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Potential applications of basalt fibre composites in thermal shielding

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Abstract
This present study demonstrates the applicability of basalt fibre-reinforced polymer (BFRP) composite materials in thermal shielding. Basalt fibres are produced from natural, sustainable sources and obtain comparable mechanical performance to commercial glass fibres. In addition to their mechanical strength, BFRPs have excellent chemical and heat resistance. Basalt fibres tend to have a higher thermal stability than their competitor glass fibres. The heat resistance of basalt fibres derives from the volcanic origin of the raw material basalt gabbro. These favourable features make BFRP composites an attractive group of materials for application in several industries. To test the fire resistance of the materials, we produced mono and hybrid composite plates from different types of basalt reinforcement structures (milled fibres, chopped fibres and woven fabric) and epoxy resin. Surface treatment with silane coupling agents significantly improved the mechanical and thermomechanical properties of BFRPs by up to 70%. Three-point bending tests were performed to determine the flexural properties of the composite specimens, and their fire behaviour was evaluated with a horizontal burning test, and a novel jet fire test assisted with infrared thermal imaging. Higher fibre content in hybrid laminates decreased the linear burning rate by 8%, and the maximum surface temperature was approximately 80 °C lower after jet fire impingement compared to woven reinforcement structure.

Keywords Basalt fibre · Functional composites · Thermal shielding · Fire protection · Sustainability

Introduction

In our research, the mechanical and thermal properties of epoxy resin matrix composites with different types of eco-friendly basalt reinforcement were analysed. The effect of short fibres (milled and chopped) and continuous fibres (plain-woven fabrics) were investigated separately and combined through hybridisation. Mechanical properties were characterized with three-point bending test and dynamic mechanical thermal analysis (DMTA), and thermal properties were investigated with thermogravimetry analysis (TGA), horizontal burning test (UL-94) and infrared thermographic camera-assisted jet fire test. Curves were analysed with a self-developed image-processing software.

According to our results, producing of the hybrid composites can be combine excellent mechanical properties with outstanding fire resistance. Woven plies with short fibres decreased the linear burning rate by up to 18%. During the jet fire test, temperature gradient and maximum temperatures could be reduced significantly with hybrid structure.

Polymer composites are suitable for manufacturing functional, structural components for various industries. The properties of composites can be tailored for increased heat, flame and chemical resistance and for improved electrical insulation and mechanical properties. The market for reinforcement fibres is dominated by synthetic fibres, such as glass and carbon fibres. Global environmental regulations urge the application of organic and inorganic fibres from natural and sustainable sources to replace conventional synthetic fibres. Therefore, in the past few decades, the applicability of these reinforcement materials and structures have been intensively researched, and there have been many innovations. Also sustainable design of composite materials and structures is gaining importance [1]. In addition to

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the mechanical design of polymer structures further safety aspects, such as fire resistance should to be taken into consideration. Traditionally, fire resistance of polymers could be achieved by flame-retardant additives or coatings, and the environmental impact of chemicals used in passive fire protection could be reduced with the application of organic intumescent retardants [2].

Volcanic basalt gabbro is easily accessible from open-cut quarries and natural reserves in most countries. It has been used mainly in the construction industry in the past few decades, due to its durability, low price and good vibration absorption properties. The chemical structure of basalt is closely related to glass as both are composed of SiO_2, Al_2O_3, CaO, MgO, Fe_2O_3 and FeO [3–6]. The main difference in the chemical composition is Fe_2O_3 content—while glass has less than 0.5 m/m% of it, basalt contains approximately 10–11.5 m/m% iron (III) oxide [6]. These oxides compose a large, cross-linked molecule; therefore, basalt and glass can be regarded as an inorganic polymer to a certain extent. Owing to this structure, fibres can be produced from molten basalt and glass. When the melt is cooled rapidly, basalt solidifies in a glass-like amorphous phase. Lower cooling rates result in a partially crystalline structure. Discontinuous basalt fibres are produced by the Junkers method [4]. For more demanding applications, spinneret technology is used to prepare continuous fibres [7]. Basalt is a good electric insulator, resistant corrosion and biologically inert, and thus, it is considered environmentally friendly. Additionally, basalt fibres are superior to glass fibres in terms of thermal stability and heat resistance. This makes them suitable for high-temperature and fire protection applications.

Basalt fibres are mainly used in the construction industry. Rock wool is a preferred thermal insulation in buildings. The load-bearing capacity of continuous fibres makes them suitable for reinforcing concrete. Meyyappan et al. [8] studied the mechanical properties of basalt fibre-reinforced concrete with a cement matrix. Unlike standard steel reinforcements used in concrete, pultruded basalt fibre rebars resist corrosion. Their lightweight and strength make them competitive in the construction industry [8, 9]. Corrosion resistance and chemical stability make BFRPs suitable for application in marine environments, for instance, in tidal and wave energy devices and offshore wind generation [10]. The intensive development of such promising renewable energy systems plays a crucial role in CO_2 emission reduction strategies. The overall performance of composite structures and mechanical properties could be enhanced with the improvement of interfacial shear strength between the fibres and the matrix interfaces. Deák et al. [11] studied the application of different silane coupling agents to enhance the interfacial properties of basalt fibre-reinforced nylon 6 matrix composites. They found that coupling agent developed for glass fibres is also applicable for basalt fibres as their structure and chemical composition is similar. Zhang et al. investigated the application of basalt fibre reinforcement in ship hulls [12]. They concluded that the heat resistance of basalt fibres makes them suitable for fire safety applications. For instance, the thermal conductivity of basalt fibres is approximately 0.030–0.038 W m^{-1} K^{-1}, which is lower compared to 1.03 W m^{-1} K^{-1} of glass fibres [13]. Structural components containing basalt fibres could provide thermal shielding and thus flame resistance without further fire-retardant additives. Attai et al. [14] have modified the surface of basalt fibres with polyaniline coating, which improved the tensile strength of EPDM rubber matrix composites by 38 and 53% compared to blank rubber and untreated basalt fibre composites. Also, the flame resistance was improved by 62 and 16% reduction in the peak heat release rate, and they managed to achieve a significant reduction in emission of CO and CO_2 gases by 65 and 58%, respectively. Gou et al. [15] studied the impact of silane surface modification of basalt fibre-based flame-retardant epoxy composites. The incorporation of basalt fibres improved mechanical properties, slightly reduced LOI value from 26.3 to 25.1%, maintained the good performance in the vertical burning test, but increased the peak of heat release rate. Wittek and Tanimoto [16] developed basalt fibre-reinforced environmentally degradable starch-based resin matrix composites. The application of silane coupling agents improved the flexural properties of the composites by 38%. Flammability resistance was improved by the application of basalt fibres, magnesium hydroxide and red phosphor. Elejoste et al. [17] studied the development of flame-retarded basalt fibre-reinforced furan resin prepregs. The developed materials achieved the R1HL3 (Requirement 1 and Hazard Level 3) classification, which is the maximum safety level according to the risk index established in railway vehicle regulations. Hao et al. [18] analysed the thermal protective performance of nonwoven basalt fabrics. Matyakiewicz et al. [19] assessed the influence of basalt powder on the thermomechanical properties of epoxy matrix composites. They concluded that basalt powder made composites resistant to fire and high temperatures. Their results have proved that high values of activation energy (Ea) and total heat release (THR) influence to the thermal degradation process. In another study [20], the research team investigated the hybrid effects of basalt powder filler in BFRPs. Zhu et al. [21] have investigated the heat and fire resistance of basalt rebars. Landucci et al. [22] developed basalt-reinforced fire protective panels, which had better thermal behaviour than other designated fire protective structures. These results prove that basalt fibres have reasonable strength, excellent thermal stability and fire protective properties, which make them an attractive material for fire-resistant structures.

Materials and methods

Three different types of basalt fibre reinforcements were used in our experiments: milled fibres (Basaltex, Belgium, average fibre length: 108.57 ± 57.09 µm), chopped fibres (Kamenny Vek, Russia, average fibre length: 12.7 mm), and plain-woven basalt fabrics (Kamenny Vek, Russia, areal density: g m^{-2}). A common laminating epoxy system of component A—IPOX MR 3010 modified bisphenol A/F resin (epoxy-equivalent: 175–190 g/equiv.; epoxy value: 0.52–0.57 equiv./100 g; viscosity at 25 °C: 800–1200 mPa s; density—1.10–1.15 g cm^{-3}) and component B—IPOX MH 3124 modified cycloaliphatic amine hardener (amine value: 450–470 mg KOH/g; viscosity at 25 °C: 40–70 mPa s; density: 0.95 g cm^{-3}); Ipox Chemicals GmbH, Germany, was used for the matrix material. These components were mixed with a ratio of 100:33 by mass, respectively. In the case of milled and chopped basalt fibres 10; 20; 30 m/m% fibre content was maintained, and dispersion of fibres was measured on five samples per material selected from different places of the plates. The short fibres were well dispersed as the standard deviation of the measured fibre content was below 0.5 m/m%. The adhesion promoter effect of the organofunctional alkoxysilane coupling agent between the basalt fibres and the matrix was also taken into account. A portion of the milled fibres and woven fabrics were treated with the GENIOSIL GF93 bonding agent (Wacker Chemie AG, Germany). During the fibre treatment, the original sizing of basalt fibres was removed by annealing at 400 °C for 3 h, and the coupling agent was dissolved in distilled water. Initially, the amount of silanes was 3 m/m% in the ratio of basalt fibres. The basalt fibres were immersed in the solution for 2 h, after dissolving for 2 h, in order to effectuate the hydrolysis of silanes and their bonding with the hydroxyl groups on the surface of BF. The water was evaporated at 80 °C in an air-circulating oven. Composite plates were produced by silicone casting, compression moulding and hand lamination. Hybrid plates were laminated with woven and milled fibres. In this case, the theoretical fibre content was calculated from the mass fraction of the milled fibres and the epoxy resin, and thus, actual fibre content was higher due to the presence of the fabric layers. Three types of hybrid structures were manufactured: H1ST consisted of treated fabric and untreated milled fibres, H2ST contained treated fabric layers and treated milled fibres and specimens marked H are the reference untreated hybrid laminates. During manufacturing, the 4 mm thickness of the composite plates was maintained. After post-curing at 80 °C for 2 h in an industrial oven (Despatch LBB2-27-1CE, Despatch Industries, Inc., Lakeville, USA), the test specimens were cut with a diamond blade saw (Mutronic Diadisc 4200, MUTRONIC Präzisionsgerätebau GmbH & Co. KG, Rieden, Germany).

A three-point bending test was performed on the composite materials according to the EN ISO 14125:2011 standard. The tests were performed on a Zwick Z005 universal tensile tester machine (ZwickRoell AG, Ulm, Germany) equipped with a 5 kN tensile head. The support span was set to 64 mm, and the testing speed was 2 mm/s. The thermal behaviour of the BFRP specimens was determined by thermogravimetry analysis (TGA) in a nitrogen atmosphere. The equipment was a TA Instruments Q 500 thermogravimetry analyser (New Castle, DE, USA), sample size was 5 mg, and the heating rate was °C min^{-1}. The dynamic mechanical thermal behaviours of the composite specimens with the size of 60 × 10 × 4 mm were tested by using a TA Instruments Q800 Dynamic Mechanical Analyser (New Castle, DE, USA) with a heat ramp from 35 to 200 °C at a rate of 5 °C min^{-1} with dual cantilever mode and a frequency of 1 Hz.

We used horizontal burning test (UL-94) to determine the burning rate of the 125 × 10 × 4 mm specimens in an arrangement according to the ISO 1210:1997 standard. The material's flame and heat spread characteristics were determined with an infrared thermographic camera-assisted (FLIR A325sc, FLIR Systems Inc., Wilsonville, OR, USA) jet fire test, inspired by the study of Landucci et al. [22]. In our laboratory-scale setup, the 80 × 80 × 4 mm specimens were fixed in place and were impinged by the flames from an Usbeck 1422 bunsen burner torch (Carl Friedrich Usbeck KG, Radevormwald, Germany). The head of the flame torch was positioned at 100 mm distance normal to the surface of the specimens, and the flames penetrated the laminates in the midpoint. The 70 m/m% butane 30 m/m% propan gas flow was 0.13 l/h, the nozzle diameter was 20 mm, and the average flame length was 175 ± 25 mm. After ignition, the specimens were impinged for 20 s, and the surface temperature of the opposite unpenetrated side was measured by the thermographic camera. The videos and thermal images captured with the IRT camera were processed with image-processing software developed in-house. The software developed in the NumPy library of Python determined the temperature distribution in the enclosed area of the specimens and produced histograms of the heat distribution for each video frame. With the aid of this software, we were able to compare the thermal images qualitatively. The temperature data were measured on the backside of the penetrated specimens, and the maximum values were measured in the centre of the specimens in 9 measurement points in the arrangement illustrated in Fig. 1. The highest temperature values were chosen for further analysis (Table 1).

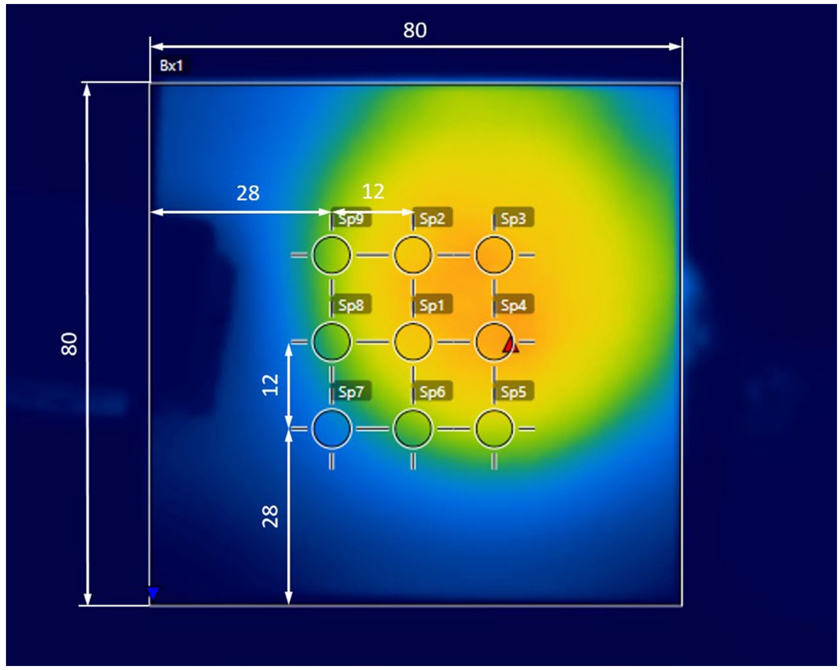

Fig. 1 Arrangement of measurement points on IR thermographic images

Table 1 Investigated materials

Name	Material	Sample preparation	Fibre diameter/µm	Fibre content	Nominal fibre content/m/m%
EP	Epoxy resin	Silicone casting	–	–	0
M10	Milled BF	Silicone casting	20.49 ± 5.81	10 m/m%	10
M20	Milled BF	Silicone casting	20.49 ± 5.81	20 m/m%	20
M30	Milled BF	Silicone casting	20.49 ± 5.81	30 m/m%	30
C10	Chopped BF	Compression moulding	16.44 ± 2.43	10 m/m%	10
C20	Chopped BF	Compression moulding	16.44 ± 2.43	20 m/m%	20
C30	Chopped BF	Compression moulding	16.44 ± 2.43	30 m/m%	30
WF6	BF woven fabric	Hand lamination	21.44 ± 2.11	6 plies	~40
H10	BF hybrid (fabric + milled)	Hand lamination	20.49 ± 5.81*; 21.44 ± 2.11**	6 plies, 10 m/m%	~43
H20	BF hybrid (fabric + milled)	Hand lamination	20.49 ± 5.81*; 21.44 ± 2.11**	6 plies, 20 m/m%	~46
H30	BF hybrid (fabric + milled)	Hand lamination	20.49 ± 5.81*; 21.44 ± 2.11**	6 plies, 30 m/m%	~49
M30ST	Milled BF + surf. treat	Silicone casting	20.49 ± 5.81*; 21.44 ± 2.11**	30 m/m%	30
WF6ST	BF woven fabric + surf. treat	Hand lamination	21.44 ± 2.11	6 plies	~40
H1ST	BF hybrid (fabric + milled) + surf. treat	Hand lamination	20.49 ± 5.81*; 21.44 ± 2.11**	6 plies, 30 m/m%	~49
H2ST	BF hybrid (fabric + milled) + surf. treat	Hand lamination	20.49 ± 5.81*; 21.44 ± 2.11**	6 plies, 30 m/m%	~49

*Milled fibres

**Fibres in a woven fabric in the hybrid layup

Results and discussion

Figure 2 shows that the continuous fibres in the woven fabric reinforcement provide outstanding flexural strength and moduli. When silane surface treatment was applied to the fibres, flexural properties further improved by 9 to 70%.

H1ST had the highest strength, with 30 m/m% hybrid reinforcement, where the fabric is surface-treated and the untreated milled fibres are between the plies. Milled fibre filling in hybrid structures and the surface treatment of the fabric decrease the modulus compared to the original woven fabric laminate. The mechanical strength of BFRP compared to E-glass fibre-reinforced composites proved to be superior.

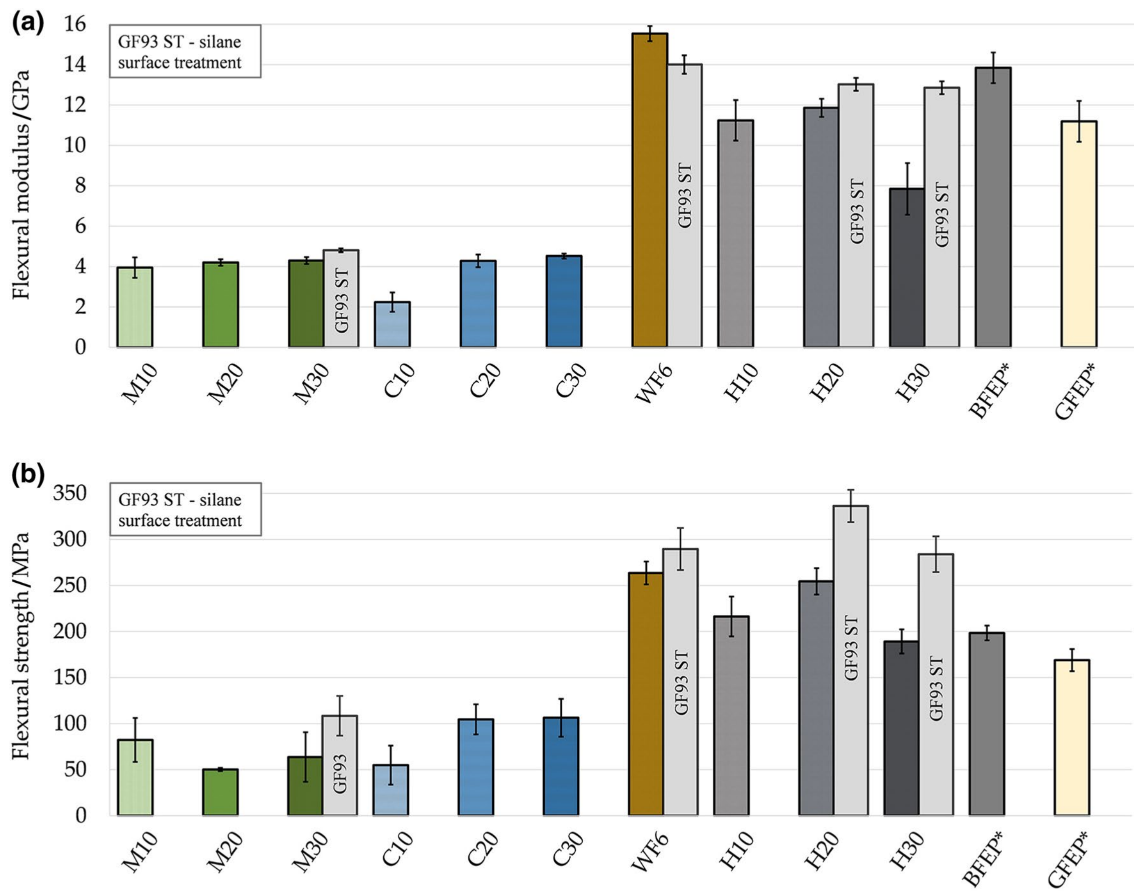

Fig. 2 Flexural properties of basalt fibre-reinforced epoxy matrix specimens, *data from literature [23]

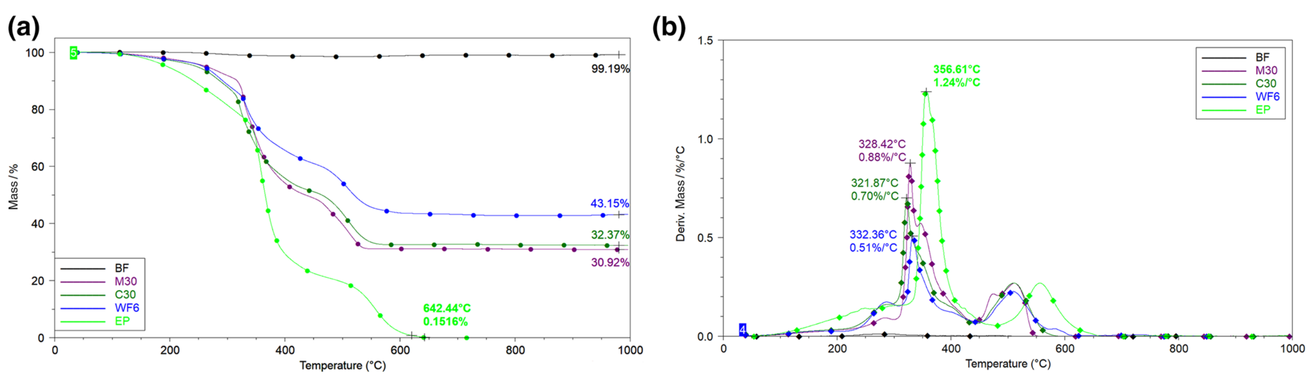

Fig. 3 **a** TG and **b** DTG curves of samples prepared from basalt fibre-reinforced epoxy composite specimens. BF, plain basalt fibre; M30, milled basalt fibre; C30, chopped basalt fibre; WF6, woven fabric; EP, Plain epoxy resin

The TGA results shown in Fig. 3a, b prove that basalt fibres have excellent thermal stability. The mass loss of the BF sample was less than 1% at the upper limit temperature of 1000 °C. The presence of basalt fibres modified the degradation temperature of epoxy resin by almost 100 °C. The nominal fibre content of the composites was calculated from the mass residue of the samples. The mass residue of the woven fabric samples was 43.15%, while for the chopped and milled fibre samples, it was 32.37% and 30.92%, respectively. This is close to the theoretical fibre content, which had been used during the mixing of the compound. According to the DTGA curves, the degradation of plain

Table 2 Data obtained from TG and DTG curves of BF, M30, C30, WF6, EP

Name	T-5%/°C	T-50%/°C	dTG_{max}/%/°C	$TdTG_{max}$/°C
BF	–	–	0.01	282.5
M30	256.8	433.2	0.88	328.4
C30	246.3	464.1	0.70	321.9
WF6	258.1	519.4	0.51	332.4
EP	188.2	365.3	1.24	356.6

T-5%, Temperature at 5% mass loss; T-50%, temperature at 50% mass loss; dTGmax, maximum mass loss rate; TdTGmax, the temperature belonging to the maximum mass loss rate

epoxy starts above 200 °C and the degradation rate peaks at 350 °C. Basalt fibres decrease the intensity of thermal degradation by 30–60% and improve the thermal stability of the composite materials up to around 300 °C. The peaks of the DTGA curves shift to the higher temperatures compared to pure epoxy due to the lower resin content of the composites. The different types of reinforcement structures have nearly the same influence on the thermal behaviour of the BFRPs (Table 2).

DMA was used to study the thermomechanical properties of the cured epoxy networks, and it allowed to evaluate the following viscoelastic parameters: storage modulus (E'), loss modulus (E''), and mechanical loss factor (tan δ). The storage modulus and tan δ values of the M30, C30, WF6, H2ST, and reference EP are shown in Fig. 4. The storage modulus of fibre-reinforced composites demonstrated considerably higher values than of the reference epoxy below 80 °C. The highest storage modulus was achieved by the H2ST hybrid laminate as it contained milled fibre additionally to the woven reinforcement fabrics and the fibres were treated. It clearly approves that the pretreatment of basalt fibres prior to the formal surface modification is vital. Short fibre M30 and C30 specimens addressed slightly higher

Table 3 Fire behaviour of BFRP composite specimens

Name	T_{max}/°C	Gradient/°C/s	Linear burning rate/mm min^{-1}	Classification
M10	141.35	2.99	17.31 ± 4.70	HB
M20	108.46	2.23	11.54 ± 1.23	HB
M30	84.30	2.00	23.23 ± 2.11	HB
M30ST	147.39	7.18	–	–
C10	114.93	1.54	21.89 ± 2.54	HB
C20	153.02	2.92	20.12 ± 1.14	HB
C30	170.35	4.32	21.62 ± 1.25	HB
WF6	273.24	9.13	25.01 ± 0.79	HB
WF6ST	252.26	12.32	–	–
H10	251.61	6.15	22.14 ± 1.15	HB
H20	152.91	4.49	21.13 ± 0.14	HB
H30	189.43	5.92	20.32 ± 0.58	HB
H1ST	206.59	8.09	–	–
H2ST	260.66	8.75	–	–

Tmax, The maximum temperature of specimens measured in the middle of the unpenetrated side; gradient, temperature gradient, derivative of the measured temperature curve by time; linear burning rate, determined by the UL-94 horizontal burning tests

storage modulus compared to the epoxy, although there was no significant difference between them. Furthermore, glass transition temperature (T_g) was determined by the peak of the tan δ curves. It was noted that the T_g values of WF6 (77.73 °C), C30 (81.37 °C) and H2ST (82.16 °C) were lower than that of pure EP (82.24 °C). It might be related to the change of cross-linking density due to the incorporation of basalt fibre. However, the presence of milled fibres in M30 increased the T_g up to 87.11 °C. Similarly, the glass transition temperature of H2ST was higher than WF6 as the hybrid laminate also contained milled fibres. The mechanism

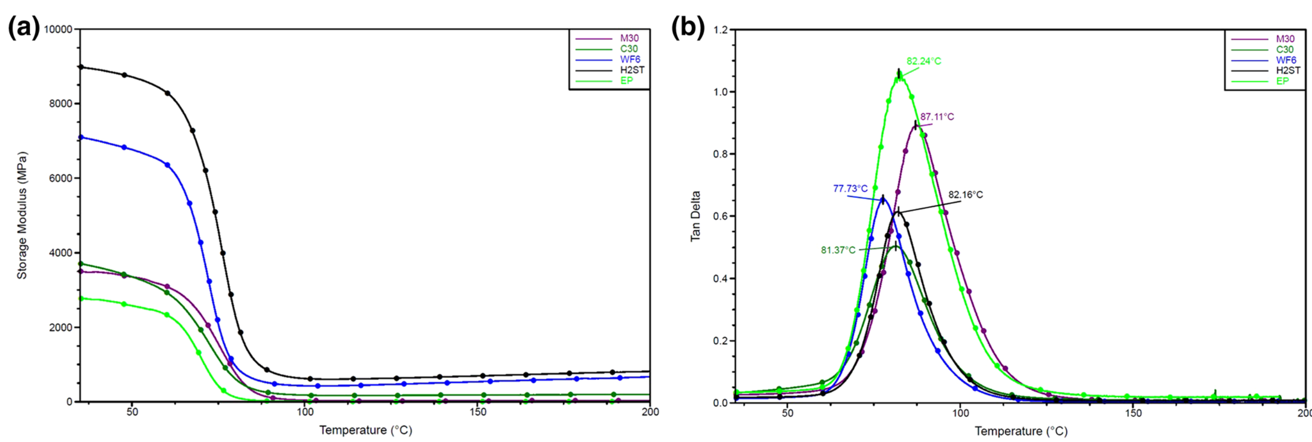

Fig. 4 Dynamic mechanical thermal behaviour of epoxy-based basalt fibre composites: **a** Plots of storage modulus versus temperature; **b** plots of tan δ versus temperature

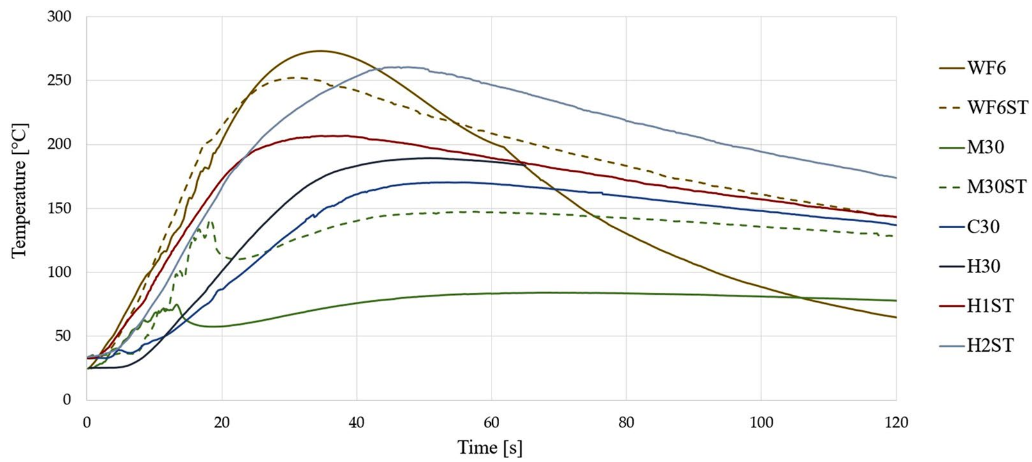

Fig. 5 Temperature measured on the back of the jet fire penetrated BFRP composite specimens

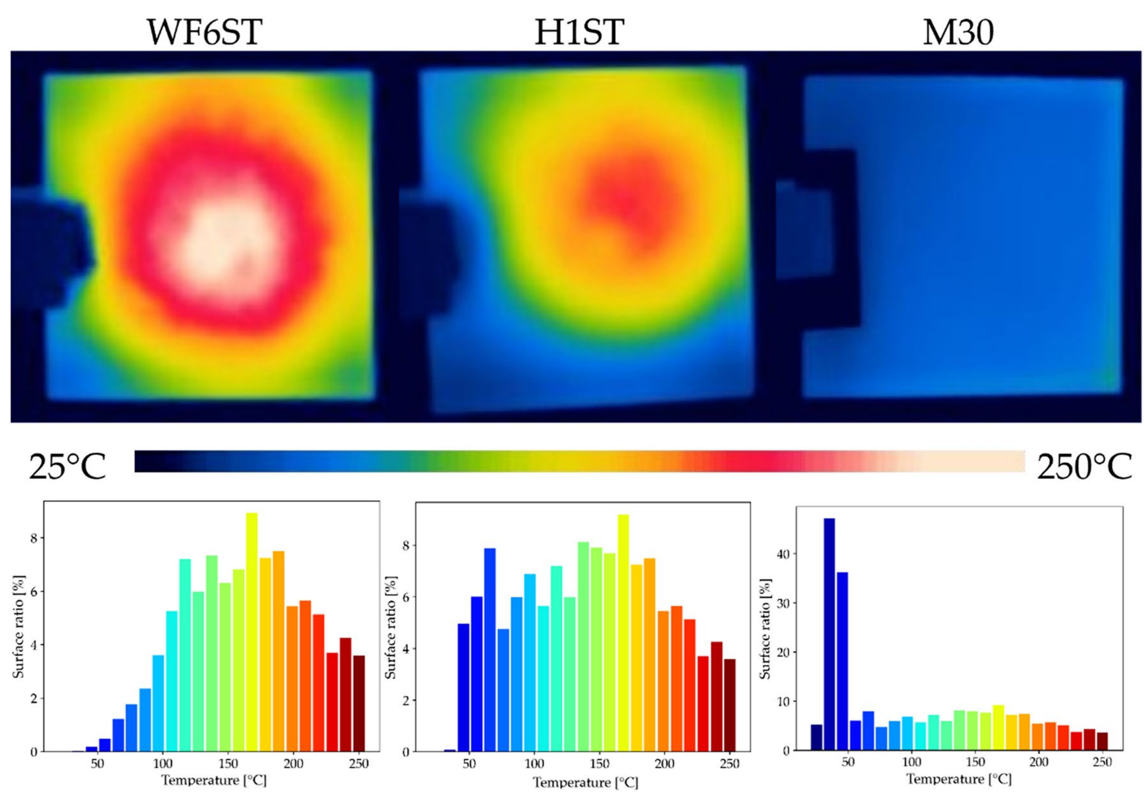

Fig. 6 Thermal camera images taken at the maximum measured temperatures

behind this phenomenon is not clarified yet and it has to be studied comprehensively in the future.

The burning rate of the test specimens is summarized in Table 3. The milled fibres had the lowest, while continuous fibres had the highest burning rate during the horizontal burning tests. This phenomenon could be explained by the so-called candle-wick effect, common among woven fabric reinforcement structures. We were able to reduce the burning rate by applying milled fibres between the fabric layers. The flame spread was slower in hybrid structures.

Thermal camera-monitored jet fire test results (Fig. 5) show that milled and chopped fibres provide excellent protection against fire impingement, as thermal shielding is the most effective in these cases. While heat resistance improved with increasing fibre mass fraction of milled basalt fibres, the chopped fibres yielded similar results, regardless of fibre content. The hybrid structures provided better fire resistance than woven fabrics. Firstly, the maximum temperatures (Table 3) reached lower values compared to the maximum temperature of WF6. Secondly, the milled fibres hindered the spread of the flames between the plies, and after the jet fire was removed, the fire stopped spreading and was extinguished by the structure, leaving a smaller penetrated area. The surface treatment of fibres improved the resistance to jet fire penetration. The treated hybrid composites achieved lower maximum temperatures and smaller penetrated areas. Thermal camera images presented in Fig. 6 show that the milled fibres in the M30 specimen are effective insulators, and the affected area of the specimen is less than 10%. In comparison, the affected area of the WF6ST and H1ST specimens is around 50%. The affected area of the H1ST hybrid structure is smaller, and the burning rate is lower due to the effective insulation of the inter-laminar milled fibres. As a result, the fire protective performance of the BFRP composite is improved with the application of the hybrid system.

Conclusions

In summary, basalt fibre-reinforced composites have not only excellent mechanical properties but also outstanding fire resistance, and therefore, they are ideal for safe structural components. While continuous basalt fibres contribute to excellent flexural properties, for example the flexural modulus of 15.54 GPa and strength of 336.4 MPa, the application of short fibres increases thermal insulation and heat resistance. Hybrid composites combine these favourable properties, creating load-bearing heat-resistant structures. Short fibres between the woven plies decreased the linear burning rate by up to 18.7% and could achieve a low flame spread speed comparable to the chopped fibre specimens. The temperature gradient and maximum temperatures at jet fire impingement test could be reduced significantly. The application of short basalt fibres in coatings could achieve the same effect for other composite materials. Since BFRP components are spreading in the construction industry, further fire and elevated temperature mechanical tests could be carried out on BFRP composite materials considering the relevant standards, such as the local ÉMI (Non-profit Organization for Construction Quality Control and Innovation) standards. The novel jet fire tests could be improved, and tests performed on larger specimens could prevent the flames from bypassing the edges of the plates. Although TGA proved that basalt fibres do not degrade at high temperatures, the dependency of the mechanical properties of BRFPs on temperature could provide further insight into the thermal stability of BFRPs. Organic flame-retardant additives from natural sources could improve the fire retardancy of the composites and could significantly improve performance during UL-94 testing. The precision of temperature measurements could be validated with the application of thermocouples inside the laminates.

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