strength as glass/polyamide.

3.2.1 Scanning Electron Microscopy (SEM) of $[90]_8$ laminates

The fracture surfaces for $[90]_8$ laminates are shown in Fig. 8. For the samples which were fabricated using process A, many bare fibers and debonding between fiber and matrix are observed. For the samples which were fabricated by process B, debonding occurred in some regions of the fiber surface, while for the majority of fibers significantly improved adhesion existed between the fibers and matrix. Many localized matrix hackles on the fiber surfaces illustrate the strength of the fiber/matrix interface (see Fig. 8b). These results further support the fact that process B results in lower matrix degradation and improved fiber/matrix interfacial strength.

Figure 8. SEM micrographs of $[90]_8$ glass/polyamide specimen fracture surfaces loaded in the transverse direction: a) fabricated with process A, and b) fabricated with process B.

3.3 $[0_n/90_n]_s$ laminates

The representative stress-strain curves for $[0_2/90_2]_s$ and $[0_4/90_4]_s$ laminates fabricated with process B, $[0_2/90_2]_s$ laminates fabricated with process A, and a $[0_2/90_2]_s$ glass/epoxy laminate are shown in Fig.

9. As shown, the mean strength Young's modulus of $[0_2/90_2]_s$ glass/polyamide laminates has increased by 24.4 % and 26.7 %, respectively which confirms the improved adhesion between fiber and matrix in process B.

Fig. 9 also shows that the Young's modulus of glass/epoxy and glass/polyamide are comparable for the $[0_2/90_2]_s$ lamination; however, the strength of glass/epoxy is 19% lower than that of glass/polyamide. This is due to the fact that transverse cracks in the glass/epoxy initiate at very low applied strain (0.25%), which notably decreases the Young modulus.

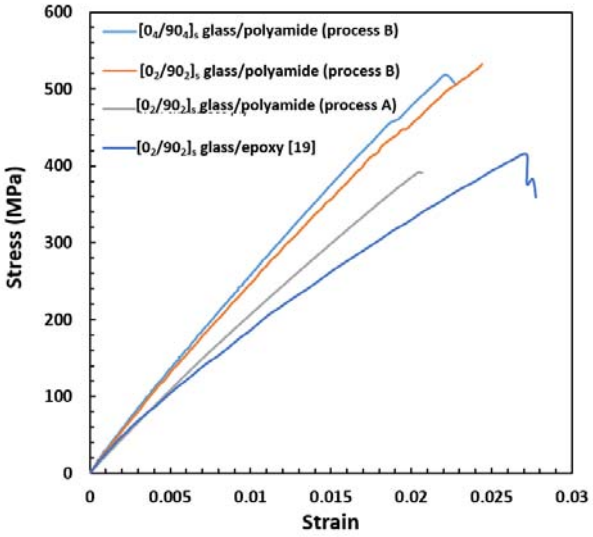

Figure 9. Tensile stress-strain curve for $[0_2/90_2]_s$ glass/polyamide and glass/epoxy, and $[0_4/90_4]_s$ glass/polyamide specimens.

3.3.1 Scanning Electron Microscopy (SEM) of $[0_2/90_2]_s$ laminates

As seen in Fig. 10 for samples fabricated with process A, in the central transverse layer there is debonding between the fibers and matrix. In the longitudinal layer there are fiber fractures and localized matrix splitting, as well as fiber matrix debonding and fiber pull out. For samples which were fabricated with process B, in the central transverse layer good bonding between the fibers and matrix were observed which is consistent with the results for the $[0]_8$ and $[90]_8$ laminates.

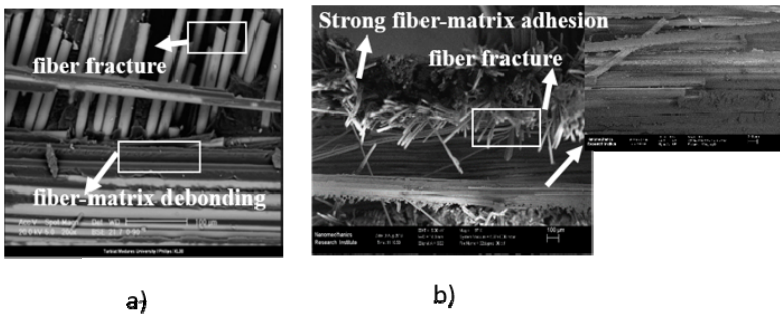

Figure 10. SEM micrographs of fracture surfaces in $[0_2/90_2]_s$ glass/polyamide specimens under quasi-static tensile loading for a) specimens fabricated with process A, and b) specimens fabricated with process B.

4 Conclusions

Continuous E-glass/polyamide composite materials were fabricated by two different process cycles, the first in which the laminate was exposed to the peak processing temperature for 10 minutes (process A), and the second without a temperature dwell (process B). For both processing cycles the cooling rate was kept constant at 10 °C/min. It was shown that a faster heating rate along with a reduced peak temperature and reducing holding time at peak temperature notably reduced the

oxidation in the laminates. To further investigate the effect of oxidation on the mechanical properties of glass/polyamide laminates, tensile static mechanical tests were performed on four different laminations including $[0]_8$, $[90]_8$, $[0_2/90_2]_s$, and $[0_4/90_4]_s$. The obtained stress-strain curves were also compared to those of glass/PP with different percentages of MA, and with that of glass/epoxy with different fiber sizings. Static tensile tests showed that glass/polyamide laminates fabricated using process B generally exhibited superior strength and stiffness when compared to the same laminates fabricated by process A, particularly for the $[90]_8$ and cross-ply laminates studied. This is attributed to observed oxidation of the matrix for the process A specimens, resulting in a reduction in the fiber-matrix interfacial strength which was confirmed though SEM micrographs revealing significant fiber/matrix debonding for all laminates studied.

Furthermore, glass/polyamide laminates fabricated using process B exhibited a low transverse strength of 20 MPa, which is less than the transverse strength of similar glass/epoxy laminates, and likely a result of stronger fiber/matrix adhesion for glass/epoxy. The longitudinal strength of nearly 885 MPa was obtained for glass/polyamide laminates, which is comparable to similar glass/epoxy and glass/polypropylene laminates with the same fiber content, demonstrating that the longitudinal strength is mainly affected by the volume fraction of glass fibers and is independent of the type of matrix and different processing conditions. For $[0_2/90_2]_s$ glass/polyamide laminates fabricated by process B, the Young modulus was the same as glass/epoxy with the same fiber content but its strength was 19% higher than that of glass/epoxy. This is attributed to a much earlier onset of transverse ply cracking in glass/epoxy cross-ply laminates when compared to the glass/polyamide cross-ply laminates studied which contain a tougher matrix.

It can be concluded that although the transverse tensile strength of continuous glass/polyamide composites is lower than that of similar glass/epoxy composites, their longitudinal strength is comparable to that of glass/epoxy. More importantly, the tensile strength of glass/polyamide cross-ply laminates is greater than that of similar glass/epoxy laminates. These characteristics suggest that glass/polyamide composites may be suitable replacements for glass/epoxy in many high performance applications. Since high quality glass/epoxy laminates can be efficiently processed, this would be highly beneficial for practical applications since these materials can be recycled at the end of the life.
Although this study has produced notable results supporting the use of glass/polyamide laminates for high performance applications, additional work is required to improve the knowledgebase for this material system. First, multidirectional glass/polyamide laminates should be studied in order to complement the results presented for cross-ply laminates, and understand the influence of damage evolution and ply constraining effects for each particular stacking sequence. Also, the fatigue performance of these materials must be considered, in particular understanding the progressive nature of failure.

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