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Influence of thermal conditioning on tensile behaviour of single basalt fibres

Fabrizio Sarasini*, Jacopo Tirillò, Maria Carolina Seghini

Department of Chemical Engineering Materials Environment & UdR INSTM, Sapienza-Università di Roma, Via Eudossiana 18, 00184 Roma, Italy

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ABSTRACT

This article presents an experimental investigation of the effects of temperature and atmosphere on the tensile behaviour of basalt fibres. The heating conditions have been chosen in order to mimic those used in thermal recycling of polymer matrix composites. The change of properties is investigated at room temperature on fibres heat-treated for 1 h up to 600 °C in air and in inert atmosphere (argon). The loss in fibre strength was found to be affected by both temperature and atmosphere with a significant strength loss occurring under the heating conditions used for high temperature incineration of polymer composites. Scanning electron images of fibre fracture surfaces after tensile tests for different environments and temperatures confirmed that failure originated from the fibre surface. The modulus of thermally-treated basalt fibres increased with conditioning temperature and these effects have been discussed in terms of decomposition of the organic sizing and structural relaxation during thermal treatment while X-ray diffraction excluded the effect of crystallization phenomena on the strength loss of basalt fibres after thermal exposure.

1. Introduction

According to a recent market report by AVK [1], Europe's production volume in glass fibre reinforced plastics (GRPs) reached 1.069 megatonnes in 2015, representing the highest level in eight years. Considering the world market, glass fibre reinforced thermoset composites account for about 90% of all the composites currently produced and these are traditionally directed to landfill at their end of life, mainly because recycling operations are often difficult and expensive [2]. It is to be emphasized that despite such technical and economical barriers, there is a growing environmental pressure to ban or at least limit the disposal of waste composites via land fill. It is therefore not surprising that the growth of a plethora of methods to recycle composites was triggered over the last years [3–6]. These methods can be classified as mechanical, thermal (pyrolysis, fluidised bed pyrolysis, microwaves assisted pyrolysis) and chemical (solvolysis) processes [7]. Pyrolysis is likely the most preferred technique to recycle composites, in particular carbon fibre reinforced composites, with the objective to reclaim fibres that represent the most valuable part of the waste. This process allows the recovery of carbon fibres largely maintaining their reinforcement capability, whilst glass fibres are quite damaged. In particular, the tensile strength is usually reduced to a great extent (~50–80%) [2,8–13] and this aspect seriously affects their potential reuse as fibrous reinforcement in new structural composite materials. Pyrolysis basically involves the matrix degradation at high temperature (typically 400–550 °C) in the absence of air to produce oil, gases and solid products (fibres, fillers and char). The fibres are usually contaminated by this char and require a post-treatment in air in a furnace at least at 450 °C to oxidize the residual char and remove surface contamination. The mechanisms responsible for the loss in fibre strength represent a much debated issue in literature and two main mechanisms have been proposed: (i) thermally-activated changes to the anisotropic silica network structure initially created by the high drawing stress during fibre manufacture [12–15] and (ii) thermally-induced diffusion of water into the fibre surface leading to increased surface area and larger micropores [11,16,17]. In an attempt to shed light on this important issue, recently Feih et al. [2] compared the tensile strength of heat-treated glass fibres with that of fibres containing a single nano-sized surface flaw of controlled size artificially created by focussed ion beam (FIB) milling. The authors concluded that bulk changes (such as a change in fracture toughness or fibre modulus) influencing the stress–flaw relationship for

* Corresponding author.
E-mail address: fabrizio.sarasini@uniroma1.it (F. Sarasini).

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E-glass fibres do not occur during thermal recycling and therefore the reduction in fibre tensile strength during thermal conditioning can be attributed solely to thermally-activated growth of surface flaws. This growth, according to the authors, is ascribed to the diffusion of water molecules in the glass fibre structure and their subsequent reaction with stressed siloxane bonds at the crack tip.

In an attempt to reduce the environmental impact of synthetic polymers and reinforcements, there has been a resurgent interest in the use of natural fibres in polymer matrices [18]. Many types of natural fibres like sisal, kenaf, hemp, flax, jute have been studied and applied but vegetal fibres are very sensitive to thermal and hygroscopic loads and show limited and variable mechanical properties due to the plant growing and harvesting conditions, the fibre extraction system, the variable fibre shape and geometry. Another drawback is the chemical incompatibility with many hydrophobic polymer matrices which results in a low fibre/matrix interface strength and limited stress transfer efficiency. A possible solution that takes into account the environmental issues is represented by the use of natural fibres but of mineral origin, like basalt [19]. The basalt fibre is an environmentally friendly material characterized by excellent sound insulation properties, resistance to heat greater than that offered by glass fibres and a good chemical inertia [20]. With regard to the mechanical properties, basalt fibres compare quite favourable with traditional E-glass fibres, at the expense of a slightly higher density [20,21]. Their suitability as reinforcement of both thermoplastic and thermoset matrices has been well documented in literature [22–30], even if no studies detailing the behaviour of such composites at their end of life can be found. Basalt fibre is superior to E-glass for high operating temperature limit (~650 °C for basalt compared to ~460 °C for E-glass), and higher softening temperature (1050 °C for basalt and 600 °C for E-glass). This often claimed higher thermal stability and resistance of basalt fibres compared to glass ones could provide in principle basalt fibres with better prospects to survive a pyrolysis treatment, thus having the chance to be re-used in valuable products. Despite the potential high temperature applications of basalt fibres, their mechanical properties retention after a thermal exposure is not known in detail. In particular, it is not completely understood whether the higher operating and softening temperatures of basalt fibres result into superior tensile strength retention properties after a thermal treatment.

While the strength loss of E-glass fibres due to exposure to high temperature has been well documented in literature, less information are available for basalt fibres. Most of these available papers addressed the thermal response of basalt tows (bundles) [31–34]. Bhat et al. [34] tested at room temperature basalt tows previously heated to temperatures ranging from 150 to 650 °C for times up to two hours and compared the results with those obtained for glass tows in the same experimental conditions. The authors found that the failure load of both tows began to decrease after exposure to 250 °C and the reduction was much more severe with increasing temperature up to 650 °C. They concluded that there were no significant differences in strength loss between basalt and glass tows. Kessler et al. [35] performed tensile tests with impregnated rovings. Before being impregnated, basalt rovings were maintained for two hours at temperatures between 100 °C and 600 °C in air. Up to 300 °C the decrease in tensile strength was limited but between 300 °C and 400 °C the tensile strength of impregnated basalt rovings was found to decrease in a significant way, exhibiting at 600 °C a lower absolute tensile strength compared to E-glass rovings. Usually the onset of crystallization phenomena is reported to be the cause of such behaviour of basalt fibres after thermal exposure [32,33,35]. Recently Jenkins et al. [36] performed a detailed experimental campaign aimed at comparing the tensile strength retention of single basalt and E-glass fibres after high temperature exposure. They found a good strength retention following a heat treatment at 300 °C (for 20 min) but a sharp decrease at higher temperatures for both fibres with strength losses of 65% and 80% for glass and basalt fibres, respectively, after a thermal exposure at 600 °C. It is to be noted that differences in tow strength could be likely due the sizing and related friction effects between fibres during testing rather than the single fibre strength. In addition, differences in sizing between different fibres can definitely play a role in determining the final mechanical properties of single fibres. In view of all these issues, the aim of the present work is to study the effect of thermal recycling processing conditions on the properties of single basalt fibres coated with two different sizings, one optimized for thermoset resins (epoxy) and the other one optimized for thermoplastics (polypropylene). The variation of the mechanical behaviour was determined following treatments at different temperatures (200–600 °C) for a total heating time of 1 h and different furnace atmospheres (air and argon). These conditions were chosen in order to simulate those used in commercial thermal recycling processes. The data were analyzed using Weibull analysis with a detailed morphological investigation of the fracture surfaces. The possible mechanisms responsible for basalt fibre strength loss with increasing temperature are discussed taking into account thermogravimetric analysis, X-ray diffraction analysis and density measurements. In this paper, for the first time, a systematic study of the effect of temperature and furnace atmosphere on the strength retention of commercially available basalt fibres was performed. The outcomes from this study provide insight into the strength loss mechanism of basalt fibres during thermal recycling, which can be used to design further experimental tests needed to resolve the causes for strength degradation.

2. Materials and methods

2.1. Raw materials

The effects of temperature and atmosphere on fibre properties were investigated using commercial grades of basalt fibres but differently sized:

- basalt fibres supplied by Kamenny Vek as a continuous roving with a nominal fibre diameter of 13 μm, linear density of 1200 tex and sized with a commercial epoxy resin compatible sizing (BCF13);
- basalt fibres supplied by Basaltex as a continuous roving with a nominal fibre diameter of 13 μm, linear density of 600 tex and sized with a commercial polypropylene compatible sizing (BCF13-PP).

2.2. Fibre heat treatment

Fibre bundles were heat treated as-received in a tube furnace (Lenton Thermal Designs Ltd) with controllable temperature and atmosphere. The effect of the furnace atmosphere was assessed by heating the fibres in high purity argon and ambient air. All fibres are thermally treated at temperatures of 200, 300, 400, 500 and 600 °C in air for 1 h, and then removed from the furnace and allowed to cool in room temperature air. The treatment in argon was performed on both fibres but at temperatures of 400, 500 and 600 °C for a duration of one hour. The argon atmosphere was chosen to replicate the initial stage of the thermal recycling process of composites when heating is performed under inert conditions in order to decompose the matrix. The ambient air reproduces the second stage when the fibres are heat-treated in air to remove residual char and contamination from the surface. The lower temperature