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Preparation and characterization of continuous basalt fibre with high tensile strength

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ABSTRACT

Continuous basalt fibres (CBFs) produced directly from natural basalt seldom achieve adequate performance with high tensile strengths. Thus, formulation optimization is a key component of technological innovation in the field of CBFs. From this perspective, a series of experiments were designed to prepare a type of CBF using two natural forms of basalt. The average tensile strength of the fabricated CBF was 4111 MPa. Moreover, it is found that the amount of glass network modifier ($Na_2O + K_2O$) has a negative correlation with the tensile strength of CBF. By combining our results with past studies, two empirical formulas were proposed to quantify the relationship between viscosity (η) and viscosity modulus (Mη) for basalt melts at 1300 °C and 1400 °C: $\eta(1300\ ^\circ C) = -91.22971 + 16.06614e^{1.25983M\eta}$ and $\eta(1400\ ^\circ C) = -30.57462 + 6.32023\ e^{1.18491\ M\eta}$. It was found that a value of Mη ranging between 2.2 and 2.6 is optimum for CBF production. We also established an important correlation between η, Mη and mix compositions, which could be an essential criterion to evaluate the characteristics and production technology of CBF, including basalt beneficiation and formulation optimization.

1. Introduction

Continuous basalt fibres (CBFs) have – for several years – been recognized for their potential as environmentally friendly fibres with the advantages of temperature and combustion resistance, heat insulation, high chemical stability and high tensile strength [1–7]. CBFs was developed firstly by Moscow Research Institute of Glass and Plastic, in 1953, but industrial production did not begin till 1985 [8]. Since then, CBFs have been widely used as reinforcing materials in the fields of transportation, aerospace and the military [9–12].

Basalt is widely distributed across the Earth's surface and in oceanic crusts, which is the primary source of raw materials for CBF production [13]. Specifically, the preparation process for CBF includes the following steps. (1) The beneficiation of basalt for available chemical compositions; (2) melt preparation within the temperature range of 1350°C-1550 °C; (3) maintaining the temperature for a long period to ensure better melt homogeneity; (4) allowing the melt with an appropriate viscosity to flow through a rhodium–platinum bushing to generate raw filaments [14,15]. It is worth noting that the production technology used and the final product quality of CBFs are strongly dependent on the basalt composition and the viscosity of the melt. From this perspective, Osnos et al. proposed a standard for basalt compositions corresponding to CBF production [16], i.e. SiO_2: 45–60 wt%, Al_2O_3: 12–19 wt%, Fe_2O_3+FeO: 7–18 wt%, MgO: 3–7 wt%, CaO: 6–15 wt%, $Na_2O + K_2O$: 2.5–6 wt% and TiO_2: 0.9–2 wt%. Further, the viscosity and crystalline properties are believed to be the most important evaluation criteria when selecting the production technology for CBFs. As Makhova et al. (1984) pointed out, a basalt melt with a medium viscosity of 180–310 dPa s at 1300 °C has the optimum parameters for CBF production, while the upper limit of the crystallization temperature is lower than 1290 °C [17,18]. In this case, the fibre-forming temperature is set to be higher than the upper limit for crystallization temperature; otherwise, the melt could crystallize when fibre drawing is conducted [2]. Further, rapid crystallization has an adverse effect on

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Table 1
Chemical composition (wt%), Viscosity modulus (Mη) Acidity modulus (Mk) and of samples.

	SiO_2	Al_2O_3	Fe_2O_3	K_2O	Na_2O	CaO	MgO	TiO_2	Mη	Mk
AH20	58.07	13.63	9.77	0.81	3.94	1.21	1.78	4.41	4.60	23.98
AH18	46.91	12.85	12.84	0.72	2.33	8.79	4.06	3.78	2.17	4.65

production technology, lowering the mechanical strength or even causing breakage of the fibre. Since CBF production has requirements on chemical composition, viscosity, fibre forming temperature and upper limit of the crystallization temperature, the basalt mine suitable for CBF production is rare.

Altering the chemical composition of basalt through extra oxides (such as SiO_2 and Al_2O_3) has proven to be effective in optimizing the production technology for CBFs. However, this also leads to some new problems: SiO_2 and Al_2O_3 have higher melting temperatures than that of basalt (usually <1550 °C). Therefore, the diffusion of oxide solid phases is relatively difficult owing to the high viscosity of the basalt melt, making the process time-consuming and energy-intensive. If the chemical composition of the melt is not sufficiently homogeneous, the fibre-drawing process and mechanical properties are affected negatively. Further, the oxide additives could introduce a large number of bubbles into the melt, which would result in fibre breakage –a significant amount of time is required to eliminate the bubbles at higher melting temperatures. Finally, it also causes a significant increase in the production cost of CBFs.

To mitigate the existing limitations in CBF production, the formulation-optimization method is proposed. In this method, different basalts – excluding oxide additives – are assigned to adjust the chemical composition of the precursor to a reasonable range. This study verified the feasibility and practicality of the method, and subsequently established a relationship between viscosity (η) and viscosity modulus (Mη) as a function of the chemical composition of the precursor. These findings provide a precise characterization and quantitative analysis for the evaluation of the mechanical properties and production technology for CBF.

2. Experiment

2.1. Starting materials

Two types of basalt obtained from different layers of the same mine in Guizhou province, China, were chosen as samples (sample number: AH18 and AH20). The chemical compositions of the samples were determined by XRF analysis, as presented in Table 1. According to the total alkali-silica classification method (recommended by the International Union of Geological Sciences Subcommission on the Systematics of Igneous Rocks), AH18 and AH20 are designated as basalt and andesite, respectively. AH18 and AH20 are not individually suitable for fabricating CBF because of the obvious mismatch between their compositions and viscosities based on trial experimental results. AH18 has a low viscosity in the range of The fibre-forming temperature, which caused the melt to stream down quickly before it solidified into a fibre. In contrast, the high viscosity of AH20 made the fibre-drawing process difficult and breakage occurred frequently.

2.2. Fibre-drawing performance estimation

Two important parameters – other than the chemical composition – are used to evaluate the type of basalt suitable for CBF production: the viscosity modulus (Mη) and acidity modulus (Mk).

The viscosity modulus (Mη) is defined as:

$$M\eta = \frac{M_{SiO2} + 2M_{Al2O3}}{2M_{Fe2O3} + M_{FeO} + M_{CaO} + M_{MgO} + M_{K2O} + M_{Na2O}} \quad (1)$$

Where M_{SiO2}, M_{Al2O3}, etc., are the molar fractions of the oxides. The Mη limit for CBF production is ≥ 1.5, as reported by Aslanova in a patent [19], according to the finding of this work, the limit of Mη for CBF production should be 2.2–2.6. The Mη of AH20 and AH18 are 4.6 and 2.17 (Table 1), respectively, which are out of the recommended range.

The acidity modulus (Mk) is defined as:

$$Mk = \frac{W_{SiO2} + W_{Al2O3}}{W_{CaO} + W_{MgO}} \quad (2)$$

Where W_{SiO2}, W_{Al2O3}, etc., are the mass fractions of the oxides. Mk denotes the ratio of acidic to basic oxides. The optimal chemical composition for CBF production is related to an Mk of 3–6 [20,21]. The Mk of AH20 and AH18 are 23.98 and 4.65 (Table 1), respectively, which are far beyond the recommended range.

2.3. Formulation-optimization model for CBFs

To fabricate high-tensile-strength CBFs, a formulation-optimization model was built using the following steps: choose the dosages (Xi) of each type of basalt as variables, set the minimum difference (MIN) between the calculated value (Ci) and the target value (Ti) of each oxide as the objective function, set the suitable ranges for the viscosity modulus (Mη) and acidity modulus (Mk), etc., as constraints. Finally, a mathematical model for CBF can be proposed, as follows:

$$\begin{cases} \mathrm{MIN}\ \ SiO_2 = |C1 - T1| \\ \ \ \mathrm{MIN}Al_2O_3 = |C2 - T2| \\ \ \ \mathrm{MIN}\ \ Fe_2O_3 = |C3 - T3| \\ \mathrm{MIN}CaO = |C4 - T4| \\ \mathrm{MIN}MgO = |C4 - T5| \\ \mathrm{MIN}K_2O = |C6 - T6| \\ \mathrm{MIN}Na_2O = |C7 - T7| \\ \mathrm{MIN}TiO_2 = |C8 - T8| \\ \ \ \sum_{i=1}^{m} Xi = 100 \\ 0 \le Xi \le 100, (i = 1, 2, \ldots, m) \\ |Ci–Ti| \le \alpha i, (i = 1, 2, \ldots 8) \\ \beta i \le Ci \le \gamma i, (i = 1, 2, \ldots 8) \\ \ \ 2.2 \le M\eta \le 2.6 \\ \ \ 3 \le Mk \le 6 \\ \ \ \vdots \end{cases} \quad (3)$$

The objective of the model is to determine the optimal mixing ratio for different types of basalt, which could ensure that the batch chemical composition is closest to the designed target values under constraint conditions. Owing to the nature of basalt ores, their chemical composition varies depending on the geographical location and source conditions. Thus, a formulation-optimization method is really necessary to solve this issue.

Data – such as raw-material chemical composition, target values and constraints – were fed into the model, and the optimization software LINGO was used to solve the model (details are presented in the supplementary material). Preliminary fibre drawing experiment had done on many basalt samples including AH18 and AH20 that were proved to be not suitable for CBF production alone, among the CBFs obtained previously, the chemical composition of the one which possess the highest tensile strength was chosen as the target values in this work. The calculation results including the mixing ratio, target values (T), and

Table 2
The results of optimizing calculation including mixing ratio, target values (T), calculated values (C) and difference between T and C.

	SiO_2	Al_2O_3	Fe_2O_3	K_2O	Na_2O	CaO	MgO	TiO_2	Mη	Mk	Mixing ratio(wt%)
AH20	58.07	13.63	9.77	0.81	3.94	1.21	1.78	4.41	4.60	23.98	16.94
AH18	46.91	12.85	12.84	0.72	2.33	8.79	4.06	3.78	2.17	4.65	83.06
Target value (T)	48.80	12.86	12.32	1.03	2.61	7.92	4.39	3.79	2.40	5.01	
Calculated value (C)	48.80	12.98	12.32	0.74	2.60	7.51	3.67	3.89	2.49	5.53	
Difference (C–T)	0.00	0.12	0.00	−0.29	−0.01	−0.41	−0.72	0.09	−0.09	0.52	

Fig. 1. Optical photograph of the monofilament (part).

calculated values (C); the differences between T and C are presented in Table 2. The optimal mixing composition under the current constraints is that AH18 and AH20 account for 16.94 wt% and 83.06 wt%, respectively. The calculated values for the oxides are close to their target values, which could be proved by the small difference between the calculated and target value. The viscosity modulus (Mη = 2.49) and acidity modulus (Mk = 5.53) of the mixture are within the limits for Mk (3–6) and Mη (2.2–2.6).

2.4. Preparation of the mixture and CBF-drawing experiment

The fibre-drawing experiment was carried out at Southeast University, China. According to the aforementioned mixing ratio for AH18 and AH20, 1000 g of mixture was prepared – composed of 169.4 g of AH20 and 830.6 g of AH18 – and crushed into particles. Then, the mixture was put into a platinum–rhodium crucible and heated up to 1500 °C for 10 h to ensure melt homogeneity. Subsequently, the as-prepared basalt melt was put into a home-made single-hole fibre-drawing crucible. By adjusting the fibre-drawing temperature, liquid level and the rotating speed, a basalt monofilament was continuously drawn out without any interruption or breakage. The fibre-drawing process lasted for 4 h without fracture. The fibre obtained is a monofilament which is golden and flexible, as shown in Fig. 1.

2.5. Characterization techniques

The phase identification of AH18, AH20 and the fibre product was conducted using powder XRD analysis and the JADE 6software. XRD patterns were collected using a Panalytical multifunction X-ray diffractometer (model: Empyrean), equipped with a 3D PIXcel detector. The XRD measurement was performed within the 2θ range of 4–70°, in continuous-scanning mode with a 0.026° step size and counting time of 30 s per step. The fibre was cut into segments, fixed in a sample holder and coated with a carbon layer for microstructural observation using a scanning electron microscope (SEM, model: JSM-6490LV, Japan). The tensile strength of the obtained CBF was determined using a fibre tensile tester (XQ-2, Shanghai New Fiber Instrument Co. Ltd., China) at Southeast University, China. The measurement method for the filament tensile strength was adopted from ISO11566:1996<Carbon fiber-

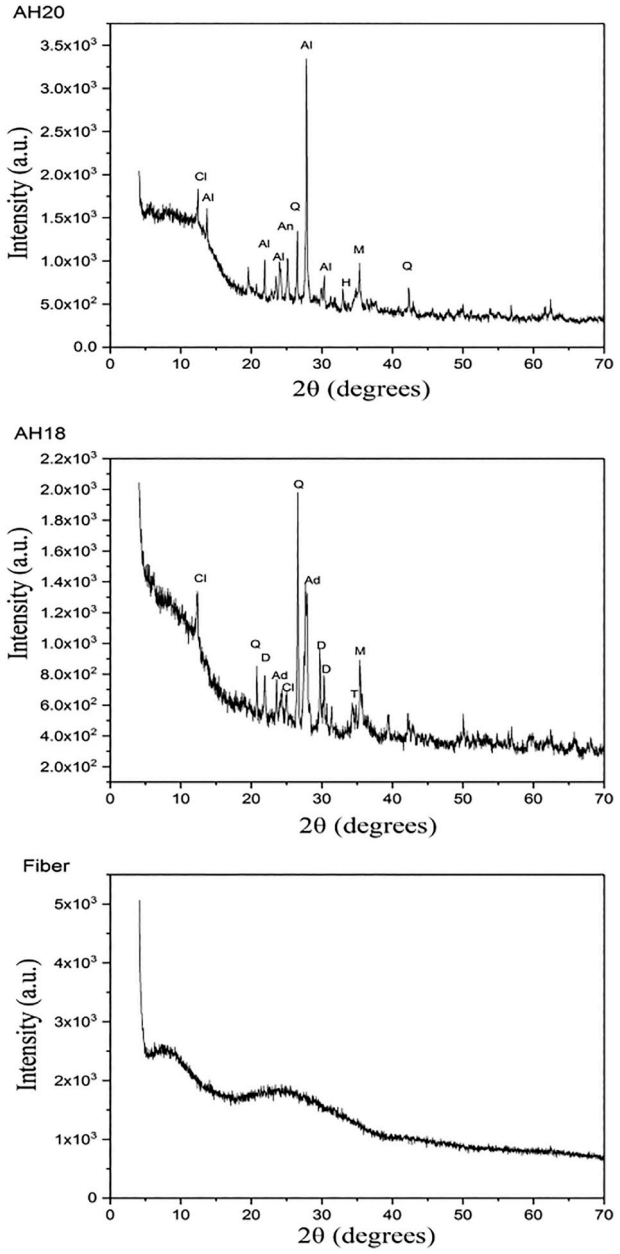

Fig. 2. XRD qualitative analysis results of raw material (AH20, AH18) and the fibre; Al: albite; An: anatase; Ad: andesine; Cl: clinochlore; M: magnetite; H: hematite; D: diopsite; T: titanite; Q: quartz; AH18: quartz, andesine, diopside, clinochlore, titanite and magnetite; AH20: quartz, albite, anatase, clinochlore, hematite and magnetite; the fiber is composed of amorphous phase.

Determination of the tensile properties of single-filament specimens>. 8 specimens were taken from the monofilament. The gauge length of specimen, loading speed and load cell capacity were 25.0 mm, 2.0 mm/min and 0–100 cN, respectively. The mean value of the tensile strength

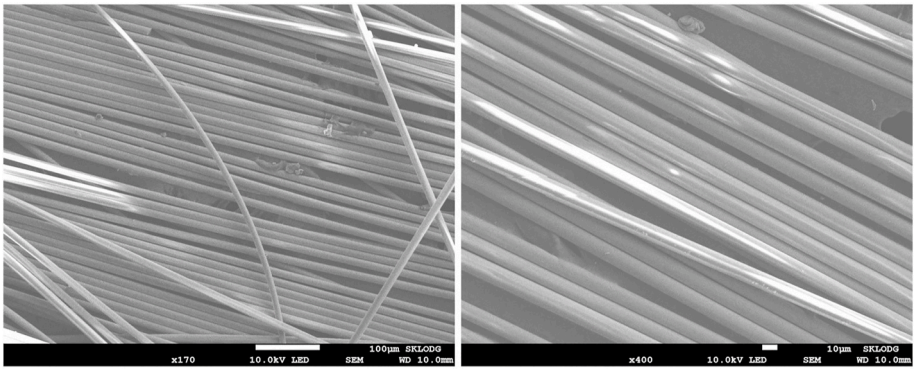

Fig. 3. SEM micrographs of the fiber.

Table 3
Tensile strength and diameter of our fibre.

									average	σ
Tensile strength (MPa)	3809	3913	4005	4007	4037	4263	4317	4543	4111	242
Diameter (µm)	12.6	11.8	12.0	11.5	12.0	12.3	12.0	12.7	12.1	0.4

with standard deviation was reported. A high-temperature viscometer (Brookfield, Germany) was employed to measure the viscosity of the two samples (YM-3, AH18) at Southeast University, China. The starting glasses used for viscosity determination were prepared by melting rocks in a platinum–rhodium crucible and maintaining the temperature at about 1500 °C for 10 h.

3. Results and discussion

3.1. XRD analysis and the morphology of CBFs

The results for the XRD patterns of AH18, AH20 and the fibre are shown in Fig. 2; subsequently, phase identification was conducted. AH18 contains a variety of minerals: quartz (PDF#85–1053), andesine (PDF#79–1148), diopside (PDF#76–0237), clinochlore (PDF#79–1270), titanite (PDF#75–1383) and magnetite (PDF#19–0629). AH20 is consist of quartz (PDF#85–1053), albite (PDF#72–1246), anatase (PDF#71–1166), clinochlore (PDF#79–1270), hematite (PDF#89–0597) and magnetite (PDF#19–0629). AH18 and AH20 do not contain refractory phases such as olivine, which are beneficial to the fibre-drawing process. The final fibre product exhibits the amorphous feature that all diffraction peaks were extinct, resulting from the quick cooling of the melt during the fibre-drawing process. This conforms with the quality requirements of CBF.

It can be seen from the SEM micrograph (Fig. 3) that the surface of the fibre is smooth and the diameter is almost uniform. The diameter of the CBF is a crucial parameter of the melt-spinning process. The diameter of the fibre was measured using the ruler tool in Adobe Photoshop on the SEM micrograph, and the average diameter was obtained: 12.1 µm (σ = 0.4) (Table 3). Here, nozzle diameter, drawing speed, melt pressure and viscosity are the main influencing factors. Generally, fine CBF (diameter<15 µm) has higher mechanical strength due to the reduction in possible flaw size in a decreased specimen volume. This fibre is fine CBF, according to classification standards.

3.2. Tensile strength

The tensile strength of the fibre is a crucial aspect for evaluating the mechanical properties of CBFs. The mean value of the tensile strength of the fibre is 4111 MPa with a standard deviation of 242 MPa (Table 3). The tensile strength of the fibre is influenced by the complex interactions of nine different oxides as well as the fibre-drawing process. Using the formulation-optimization model proposed above, the chemical composition of the fibre was controlled.

Furthermore, it should be noted that the tensile strength of the fibre is relatively higher than the tensile strength of the CBFs proposed in Russia, Israel, and Ukraine, reported by Deák and Czigány [14]. Table 4 shows the chemical composition, Mk, Mη and the tensile strength of the four fibres. Many factors can affect the tensile strength of a fibre, such as the filament diameter, chemical composition and homogeneity of the melt. The amount of $Na_2O + K_2O$ has a negative correlation with the tensile strength of fibres, as shown in Table 4. The tensile strength of a fibre – to some extent – depends on the chain of the $[SiO_4]$ and $[AlO_4]$ tetrahedron; in contrast, Na_2O and K_2O are glass network modifiers that could break the chain structure, reducing the tensile strength of the fibre. Our fibre has the highest tensile strength among the four fibres because of the least amount of $Na_2O + K_2O$. Overall, the total amount of $Na_2O + K_2O$ in the precursor is a critical factor and should be controlled to a low value to ensure higher tensile strengths.

3.3. Relationship between viscosity (η) and viscosity modulus (Mη)

Viscosity is a key parameter in current CBF production technology. Specifically, SiO_2 and Al_2O_3, as glass network-forming compounds, can dramatically increase the viscosity of the melt and the strength of fibre. In contrast, CaO and MgO can reduce the melt viscosity and chemical

Table 4
Comparison between our fibre and three CBFs from RUSSIA, ISRAEL and UKRAINE.

	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	TiO_2	Mk	Mη	SiO_2+ Al_2O_3	$Na_2O + K_2O$	tensile strength (MPa)
Our fibre	48.80	12.98	12.32	3.67	7.51	2.60	0.74	3.89	6.19	2.49	61.78	3.34	4111
RUSSIA	55.69	15.44	10.80	4.06	7.43	2.40	1.51	1.23	5.16	2.90	71.13	3.91	2016
UKRAINE	50.62	17.97	11.11	5.19	8.85	2.38	1.73	1.10	4.89	2.47	68.59	4.11	1811
ISRAEL	53.36	14.21	10.98	5.35	7.74	3.79	1.06	1.73	5.53	2.43	67.57	4.85	1608

Table 5
Chemical composition (wt%), Mη, reference, η(d Pa·S, 1300 °C) and η(d Pa·S, 1400 °C) of samples.

	SiO_2	Al_2O_3	Fe_2O_3	MgO	CaO	Na_2O	K_2O	FeO	TiO2	Mη	η(1300 °C)	η(1400 °C)	Reference
YM-3	50.32	12.95	14.40	4.32	8.62	2.25	1.54		3.79	2.21	144.7	48.64	
AH18	46.91	12.85	12.84	4.06	8.79	2.33	0.72		3.78	2.17	159.0	56.03	
AN-1	61.09	17.97	1.74	1.38	4.75	3.15	2.30	3.99	0.66	5.04	9103.0	2452.17	[3]
AN-2	58.69	13.45	4.30	4.61	4.95	0.79	2.18	2.58	0.74	3.77	1400.6	401.77	[3]
AN-3	57.50	16.85	3.86	2.64	3.75	3.27	2.58	3.22	1.16	4.21	3191.3	959.13	[3]
ANB-1	56.7	14.47	1.53	5.25	6.93	3.42	0.73	7.72	1.33	2.77	606.4	181.61	[3]
ANB-3	52.22	17.27	4.05	3.42	4.95	3.75	1.60	4.38	1.11	3.33	574.9	192.2	[3]
TB-2	50.76	15.2	4.95	5.58	6.21	3.18	1.79	4.71	2.03	2.55	257.2	89.07	[3]
TB-3	49.64	13.18	3.72	5.60	8.61	2.24	0.75	6.69	2.10	2.27	149.3	53.49	[3]
CBF2	54.12	13.98	6.81	4.88	6.00	3.24	2.85	3.19	0.90	2.67	557.9	178.53	[23]
CBF3	51.79	16.54	4.47	5.25	8.37	2.29	0.90	4.40	1.63	2.67	269.8	87.51	[23]
ANBF5050	54.55	17.42	2.57	3.27	6.18	2.94	1.67	6.08	1.67	3.35	1312.1	370.18	[22]
ANBF6040	55.86	17.53	2.40	2.89	5.89	2.98	1.80	5.66	1.47	3.61	1768.0	469.95	[22]
BF	48.01	16.86	3.4	5.15	7.60	2.73	1.04	8.17	2.68	2.38	165.3	57.51	[22]
ANBF2080	50.63	17.08	3.07	4.40	7.03	2.81	1.29	7.33	2.28	2.71	298.8	89.51	[22]
ANBF3070	51.93	17.19	2.90	4.02	6.75	2.86	1.42	6.92	2.07	2.90	606.4	223.45	[22]

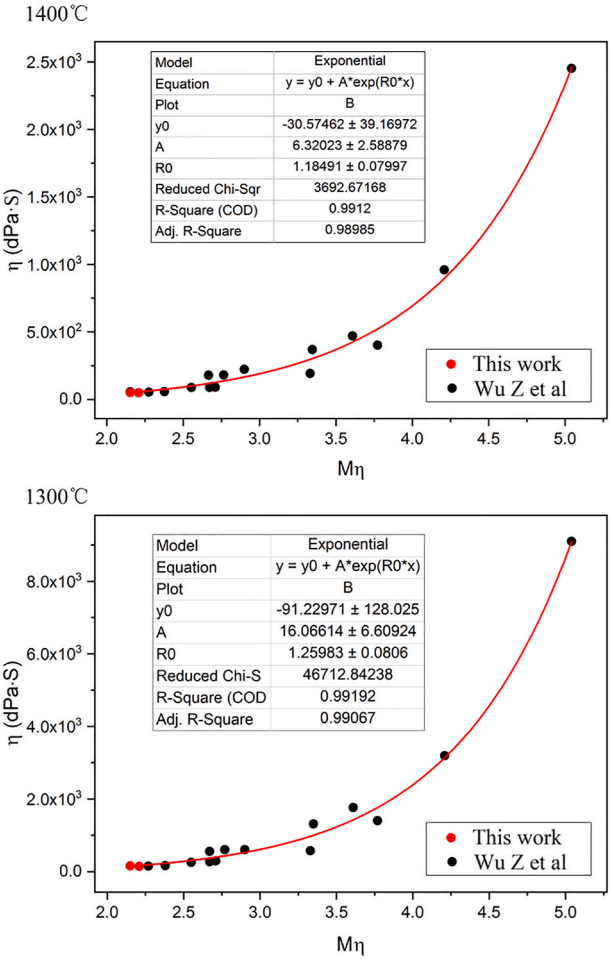

Fig. 4. Exponential fitting between viscosity and viscosity modulus at 1300 °C and 1400 °C.

stability of the fibre. Moreover, K_2O and Na_2O can reduce the melting temperature and the viscosity of melt. The viscosity of melt is influenced by the complex interactions of nine different oxides. During the production process of CBF, the viscosity of basalt melt must be modest at all working temperatures. A highly sticky melt is difficult to control during the drawing process and can lead to fibre breakage and damage. In contrast, a low-viscosity melt could flow down quickly, before it solidifies into a fibre. In this regard, Makhova et al.(1990) suggested that the basalt melt for the production of superfine CBF should have a medium viscosity, ranging from 180 to 310 dPa S at 1300 °C [18]. However, direct viscosity measurements for basalt melts are time-consuming, energy-intensive and expensive. Thus, an alternative method is required to establish the quantitative relationship between viscosity and chemical composition. Here, introducing the viscosity modulus as a suitable single variable is proposed.

In this work, we have finished viscosity measurement of two basalts (YM-3, AH18) and collected 14 basalts' chemical composition and viscosity (at 1300 °C and 1400 °C) data (Table 5) reported by Wu Z et al. [3, 22,23], and worked out the viscosity modulus of each basalt (Table 5) with their chemical composition and the expression (1) above. As shown in Fig. 4, exponential relationships between viscosity (η) and viscosity modulus (Mη) at 1300 °C and 1400 °C were found (fitting $R^2 = 0.99$), and are presented as follows:

$$\eta(1300\ ^\circ C) = -91.22971 + 16.06614e^{1.25983M\eta} \quad (4)$$

$$\eta(1400\ ^\circ C) = -30.57462 + 6.32023\ e^{1.18491\ M\eta} \quad (5)$$

Furthermore, an exponential relationship also exists between viscosity and viscosity modulus at other temperatures, such as 1350°Cand 1450 °C, which makes it possible to predict the viscosity of the melt using the chemical composition of the precursor. Using the formulation-optimization method, the viscosity of melt can be adjusted to an optimal range using only the viscosity modulus. Using the reference of the viscosity range (180–310 dPa s) at 1300 °C, reported by Makhova et al. [18], and equation (4), the optimal range of the viscosity modulus for fabricating CBFs is found to be between 2.2 and 2.6. This is an essential constraint condition in the formulation-optimization model. The viscosity modulus (2.49) of our fibre is well within the acceptable limits, indicating that we fabricated a fibre with high tensile strength. Therefore, the empirical formula proposed here is reasonable for directly estimating whether a precursor is suitable for CBF production.

4. Conclusion

Considering the limitations of traditional CBF production technology, we verified that the formulation-optimization method is feasible and practical for fabricating CBFs. A high tensile strength (4111 MPa) CBF was successfully produced from two different types of basalt that are not individually suitable for making CBFs. Subsequently, the morphology and tensile strength of the produced CBFs were characterized. Our results, along with existing literature, were used to propose two empirical formulas to quantify the relationship between viscosity (η) and viscosity modulus (Mη) for basalt melts at 1300 °C and 1400 °C: $\eta(1300\ ^\circ C) = -91.22971 + 16.06614e^{1.25983M\eta}$ and $\eta(1400\ ^\circ C) = -30.57462 + 6.32023\ e^{1.18491\ M\eta}$. It was found that a value of Mη

ranging between 2.2 and 2.6 is optimum for CBF production. We established an important correlation between η, Mη and mix compositions, which could be an essential criterion to evaluate the characteristics and production technology of CBF, including basalt beneficiation, formulation optimization and the strength performance of CBFs.

Declaration of competing interest

We declare that we have no financial and personal relationships with other people or organizations that can inappropriately influence our work, there is no professional or other personal interest of any nature or kind in any product, service and/or company that could be construed as influencing the position presented in, or the review of, the manuscript entitled, "Preparation and characterization of continuous basalt fibre with high tensile strength".

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