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## Impact of fiberizing method on physical properties of glass wool fibers

Yanfei Zhang<sup>a</sup>, Yuri Vulfson<sup>b</sup>, Qiuju Zheng<sup>a</sup>, Jiawei Luo<sup>c</sup>, Seong H. Kim<sup>c</sup>, Yuanzheng Yue<sup>a,d,\*</sup><sup>a</sup> School of Materials Science and Engineering, Qilu University of Technology, Jinan 250353, China<sup>b</sup> Hollingsworth and Vose Company, Corvallis, OR 97333, USA<sup>c</sup> Department of Chemical Engineering, Materials Research Institute, Pennsylvania State University, University Park, PA 16802, USA<sup>d</sup> Department of Chemistry and Bioscience, Aalborg University, 9220 Aalborg, Denmark

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## ABSTRACT

The physical properties of the glass fibers and their woven and non-woven products are affected by many factors, such as glass composition, thermal history, melting conditions, and fiberizing method. In this work, we have investigated the impact of fiberizing method on dynamic properties and relaxation behavior of the glass wool fibers, and inferred its effect on the mechanical properties of the filtration mats consisting of glass wool fibers. We have fabricated the glass wool fibers using the rotary (R) and flame (F) attenuation processes, which are denominated as R- and F- fibers, respectively. Both fibers have the same chemical composition and hence similar structural features, surface quality, glass transition temperature ( $T_g$ ), and sub- $T_g$  enthalpy relaxation behaviors. However, two fibers show striking differences in the surface hydroxyl (OH) content, fictive temperature ( $T_f$ ) and cooling rate ( $q_c$ ). F-fibers have weakly hydrogen-bonded surface hydroxy (OH) groups, while R-fibers have free OH groups and strongly hydrogen-bonded OH groups. F-fibers have higher  $T_f$  and  $q_c$  than R-fibers. We argue that the higher tensile strength of the F-fiber mat (compared to the R-fiber mat) could be attributed to both the weaker hydrogen-bonded surface OH groups, and the higher  $T_f$  and  $q_c$  values that may increase the tensile strength of fiber itself. This work gives guidance for designing the fiber mat products with high tensile strength.

## 1. Introduction

Glass fibers have wide applications such as filtration mat, fiber communications, fiber-reinforced composites, battery separator, thermal and sound insulation [1–6]. Recently, the fibrous mat for high-efficiency fuel filtration has attracted attentions [7–9]. The nanofibers used in membranes for environmental remediation provide high efficiency than conventional fiber mats [7]. In addition, the nanofibrous filters exhibit high efficiency in filtering large particles and causes a moderate rise in the pressure drop [8]. The fibers play an important role in removing the contaminants of air and liquid via fiber entanglement and interlocking [2–4].

The properties of glass fibers are influenced by chemical composition, thermal history, and fiberizing conditions [1,3,10–12]. For instance, three types of filtration mat, which are composed of B-glass fibers, E-glass fibers and a mixture of E and B glass fibers, respectively, exhibit different performances, e.g., in affecting the exiting air concentration, the mat flexibility and the ability to maintain the mat hardness over longer periods of time after exposure to oil [3]. In the fabricating process of wet-laid mat, it is found that beating the flame attenuated glass wool in water at 8000 revolutions benefits increasing

both the uniformity of glass wool suspensions and the tensile strength [13].

The properties of filtration mat are, to a large extent, determined by those of glass fibers. Glass fibers are usually drawn or spun from a homogeneous glass-forming melt using different fiberizing methods, e.g., continuous fiber drawing [1], glass preform drawing [14], extrusion drawing [15], and centrifugation fiber spinning [16]. In this study, we draw silicate glass fibers by using both rotary and flame attenuation processes, respectively. The chemical composition of the glass fibers and the details of the fiberizing processes are given in the experimental procedure (Section 2.1). The fibers drawn by both rotary process and flame attenuation process are denominated as R- and F-fibers, respectively. Both kinds of fibers are used to manufacture filtration mats. Despite same fiber chemical composition and same mat-making condition, the F-fiber mat has approximately double tensile strength of R-fiber mat.

In this work, we characterize the differences in structure, surface quality, thermal history, and relaxation behavior between F- and R-fibers and thereby infer the possible reasons of the tensile strength difference between the two fiber mats. To the best of our knowledge, there are relatively few studies addressing the impact of fiberizing method on

\* Corresponding author at: Department of Chemistry and Bioscience, Aalborg University, 9220 Aalborg, Denmark.  
E-mail address: [yy@bio.aau.dk](mailto:yy@bio.aau.dk) (Y. Yue).

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physical properties of glass wool fibers. Here, we found significant differences in the surface hydroxyl (OH) groups, fictive temperature ( $T_f$ ) and cooling rate ( $q_c$ ) between the two kinds of fibers. The results of this study will be important for optimizing the wool fiber spinning process to increase the tensile strength of the fiber mats for oil and gas filtration.

## 2. Experimental procedure

### 2.1. Fiber drawing

The glass fibers used in this work were provided by Hollingsworth & Vose Company, which were drawn by two processes, i.e., rotary and flame attenuation processes, respectively. The derived fibers are here denoted as R- and F-fibers. Both fibers have the same nominal composition (mol%): 63.6 SiO<sub>2</sub>, 5.6 Al<sub>2</sub>O<sub>3</sub>, 5.6 CaO, 3.8 B<sub>2</sub>O<sub>3</sub>, 15.1 Na<sub>2</sub>O, 1.1 K<sub>2</sub>O, 3.8 MgO and 1.4 F<sub>2</sub>. In rotary process, a stream of glass melt, which was stabilized at about 1428 K, falls into a rotating spinner. The rotating spinner is a metal bowl with perforated sidewalls. The glass melt then was extruded through sidewall's tiny holes (diameter: about 50 μm) into many streams. Subsequently, the streams were attenuated by a combination of centrifugal forces and aerodynamic drag forces, and then cooled to form fibers much thinner (about 3 μm) than the extrusion holes [16]. In parallel, the flame attenuation process for manufacturing glass fibers is a two-step process. At first, the stabilized viscous silicate melt at about 1428 K flowing out through many bushing in the melting pot is drawn into a suite of coarse primary filament. Then the coarse filament is remelted and attenuated into many fine fibers with a high temperature gas flame, normally impinging at right angles to the primary filament [16]. Both R- and F-fiber samples were spun from the same melt composition.

### 2.2. Fiber characterization

The filtration mat is generally made using paper manufacturing technology [17], which is regarded as the most prominent preparation technique of nonwoven mat. The pulp is made by mixing glass fibers, water and sulfuric acid and then the mat is produced upon drying and pressing the pulp. The tensile strength of the two filtration mats made from R- and F-fibers were measured by a Universal Materials Tensile Tester. The tensile strength data cannot be shown here since it refers to the company confidential information.

To confirm the amorphous nature of the studied R- and F-fibers, X-ray diffraction (XRD) characterizations were performed using an X-ray diffractometer (Shimadzu LabX XRD-6100) with CuKα radiation at room temperature. A scanning electron microscope (SEM) (FEI Quanta 200) was used to observe the surface morphology of the fibers.

In order to compare the distribution of hydroxyl groups at the R- and F-fiber surfaces, vibrational sum frequency generation (SFG) spectroscopy was employed. The details of experimental setup of SFG spectrometer were described elsewhere [18]. To maximize the signal intensity, fibers were pressed into a flat sheet with ~1 mm thickness and the scattered SFG signals were collimated using a pair of lenses. The polarization combination was *s* for SFG signal, *s* for 532 nm laser pulses, and *p* for IR pulses (*ssp*). The visible and IR pulse energies at the samples were 120 and 136–220 μJ, respectively. The IR frequency was calibrated with SFG spectra of DMSO/air interface in *ssp* polarization combination. All the spectra were collected at room temperature with a relative humidity (RH) of ~30%.

Viscosity measurements were performed to obtain the viscosity-temperature relation and the melt fragility index (*m*) [19] of the studied glass. The high temperature viscosities (10–10<sup>4</sup> Pa s) were measured using a concentric cylinder viscometry (model SRV-1600, Orton Company, USA) under atmospheric condition. The standard glass NIST SRM 717A was used for calibration of the viscometers [20]. The error ranges for the high temperature viscosity measurements was  $\Delta \log \eta = \pm 0.02$

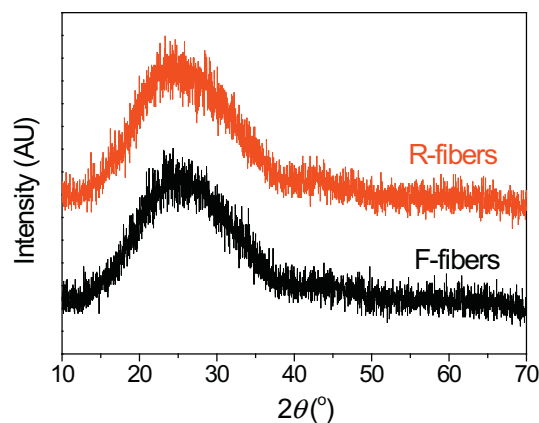


Fig. 1. XRD patterns for R- (red curve) and F- (black curve) glass fibers. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

( $\eta$  in Pa s). The low temperature viscosities between 10<sup>10</sup> and 10<sup>14</sup> Pa s were measured using beam bending viscometer (Orton, USA). The errors for the high viscosity measurements were found to be within  $\pm 1\%$ .

The standard glass transition temperature ( $T_g$ ) and the isobaric heat capacity ( $C_p$ ) for both fibers were measured using a differential scanning calorimeter (DSC) (Netzsch Jupiter STA 449F3, Selb, Germany) at the upscan rate of 10 K/min equal to the prior downscan rate in N<sub>2</sub> atmosphere [21–23]. For oxide glasses, the standard calorimetric  $T_g$  corresponds to the shear viscosity of 10<sup>12</sup> Pa s, at which the average relaxation time  $\tau$  is about 30–40 s according to the Maxwell relation  $\tau = \eta/G_\infty$ , where  $G_\infty$  is the unrelaxed shear modulus and is about 25–35 GPa at  $T_g$  for oxide liquids [24]. In order to compare the sub- $T_g$  enthalpy relaxation behavior between R- and F-fibers, the as-drawn fibers were annealed at a specific temperature ( $T_a$ ) below  $T_g$  for various durations ( $t_a$ ). Isothermal annealing was performed in air atmosphere in a muffle furnace.

## 3. Results

The XRD patterns obtained for R- and F-glass fibers are depicted in Fig. 1. It can be seen that the XRD curves of the two samples exhibit almost the same broad hump in the  $2\theta$  range from 12 to 38°, confirming that R- and F-fibers were in the glassy state.

Fig. 2. shows the SFG spectra of R- and F-fibers, respectively. Two fibers show distinct OH stretch peaks in SFG. Because it is a nonlinear optical process, SFG is not sensitive to bulk OH groups randomly distributed in the fiber, but highly sensitive to the OH groups at the surface [25]. The SFG spectra shown in Fig. 2 indicate that the OH groups exposed at the fiber surface can be drastically different or affected by the fiberizing process, i.e., rotary and flame attenuation processes. The C–H stretch peaks in the 2800–3000 cm<sup>-1</sup> region originate from adventitious organic species.

In the vibrational spectroscopy literature, it is well known that the free OH groups without hydrogen bond interactions with surrounding molecules appears at  $\geq 3650$  cm<sup>-1</sup> with a narrow band width [26,27]. As the hydrogen-bonding interaction increases, the stretch band position is red-shifted; the broadness of the OH stretch band reflects the distribution of hydrogen bonding dynamics and strength [28–30]. Thus, the two sharp peaks observed at 3620 cm<sup>-1</sup> and 3700 cm<sup>-1</sup> for R-fibers can be assigned as free SiOH or free OH of physisorbed H<sub>2</sub>O. Since the SFG spectra were taken in humid air (30% RH), there must be physisorbed water layers on the fiber surface. Thus, all free OH groups accessible by the gas phase water molecules must be involved in hydrogen bonding with physisorbed water molecules. The presence of prominent free OH peaks implies that a large fraction of OH groups are

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