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Strength degradation of glass fibers at high temperatures

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Abstract This article presents an experimental investigation into the effects of temperature and heating time on the tensile strength and failure mechanisms of glass fibers. The loss in strength of two glass fiber types (E-glass and Advantex®, a boron-free version of E-glass) was investigated at temperatures up to 650 °C and heating times up to 2 h. The tensile properties were measured by fiber bundle testing, and the maximum strength was found to be temperature and time dependent. The higher softening point of the Advantex® fibers is reflected in superior high-temperature performance. A phenomenological model is presented for calculating the residual strength of glass fiber bundles as functions of temperature and time. The strength reduction mechanism was determined by single-fiber testing. Fracture mirror sizes on the E-glass fibers were related to the fiber strength after high-temperature treatment. Based on fracture mirror measurements, it was established that (1) the mirror constant of the glass, which reflects the network structure, does not change during heat treatment and (2) the strength degradation is a result of larger surface flaws present after heat treatment.

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Introduction

The most common reinforcement used in polymer matrix composites is fiberglass. Approximately 90–95% of all composite products contain fiberglass. Application areas include aircraft and helicopter components, marine structures, land transport vehicles, electrical/electronic appliances, and many types of consumer goods. It is expected that the use of fiberglass composites will grow in coming years as these materials penetrate deeper into established markets (e.g., marine, transportation) and find new applications in emerging markets (e.g., bridges and other civil infrastructure). In comparison to metals, one challenging issue when using composites remains their high flammability. Many composite materials, whether reinforced with glass or another fibrous material such as carbon, have a flammable polymer matrix that combusts at high temperature. Composites can smolder or burn with the release of significant heat, smoke, and fumes, which may pose a serious safety hazard. Composites may also soften, buckle, or collapse in a fire. This is a concern in most structural applications, especially when failure can cause injury and death.

Recent fire research by Feih et al. [1, 2] has shown that tensile failure of fiberglass composites is dependent on thermally induced strength reduction of *both* the polymer matrix and the fibers. Composites in fire generally retain significant tensile strength after the matrix has fully

softened and decomposed because of the strength provided by the glass fibers. However, glass fibers also lose their strength, albeit at a slower rate than the matrix, and eventually fail by thermally induced embrittlement and rupture under certain fire and load conditions. Strength reduction of glass fibers after high-temperature exposure is also an important issue for the recycling of glass fiber composites. During recycling, the resin matrix may be removed by heating at around 500 °C for up to 2 h [3]. Although glass fibers appear unaffected on visual inspection after heating, the treatment can significantly decrease the fiber strength and remains one of the major issues preventing the use of recycled glass fibers in load-bearing structures. This strength reduction is also an issue in cases where thermal processes are used to remove the size coating on certain types of fiberglass product.

The tensile strength of glass fibers decreases with increasing temperature and heating time [4]. The cause of this strength reduction has been much debated [5] and fully satisfactory conclusions have yet to be drawn. The available results to date, and various interpretations of them, were summarized by Gupta [5] in 1988 and there has been little significant new information since. The loss in strength has been attributed to several mechanisms, which may include (1) annealing of compressive residual stresses, (2) re-orientation or loss of orientation of a molecular network structure, (3) presence of a surface layer with different properties to the fiber core, and (4) development of surface flaws due to high-temperature attack, probably involving water. Mechanism (1) has now been discredited, but mechanisms (2)–(4) are still regarded as plausible, despite little evidence of either network orientation or differences between the surface and core properties of the fiber.

The present article examines the effect of temperature and heating time on the tensile strength properties and failure mechanisms of glass fibers. Heating of glass fibers is performed in air to mimic the atmospheric conditions during fire exposure. The atmosphere is assumed to play an important role during strength degradation. The tensile properties measured for treatment temperatures in the range 150–650 °C and times of 5–120 min have been used to model the kinetics of strength loss in air. Two types of glass fibers—E-glass and Advantex®—were used to investigate the effect of heat treatment on strength degradation. Advantex® is a boron-free variation of E-glass. Fractographic and chemical analysis were undertaken to identify the mechanism of embrittlement at high temperature.

Fiber materials and experimental methods

Materials

Two types of glass fibers were supplied by Owens Corning: conventional E-glass with 111A sizing and Advantex® glass fibers with SE1200 sizing. The fiber diameters were 13 μm for the E-glass and 17 μm for the Advantex® fibers. Advantex® is a boron-free glass fiber with a higher softening temperature, 916 °C, than standard E-glass, 830–860 °C [6]. The bundle strength degradation for Advantex® fibers is compared to standard E-glass in this article to highlight the effect of changes to the glass network composition on embrittlement. Table 1 shows the differences in chemical compositions as established by energy dispersive X-ray (EDX) analysis. The EDX spot analysis was performed on the cross-section of polished fibers to evaluate the chemical composition of the fibers. The composition was determined with three scans per fiber type and validated against a copper standard. E-glass fibers were shown to have a higher sodium (Na) and aluminum (Al) content, while the magnesium (Mg) content was lower than for the Advantex® fibers. The elemental changes (decrease in boron and increase in magnesium content) increase the fibers' resistance to corrosion and stress rupture in all environments. The performance of Advantex® fibers is comparable to E-CR glass. Table 1 also shows that no significant changes in elemental composition were observed following heat treatments.

Table 1 Comparison of chemical composition of glass fibers as measured by EDX

Fiber	O (%)		Na (%)		Al (%)		Si (%)		Ca (%)		Mg (%)	
	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max	Min	Max
E-glass												
As-received	47.1	47.6	1.13	1.15	7.22	7.37	27.9	28.2	16.0	16.0	0.14	0.15
Heat treated	46.8	48.46	1.07	1.18	7.38	7.53	27.18	28.05	15.70	16.21	0.15	0.26
Advantex®												
As-received	46.7	51.1	0.43	0.56	6.30	6.71	26.3	28.5	13.7	15.6	2.04	2.06
Heat treated	40.83	45.6	0.32	0.52	6.68	6.83	29.15	30.58	16.09	19.53	1.91	2.0

Heat treatment

Fiber bundles of E-glass and Advantex® were heat treated in air using a muffle furnace. No load was applied during the heat treatment. The bundles were subsequently cooled in air, prior to testing. The possibility of strength recovery during cooling was investigated by comparing the residual strength of a heat-treated bundle measured at room temperature against the bundle strength measured at high temperature [2]. The residual strength and high-temperature strength were found to be the same, so strength recovery was discounted.

Fiber bundle testing

The tensile strengths of 300 tex fiber bundles of E-glass and Advantex® were measured at room temperature following heat treatment. The fiber bundles were tested under tension using a 10 kN Instron machine. The ends of the fiber bundle were wrapped around circular rollers so that the stress is introduced into the specimen gradually via friction. This ensured bundle failure in the gage section. The maximum force was measured, which can be related to the maximum strength of the fiber bundle through the weight per unit length of the bundle ($W_f = 300$ tex) and the density of glass ($\rho_f = 2560$ kg/m^3):

$$\sigma_{max} = \frac{F_{max}\rho_f}{W_f} \quad (1)$$

A gage length of 150 mm was used for the fiber bundles. At least five samples were tested for each heat-treatment condition. The test method allows for efficient testing of large sample numbers as no end tabs are required for gripping of the fiber bundles. However, the strain in the gage section cannot be accurately determined with this test method, and result discussions are therefore limited to the decrease of the maximum strength. Weibull statistics can therefore only be derived from single-fiber test results (described below) and not bundle test results. Furthermore, no effort was made to lubricate or resize fiber bundles following heat treatment to minimize friction between fibers as the strength loss data were required as input for a tensile failure model during fire [2].

Single-fiber testing

Single-fiber testing was performed on the E-glass fibers for two selected heating conditions (450 °C for 15 and 30 min). The results from this test are compared to the bundle test results for the same heat-treatment conditions. Single-fiber testing excludes the effect of interfiber friction and any other physical interactions between fibers which could introduce damage during handling and loading.

A single fiber was mounted on a cardboard frame by applying re-usable adhesive to both ends to obtain a vertically positioned fiber with a prestress defined by the weight of the adhesive. The fiber was then glued onto the cardboard frame with Loctite 406 adhesive. The cardboard frame had holes at a fixed spacing of 22 mm, thereby defining the gage length. A 2.5 N Instron 4501 machine operated at 0.5 mm/min was used to measure the tensile strength of the single fibers. The broken fibers were collected, and their fracture surfaces examined and measured using a scanning electron microscope (LEO 1530VP-21-32) operated at 5 kV. The SEM was calibrated with a graticule with a tolerance of 3%.

Results

Comparison of heat-treatment effect for single-fiber strength and fiber bundle strength

Tensile stress–strain/displacement curves for the as-received and heat-treated E-glass measured by single- and fiber bundle testing are shown in Fig. 1. Both tests revealed that the elastic stiffness of the fibers was unaffected by heat treatment, while the strength reduction was significant. Failure in the single-fiber test involved sudden brittle fracture with complete loss of load, regardless of the heat-treatment temperature or time. Rupture of the fiber bundles without temperature exposure was gradual due to the variability in fiber strength. Following temperature exposure, the fiber bundle tests generally showed a less gradual failure, which was attributed to the loss of sizing and resulting increase in friction between fibers.

It is well known that the tensile strength of single fibers and fiber bundles is governed by the maximum size of pre-existing flaws (usually surface cracks). A large scatter in strength values is often observed due to variations in the maximum flaw size between fibers. The effect of heat treatment on the scatter in fiber strength was evaluated by performing single-fiber tests each on as-received (20 samples) and heat-treated E-glass filaments (30 samples each). Figure 2 shows Weibull plots of tensile strength for the original fiber and the fiber following two heat-treatment conditions. The results suggest two main effects:

1. The fiber strength decreases significantly with increasing heat-treatment time, even though the temperature (450 °C) is well below the annealing temperature for E-glass, and
2. The heat-treated results show a larger scatter in strength than the as-received fibers, which suggests a greater variation in flaw sizes caused by heating.

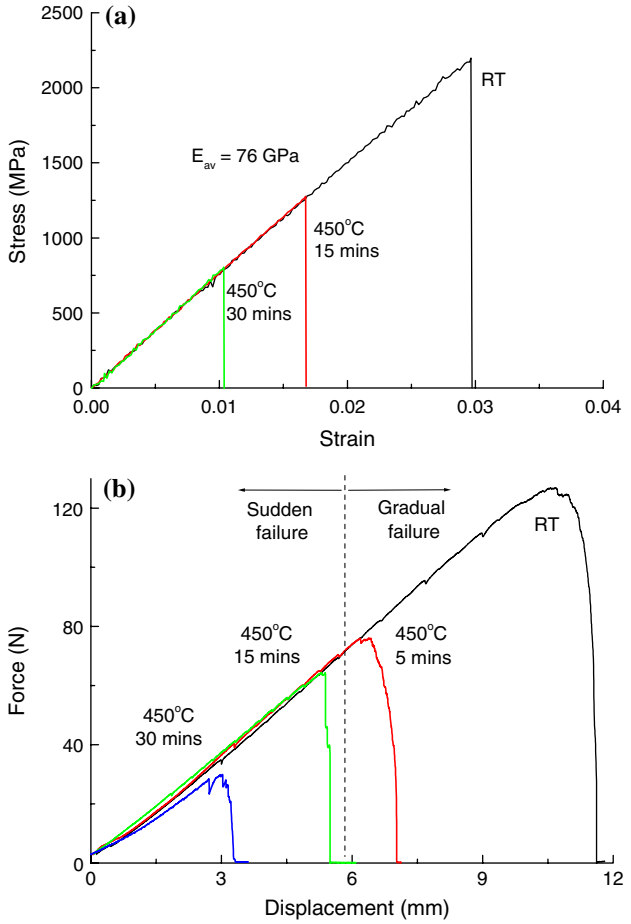

Fig. 1 Variation in single-fiber test (**a**) and fiber bundle test (**b**) results for E-glass fibers due to heat treatment

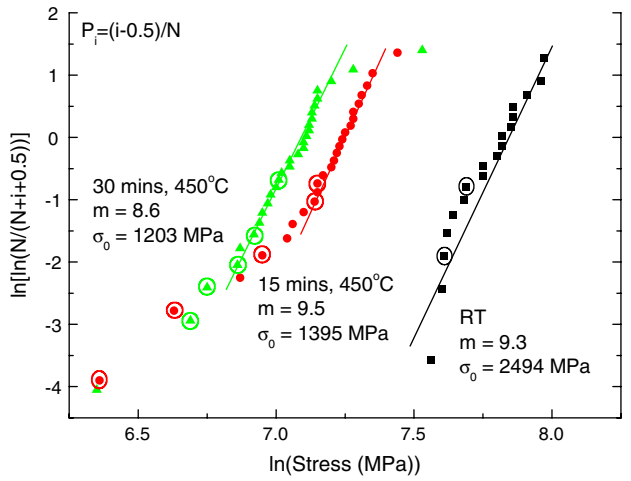

Fig. 2 Weibull plot of single-fiber test results for E-glass (*circled data points* indicate investigated fracture surfaces)

In fact, the heat-treated fibers show two distinct regions with different slopes in the Weibull distribution. This type of Weibull distribution has been attributed to both bi-modal fiber strength distribution due to different types of flaws (i.e., bulk and surface flaws [7]) or end effects from the single filament test [8]. While end effects may play a role for some fibers, several low-strength failure locations were identified toward the middle of the fiber. To identify the flaw origin, the fiber fractures were investigated from both regions (circled data points) and are discussed later in the article. The Weibull parameters in this study were not calculated based on the bi-modal distribution as the number of test samples was considered too small for accurate results and should ideally approach about 100 samples per heating condition [9]. For the purpose of a comparison of test methods, fiber strength values were established from the linear region, where the bulk portion of fibers failed (see linear fit lines). It should be noted though that a fit through all data points results in similar average strength values as outliers away from the linear fit occur in both the low- and high-strength region.

Figure 3 shows the normalized tensile strength values of single fibers and fiber bundles in the as-received and heat-treated conditions. The strength values were normalized to their room temperature (as-received) strength to allow a comparison between single fibers and bundles of fibers. Despite the scatter, the results show that the bundles experience a greater percentage loss in strength after heat treatment than the single fibers. This was attributed to the removal of organic sizing from the filaments, which decomposes during heat treatment above ~350 °C. The size provides lubrication between fibers during bundle testing, so its removal will result in greater frictional forces. Bundle test results can only be related to single-fiber test results when the assumption of independent fracture events can be justified [10]. Up to 40% variation in bundle strength has been previously observed for identical glass fiber bundles (i.e., the same tex and fiber diameter) with different lubricants [10] and sizings (personal communication with Owens Corning).

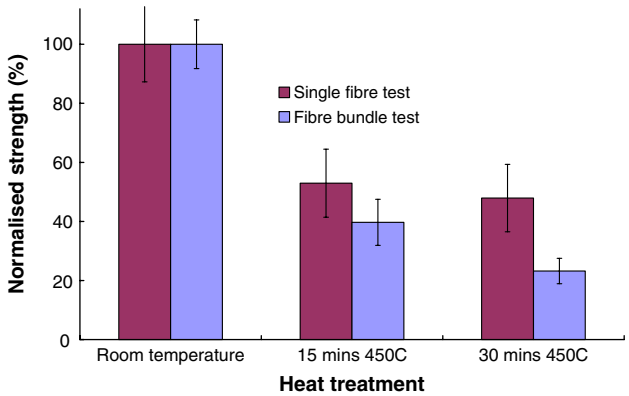

Fig. 3 Comparison of strength reduction from single-fiber and fiber bundle tests for E-glass

For the purpose of strength degradation in composites during fire, the friction effect in fiber bundles is important once the resin has burnt completely. Due to the original purpose of fire research, the following detailed investigation of strength loss during heat treatment was therefore performed on fiber bundles, rather than single fibers. The cause for strength degradation was evaluated based on the single-fiber test results.

Effect of heat treatment on fiber bundle strength

Figure 4 shows the effects of heat-treatment temperature and time on the tensile strength of the E-glass and Advantex® fiber bundles. Using Eq. 1, the maximum bundle strengths for E-glass and Advantex® were found to be σ_{max} = 1058 MPa (F_{max} = 124 N) and σ_{max} = 888 MPa (F_{max} = 104 N), respectively. The difference in room temperature strength was attributed to the different sizings as the single filament strength is similar for both fibers [6]. The failure strengths of the heat-treated bundles were normalized to their respective room temperature strengths (fiber area remains constant during heat treatment) to allow a direct comparison of the strength loss for both fiber types. The strength of both bundle types decreases with temperature (above ~150 °C) and time, until a steady-state strength value is reached for each temperature. The heating time required to reach steady-state decreases rapidly with increasing temperature. The gradient of the stress–displacement plots for both the fiber types remained unchanged under all conditions, showing that the damage caused by the heat treatment affected the strength but not the elastic modulus. It can also be seen from Fig. 4 that both fiber types showed similar strength degradation behavior, thereby indicating that the boron content does not affect the embrittlement mechanism.

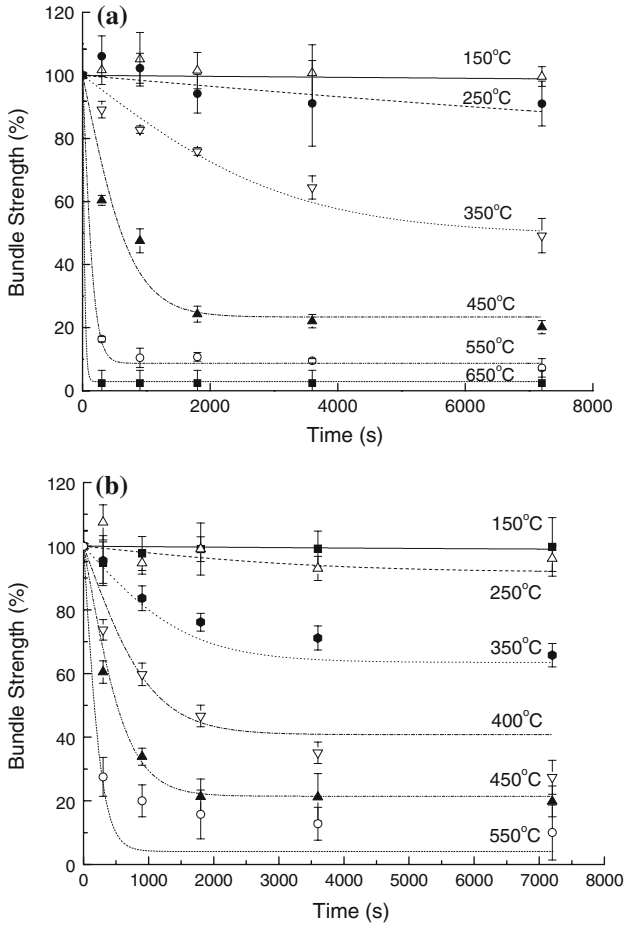

Fig. 4 Normalized experimental results for fiber strength as a function of time and temperature for: **a** E-glass (σ_{max} = 1058 MPa). **b** Advantex® fibers (σ_{max} = 888 MPa). The *lines* indicate modeling results

A phenomenological model was developed to describe the strength loss as a function of time and temperature to enable modeling of the results of fire tests under load on E-glass composites [2]. The curves shown in Fig. 4 follow a profile that is represented in Fig. 5 and can be described mathematically using a tanh function. The tanh function relating fiber bundle strength (σ_{fb}) to the temperature (T) and heating time (t) is given by:

$$\sigma_{fb}(t,T) = \sigma_{fb(0)} - \sigma_{loss}(T)\tanh[k_{fb}(T)t] \quad (2)$$

where $\sigma_{fb(0)}$ is the maximum tensile stress at 20 °C. $\sigma_{loss}(T)$ describes the strength loss and $k_{fb}(T)$ describes the rate of strength loss, and both these material parameters are a function of temperature. The strength loss, $\sigma_{loss}(T)$, is determined by curve fitting the function:

$$\sigma_{loss} = \frac{\sigma_{fb(0)}}{2} + \frac{\sigma_{fb(0)} \cdot \tanh[p_{fb}(T - T_{50\%})]}{2} \quad (3)$$

with $T_{50\%}$ and p_{fb} being curve-fitting constants. $T_{50\%}$ is defined as the temperature at which the fiber bundle loses

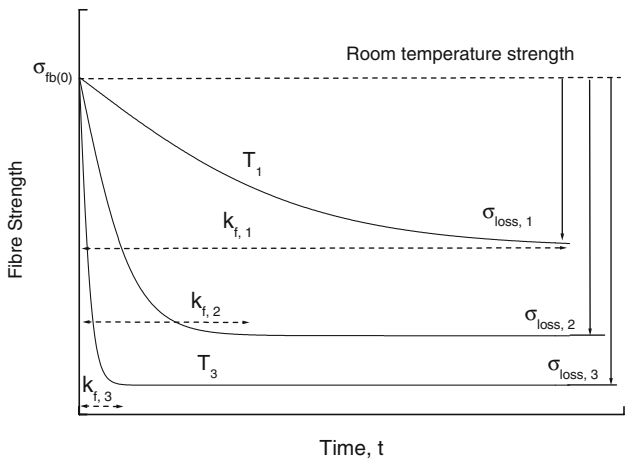

Fig. 5 Representation of the typical relationship between temperature, time, and tensile strength of glass fibers

Table 2 Fitted data for glass fiber strength reduction

Parameter	E-glass		Advantex®	
	Fitted value	Standard error (%)	Fitted value	Standard error (%)
$T_{50\%}$ (°C)	347.6	3.9	379.9	1.7
p_{fb} ($^{\circ}\mathrm{C}^{-1}$)	5.83E − 3	11.1	9.26E − 3	11.2
k_1 (s^{-1})	1.81E − 6	58.2	2.98E − 5	68.4
k_2 ($^{\circ}\mathrm{C}^{-1}$)	1.45E − 2	8.8	8.65E − 3	17.5

50% of its original tensile strength after long-term heat exposure. The rate of strength loss, $k_{\mathrm{fb}}(T)$, is given by:

$$k_{\mathrm{fb}}(T) = k_1 e^{k_2 T} \tag{4}$$

where k_1 and k_2 are constants.

A curve-fitting method was used to establish the model parameters, which are given in Table 2 for E-glass and Advantex®. The curves in Fig. 4 show the predicted reduction in strength of the E-glass and Advantex bundles with increasing temperature and time, and there is good agreement with the experimental data. Apart from the value for k_1, all parameters show a low standard error, which indicates a good fit for the data. It should be noted that $T_{50\%}$, which describes the temperature at which a fiber bundle has lost 50% strength, is significantly lower for E-glass than for Advantex®, indicating inferior high-temperature performance. The increase in $T_{50\%}$ from 347 to 380 °C reflects the increase of the annealing temperature of the Advantex® fibers (916 °C) compared to E-glass (830–860 °C) [6].

Discussion

Fracture surfaces and flaw origin

Failure of glass and glass fibers commonly originates from small surface flaws. Fracture surfaces after single-fiber testing were examined using SEM. Samples for which fracture surfaces were obtained are indicated in Fig. 2 by the circled data points. Figure 6 shows representative fracture surfaces. In some cases it was also possible to compare both surfaces from a fracture event, which were found to match (see Fig. 6d, e).

Distinctive markings were found on all fracture surfaces. The fracture surface of brittle materials is generally characterized by three distinct regions: the mirror, mist, and hackle regions [11]. These regions are caused by changes in the crack growth rate and are shown schematically in Fig. 7. The mirror zone is the smooth, featureless region surrounding the original flaw in which the growth rate of the crack accelerates under load. As the crack accelerates it becomes increasingly unstable, which creates a dimpled surface known as the mist. The crack eventually branches out and produces the rough hackle region. The fracture mirror boundaries are indicated by the dotted line in Fig. 6. For all fracture surfaces investigated, the location of the fracture mirror shows that fracture originated from the surface (rather than within the bulk), regardless of the heat-treatment temperature or time.

It has been demonstrated [12, 13] that the strength of silicate glasses is related to the mirror radius. For optical fibers, it was previously shown that the mirror size is not semicircular as in the case of rectangular bulk glass specimens. For larger mirrors in the order of a fifth of the fiber diameter, the width of the mirror is interrupted by the curvature of the fiber edge [14]. In this case, the mirror depth, rather than the mirror width, was shown to result in a linear relationship with the fiber strength:

$$\sigma_{\mathrm{TS}} = A_{\mathrm{m}} (d_{\mathrm{m}})^{-1/2}, \tag{5}$$

where d_{m} is the mirror depth and A_{m} is the so-called mirror constant. Figure 8 shows a plot of the reciprocal square root of the mirror depth against E-glass fiber strength to determine the mirror constant. The value for the mirror constant is $A_{\mathrm{m}} = 2.1$ MPa $\mathrm{m}^{1/2}$, which is in very good agreement with the range of $A_{\mathrm{m}} = 1.8$–2.5 MPa $\mathrm{m}^{1/2}$ given in the literature for glasses [13] and optical glass fibers [14]. These bounds are also indicated in Fig. 8. The data shows a good linear fit ($R^2 = 0.944$), which confirms the relationship between strength and mirror depth described mathematically in Eq. 5 for both original and heat-treated fibers.

The findings from the fracture surfaces lead to several important conclusions:

1. The mirror constant of the glass does not change, so it is unlikely that the glass network structure and mode I fracture toughness change during heat treatment. This is also indicated by the fact that the Young's modulus of the fibers remains constant.
2. The mirror size increases during heat treatment; suggesting that the flaw size increases during heat treatment.
3. The apparent bi-modal Weibull distribution for single-fiber testing discussed earlier is not caused by the

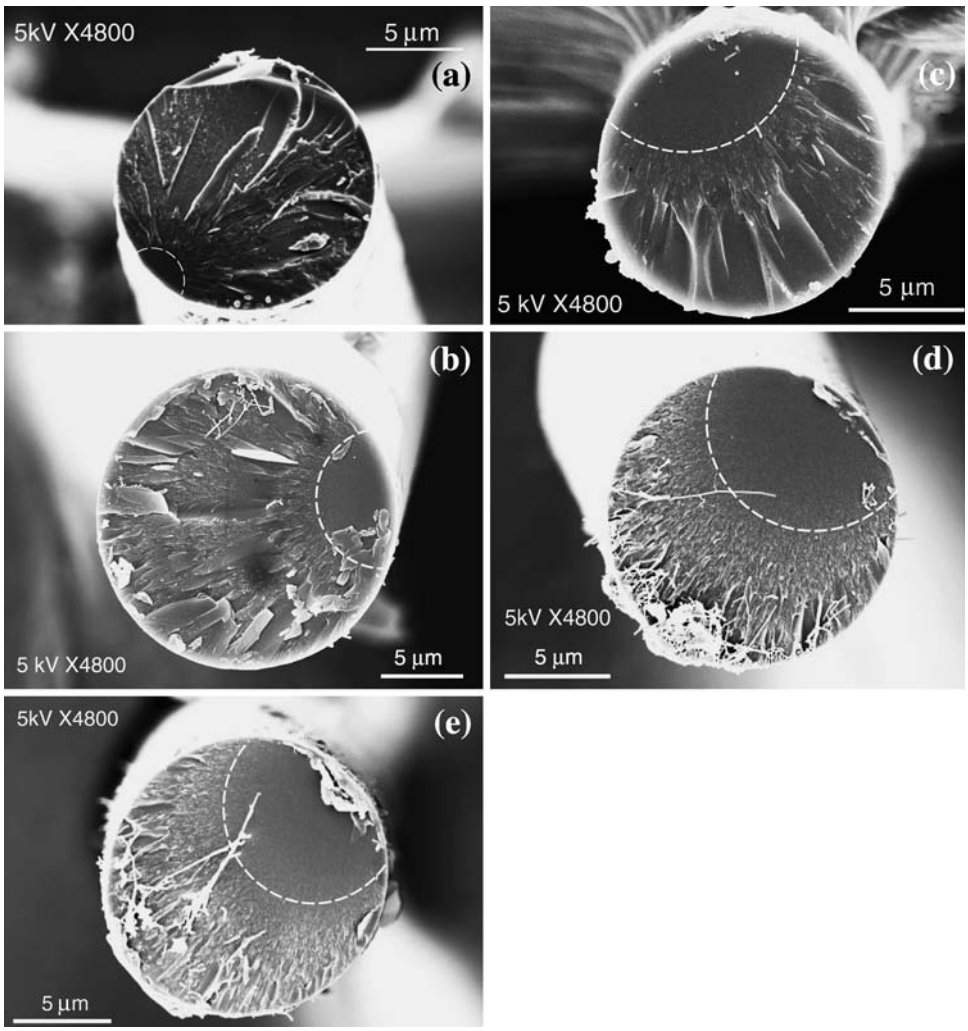

Fig. 6 Fracture patterns with surface flaw origin for E-glass fiber for: **a** Room temperature with $\sigma_{TS} = 2020$ MPa. **b** Heat treated at 450 °C for 30 min with $\sigma_{TS} = 1100$ MPa. **c** Heat treated at 450 °C for 30 min with $\sigma_{TS} = 950$ MPa. **d**, **e** Heat treated at 450 °C for 15 min with $\sigma_{TS} = 750$ MPa. The fracture mirror depth is indicated by the *white line*

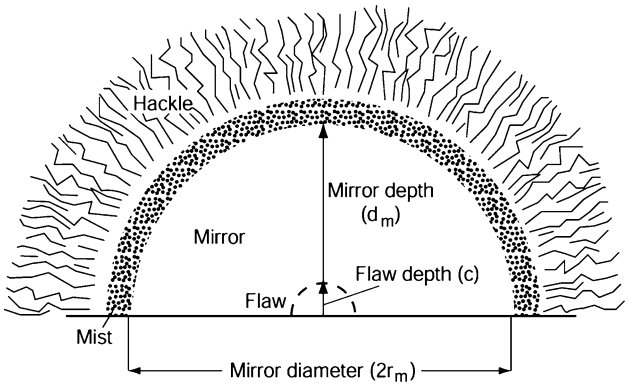

Fig. 7 Schematic showing of typical glass surface features that form during failure from surface flaws

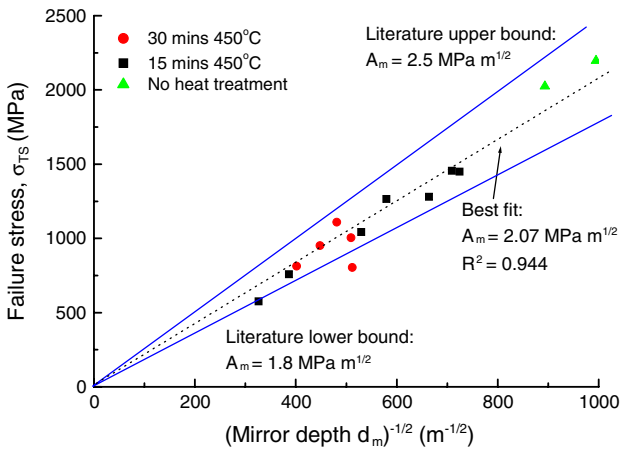

Fig. 8 Fracture stress versus reciprocal square roots of mirror depth with least-squares linear fit according to Eq. 6 for E-glass

presence of surface and bulk flaws. All observed fractures start from the surface. Furthermore, several fibers with significantly lower strength and failure at the center of the gage length are encountered. The growth and creation of flaws therefore appears to be more severe for selected flaw locations as the Weibull modulus decreases after heat exposure.

Flaw size

The depth of the surface flaws can be estimated from fracture mechanics. A planar crack perpendicular to the fiber axis is the simplest idealization of the crack shape, and the dependence of fiber strength on flaw size (c) is governed by the well-known relationship:

$$\sigma_{TS} = \frac{1}{Y(c,R)} K_{IC} (\pi c)^{-1/2} \quad (6)$$

K_{IC} is the fracture toughness. The fracture toughness cannot be measured for single fibers and was therefore assumed to be similar to the value for borosilicate glass ($K_{IC} = 0.76$ MPa $m^{1/2}$ [15]). The constant mirror constant A_m warrants the assumption of a constant fracture toughness value. R is the fiber radius and $Y(c,R)$ is a factor generally dependent on the crack geometry and its size in relation to the fiber radius. Assuming small and semicircular flaws, Y can be considered constant with a value of $Y = 2/\pi$. Table 3 shows the required flaw size, c, to obtain the measured average fiber strengths as calculated using Eq. 6. When measured immediately after manufacture, pristine fibers have a tensile strength of 3000–3500 MPa [6]. Bundle formation and handling reduces the strength due to fiber damage. Griffith's fracture model predicts that the average flaw size increases from around 37 nm (pristine), to 73 nm (as-received fibers), to 313 nm (after heat treatment at 450 °C for 30 min).

Cause of strength degradation

Fracture was shown to occur from the surface. A possible cause needs to be determined for the growth or generation of surface flaws up to 500 nm at high temperature. Moisture or water vapor is generally found to be the driving factor for static fatigue of glass and results in surface pits. Pit formation generally occurs over long exposure times, but is accelerated by elevated temperature [16, 17]. Fiber pits preferentially develop on pre-existing structural flaws representing weak sites along the glass fibers [16]. Fiber pitting with flaw sizes in the order of 500 nm was, for example, observed after exposure to deionized water after 500 days at 60 °C [18]. The high-temperature exposure acts as an accelerator with water vapor being present in the atmosphere during the heat-treatment process [16]. Further SEM investigations were conducted on the surfaces of the heat-treated fibers; however, no obvious surface flaw population of a size between 200 and 500 nm could be detected. It was concluded that flaws are either extremely random or hard to detect on the surface of unstressed fibers due to the cracks being closed. Further investigation is currently underway to identify the effect of various atmospheric conditions and preload on fiber strength degradation.

Table 3 Flaw size calculation for average E-glass fiber strength for semicircular flaw shape

Condition	Strength (MPa)	Flaw depth (nm)
Pristine condition [6]	3500	37
As-received	2494	73
450 °C, 15 min	1395	233
450 °C, 30 min	1203	313

Conclusion

The heat treatment of E-glass fiber and boron-free E-glass (Advantex®) fiber causes an irreversible loss in tensile strength even at moderately low temperatures and short heating times. The strength decreases rapidly with increasing temperature above ~200 °C, which is well below the annealing temperatures of the fibers. At the highest treatment temperature (650 °C) the fiber strengths have dropped to only a few percent of their original (as-received) strength. While the strength is adversely affected, the elastic stiffness does not change during heat treatment. The experimental data suggests that the strength of glass fibers used as reinforcement in polymer matrix composite materials will be irreversibly degraded even under moderately low-temperature fire conditions. The results also reveal that the recycling of fiberglass composites by thermal treatment methods (e.g., pyrolysis of the polymer matrix) will reduce the strength of the recovered fibers.

The reduction in glass strength can be attributed to the growth of pre-existing surface flaws or creation of new flaws during the heat-treatment process, and is not caused by changes to the fracture toughness due to physical changes to the glass network structure or leaching of metallic ions. Examination of the fracture surfaces of fibers revealed the size of the critical surface flaws (as measured by the mirror size) increased with the temperature and duration of the heat treatment. Although the mechanism of flaw growth and creation remains to be determined, the most likely explanation is a corrosive reaction with water present in the atmosphere.

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