

MECHANICAL STUDY ON SURFACE TREATED GLASS FIBRES AFTER THERMAL CONDITIONING

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Abstract

The study reported the mechanical properties of glass fibres conditioned at typical temperatures for thermoplastic processing. Single fibre and bundle tensile tests were used to characterise mechanical performance on water sized (known as unsized) and γ -aminopropylsilane (APS) sized glass fibres thermally treated up to 400°C, respectively. The results from both tests showed that the strength of the silane sized fibres drops significantly when the fibres are heated above 300°C for 15 minutes. On the other hand, unsized fibres exhibited a gradual linear decrease with increasing temperatures. The failure mechanism of the fibres was discussed in this study by further analysing the results in single fibre test using Weibull cumulative function.

1 Introduction

Glass fibres are attractive materials due to their excellent mechanical properties with respect to their production costs. The materials incorporated with various polymeric resins have therefore been widely used in automotive, wind turbine and leisure industries. Due to its tremendous industrial applications, the disposal of end-of-life products becomes a serious issue for decades. With increasing landfill costs, demands for light-weight structured materials, it increases the pressure on manufacturers to pursue the recycling and reuse of glass fibre composites. Various techniques, such as mechanical grinding, thermal, and thermo-chemical degradation processes, have been developed to recycle glass fibre composites [1-5].

Although it has been demonstrated that the reinforced glass fibres can be recovered with small amount of resin remaining on the surface after recycling process, the fibres are subjected to great decreases in strength. For example, the fibre strength degrades 50% compared to pristine one after fluidised-bed process at 450°C and the strength reduces up to 90% at processing temperature of 650°C [4-5]. Due to this, the reapplication of the recycled glass fibre is limited to small amount of replacement for pristine fibres or uses as fillers for composites.

In order to recycle glass fibres as reinforcement for composite system, one of the concerns is to decrease the temperatures during recycling. However, lower processing temperature may introduce low conversion rate of glass fibres recovered and large amount of resin left on the

fibre surface which may influence the interfacial properties of fibres leading to low mechanical performance on the composites. On the other hand, it is reasonable to suspect that the low mechanical performance on recovered glass fibre may be due to damaged or missed silane coating on the surface. It has been well known that silane coating provides protective layer for glass fibre not only to retention of their strength and increase of interfacial properties to polymeric resin but also resistance to potential damage from environment such as moisture. In addition, it has been also suggested that silane coating provides flaw “healing” on the glass fibre surface up to certain level [6]. Therefore, re-silanisation process after recycling may increase the reinforcement level for composite applications.

Prior to regenerating mechanical properties of the recovered glass fibres from silane recoating, it is important to understand the mechanism of the fibres and the coating materials degraded under thermal environment. In this study, we present initial results of unsized and γ -aminopropylsilane (APS) sized glass fibres from single and bundle fibre tests. The origin of fibre failure, internally from fibre or externally from silane coating, was discussed from the Weibull analysis on the single fibre testing results.

2 Experiments

2.1 Materials

The glass fibre rovings used in this study were boron-free glass supplied from Owens Corning Vetrotex. The rovings were produced using conventional, but pilot scale, bushing process. For water sized fibres, only water spray has been applied to the fibre surface once the fibres have been produced. These samples will be denoted as “unsized” fibres at followings of the study. As to sized sample, 1 wt.% of γ -aminopropylsilane (APS) was firstly hydrolysed in distilled water and the glass fibres produced from melt were then coated with the hydrolysed silane solution using industrial rotating cylinder sizing applicator. Subsequently, both of the unsized and sized samples were dried at 105°C for 24 hours.

2.2 Heat treatment process

The as-received samples were heat treated using Carbolite LHT6 high temperature oven. Prior to the heat treatment process, the glass fibre strands, with 300 mm in length, were first suspended using customised holding grips in order to isolate the sample from any physical contact which may result in fibre damage. Both of the gripped fibres were heat treated simultaneously in the same oven in order to be consistent with the identical thermal history. The oven was firstly stabilised for 2 hours before inserting samples. A total of 10 minutes was allowed to reach set temperature before carrying out thermal treating the sample for 15 minutes. Apart from room temperature, 3 temperatures of 300, 350 and 400°C were performed, respectively, to study the heating effect on the unsized and sized glass fibres.

2.3 Tensile tests

2.3.1 Fibre bundle test

The sample preparation and tensile methods for glass fibre strand follow ASTM 578 [7] and ASTM 2256, respectively [8]. Araldite Rapid epoxy was firstly applied on the fibre strand for tabbing purpose. The sample was then carefully clamped in two aluminum grips, with gauge length of 250 mm, mounted in Instron 3342 tensile testing equipment. The load was measured using a 1 kN load cell and the displacement were recorded using a linear potentiometer. The

cross head speed for the test is 3.75 mm/min which gives 1.5% strain/min. It should be addressed that the samples broke at the distance longer than 3 mm away from aluminum grips are accountable for successful test in this study. The result shows the average of 10 successful samples for each thermal condition.

2.3.2 Single Fibre tensile test

The method for single fibre testing is according to ASTM C1557-03 [9]. The samples were firstly separated from a fibre bundle with extra care to minimise any further physical damage to the fibre. The isolated single fibre was centred in a window card and secured by applying Locite Gel superglue at the ends the window frame. The test was carried out using the same tensile testing equipment but different clamping system and a load cell of 10 N. Once the sample was mounted on the grips the edge of the window card was carefully cut away before undergoing the test. In our study, each type of thermal treated fibres was tested using various gauge lengths of 10, 20 and 40 mm, respectively. The cross head speed was adjusted following different gauge lengths in order to give identical stain rate of 1.5%/min. The fibre diameter was measured using Nikon Epiphot Inverted optical microscopy (OM). The diameter was averaged from 5 areas on the same fibre and the average value was used to determine the mechanical properties of the fibre. The mechanical results showed in this study are the average values of 70 – 100 specimens for each type of fibres (different gauge lengths and thermal conditioned fibres). The error bars showed in this study indicate 95% confidence limits on the average values. In addition, the bundle and single fibre tests were carried out at room temperature with 50% relative humidity to eliminate environmental factors to the mechanical results obtained.

3 Results and discussion

3.1 Fibre bundle test

Typical load-extension curve from fibre bundle tests are shown in Figure 1. It can be clearly seen from Figure 1 (a) that the mechanical performance of silane coated fibres increases about 4 times than unsized ones. The observation is in a good agreement with the findings from Zinck et al. [6, 10] which indicates that silane coating could act as “healing” agent to recover flaws on fibre surface. After post-heat treatment on the fibres, the fibre strength decreases with increasing temperatures as the load-extension curve shown in Figure 1 (b).

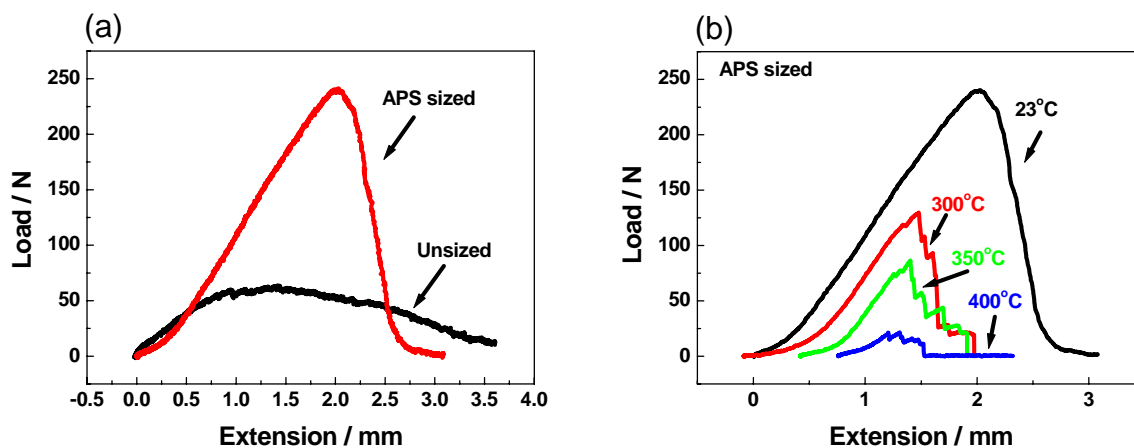


Figure 1. Load-extension curves from fibre bundle test for (a) unsized and sized fibres (b) sized fibres at various heat treatment temperatures. The x-axis values in Figure (b) are offset for clarity.

In addition to the Figure 1 (b), the initial part for each curve seems to be similar. However, as the load increases, the jagged shape is observed apart from the results at room temperature and the behaviour is repeatedly obtained after the maximum load. The jagged shape observed before maximum bundle strength is an indication of small amount of fibre breaking at early stage. Subsequently, majority of fibres breaks at maximum load and the fibre-fibre interfacial fraction and breakage of remaining intact fibres are then taking place until the sample fails completely. It is interesting to note that the jagged behaviour is more apparent with increasing temperature.

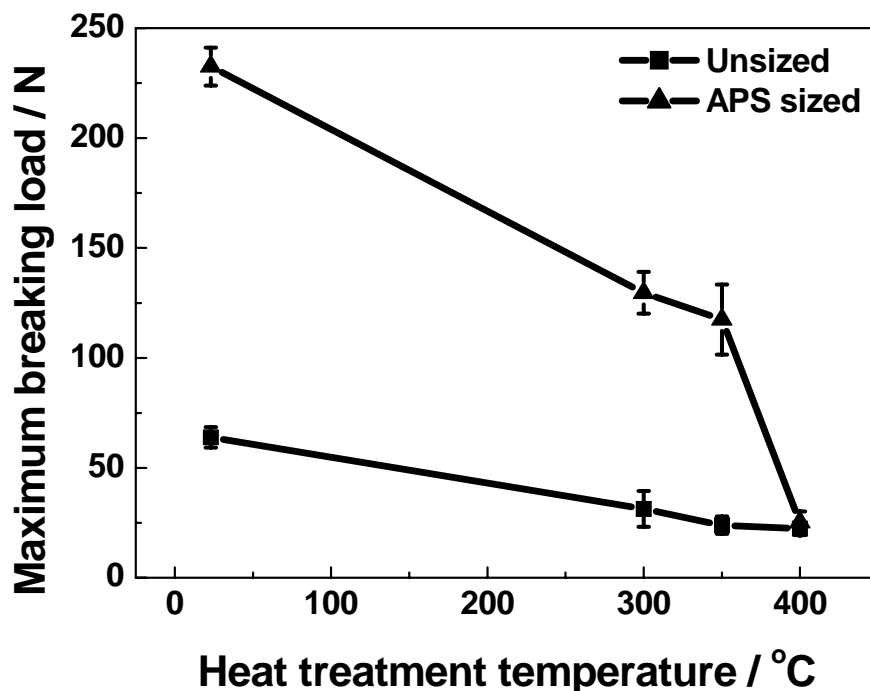


Figure 2. Fibre bundle testing result of unsized and sized fibres under various heat treatment temperatures

Figure 2 shows the average value of maximum bundle strength as function of temperatures for unsized and sized fibres. It can be seen from the figure that for unsized fibres the bundle strength shows linear dependence on heating temperatures. In contrast to sized fibres, a threshold is found at 350°C where the bundle strength drops significantly with increasing temperature. For both of the fibres, the bundle strength reduces over half of original strength at 400°C for 15 minutes.

3.2 Single fibre test

The single fibre strength (at 20 mm gauge length) with various heat treatment temperatures for unsized and sized fibres was shown in Figure 3 (a). The results are similar to the one from bundle test that thermal treatment leads to significantly strength reduction for both of unsized and sized fibres. However, a threshold temperature was found at 300°C for sized sample which is slightly lower than the one in bundle testing. In addition, it is interesting to note that a large deduction in strength is observed at lower temperature region for bundle test. Figure 3 (b) shows the results tested using various gauge lengths. It appears that the average fibre strength varies with gauge length used for single fibre testing. As to the post-heat treatment,

tensile testing results at 10 and 40 mm gauge lengths exhibit similar trends to 20 mm which a threshold temperature accompanying with large reduction in fibre strength observed above 300°C.

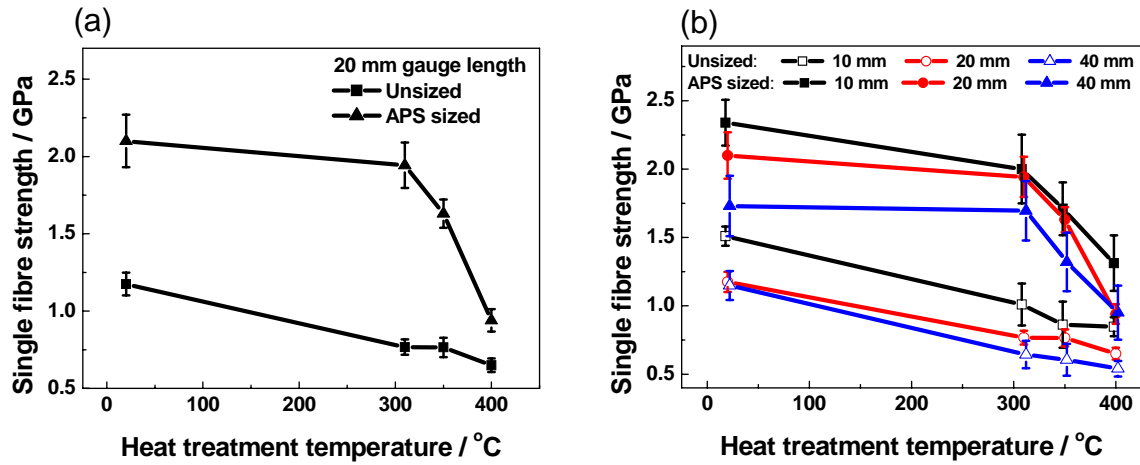


Figure 3. Single fibre strength as a function of heat treatment temperatures for testing gauge lengths of (a) 20 mm and (b) 10, 20 and 40 mm

3.3 Weibull analysis for single fibre testing results

The results from single fibre test at 20 mm gauge length are further investigated using Weibull cumulative distribution function [11] in order to assess single fibre failure mechanism. The three parameter Weibull distribution is given by following equation:

$$P = 1 - \exp \left[-L \left(\frac{\sigma - \sigma_u}{\sigma_0} \right)^m \right] \quad (1)$$

where P is the cumulative probability of failure of a fibre of length L at apply stress σ . In our case, L is equal to gauge length of 20 mm. m is a shape parameter which is known as Weibull modulus. σ_0 and σ_u is a scaling parameter and a threshold stress below which the failure probability is zero.

Equation (1) is established based on an assumption single flaw population, unimodel function. It is recommended that σ_u is 0 for brittle materials [12] so that rewriting Equation (1) gives:

$$\ln \left[\ln \left(\frac{1}{1-P} \right) \right] = m \ln(\sigma) + (\ln L - m \ln \sigma_0) \quad (2)$$

Weibull plots according to Equation (2) for unsize and size fibres under heat treatment were shown in Figure 4, respectively. It should be addressed that the probability of failure, P_i , for the i^{th} strength is calculated using the following equation:

$$P_i = \frac{i-0.5}{N} \quad (3)$$

where N is the sample number tested for the same condition (silane coated, thermally treated).

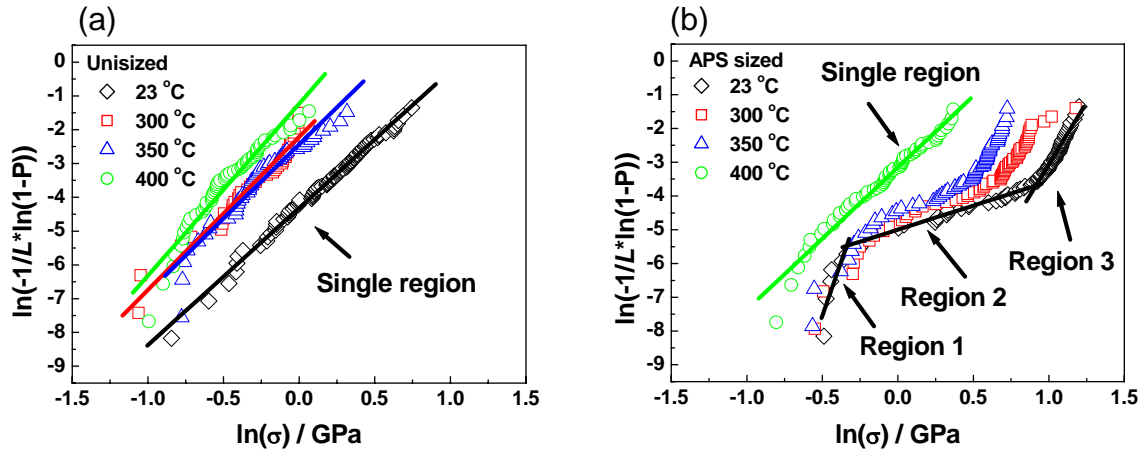


Figure 4. Weibull distribution for (a) un-sized glass fibres and (b) sized fibres under various thermal treatment temperatures

Temperatures	23°C	300°C	350°C	400°C
Un-sized fibres				
m	4.06	4.53	4.39	5.09
$\ln(\sigma_0)$	1.06	0.49	0.56	0.24
R^2	0.99	0.98	0.96	0.97
APS sized fibres				
m	-	-	-	4.22
$\ln(\sigma_0)$	-	-	-	0.75
R^2	-	-	-	0.99
m_1	12.83	5.67	6.21	-
$\ln(\sigma_{01})$	0.09	0.77	0.61	-
R^2	0.86	0.96	0.97	-
m_2	1.43	1.71	1.82	-
$\ln(\sigma_{02})$	3.51	2.75	2.46	-
R^2	0.99	0.99	0.98	-
m_3	7.34	5.05	7.37	-
$\ln(\sigma_{03})$	1.42	1.34	0.97	-
R^2	0.98	0.96	0.98	-

Table 1. Weibull parameters analysed from the single fibre testing results for un-sized and sized fibres with respect to post-heat treatment temperatures. The un-sized fibres and sized fibre at 400°C were linear fitted using unimodal Weibull function. The subscripts 1, 2 and 3 represent the linear fitting results in region 1, 2 and 3, respectively, for sized fibres at 23, 300 and 350°C as shown in Figure 4 (b).

In general, for un-sized fibres, a linear dependence is observed throughout whole set of data which indicates that the fibres are failed with one flaw population. The linear relationship and the slopes obtained are slightly up-shifted with increasing temperatures which means that the tensile strength of the fibres decreases at higher heat treating temperatures being employed. In terms of sized fibres, the results exhibit three distinct linear regions (Figure 4 (b)) which may be described the multimodal Weibull distribution. The failure of the fibre may be categorised into three flaw populations or in a combination of two. As heating temperature increases, the three linear relationship turns towards one single linear region for sized fibres at 400°C which can be described by unimodal Weibull distribution used for un-sized fibres. The behaviour may imply that the fibres reach a transition point at above 350°C leading to the observation.

The origin of the behaviour is unclear up to best of our knowledge at this stage. However, it may be due to the thermal degradation of silane coating.

The results for both the fibres were fitted using least squares method and were listed in table 1 with R^2 value provided. The Weibull modulus for unsized fibres is about 4 – 5 which falls in a typical range found in literature. In addition, intercept ($\ln(\sigma_0)$), which could represent the tensile strength of the fibre, decreases with the increase of temperature. From previous study on qualitative investigation on fibre strength using Weibull distribution [6], the failure could be attribute to internal flaw on the fibre surface. Furthermore, it is interesting to note from the result of sized fibres at 400°C that the Weibull modulus and the scaling parameter are falling in similar range to unsized fibre from 23 to 400°C. It may imply that the fibres (sized, heat-treated at 400°C) fails the same type as for unsized fibres which could be an evidence that the silane coating is no longer supporting the flaw recovery on fibre surface.

In addition to sized fibres at 23, 300 and 350°C, the results in region 1 and 3 are similar apart from for sized fibre at 23°C which may be resulted from the low density of data in region 1. However, the results in region 1 overlapped at these three heating temperature indicates that the fibres fails from severe flaw on surface so low tensile strength is obtained. Further investigating results in region 2 and 3 and following previous study [6] on bimodal Weibull analysis, it may be reasonable to suspect that the failure type for region 3 is due to internal flaw as the intermediate region 2 is introduced after silane coating is applied to the fibres.

4 Conclusions

Mechanical properties of the unsized and the sized fibres at various heating temperature was carried out using single and bundle fibre tests. The results in both tests showed that the unsized fibre exhibits an approximate linear decrease with increasing temperature. A threshold temperature of around 300°C was found for sized fibres that the strength reduces significantly with increasing temperatures. The behaviour was double confirmed by the single fibre tests at various gauge lengths. From Weibull analysis on the single fibre testing results, the failure mechanism was investigated. Single linear region was found for unsized fibres whereas multi linear region was observed for sized fibre apart from the one at 400°C. Assignment of the linear regions associated with the fibre failure mechanism (internally or externally) was discussed in this study.

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