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Lund, Majbritt; Yue, Yuanzheng

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Influences of thermal and chemical ageing on the surface morphology and crystallization behaviour of basaltic glass fibre

Majbritt Lund[a] *and Yuanzheng Yue*[b]
[a] Aalborg University, Section of Chemistry, Sohngaardsholmsvej 57, Aalborg, Denmark. Fax: +45 9635 0558; Tel: +45 96357246; E-mail: ml@bio.aau.dk
[b] Aalborg University, Section of Chemistry, Sohngaardsholmsvej 57, Aalborg, Denmark. Fax: +45 9635 0558; Tel: +45 9635 8522; E-mail: yy@bio.aau.dk

ABSTRACT: Basaltic glass fibres exposed to thermal and chemical ageing have been studied using calorimetry, scanning electron microscopy, and atomic force microscopy concerning the impact of the ageing processes on the morphology, relaxation, and crystallization of fibre surfaces.

Introduction

Mineral glass fibre products have widespread applications in the modern world, including heat and sound insulation and reinforcements in composite materials. The application possibilities of the glass fibres are determined by their chemical and mechanical properties. Basalt-like fibres have excellent heat and sound insulation properties and such wool is used for insulation products. The thermal and mechanical performance of the wool is affected by the interactions between the fibres and the surrounding media, i.e. humidity, temperature field, and atmosphere. Wool products have large specific surface areas, and interactions between glass fibre surfaces and their surroundings have not yet been clarified, including possible mechanisms and relations to mechanical degradation.
This study aims to provide experimental information about interactions between fibre surfaces and their surrounding media, which are expected to be crucial factors for mechanical and chemical properties. To do so, ageing experiments have been performed that include chemical ageing (i.e. fibres exposed to humidity or immersed in water), and thermal ageing (i.e. annealing at various temperatures between 250°C and the glass transition temperature [T_g]).

Experimental

Samples analyzed in the present work are basaltic fibres produced by cascade spinning [1], with an averaged fibre diameter of 4-6μm. Two types of basaltic fibres are investigated: pure basaltic fibres and Rockwool HT fibres. The Rockwool fibres are high temperature heat resistant and biosoluble [2], and spun from a melt produced under reducing conditions. Both samples are exposed to similar ageing conditions.
Humidity ageing of the samples was performed in a climatic chamber of controlled 95% relative humidity and 70˚C, ageing was also performed immersed in water at 70˚C. Thermal ageing was performed in an annealing furnace in atmospheric air at various temperatures between 250°C and T_g.
Differential scanning calorimetry (DSC) and thermogravimetry (TG) measurements were performed in a NETZSCH STA 449C calorimeter, at a rate of 20˚C/min and under argon atmosphere. SEM analyses were performed in a Leo-1550, Gemini at low accelerating voltage to avoid conductive coating of the fibres prior to measurements.

Results and Discussion

Chemical attack in the form of water adsorption on fibre surfaces may be concentrated at defects or micro-cracks [3], or at highly strained chemical bonds [i.e. 4, 5]. Surface appearances are highly important clues for mechanical and thermal performances of the glass fibres.

Surface appearances in SEM

Distinct differences in surface appearances of humidity and water aged Rockwool fibres are observed in SEM images of the analyzed samples. In the resolution of the SEM the biosoluble Rockwool samples show no indication of apparent surface changes due to humidity exposure; whereas samples immersed in water showed pronounced formation of particles/leaching products at the surface after prolonged ageing times (figure 1).
AFM analyses of the pristine and aged fibre surfaces are in progress to evaluate the surface morphology at a higher resolution as well as characterization of the surface defects present.

Crystallization behaviour

Fibre surfaces and glass structure exposed to thermal and chemical ageing may over time change the crystallization behaviour of the basaltic glasses. Studies of crystallization and oxidation of basaltic bulk glass [6] show difference in crystallization temperatures for surface crystallization and bulk crystallization. No such evidence is found in DSC data of this study. The present study shows pronounced changes in the crystallization behaviour due to humidity and water exposure, which are reflected by an increase of the crystallization temperature and a decrease in crystallization enthalpy upon prolonged ageing times (see figure 2). The

increase in crystallization temperature is more evident in samples aged in water than samples exposed to humidity at similar temperatures. X-ray diffraction of crystallized humidity samples shows the presence of phases of pyroxene and feldspar [7].

Upon thermal ageing at T_g for one hour, the crystallization behaviour of the samples significantly changes in the manner that two enthalpy peaks occurs on the DSC upscan curve, in stead of only one peak as observed in upscans of non aged samples and the sample aged at 350°C. This indicates that two crystallization events occur in the samples aged at T_g.

A previous study [8] on thermally aged basaltic fibres containing only Fe^{2+} ions shows changes in oxidation of Fe^{2+} at ageing temperatures above $0.8 \cdot T_g$, but no changes in the oxidation of Fe^{2+} below this temperature. Thermal ageing in the present project is performed both above and below "Fe-oxidation temperature". The observed difference in crystallization behaviour of these samples may be attributed to oxidation or recrystallization phenomena at the surface. Thermal ageing below $0.8 \cdot T_g$ reduces the crystallization enthalpy, the crystallization temperature falls in between the two enthalpy peaks produced upon crystallization of thermal aged samples at T_g (figure 2). This implies that thermal ageing affects the crystallization of Fe^{2+} rich basaltic fibres measured by DSC at heating rates of 20°C/min.

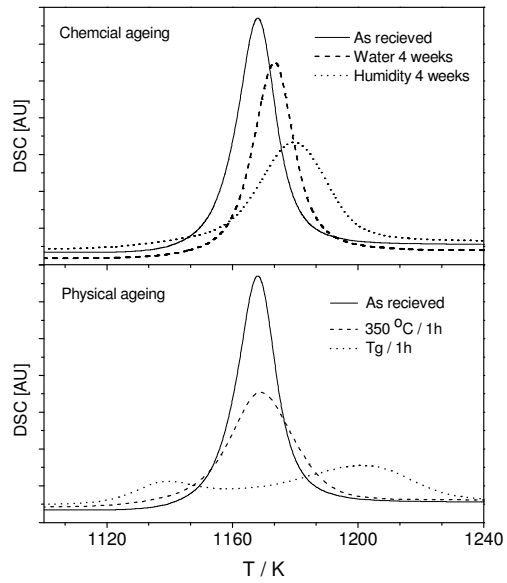

Figure 2. Crystallization behaviour measured during DSC upscans at 20°C/min. a) Chemical ageing; in humidity and immersed in water, and b) thermal ageing; heat treatment in air.

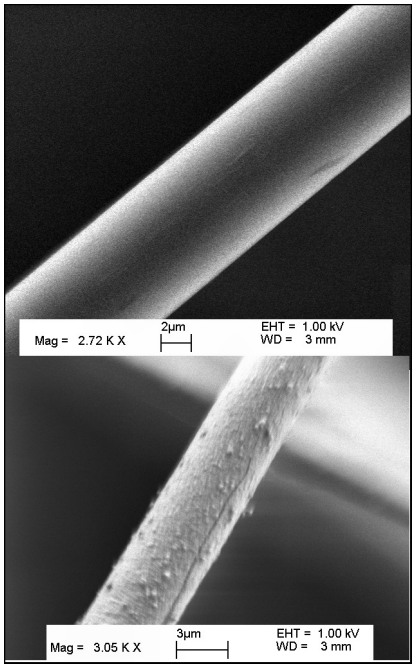

Figure 1. SEM images of basalt like fibres as received from cascade spinning and aged immersed in water.

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