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A simple chemical approach to regenerating the strength of thermally damaged glass fibre

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ABSTRACT

Process-induced strength loss is a major technical barrier to the effective reuse of thermally recycled glass fibres in composite applications. We have developed a novel approach to effectively restore strength in glass fibres through treatment in alkaline solutions. Glass fibres were treated at elevated temperature and experienced significant strength loss found typically after thermal recycling processes. Different alkaline treatments were then applied to the thermally damaged fibres in an attempt to restore strength which had been lost as a result of the heat conditioning procedure. Results indicated that these treatments were able to generate considerable fibre strength recovery. The degree of strength regeneration was found to be highly dependent on reaction conditions, which were investigated and optimised. The positive effect of these simple chemical treatments demonstrated great potential for facilitating the reuse of thermally recycled glass fibres in composite applications.

1. Introduction

There is growing concern of the negative effect current disposal methods of composite waste are having on the environment. It is estimated that by 2020 the composites market will reach almost £80 billion globally, and as a result, there will be a high volume of waste once these materials reach the end of their life cycle [1]. Glass fibre is currently used as reinforcement in over 90% of all fibre-reinforced composites produced, and production waste represents 5–10% of composites production. The high rigidity and chemical resistance of these composites, particularly glass fibre reinforced thermosetting polymers (GRP), are required for optimum performance but unfortunately result in poor recyclability; when such materials are no longer fit for purpose, they are deposited frequently in landfill sites. The rising costs associated with landfill together with increasingly stringent legislation means this disposal route is becoming ever more undesirable. Consequently, alternative methods for dealing with GRP manufacturing waste are needed [2]. In addition, the accelerating growth in use of GRP materials such as in the production of wind turbine blades [3] means it is imperative that a long-term, cost-effective, recycling solution be developed for end-of-life composites.

In an effort to reduce the environmental damage caused by disposal of end-of-life composite materials, a range of recycling techniques have been investigated, some of which are now exploited on an industrial scale [4,5]. Thermal treatment is one of the most widespread recycling technologies; by subjecting the composite to elevated temperatures, degradation of the polymeric matrix is achieved, facilitating subsequent extraction of any fibrous reinforcement. Due to the harsh conditions employed in this procedure the glass fibres suffer from a severe loss in strength, and therefore cannot be reused in many forms of composite applications [6–9]. These damaged filaments can however be reused as reinforcements if their strength is restored by means of chemical treatment. Such an example has recently been given in [10] where the strength of glass fibres heat conditioned at 450–600 °C can almost triple after a few minutes of immersion in dilute hydrofluoric acid (HF). HF is proven to be an effective chemical etchant, and is thought to strengthen glass by smoothing out sharp, severe surface flaws [11]. However as HF is highly toxic in nature, its commercial use for regenerating strength of thermally weakened glass fibres is problematic. Consequently there is a need to find less challenging chemical routes to enable a solution to the problem of cost-effective recycling and reuse of the glass fibres in GRP waste.

The present work presents the results of an investigation of the use of hot alkaline solutions in regenerating the strength of thermally degraded E-glass fibres. The dissolution of glass in alkali is well documented in literature [12–14], however the use of these

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corrosive substances for strengthening thermally damaged glass fibres is a novel concept, given that alkaline treatments are shown to have a detrimental effect on virgin glass fibre strength [15]. It is believed that the reduction in strength of fibres after heat treatment can be attributed to the creation of new flaws on the surface and/or the growth of pre-existing flaws [16,17]. The mechanism by which these flaws develop is not yet fully understood, however it has been postulated that it could involve the interaction of water with the fibre surface during the heat treatment process [16,17]. It can be hypothesised that the reaction of silica (SiO_2) in the glass fibre with hydroxide ions (OH^-) from the alkaline solution [18] leads to the smoothing of the sharp, severe surface flaws, and thus increases the tensile strength of the fibre. The modification of surface flaws has been reported previously on bulk glass with HF as the corrosive medium [11].

We have recently discovered that sodium hydroxide (NaOH), prepared at high temperatures and at concentrations of 1.5 M and above, can significantly improve the strength of thermally damaged glass fibre [19,20]. To further our understanding of the reaction of glass with alkaline solutions, hydroxides based on other alkali metals were surveyed: lithium hydroxide (LiOH) and potassium hydroxide (KOH). It is thought the effect of etching of the glass surface by alkaline treatment is strongly dependent on various reaction conditions including nature of alkaline solution, temperature, molarity and treatment duration. In this research study, we provide initial evidence of glass fibre etching by alkaline solutions, and the resulting deposit formed on the fibre surface was analysed through various techniques. The main aim of this research study is to validate our hypothesis that alkaline treatment can regenerate the strength of thermally degraded glass fibres, offer a potential cost-effective route to GRP recycling, and ultimately reduce the negative environmental impact from landfill disposal. Additional objectives include generating further understanding of the etching mechanism and how differences in chemical properties of alkali metal hydroxides affect their reaction with glass fibre.

2. Experimental

2.1. Materials

Boron-free E-glass fibres supplied by Owens Corning (OC) were used in this study. These OC fibre rovings were manufactured on a pilot scale bushing and received as 20 kg continuous single end square edge packages. Each roving had a nominal tex of 1200 and a nominal fibre diameter of 17 μm. During production, fibres were coated with a 1% volume γ-aminopropyltriethoxysilane (APS) hydrolysed solution in deionised water. The purpose of this APS sizing is to functionalise and protect the fibre surface. One of the experiments described in Section 2.8 involved the use of unsized glass fibres; APS solution was not applied to these fibres and they were water sprayed only. Mechanical properties of these fibres at room temperature are reported elsewhere [21]. The chemicals used in this project were purchased from Sigma Aldrich and included NaOH pellets, LiOH powder, KOH flakes (all at commercial grade), and standard 37% concentrated hydrochloric acid (HCl).

2.2. Thermal treatment

Fibre bundles were arranged in a steel rig for thermal conditioning, which was carried out in air. A Carbolite furnace was used to treat the fibres at 450 °C for 25 min, as these conditions were severe enough to result in the amount of strength loss representative to that of fibres recycled from pyrolysis and thermal oxidative processes. The rig was then extracted from the furnace and left to cool at room temperature, before fibre bundles were removed and treated in various alkaline solutions. Further details of the heat conditioning procedure are given in [6,7].

2.3. Alkaline treatment

NaOH, LiOH and KOH solutions were prepared according to the following molarities: 1.5, 2, 3 and 5 M. Solutions were heated to 95 °C before treating the fibre bundles. The standard treatment duration was 10 min; however this was varied in a subsequent experiment (at 2, 5, 20 and 30 min) to investigate its effect on fibre properties. After fibres were treated in alkaline solution, they were rinsed in 5% HCl solution for 7 min followed by rinsing with deionised water for 1 min. The purpose of this rinsing procedure was to ensure the effective removal of residual deposits which developed on the fibre surface as a result of interaction with alkaline solution [15]. In addition, previous experimentation did not show any significant change in mechanical properties of glass fibres after a short period of acid rinsing alone. Once the fibres were rinsed after alkaline treatment, they were dried out in an oven at 110 °C for 15 min.

2.4. Single fibre tensile testing

Single fibre tensile testing was performed following the standard ASTM C1557-03. After heat and chemical treatment, glass filaments were carefully separated from the bundle and mounted on card tabs with the central window matching the desired gauge length of 20 mm. Fibre diameters were measured using an optical microscope before testing for tensile strength using a Testometric tensile testing machine at ambient environment. The load cell was 5 N with a strain rate of 1.5%/min applied to the samples, and at least 30 samples were tested for each condition; the strength values were then averaged to give the average strength. Error bars associated with the strength measurements represent 95% confidence limits. The tensile test procedure is described in detail elsewhere [21].

2.5. Scanning electron microscopy (SEM)

A HITACHI SU-6600 field emission scanning electron microscope (FE-SEM), equipped with an energy dispersive X-ray spectrometer (EDS), was used for surface morphology and compositional analysis of the fibres following chemical treatment. Samples were coated in gold using an Edwards S150 sputter coater in order to prevent charge build-up since glass fibres are non-conductive. Images were captured at an accelerating voltage of 15 kV and extraction voltage of 1.8 kV.

2.6. Atomic force microscopy (AFM)

A Bruker Innova atomic force microscope was used for analysing the roughness of fibres following alkaline treatment. Tapping mode was used with a visible apex Si tip that had a mean resonance frequency of 70 kHz and a low spring constant (2 N/m) ideal for fibrous samples. AFM images were acquired at 128 × 128 pixel resolution and a low scan rate (0.1 Hz). For each treatment condition three individual fibres were selected at random and mounted on a metal plate. Two areas of each fibre were scanned in a 3 × 3 μm region. Height and tapping phase images were flattened to remove curvature by using the 'Flatten' function in NanoScope Analysis at 2nd order, and roughness values were measured and plotted as a function of treatment time.