RECYCLED BASALT FIBRES: FRACTURE TOUGHNESS EVALUATION AND STRENGTH REGENERATION

J. Tirillò[1], F. Sarasini[1], L. Di Fausto[1], C. Gonzalez[2], A. Fernandez[2], C.S. Lopes[2]

[1] Department of Chemical Engineering Materials Environment, Sapienza – Università di Roma and UdR INSTM, Via Eudossiana 18, 00184 Rome, Italy

[2]IMDEA Materials, C/Eric Kandel 2, 28906 - Getafe, Madrid, Spain

Keywords: Basalt fibres, Mechanical properties, Recycling, Surface treatments, Fracture toughness

Abstract

Basalt fibre is characterized by excellent sound insulation properties, thermal resistance, good chemical inertia and Its role in replacing glass as reinforcement in polymer matrices has been confirmed over the past years For basalt fibres, a severe loss in strength after thermal treatment has been reported, thus preventing their re-use in structural composite materials at the end of a recycling process. Studies on glass fibres have recently confirmed the possibility to restore the strength of thermally damaged filaments by means of chemical treatments. The present work investigates how chemical treatments can be used to regenerate the strength of thermally degraded basalt fibres. In an attempt to shed light on the mechanisms controlling basalt fibre strength loss, FIB milling has been used to determine the fracture toughness of single basalt fibres. This evaluation, never reported previously on basalt fibres, is important to assess any changes occurring in the flaw-strength relationship caused by thermal exposure. To regenerate the fibre strength, the thermally conditioned fibres were immersed in a NaOH and in a HF solution. The fracture stress of the notched fibres was determined using the single filament test to determine whether the mode I fracture toughness changed due to heat treatment.

1. Introduction

Thermal processes, among all the recycling techniques, are used on an industrial scale as they allow the glass fibres recovery [1]. However, after the thermal treatment process, tensile strength of the single fibre quickly decreases [2], and fibres are quite damaged, not being suitable for a new structural composite [3]. The use of mineral fibres, such as basalt, is a valid option for the reduction of the environmental impact of composite materials. Basalt fibres can be successfully applied in reinforced composite materials instead of glass fibres [4,5]. As already shown in a previous paper [6], also basalt fibres exhibit a significant decrease of the mechanical properties after thermal exposure, limiting the competitiveness with pristine fibres after the typical thermal recycling process of composite materials. Recent studies have demonstrated the remarkable increase of glass fibres tensile strength by a post treatment in acid [7] or alkaline [8] solution. In this framework, the aim of the present experimental work is to investigate the effect of two different chemical treatments on mechanical properties of basalt fibres in order to confirm the possibility of recovering the previously lost strength and obtain fibres ready for a new use. These treatments can in principle allow to obtain a closed-loop recycling process, resulting in a considerable reduction of the CO_2 produced [9]. Chemical treatments were carried out on the fibres that had been previously heat treated at three temperatures, namely 400°C, 500°C and 600°C. It was possible to determine the best chemical treatment condition able to regenerate the basalt fibres tensile strength, in terms of: chemical agent, concentration and treatment time. At the same time, fracture toughness (K_{Ic}) of the as-received, heat-treated and regenerated basalt fibres was analyzed to better understand the phenomena controlling basalt strength behavior after different treatments. This fundamental property has not yet been widely studied in literature due to the great difficulty in carrying out mechanical tests on small-diameter samples, such as fibres. An empirical method, used in the past to estimate K_{Ic}, involves the determination of the initial crack size [10]. The size of initial flaws can be estimated to obtain a relationship between the fibres fracture toughness and the radius of the mirror area on the fracture surface [11]. To avoid a large scatter of fracture toughness values owing to this

method, the use of focused ion beam technique (FIB) allows to introduce a known-size artificial defect in the fibre plane, to more accurately estimate the fracture toughness of the fibres. This methodology has already been used for carbon [12,13,14], silicon carbide [11], Kevlar [12] and glass fibres [12,15]. Fracture properties of these fibres have already been discussed in literature, but no other types have been investigated with this technique so far. For the first time, focused ion beam milling is used to create a nano/micro-sized deep notch in the perpendicular plane to the drawing direction of the basalt fibre. Fibre fracture toughness was calculated based on the residual strength value obtained by tensile test, assuming linear elastic fracture mechanics (LEFM), as a correct approximation of the brittle materials behavior, as previously done both on carbon and glass fibres. In addition, the fracture behaviour of heat-treated and regenerated basalt fibres containing a single nano/micro-sized notch was evaluated. This investigation can help in clarifying the mechanisms responsible of the loss of fibre strength, between the two main mechanisms proposed: (i) thermally-activated changes to the anisotropic silica network structure initially created by the high drawing stress during fibre manufacture [16,17,18] and (ii) thermally-induced diffusion of water into the fibre surface leading to increased surface area and larger micropores [19,20,21]. Hypotheses on the mechanisms able to cause at least a partial regeneration of the fibres mechanical properties were formulated on the basis of the results of tensile tests on chemically-treated notched fibres. A correlation with the conclusions obtained from SEM analysis of lateral and fracture surfaces of fibres is introduced in the discussion of the results.

2. Materials and methods

2.1 Raw Materials

A continuous roving of non-impregnated basalt fibres supplied by Kamenny Vek with a nominal fibre diameter of 13µm, linear density of 1200 tex and sized with a commercial sizing compatible with epoxy resin, was used in this work.

2.2 Fibre heat treatment

A tube furnace (Lenton Thermal Designs Ltd) was used to heat-treat as-received fibre bundles. Thermal treatments were carried out at 400°C, 500°C and 600°C in air for 25 minutes and then fibres were cooled at room temperature .

2.3 Fibre chemical treatment

The thermally treated basalt fibres were immersed in a solution of sodium hydroxide (NaOH), or hydrofluoric acid (HF). Different types of chemical treatments have been carried out, modifying the chemical agent, the concentration or the immersion time of the treatment, for each temperature.
NaOH - The heat-treated fibre bundles weighing about 0.1g, were subjected to a chemical treatment with sodium hydroxide. To recover the mechanical properties, these bundles were immersed in NaOH solutions at different concentrations (1.5-3M) for 5 or 10 minutes, always at 90 ± 5 °C. All the bundles were drained and rinsed for 1 minute in 150 ml of 37 w% hydrochloric acid (HCl), to remove residual deposits on the fibres surface.
HF - The heat-treated fibres were immersed in 1v% HF aqueous solution for 2.5 minutes at room temperature.
Finally, all the fibre bundles were rinsed 3 times for 30s in 150ml of deionized water. Once the chemical treatment was completed, the NaOH chemically-treated bundles were allowed to dry for 6 hours at 105 ± 5 °C, while the HF chemically-treated bundles were dried in a furnace for 20 minutes at 105 ± 5 °C.

2.4 Single fibre tensile testing

A Zwick/Roell Z010 machine equipped with a 100 N load cell was used to perform tensile tests at room

temperature. The experiments were carried out under displacement control at a cross-head speed of 2 mm/min. At least 60 fibres for each type were glued onto cardboards with a cut-out central window to match the gauge length. The average diameter for each fibre was obtained by measuring the diameter in six points along the gauge length using a Nikon Eclipse 150L optical microscope equipped with Lucia Measurement image analysis software. By subtracting the displacement associated to the system compliance from the total cross-head displacement, it was possible to determine the effective specimen elongation in the gauge length. The system compliance was measured according to ASTM C1557 [22] by obtaining the force versus displacement behaviour of as-received fibres at three-gauge lengths, namely 20,30 and 40 mm. The scatter of the tensile properties was statistically explored using a two-parameter Weibull distribution, according to the expression (equation 1):

$$F(x) = 1 - exp\left[-\left(\frac{x}{x_0}\right)^{\alpha_x}\right] \quad (1)$$

where $F(x)$ is the probability of failure of the parameter x, α_x is the Weibull modulus and x_0 represents a scale parameter. The probability of failure was estimated using equation 2:

$$F_j = \frac{j-0.5}{N} \quad (2)$$

where N is the number of tested fibres and j is the rank of the jth data point [23]. A single set of parameters for the tensile strength property, x_0 and α_x was obtained from the intercept and slope, respectively, of the plot $ln\{ln[1/(1- F(x))]\}$ against $ln(x)$. Moreover, at least 10 virgin, heat-treated and regenerated fibres, containing an artificial notch, were subjected to tensile tests. The equipment Favimat+, supplied by Textechno Textile Testing Technology, was used to perform the single fibre tensile test working with a cross-head speed control of 2 mm/min. During the FIB procedure, it was possible measure the diameter of each fibre from SEM images.

2.5 Focused ion beam (FIB) milling and fracture toughness test

A FEI Helios NanoLabTM DualBeamTM 600i system equipped with a FIB was used to introduce an artificial notch on a plane perpendicular to the single fibre axis, by means of a scanning electron microscope equipped with the focused ion beam (FIB). A cardboard able to hold up to 5 fibres was connected to a metal support using a copper tape to provide the appropriate electrical paths during the fibre milling operation. The appropriate beam current was selected to limit microstructural changes due to ion impact during milling operations. Straight and sharp notches perpendicular to the fibre axis were introduced into the fibres with a depth (a_0)/diameter (D) ratio of approximately 0.2. The notched fibres were then subjected to a tensile test up to failure using the experimental set-up previously described.

2.6 Scanning electron microscopy (SEM)

The lateral surfaces of all the fibre types were examined in order to understand if any superficial changes occurred after the thermal and chemical treatments. Furthermore, all the fracture surfaces of notched fibres were observed to confirm the position of the fracture and verify the quality of the notch. All samples were analysed using an high resolution field emission scanning electron microscope (Zeiss Auriga).

3. Results and discussion

Mechanical tests on heat-treated fibres in the temperature range commonly used during the second stage of the thermal recycling process of a fibre-reinforced composite material allowed to establish the effect of temperature on the mechanical properties of basalt fibres. The results on untreated and thermally conditioned fibres show a considerable tensile strength loss with increasing temperature. From Table 1, it is evident that basalt fibres heated from room temperature to 400°C undergo a strength loss of ~50%, and up to ~75% at the maximum temperature investigated (600°C), as confirmed in a previous study [6]. By accounting for the similarity between basalt and glass fibres, it is reasonable to suppose that the tensile strength drop mechanism will be similar [5,24]. High temperature thermal treatments remove the fibre sizing causing exposure of surface flaws [25]. The phenomenon of sizing degradation cannot be solely responsible for the

strength drop of the fibres. In addition, an oxidation reaction of Fe^{2+} to Fe^{3+} ions is established to occur at high temperature, which is able to reduce the fibre strength. Moreover, the action carried out by the diffusion of the water present in the environment during the thermal treatment is able to reduce the static fatigue life of the silica glass [21]. Thermally-induced water diffusion from the fibre surface to the bulk leads to the increase in number and size of pores on the fibre surface [20].

Temperature [°C]	Diameter [μm]	Tensile strength [MPa]	Young's modulus [GPa]	Strain at failure [%]
R.T.	12.8 (0.68)	2514.85 (276.67)	81.13 (6.24)	2.41 (0.32)
400	11.02 (1.20)	1304.77 (304.96)	104.20 (5.43)	1.24 (0.28)
500	11.88 (1.74)	1056.67 (305.80)	104.16 (4.59)	1.01 (0.30)
600	11.83 (1.66)	663.96 (166.22)	107.37 (4.28)	0.65 (0.20)

Table 1. Tensile tests data for untreated and thermally-treated basalt fibres.

The mechanisms responsible for the fibre strength loss are ascribed to superficial phenomena, but studies related to hyperquenched glass fibres, suggested to take into account also relaxation mechanisms of bulk fibre [26]. Relaxation involves both the local structural rearrangements and the change in the orientation of the flaws towards a random state with a much greater probability of leading to a catastrophic failure [6]. The hyperquenched fibres, subjected to a thermal treatment close to the T_g, are subjected to structural relaxation phenomena of two types: (i) enthalpy relaxation and (ii) anisotropic relaxation owing to the long-range movement of silica networks [18]. The fracture surfaces of the untreated and thermally treated fibres appear to be the same under SEM analysis. Surface pattern presents the 3 typical regions: mirror, mist and hackle, as typically identified for brittle fibres [27]. The previous results seem to indicate that the increase in temperature does not involve a change in the nature of the flaws, thus the increasing number of the surface flaws is the mechanism controlling the strength loss in basalt fibres At the same time the results in Table 1 show that another effect of temperature is the increase of the Young's modulus of basalt fibres. This thermal compaction phenomenon, also present in glass fibres [27,28], corroborates the assumption that the heat treatment induces structural changes in the basalt fibres. The effect of thermal recycling process on fracture toughness, evaluated by means of tensile tests on notched fibres, can help in clarifying if surface flaws are the controlling mechanism for the strength decrease or if structural relaxation plays the crucial role. As previously mentioned, focused ion beam milling was used to introduce artificial nano/micro-sized defects (0.8-2 μm) on single fibres (Fig.1).

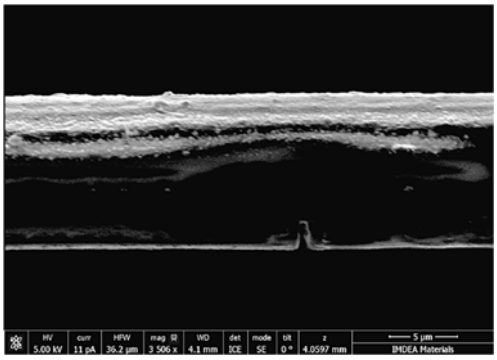

Figure 1. SEM detail of an artificial notch introduced using focused ion beam milling (FIB), on heat-treated basalt fibres.

Fracture toughness and residual strength (σ_f) of basalt fibres have never been investigated up to now. The apparent fracture toughness is expressed in terms of mode I fracture toughness according to the equation 3:

$$K_{Ic} = Y[a/D] \cdot \sigma_f \sqrt{\pi a} \quad (3)$$

where "a" represents the notch depth. The stress intensity factor (SIF) or geometric factor "Y" was determined for each fibre by Valiente's equation, as previously done in literature for surface cracks in round bars subjected to tension loading [30]. This equation is based on the hypothesis that a straight-fronted edge crack is introduced in a cylinder subjected to uniaxial tension along its axis. As can be seen from the examination of the fibres fracture surface (Fig. 2), the formation of a linear fracture front originated from the artificial flaw confirms the fundamental hypothesis of the Valiente's equation.

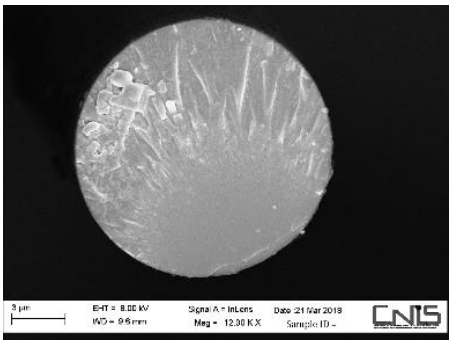

Figure 2. Fracture surface where an artificially notch was introduced by FIB.

The mode I fracture toughness for untreated fibres was found to be equal to 0.94 ± 0.24 MPa·m$^{0.5}$. A higher value of fracture toughness was been obtained from tensile tests of thermally treated fibres, K_{Ic}=1.65±0.52 MPa·m$^{0.5}$.
The results from the tensile tests reported in Table 2 must be evaluated underlining that the geometric factor and the fracture toughness are a function of the ratio "notch depth/fibre diameter" (a/D).

Temperature [°C]	Chemical treatment	a/D	SIF - Y	Residual strength [MPa]	Fracture toughness [MPa·m$^{0.5}$]
R.T.	-	0.15 (0.06)	1.28 (0.16)	338.15 (157.91)	0.94 (0.24)
600	-	0.13 (0.05)	1.30 (0.17)	582.58 (226.24)	1.65 (0.52)
600	NaOH 1.5M 5min	0.10 (0.04)	1.26 (0.03)	893.86 (166.26)	2.14 (0.57)
600	NaOH 3M 10min	0.13 (0.04)	1.24 (0.02)	984.89 (295.63)	2.53 (0.59)
600	HF 1%v. 2.5min	0.16 (0.03)	1.24 (0.02)	991.16 (315.94)	2.88 (0.53)

Table 2. Fracture toughness evaluations of untreated, heat-treated and regenerated basalt fibres, notched using the focused ion beam technique (FIB.)

The results reported by Feih et al. [15] on glass fibres fracture properties seem not to be suitable to basalt fibres. The data of untreated and heat-treated fibres, arranged in the graph ($\sigma_f \cdot Y(a/D)$ versus $(\pi a)^{-1/2}$) are not in agreement with a single linear regression line to guarantee a sufficient correlation ratio (R^2). The impossibility of pursuing this approach confirms that the theory expressed for glass fibres cannot be extended to basalt fibres. The increase in fracture toughness corroborates the hypothesis that structural changes occur and this supports the idea that the strength decrease is not due solely to a superficial degradation. As demonstrated by Bashir et al. [31] in their studies on glass fibres, the results for the basalt fibre strength recovery obtained after a treatment with hot NaOH are noteworthy, with a strength regeneration after exposure to all three temperatures. The recovery of the thermally treated fibres is remarkable: 95% of the strength loss after the heat-treatment at 400°C can be recovered with the more aggressive NaOH treatment (3M, 10 min). The ability to regenerate the fibre strength decreases with the increase of the conditioning temperature. Chemical treatments on heat-treated fibres based on an acid

solution (HF) seem to show a lower tendency to recover the strength of basalt fibres, but this could be indicative of the fact that 2.5 min may not be enough to regenerate fibre properties (Fig.4).

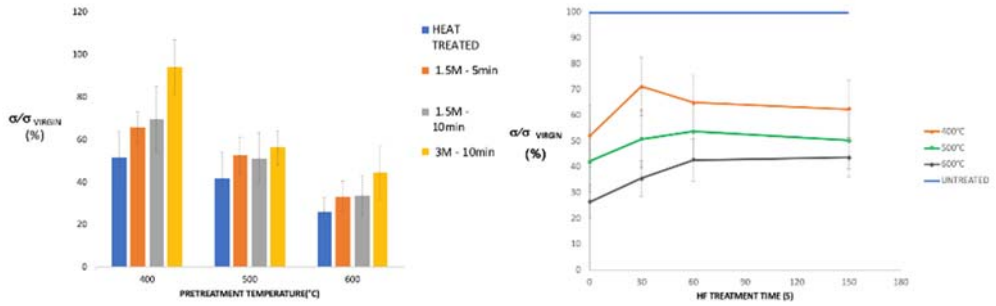

Figure 4. Influence of the (left) NaOH- and (right) HF-based chemical treatments on heat-treated basalt fibre strength.

The reasons responsible of the increase in fibre strength after the chemical treatments have not been addressed in detail in this work, but it is possible to refer to previous studies on glass fibres. In fact, glass fibres strength increase obtained after treatment with hydrofluoric acid or alkaline solutions, seems to depend on superficial dissolution (chemical etching) or bulk structural changes in heat-conditioned fibres. It is known that the aqueous solution of HF can dissolve glass and it has been widely used to remove the damaged surface through wet HF-etching and in turn increase glass strength. This implies that HF-treatment does not just simply remove the material from the surface but also significantly change the characteristics of existing cracks on etched surface [32]. The same mechanism could also be proposed in the case of basalt fibre, as the SEM analysis shows superficial change compared to heat-treated fibres, with a major homogeneity of the lateral surface, as shown in Fig. 6.

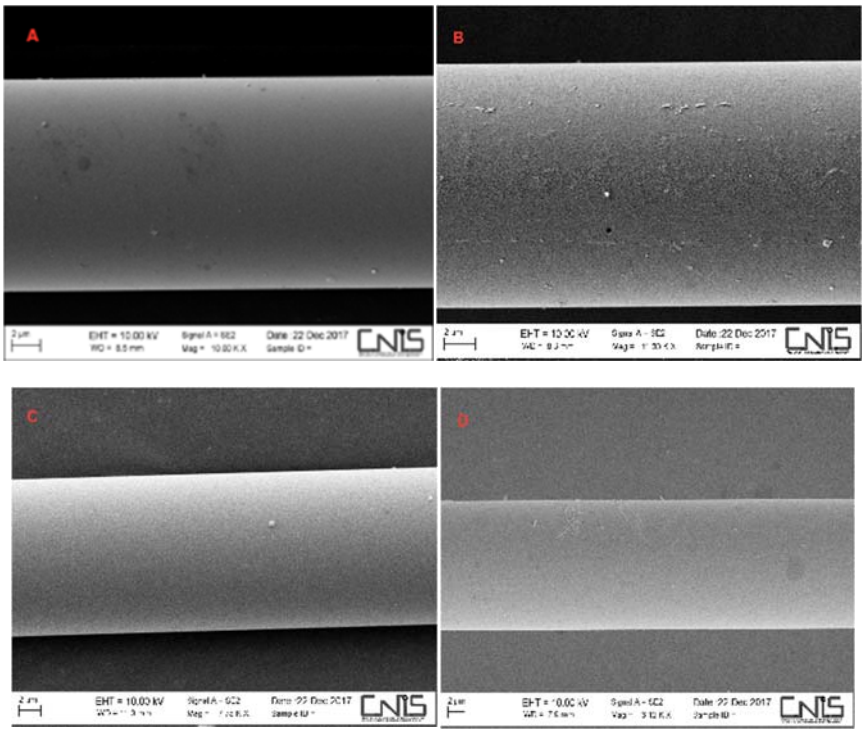

Figure 6. SEM images of the basalt fibres lateral surface where the effect of chemical etching is evident. (A) as-received basal fibres; (B) thermally treated at 600°C; (C) basalt fibres chemically regenerated with NaOH 3M,10min and (D) with HF 1%v. 2.5 min.

Once the regenerative effect of chemical treatments on heat-treated basalt fibres has been established, the fracture properties of these fibres have been characterized. Especially, the K_{Ic} of the regenerated fibres is higher than the values previously obtained for virgin and thermally treated fibres, as shown in Table 2. This behavior is related to the positive effect on fibres residual strength after the chemical treatment. Further studies will be carried out to determine the controlling mechanism associated with superficial etching and structural changes that are responsible for the regeneration of the strength of basalt fibres .

4. Conclusions

The re-use of recovered fibres by matrix pyrolysis in a new fibre-reinforced composite is limited by the weakening of the fibre reinforcement after the thermal exposure. The results of tensile tests on thermally treated basalt fibres reveal a large strength decrease up to 75% with increasing temperature (400–600°C). Hence, a difficult challenge for the academic and scientific world is to ascertain the fibres strength loss controlling mechanism during the recycling process. Focused ion beam (FIB) milling was used to introduce an artificially single nano/micro-sized deep notch (0.8-2μm) on single fibres, to understand if any bulk change occurs at high temperature. Fracture toughness has been studied to outline a strength-flaw relationship for basalt fibres. Tensile tests on notched basalt fibres were performed before and after thermal treatment to determine residual strength and fracture toughness. The fracture toughness of the untreated basalt fibres (0.94±0.24 $MPa \cdot m^{0.5}$) is close to the values reported in literature for E-glass fibres obtained using the same techniques (FIB). Fracture toughness value of the thermally treated fibres at 600°C increased up to 1.65±0.52 $MPa \cdot m^{0.5}$ due to the high temperature exposure. The heat treatment-induced alterations of the fibres mechanical properties in terms of fracture toughness and Young's modulus provide the experimental evidence of basalt fibres structural changes. In the present work, it has been shown as a short treatment of thermally degradated basalt fibres in NaOH allows to recover up to 95% of the strength previously lost. Although also HF has regenerative effects, this treatment is only able to partially regenerate the fibres, with a maximum recovering of 70%. This lower effectiveness, coupled with the numerous problems and toxicity related to its use, simply suggest its replacement with more benign chemicals such as alkaline solutions. For a given chemical agent, the regenerative effect is influenced by the molarity and the treatment time. The main mechanism supposed to be responsible for the regeneration of the mechanical strength of the fibres is chemical etching. The fracture properties of the regenerated fibres were analysed to understand the possible changes in strength-flaws relationship during the chemical treatment. The regenerated fibres (NaOH and HF) fracture toughness increase seems to confirm that structural changes occur in the bulk due to the chemical treatment, but further investigations are necessary to confirm this statement.

Acknowledgments

A. Fernandez and C.S. Lopes acknowledge the support of the Spanish Ministry of Economy and Competitiveness via the project HYDTCOMP (MAT2015-69491).

References

[1] G. Oliveux, L. O. Dandy, and G. A. Leeke, “Current Status of Recycling of Fibre Reinforced Polymers: review of technologies, reuse and resulting properties,” *Prog. Mater. Sci.*, vol. 72, pp. 61–99, Mar. 2015.

[2] P. G. Jenkins, S. Riopedre-Méndez, E. Sáez-Rodríguez, L. Yang, and L. James, “Investigation of the Strength of Thermally Conditioned Basalt and E-Glass Fibres,” *20th Int. Conf. Compos. Mater. Copenhagen, 19-24th July 2015*, no. July, pp. 19–24, 2015.

[3] J. Kennerley, “Recycling fibres recovered from composite materials using a fluidised bed process,” no. May, 1998.

[4] V. Fiore, T. Scalici, G. Di Bella, and A. Valenza, “A review on basalt fibre and its composites,” *Compos. Part B Eng.*, vol. 74, pp. 74–94, Jan. 2015.

[5] Tamás Deák and Tibor Czigány1, “Chemical Composition and Mechanical Properties of Basalt and Glass Fibers : A Comparison,” vol. 79, no. 7, pp. 645–651, 2016.

[6] M. C. Seghini, F. Sarasini, and J. Tirillò, “Influence of thermal conditioning on tensile behaviour of single basalt fi bres,” vol. 132, pp. 77–86, 2018.
[7] L. Yang, E. R. Sáez, U. Nagel, and J. L. Thomason, “Can thermally degraded glass fibre be regenerated for closed-loop recycling of thermosetting composites?,” *Compos. Part A Appl. Sci. Manuf.*, vol. 72, pp. 167–174, 2015.
[8] J. L. Thomason, U. Nagel, L. Yang, and E. Sáez, “Regenerating the strength of thermally recycled glass fibres using hot sodium hydroxide,” *Compos. Part A Appl. Sci. Manuf.*, vol. 87, pp. 220–227, 2016.
[9] J. L. Thomason, L. Yang, and R. Meier, “The properties of glass fibres after conditioning at composite recycling temperatures,” *Compos. Part A Appl. Sci. Manuf.*, vol. 61, pp. 201–208, Jun. 2014.
[10] S. Ogihara, Y. Imafuku, R. Yamamoto, and Y. Kogo, “Application of FIB technique to introduction of a notch into a carbon fiber for direct measurement of fracture toughness,” *J. Phys. Conf. Ser.*, vol. 191, pp. 0–6, 2009.
[11] K. Morishita, S. Ochiai, H. Okuda, T. Inshikawa, M. Sato, and T. Inoue, “Fracture toughness of a crystalline silicon carbide fiber (tyranno-SA3??),” *J. Am. Ceram. Soc.*, vol. 89, no. 8, pp. 2571–2576, 2006.
[12] M. Herrez, A. Fernandez, C. S. Lopes, and C. Gonzalez, “Strength and toughness of structural fibres for composite material reinforcement,” *Philos. Trans. R. Soc. A Math. Phys. Eng. Sci.*, vol. 374, no. 2071, p. 20150274, 2016.
[13] M. Kant and D. Penumadu, “Fracture behavior of individual carbon fibers in tension using nano-fabricated notches,” *Compos. Sci. Technol.*, vol. 89, pp. 83–88, 2013.
[14] K. Naito, “Stress analysis and fracture toughness of notched polyacrylonitrile (PAN)-based and pitch-based single carbon fibers,” 2017.
[15] S. Feih, A. P. Mouritz, and S. W. Case, “Determining the mechanism controlling glass fibre strength loss during thermal recycling of waste composites,” *Compos. Part A Appl. Sci. Manuf.*, vol. 76, pp. 255–261, Sep. 2015.
[16] J. L. Thomason, C. C. Kao, J. Ure, and L. Yang, “The strength of glass fibre reinforcement after exposure to elevated composite processing temperatures,” *J. Mater. Sci.*, vol. 49, no. 1, pp. 153–162, Sep. 2013.
[17] M. D. Lund and Y. Yue, “Impact of Drawing Stress on the Tensile Strength of Oxide Glass Fibers,” *J. Am. Ceram. Soc.*, vol. 93, no. 10, pp. 3236–3243, Oct. 2010.
[18] M. Ya, J. Deubener, and Y. Yue, “Enthalpy and Anisotropy Relaxation of Glass Fibers,” *J. Am. Ceram. Soc.*, vol. 91, no. 3, pp. 745–752, Mar. 2008.
[19] S. Feih, E. Boiocchi, G. Mathys, Z. Mathys, A. G. Gibson, and A. P. Mouritz, “Mechanical properties of thermally-treated and recycled glass fibres,” *Compos. Part B Eng.*, vol. 42, no. 3, pp. 350–358, Apr. 2011.
[20] M. Tomozawa and R. W. Hepburn, “Surface structural relaxation of silica glass: a possible mechanism of mechanical fatigue,” *J. Non. Cryst. Solids*, vol. 345–346, pp. 449–460, Oct. 2004.
[21] K. M. M. Davis and M. Tomozawa, “Water Diffusion Into Silica Glass - Structural-Changes in Silica Glass and Their Effect on Water Solubility and Diffusivity,” *J. Non. Cryst. Solids*, vol. 185, pp. 203–220, 1995.
[22] ASTM C 1557-03, “Standard Test Method for Tensile Strength and Young ’ s Modulus of Fibers 1,” vol. 03, no. Reapproved, pp. 1–10, 2013.
[23] J. D. Sullivan and P. H. Lauzon, “Experimental probability estimators for Weibull plots,” *J. Mater. Sci. Lett.*, vol. 5, no. 12, pp. 1245–1247, Dec. 1986.
[24] K. Singha, “A Short Review on Basalt Fiber,” *Int. J. Text. Sci.*, vol. 1, no. 4, pp. 19–28, 2012.
[25] T. Förster, G. S. Sommer, E. Mäder, and C. Scheffler, “Surface, interphase and tensile properties of unsized, sized and heat treated basalt fibres,” *IOP Conf. Ser. Mater. Sci. Eng.*, vol. 139, no. 1, 2016.
[26] M. D. Lund and Y.-Z. Yue, “Fractography and tensile strength of glass wool fibres,” *J. Ceram. Soc. Japan*, vol. 116, no. 1356, pp. 841–845, 2008.
[27] S. Feih, A. Thraner, and H. Lilholt, “Tensile strength and fracture surface characterisation of sized and unsized glass fibers,” *J. Mater. Sci.*, vol. 40, no. 7, pp. 1615–1623, Apr. 2005.
[28] Y. Z. Yue, J. Christiansen, and S. L. Jensen, “Determination of the fictive temperature for a hyperquenched glass,” vol. 357, no. May, pp. 20–24, 2002.
[29] C. A. Angell, Y. Yue, L. M. Wang, J. R. D. Copley, S. Borick, and S. Mossa, “Potential energy, relaxation, vibrational dynamics and the boson peak, of hyperquenched glasses,” *J. Phys. Condens. Matter*, vol. 15, no. 11, pp. 1051–1068, 2003.
[30] J. Toribio, N. Álvarez, B. González, and J. C. Matos, “A critical review of stress intensity factor solutions for surface cracks in round bars subjected to tension loading,” *Eng. Fail. Anal.*, vol. 16, no. 3, pp. 794–809, 2009.
[31] S. T. Bashir, L. Yang, R. Anderson, P. L. Tang, J. J. Liggat, and J. L. Thomason, “A simple chemical approach to regenerating the strength of thermally damaged glass fibre,” *Compos. Part A Appl. Sci. Manuf.*, vol. 102, pp. 76–87, 2017.
[32] G. A. C. M. Spierings, “Wet chemical etching of silicate glasses in hydrofluoric acid based solutions,” *J. Mater. Sci.*, vol. 28, no. 23, pp. 6261–6273, 1993.