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THE EFFECT OF TEMPERATURE CHANGES ON TO QUASI-STATIC TENSILE AND FLEXURAL PERFORMANCE OF GLASS FIBRE REINFORCED PA66 COMPOSITES

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ABSTRACT

A significant method of reducing CO_2 emissions in road vehicles is to reduce the vehicle mass. One means in which this can be achieved is to adopt lightweight materials such as thermoplastic composites. Thermoplastics offer advantages in term of weight when compared to conventional steel and aluminium casting. In this study thermal mechanical testing has been conducted on two types of commercial polyamide 66 (PA66) with 35 wt.% short glass fibre reinforcement. One of the materials was impact modified with an elastomer to increase material toughness. Experimental results showed both the reinforced PA66 materials to be temperature dependent. All test results demonstrated the trade-off in the mechanical properties of the two materials especially the impact modified. PA66 with 35 wt.% short glass fibre exhibits the best tensile strength, flexural strength and modulus for each temperature tested. Whereas the impact modified PA66 with 35 wt.% short glass fibre exhibits the higher strain and toughness for each temperature tested.

Keywords: Thermoplastic; Polyamide 66; Short Glass Fibre Reinforced.

1 INTRODUCTION

Recent interest in developing highly fuel efficient vehicles with low emissions has focused efforts toward materials. Materials used for automotive components traditionally consisted of metals in regard to their established use in most engineering industries, better understanding of application and manufacturing processes. As weight reduction is now seen as vital area that automotive industries can now exploit to achieve fuel efficiency and reducing CO_2 emissions (Park et al. 2012). Lightweight polymers have been used to replace metal counter parts over the last few decades from interior trim to external body panels (Araújo et al. 2003, Mouti et al. 2010). Under bonnet fluid reservoirs and tubing is common place for polymers but a need for structural under bonnet components that perform better

and longer at less weight and cost to metal counter parts has opened a growth area for injection-mouldable fibre-reinforced thermoplastics. Among the most challenging of under bonnet applications is the engines oil pan. As it is mounted low on the undercarriage where it is subject to stones and gravel kicked up by tires. Most oil pans have a structural requirement needing fairly complex internal and external geometries, which in metals can translate to heavy multi-piece assemblies produced in numerous manufacturing steps. Work has already been done to optimised material properties for an oil pan using polyamide 66 (PA66) 35 wt.% short glass-reinforced with an elastomer modifier to improve impact resistance (Mouti 2012, Mouti et al. 2010). But the toughness of PA materials are temperature dependent and characteristic of brittle tough transition temperatures have been identified of several PA6 compounds containing glass fibres (Araújo et al. 2003, Kroll et al. 2013). The next step is to understand the effects of different temperatures conditions that the polyamide 66 material systems need to cope with for a range under the bonnet applications that metals have yet to be replaced. Therefore this study aims to investigate the properties of two PA66 35 wt.% short glass fibre reinforced materials one of which has been impact modified with an elastomer. The test strategy in this work is to use temperature as the variable to determine effects of different thermal conditions of each material at an experimental level. The results can then be used to develop new material grades to address deficiency's based on application requirements.

2 EXPERIMENTAL

2.1 Materials and Sample Manufacturing

Two commercial grades of polyamide 66 with 35 wt. % of discontinuous glass fibre, Ultramid® A3HG7 and Ultramid® A3ZG7, (henceforth denoted as Material A and B respectively) were supplied by BASF. Material B was elastomer toughened and has been blended as co-polymer into the base PA66 material full details of this process are kept as a trade secret. The rubber toughening is also kept as a trade secret by BASF although using literature three elastomers have been narrowed down that can be compounded with polyamide and are cost effective which are Acrylonitrile Butadiene Styrene (ABS), Ethylene Propylene Rubber (EPR) and Ethylene Propylene Diene Monomer (EPDM) (Laura et al. 2000; Karayannidis et al. 2002; Ren et al. 2009; Wong et al. 2002). The graft copolymers produced aids dispersion, creating separate phases in the solid enhancing interfacial adhesion. The glass fibres have an average length (l) of 300 µm and diameter (d) of 13 µm, thus yielding an average aspect ratio (l/d) of 23. Test samples where manufactured using injection moulding to suit ISO standard 527 (tensile) and 178 (flexural) for shape and size. **Figure 1**a illustrates specimens used for the testing with attached retro-reflective tape. Flexural test specimens where fabricated by injection moulding which complied with ISO 178 for shape and size, see **Figure 1**b for specimens used in the testing. All test samples in this study come from the same batch of material.

Figure 1: Sample types used: a) tensile dumbbell; b) flexural bar

2.2 Tensile and Flexure Tests

Tensile testing was conducted following ISO standard 527, testing speed for all specimens was 1 mm/min and sample shape was type 1A and the samples were tested unconditioned. To gain accurate strain data for tensile testing retro-reflectors where attached to the specimens 50 mm apart ±2 mm to suit the specimen type 1A gauge length in ISO 527. The flexure testing was conducted following ISO standard 178 and all specimens were tested at 2 mm/min with a size of 80x10x4mm. Testing conditioning was done using different temperatures as follows 23°C, 65°C, 90°C and 120°C. Temperature selection: The typical standard air temperature of 23°C in ISO 291 is the most common test temperature conducted which allows other work to be compared and can represent the temperature of an engine before it has started on a normal day. 65°C was selected as a middle ground

between 23°C and 90°C. 90°C was selected based on a typical oil operating temperature. An IC engine runs around 90°C to 100°C which is based on SAE J300 standards test Kinematic Viscosity of engine oil at 100°C. Also the temperature of the oil in a big end bearing is around 100°C which is the most highly stressed part of an ordinary car engine. 90°C was selected as a nominal for high oil film temperature that materials should be tested to as oil will start to cool once it has passed from the top to the bottom of the engine. 120°C was selected in a case of engine overheating and if accumulative temperatures were to build up. Each temperature was checked using a K-type thermocouple on the specimen surface before starting each test. Testing was conducted on an Instron 5500R screw driven universal test machine using a 30 kN load cell and a laser extensometer for tensile testing.

3 RESULTS AND DISCUSSION

Tensile test data was captured from each material tested. Tensile test data was then used to plot stress strain relations. Data captured from the universal testing machine and the laser extensometer was plotted in **Figure** showing the relationship between stress and strain.

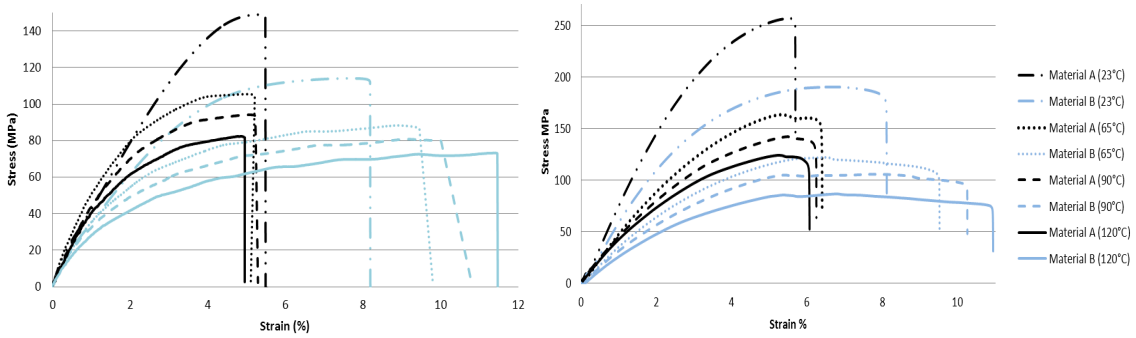

Figure 2: Plotted stress and strain results Left tensile data; Right flexure data.

There is a clear difference on how the elastomer modifier in material B affects toughness by allowing the material to achieve higher strain before failing but loses maximum stress when compared to material A. Results for material A at 23°C are in agreement with the literature (Mouhmid et al. 2006) for PA66/short glass fibre 35 wt.% tested at 20°C. There is complete disagreement when comparing both materials A and B to work done by Mouti under similar conditions. Explanations for some of the discrepancies could have come from the storage of the test specimens which could have degraded from a two year shelf life including thermal degradation (Pielichowski and Njuguna 2005); moisture uptake (Mouti 2012); and strain rate dependency due to difference in testing speeds of 2 mm/min (Mouti 2012) against 1 mm/min in present work. Material A results at 65°C, 90°C and 120°C showed consistency with each other for their curve characteristic with a 10% stress step between each temperature increase when comparing to 23°C. Strain results for all tests done on material A failed with 11% range of 23° to 120°C suggesting material A has good stiffness across a range of different temperatures. Material B's curves share similar shape characteristics with each other with decreases in stress and strain in expansion related to increases in heat. At 65°C, 90°C and 120°C test, the plotted results displays wavy curve. This could relate to the thermal chamber turning on and off to regulate the temperature for the experiment and which could introduce an oscillation effect in the results. With increases of temperature material B becomes more ductile and less stiff with a decrease in tensile strength. Temperature clearly affects properties of the Material A. This can be seen in **Figure** left stress versus strain. 23°C results perform the best out of all the temperatures tested demonstrating better toughness. Temperatures 65°C, 90°C and 120°C show a 28% to 46% in tensile strength reduction to 23°C which means a loss in toughness is affected by the increase of temperature. The results from material A testing do go in line with expectations where an increase in temperature is likely to reduce tensile strength. Load and stress were likely to decrease with an increase in temperature and extension and strain will react less as the material is stiffer without being impact modified. Temperatures also clearly affect the properties of material B as well when looking at **Figure** left stress versus strain. 23°C results perform the best out of all the temperatures tested for

strength but not for extension as this increased the most at 120°C offering about the same level of toughness for each temperature tested. Temperatures 65°C, 90°C and 120°C show a 23% to 36% in tensile strength reduction to 23°C which means a loss in toughness is affected by the increase of temperature. The results from material B testing do go in line with expectations where an increase in temperature is likely to reduce tensile strength. Tensile stress was also likely to decrease with an increase in temperature and extension and strain will increase from the material becoming more ductile. This information also demonstrates the trade-off's in the mechanical properties of the two materials when one of the materials has been impact modified with an elastomer. Flexural test data was captured from each material tested. Flexural test data was then used to plot stress strain relations. Plots showed a clear difference between the two materials tested across the range of temperatures tested. **Figure** right showing the relationship between stress and strain. There is also an obvious stress difference for material A and B tests done between 23°C and 65°C which could be put down to the temperature increase of 42°C between the 23°C and 65°C set of tests. Material A showed little variation to strain from each temperature tested compared to each temperature tested on material B. This can be put down to the elastomer filler which allows material B to strain further when temperature is increased, whereas material A has some stability most likely gained for the addition of glass fibre in a PA66 matrix. Temperature clearly affects properties of the material A. This can be seen in **Figure** right stress versus strain. 23°C results perform the best out of all the temperatures tested demonstrating better stiffness. Temperatures 65°C, 90°C and 120°C shows a 36% to 52% in flexural strength reduction to 23°C which means a loss in toughness and is effected by the increase of temperature. Material stiffness can be considered good as the average strain works out at 6.1% with a deviation of ±0.3%. The results from material A testing does go in line with expectations where an increase in temperature is likely to reduce tensile strength, load and stress were likely to decrease with an increase in temperature and extension and strain will react less as the material is more stiff without being impact modified. Temperatures also clearly affect the properties of material B as well when looking at **Figure** right stress versus strain. 23°C results perform the best out of all the temperatures tested demonstrating better toughness and stiffness. Temperatures 65°C, 90°C and 120°C show a 37% to 46% in flexural strength reduction to 23°C which means a loss in toughness is affected by the increase of temperature. The results from material B testing do go in line with expectations. The expectation was increases of temperature were going to make the material more ductile reducing flexural strength load and stress but increasing extension and strain before failure. Material A shows better strength to material B but has much lower strain values making material A less tough then material B. This can be seen in **Figure** right. The test results also demonstrate the trade-off's in the mechanical properties of the two materials when one of the materials has been impact modified with an elastomer. Using results from (Mouti 2012) flexural testing at 23°C shows much closer similarities for both materials when comparing to this work, unlike the tensile testing. There is some discrepancy between maximum stresses but strain could be considered a lot more similar. A reason for this is that the test speeds are both the same at 2 mm/min.

3.1 Failure Mechanism

The tensile failure mechanism is a single brake with no necking, chipping or strain marks at the brake point. The failure itself can be defined as a brittle fracture in agreement with (Mouhmid et al. 2006) and can be explained by the contribution of the glass fibre as a brittle tough material. Each material and temperature tested showed consistent peek loads for each specimen but both materials show deviation on the extension by ±0.4 mm. The flexure failure mechanism for the bar samples all failed at the mid-point of the 3-point bending test as to be expected. Samples remained intact with a rupture failure on the corresponding surface to the central point which was coursed from a tensile function.

3.2 Morphology Studies

SEM micrographs were taken of the fracture surface of the samples represented from the tensile and flexure tests. The predominant failure mechanisms are fibre pull out and matrix plastic deforming (ductile pulling/tearing). Fibre pull-out, matrix plastic deforming and matrix brittle fracture is evident across all micrographs taken of material A (**Figure 3**a-b). Matrix pull away, fraying, voids and plastic deforming is evident across all micrographs taken of material B (**Figure 3**c-d). Temperature increases

for material A would also appear to affect the extent fibre pull out length. A course for this is poor matrix surface adhesion to the glass fibres as the fibres been pulled out are cleaner at higher temperatures. This could be linked to why flexural strength is reducing for each increase in temperature. This completely differs when an elastomer is used to reinforce a matrix (material B) as at low temperatures there is less adhesion to the fibre surface but increases in temperature sees the matrix holding and maybe pulling out with the fibres together also allowing the material to flex much further before failing. As for material B reduction in mechanical strength which contradicts the statement made for material A it is likely the elastomer in the matrix is becoming more ductile for each temperature increase, reducing its maximum flexural strength so even though the elastomer is offering better fibre adhesion in the matrix material the elastomer is becoming more ductile reducing maximum flexural strength. **Figure 3**a shows little pull out and brittle fracture at lower temperatures ware as **Figure 3**b show longer fibre pull out and matrix plastic deformation of material A at higher temperatures. All micrographs taken of material B shows the matrix having strong bonding to the glass fibres in each test temperature. Voids in the matrix are easily found in material B temperatures at higher temperatures which be seen in **Figure 3** d typical surface found at 90°C and 120°C. This could be linked to the specimen fabrication process as micro defects in the injection moulding process. But the voids could also be present for specimens tested at 23°C and 65°C but are hidden due to matrix plastic deforming but propagated as fraying instead **Figure 3**c typical surface found at 23°C and 65°C. Assuming voids are present in tests conducted at 23°C and 65°C where the fraying does look fibrous which maybe causing its own elastomer fibrous pull out leaving voids from a fibrous ripping function. The fibrous ripping function producing voids which can be summed up as dispersion of soft inclusions in a rigid matrix and as a result of dilatation and elongation processes the post-failure fracture surfaces exhibit holes or cavities where the deformed and ruptured particles have relaxed back into holes larger than the original particle size.

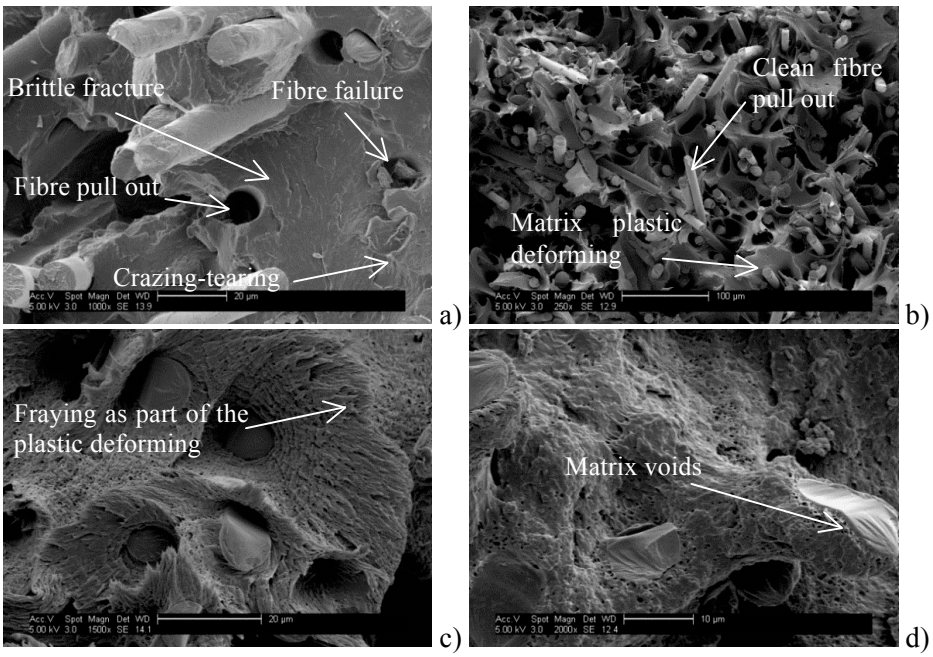

Figure 3: SEM micrographs of the typical fracture surfaces of material A (a-b); material B (c-d)

4 CONCLUSION

In this study, the influence of temperature on quasi-static tensile and flexural properties of 35 wt.% short glass fibre reinforced PA66 have been investigated. The uses of material A would be better suited to structural applications as it demonstrated the least amount of deflection and remained close to the 5.2% strain range for each temperature tested for tensile and a 6% strain range for flexural. Whereas material B strain increased with an increase in temperature as to be expected due the addition of an elastomer, removing some of the stability that glass fibre offered to PA66. This would make material B better suited to applications that requires better impact resistance. Material A

exhibits the best strength and modulus across each temperature tested. Testing conducted at 23°C gained the best toughness properties for both the materials whereas increases in temperature from 23°C up to 120°C reduced toughness properties for material A. Material B would have lost toughness when stress reduces, but levels out as strain increases with every temperature increase so toughness remains almost the same for each temperature increase. The test results also demonstrate the trade-off's in the mechanical properties of the two materials when one of the materials has been impact modified with an elastomer. In this case mechanical strength is reduced for material B but gained much higher strain across all temperatures tested. Clear differences in material properties and plots can be observed in all results. Both materials show the same failure mechanisms for both tensile and flexural studies. SEM micrographs from tensile and flexure testing demonstrated the elastomer in material B had better glass fibre adhesion allowing higher strain and extension which give the material system more toughness and which also improved with each increases in temperature as the elastomer became more effective. The addition of elastomer weakened material B's total strength compared to material A. Material A loses surface adhesion as temperatures were increased shown as long fibre pull out as well as cleaner fibres from pull out. The edition of elastomer effected the plastic deformation on the matrix material as it was more fibrous.

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