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RESEARCH

Influence of Fiber Orientation on the Strength Properties of Paper-Epoxy Composites

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ABSTRACT

An important advantage of paper composites is their sustainability. Natural fibers store CO_2 during growth, they are recyclable and may be safely thermally recycled. In this work, composites out of laboratory paper and epoxy resin are generated in a hand lay-up process. The laboratory paper varies in the degree of fiber orientation. As a comparison, paper with isotropic fiber orientation is also used. The tensile strength for the isotropic paper composite is about 120 MPa. It can be observed that the tensile strength of the composite tends to increase with the increase of fiber orientation in the paper. The measured tensile strength of the oriented paper composites in the fiber direction is about 150 MPa and in cross fiber direction about 50 MPa. The strength characteristics are comparatively lower than for carbon or glass fiber reinforced composites, but the density of the paper composites investigated here is only about 1.26 g/cm^3 and the raw material price is significantly lower making paper composites economically attractive. At the end, strength values are modeled with the rule of mixture as well as with Kröling's strength model. In conclusion, tensile strength of oriented paper composites are higher than of isotropic paper.

KEYWORDS

Bio-based composite, paper composite, fiber reinforced composite, lightweight construction, fiber orientation.

INTRODUCTION

Waste from the construction sector accounts for 35.7 % of the total waste generated in the European Union in 2018 [**1**]. To counteract climate change, it is therefore essential to improve sustainability in the construction sector. This can be achieved, among other things, by using building materials made from renewable raw materials that are ideally recyclable or biodegradable after use. With the right choice of raw materials, fiber-reinforced composites can be increasingly used.

Fiber-reinforced composites are used in mechanical, construction, aerospace, automotive, and many other manufacturing industries due to their high strength to weight ratio. Composite materials consist of the base material as well as the filler material. The base material holds the filler material in its structure and is called the matrix. The filler material can consist of particles, fibers, or sheets. In fiber reinforced composites, the filler material consists of synthetic or natural fibers, which can have different lengths (continuous vs. discontinuous composites) and arrangements (random vs. aligned, unidirectional vs. bidirectional, etc.) [**2**].

Fiber reinforced composites made of paper have already been produced. On the one hand, industrially produced papers such as copying paper or filter paper were taken as raw material, e.g. in Prambauer *et. al.* [**3–6**], Kröling *et. al.* [**7–9**] and by others [**10–13**]. On the other hand, papers produced in the laboratory were also used. Among the laboratory papers, on the one hand, isotropic papers, which do not have a preferred fiber direction within the sheet plane, were used for composite production, e.g. by Kröling *et. al.* [**7–9,14**] as well as in [**15–17**]. On the other hand, anisotropic papers have been used for composite fabrication by Kröling *et. al.* [**7,9**], Zadorecki *et. al.* [**13, 18**], and Du *et. al.* [**17**].

Cordin *et. al.* [**19**] investigated the influence of fiber orientation in Lyocell-polypropylene composites. For this purpose, five different composite types with different orientation angles of the Lyocell fibers in the matrix were produced. According to their results, the strength properties of the fiber composite strongly depend on the fiber orientation. The further the stress direction deviates from the fiber direction, the more the strength characteristics decrease. The highest strength values were measured for fiber composites with fibers parallel to the stress direction. With a deviation of the fiber orientation of ± 22.5 °, the tensile strength already decreases by 46 %.

Kröling [**7**] compares the cost and density of paper epoxy composites to other neat and reinforced plastics. According to this analysis, paper composites have the highest modulus of elasticity of the materials considered in terms of cost and density. However, foams have a higher absolute strength.

In summary, the use of paper fibers in fiber composites can increase the proportion of sustainable raw materials. In order for these fiber composites to be able to withstand high loads, the individual fibers in the original paper or in the later composite must be highly oriented or approximately unidirectional. In this work, the influence of fiber orientation in the paper on composite strength is investigated, with the assumption that tensile strength characteristics of the composite increase as fiber orientation increases, followed by the modeling of these strength characteristics.

BACKGROUND

The fiber volume fraction V_f in the composite, assuming that there are no air voids, can be calculated via the density of the fibers ρ_f and of the composite ρ_c as well as the weight fraction of the fibers W_f , as equation (1) shows [**20**]. Kröling *et. al.* [**14**] adapted the calculation of the fiber volume fraction to paper as raw material as shown in equation (2). Thus, the paper basis weight BW_p, the number of sheets n_p used for the composite production, the fiber density ρ_f and the thickness of the composite t_c are taken into account. Due to the collapse of the lumen, the density of the fibers must be approximately equal to the density of cellulose, which is about 1.5 g/cm^3 [**21**].

$$V_f = \frac{\rho_c}{\rho_f} \cdot W_f \tag{1}$$

$$V_f = \frac{BW_p \cdot n_p}{\rho_f \cdot t_c} \tag{2}$$

There are various models for describing the strength properties of composite materials. Probably the simplest micromechanical model to describe the tensile modulus of elasticity is the rule of mixtures (ROM). The model is based on the assumptions that the fibers are homogeneous, uniformly distributed, linearly elastic, and unidirectional oriented. It is also assumed that the adhesion between matrix and fibers is ideal and the matrix is homogeneous as well as isotropic. Furthermore, possible air cavities in the composite material are not taken into account [**19**].

The model is based on the idea that the fibers and the matrix have the same stress in the direction of the fibers. Consequently, the elastic modulus of the composite E_c is composed of the elastic modulus of the fibers E_f and the matrix E_m multiplied by the respective volume fractions V_f and V_m, as equation (3) shows [**22–25**]. Since air cavities are not taken into account, the relationship $V_m = 1 - V_f$ applies [**20,23**].

$$E_c = V_f \cdot E_f + V_m \cdot E_m \tag{3}$$

If the stress is not in fiber direction but transverse to it, the inverse mixing rule is used, as equation (4) shows [**23-25**].

$$E_c = \frac{E_f \cdot E_m}{V_f \cdot E_m + V_m \cdot E_f} \tag{4}$$

Similarly, the tensile strength in stress direction can be modelled with equation (5) [**24,25**] or transversely to the stress direction with equation (6) [**24,25**] using the same idea and assumptions. Equation (5) can be improved, among other things, by using a correction factor for the consideration of the fiber orientation η_θ in the first term [**7**].

$$\sigma_c = V_f \cdot \sigma_f + V_m \cdot \sigma_m \tag{5}$$

$$\sigma_c = \frac{\sigma_f \cdot \sigma_m}{V_f \cdot \sigma_m + V_m \cdot \sigma_f} \tag{6}$$

The fiber strength of paper fibers is usually calculated with the zero span tensile index $ZSTI$ [**26**] as well as the fiber density ρ_f and orientation efficiency η_θ, as equation (7) shows [**27**].

$$\sigma_f = \frac{\rho_f}{\eta_\theta} \cdot ZSTI \tag{7}$$

Kröling *et. al.* add a correction factor in the modelling of the composite strength taking into account the comparatively small composite fracture strain ε_c in contrast to the fracture strain of the matrix ε_m. By inserting the two correction factors as well as the calculation of the fiber strength from equation (7), the tensile strength can be modeled according to Kröling as equation (8) states [**7,9**].

$$\sigma_c = ZSTI \cdot c_f + (1 - V_f) \cdot \frac{\varepsilon_c}{\varepsilon_m} \cdot \sigma_m \tag{8}$$

EXPERIMENTAL

Materials/chemicals details

Northern Bleached Softwood Kraft (NBSK, Mercer International Inc., Berlin, Germany) was used for paper production. The pulp was refined in a laboratory refiner (Voith LR40, SEL 1.5 J/m, set 3-1.6-60, 170 kWh/t). The drainability after refining is 27.0 ± 0.5 SR according to ISO

5267-1 [28]. CMC (carboxymethylcellulose sodium salt, Sigma-Aldrich, St. Louis, MO, USA, 55 mg/L) was added to the fiber suspension as an additive for papermaking to prevent flocculation.

For the production of the fiber composites, the epoxy resin Larit L-285 and the hardener Larit L-287 in blue (Lange+Ritter GmbH, Gerlingen, Germany) were used.

Material synthesis

Isotropic Rapid Köthen laboratory sheets according to ISO 5269-2 [29] were produced as reference material. In isotropic laboratory sheets, there is no preferred fiber direction within the sheet plane. The thickness of the single sheet is 85.60 ± 2.83 µm and the basis weight 52.08 ± 1.28 g/m^2 (measured at 23 °C and 50 % humidity, in accordance to [30]).

The production of the oriented paper with an experimental sheet former is based on Götzinger *et. al.* [31–35]. Therefore, a 0.05 % fiber suspension was mixed from the refined fibers and CMC. In the sheet former, the fiber suspension flows at constant hydraulic pressure through a tube into a nozzle. Already in the tube, the fibers are aligned in the direction of flow. This effect is further enhanced by the extensional flow in the conical nozzle pipette. The suspension jet falls onto a rotating wire. Behind the wire, there is a suction zone, which directly dewaters the suspension and fixes the fiber on the wire. The nozzle and the suction zone traverse synchronously across the wire width. If the speed of the suspension jet and the rotating wire is unequal, a shear flow is created. As a result, one end of the fiber is fixed on the wire and the remaining fiber part aligns itself in the direction of the shear field, which further increases the fiber orientation. The degree of fiber orientation can consequently be adjusted by the difference in speed between the wire and the jet. The paper is thus built up layer by layer. As the thickness of the paper web increases during production, the dewatering capacity decreases and consequently the fiber orientation also decreases. Hence, the fiber orientation of the paper is higher on the wire side than on the top side of the paper facing away from the wire. After production, the paper was dried in the Rapid-Köthen dryer for 10 minutes.

Götzinger [31] investigated the effect of CMC on the stress-strain behavior of laboratory papers produced by the Rapid-Köthen method. However, no discernible changes in stress-strain behavior were observed when CMC was introduced. In addition, conductivity titration tests according to Eyler *et. al.* [36] and complex titration according to Kessel [37] on both oriented papers with and without CMC showed no difference between the samples. These results suggest that most of the CMC is removed with the white water during production. Therefore, while CMC was included in the suspension for the production of oriented papers to prevent flocculation, it was not used in the production process of Rapid-Köthen laboratory sheets, as it was unnecessary.

In this work, oriented papers were produced with a wire-jet speed difference of 45, 75, and 105 m/min. The average thickness of the single sheet is 86.05 ± 3.10 µm and the average basis weight 52.62 ± 1.55 g/m^2 (measured at 23 °C and 50 % humidity, in accordance to [30]). In addition, papers were formed at 105 m/min with a thickness of 65.87 ± 3.51 µm and a basis weight of 33.27 ± 1.01 g/m^2. Due to the lower thickness, the proportion of the highly oriented wire side is higher with these papers.

The production and testing of the paper composites is based on Kröling *et. al.* [7–9,14]. To produce the composites, the resin and hardener were mixed in the ratio of 100:40 as recommended by the manufacturer. The air bubbles in the prepared resin were removed in a vacuum chamber to minimize air cavities in the later composite. The production was done in a hand lay-up process. The paper stacks used had a thickness between 1.96 and 2.05 mm resulting in a fiber content in the composite of approx. 47 %. The single paper sheets were gradually coated with the epoxy system using a brush and placed on top of each other. Air pockets were removed with a pressure roller. The laminated paper stacks were placed between two coated metal plates, treated with release wax (R&G Faserverbundwerkstoffe GmbH, Waldenbuch, Germany) and the release agent Mold Sealer S31 (Ing. R. Konitzer Ges.m.b.H., Wals-Siezenheim, Austria) and were vacumised. The paper stacks were placed in the vacuum bag in the press and pressed for 5 hours at 50 °C and 50 kN. This was followed by tempering at 60 °C and 50 % humidity.

Characterizations

The fiber orientation in the oriented paper was analyzed using the L&W TSO Tester (TSO = tensile stiffness orientation, Lorentzen & Wettre, ABB Group, Kista, Sweden, type: 973574, no.: 380). The time of an ultrasonic pulse through the paper was measured, which allows conclusions to be drawn about the fiber orientation.

Tensile tests in accordance with ISO 1924-2 [38] and ZeroSpan measurements in accordance with T 231 cm-96 [39] were also carried out from the paper samples.

The composites were cut to a width of 20 mm and a length of over 150 mm using a guillotine shear. Cap strips were attached to the composites. The composites were sprinkled with black acrylic varnish. The change of the pattern was recorded by a camera system during the tension measurement and can be used to measure the elongation.

The tensile test was carried out with a servo-hydraulic testing machine MTS Bionix with axial tension and a 15 kN load cell. The composite specimens were clamped between pyramid jaws with a contact stress of 5 MPa. The test speed was 2 mm/min and the free clamping length was 100 mm. The comparatively short free clamping length was due to the paper size, limited by the size of the Rapid-Köthen dryer.

The breaking stress is the largest measured stress value before the longitudinal strain decreases. The stress is related to the initial surface. The elastic modulus was determined by a linear regression between 0.05 % and 0.25 % strain oriented to ISO 527-4 [**40**]. By changing the specific pattern, the arithmetic mean of the strain can be determined with the software GOM Correlate. The elongation at break is the largest optically determined longitudinal elongation before the force decreases significantly.

A schematic diagram of sample production and measurement can be found in **Fig. 1**.

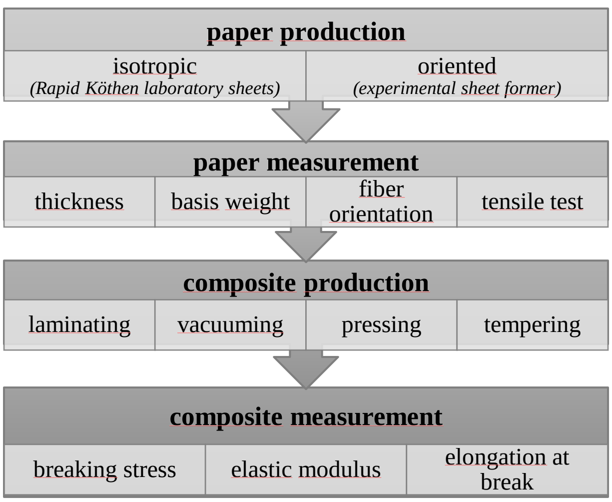

Fig. 1. Schematic diagram of sample production and measurement.

RESULTS AND DISCUSSION

Experiment

Considering the tensile stiffness index (TSI), it can be seen in **Fig. 2** that the TSI of the oriented paper tends to increase with an increase in the wire jet velocity difference (WJSD) in the main fiber direction (machine-direction, MD) and to decrease transverse to the main fiber direction (cross-direction, CD). The values are in each case mean values of the measurement of the wire side and the top side. Due to the reduction of the basis weight to approx. 30 g/m^2, the TSI decreases in CD, but does not increase further in MD. Since the isotropic papers do not have a preferred direction of the fibers, no fiber orientation measurements were made.

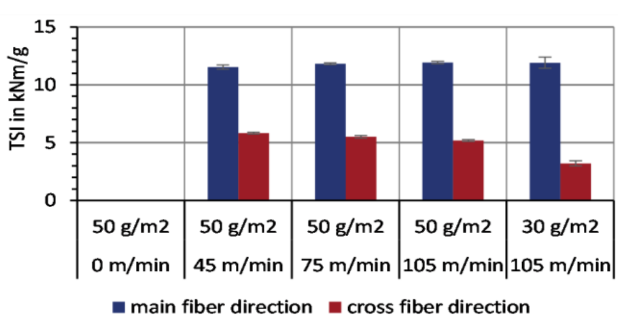

Fig. 2. TSI measurement results from oriented paper. The error bars indicate the standard deviation.

The zero span tensile index (ZSTI) provides information about the fiber strength in test direction (**Fig. 3**). The more fibers are oriented in test direction, the higher the ZSTI should be. The ZSTI of isotropic papers with approx. 0.14 kNm/g is smaller than the ZSTI of oriented papers in MD with approx. 0.18 kNm/g. However, there is no significant difference within the oriented papers. The ZSTI in CD is significantly lower at about 0.07 to 0.08 kNm/g because few fibers are oriented in test direction.

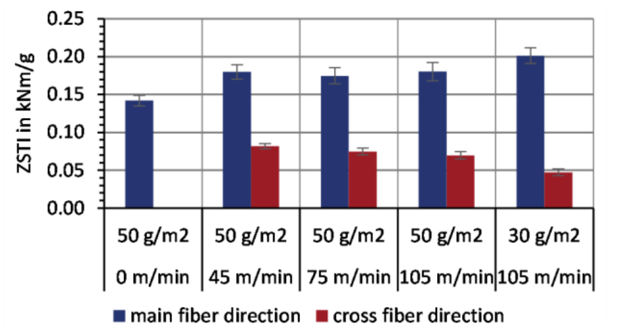

Fig. 3. ZSTI measurement results. The error bars indicate the standard deviation.

Comparable results are shown by the tensile test of the papers in **Fig. 4(a)**. The isotropic papers have a breaking stress of about 40 MPa, whereas the oriented ones in MD have a breaking stress between 50 and 56 MPa. Apparently, there is an increase in the breaking stress with increasing WJSD, but not significant. The fracture stress in CD decreases with increasing WJSD and is in the range of about 14 to 18 MPa. The values for the 30 g/m^2 paper are smaller due to lower basis weight. The same findings are obtained when considering the elastic modulus (**Fig. 4(b)**).

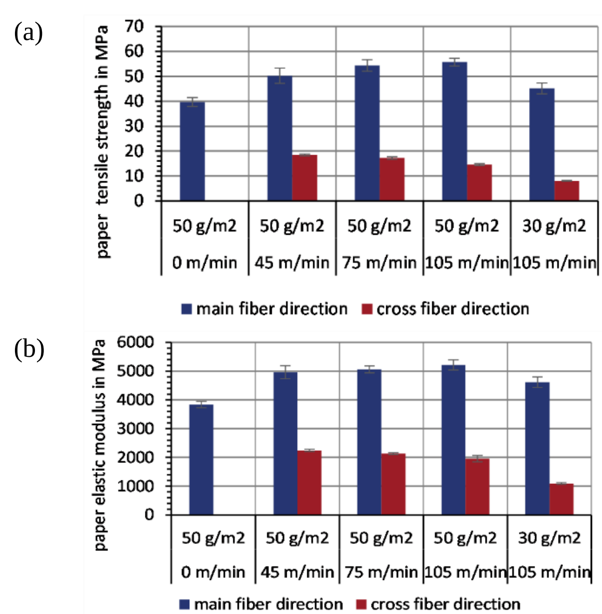

Fig. 4. Paper tensile test results, **(a)** tensile strength and **(b)** elastic modulus. The error bars indicate the standard deviation.

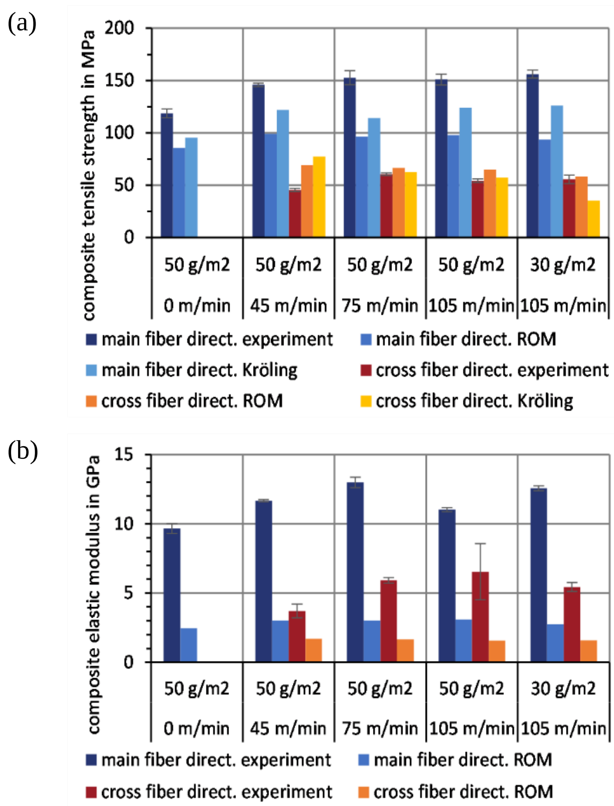

Fig. 5. Composite tensile test results and modeled values, **(a)** tensile strength and **(b)** elastic modulus. The error bars indicate the standard deviation.

The paper composites have an average density of 1.25 ± 0.03 g/cm^3 and an average fiber volume fraction of 47.7 ± 1.7 %. **Fig. 5(a)** shows the strength characteristics of the composite tensile test. The breaking stress of the composite from isotropic paper is about 119 MPa. The breaking stress of the oriented composites is in the range of 145 to 153 MPa. No clear increase in breaking stress with the WJSD or with the TSI in MD can be seen. The fracture stress of the composite from the 30 g/m^2 paper has the highest value with about 156 MPa. This paper should have the highest fiber orientation due to its low thickness, but this was not quantifiable in the TSI. However, the higher fiber orientation would explain the high composite tensile strength. The breaking stress transverse to the main fiber direction is between 45 and 60 MPa.

The elastic modulus of the composite of isotropic paper is about 9.7 GPa. The elastic modulus of the oriented papers in the main fiber direction are between 11 and 13 GPa and transverse to the main fiber direction between 3.7 and 6.5 GPa. Here, no clear correlation with the fiber orientation in the paper is visible (**Fig. 5(b)**).

The elongation at break of the composite (**Fig. 6**) of isotropic paper is approx. 3.4 %. The elongations at break of the oriented papers in the main fiber direction are between 3.5 and 4.9 %. Cross to the main fiber direction, the values vary greatly and the standard deviations are large. The composite of the 30 g/m^2 paper has the smallest value with only 1.8 %. The paper from a WJSD of 45 m/min has the largest elongation at break with about 6.8 %. All data can be found in **Table 1**. The differences in the TSI as well as the measured strength properties between the samples are significant according to a one-factorial analysis of variance with an alpha error of 0.05.

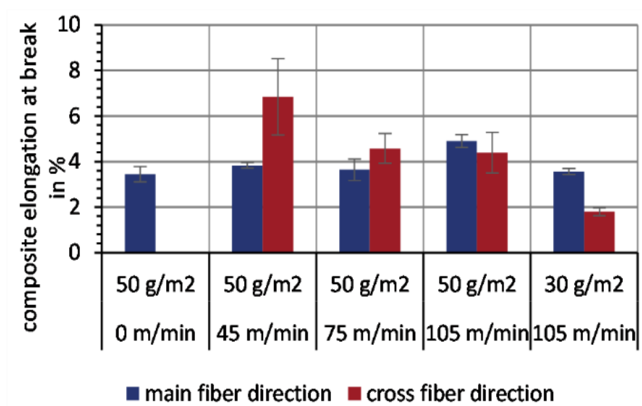

Fig. 6. Composite elongation at break. The error bars indicate the standard deviation.

Table 1. Determined values of the paper and composite tests.

Orientation	WJSD	Basis weight	TSI	fiber tensile strength	ZSTI	paper tensile strength	paper elastic modulus	fiber volume fraction	fiber weight concentration	composite elongation at break	composite tensile strength	modeled tensile strength (ROM)	modeled tensile strength (Kröling)	composite elastic modulus	modeled elastic modulus (ROM)
/	m/min	g/m^2	kNm/g	MPa	kNm/g	MPa	MPa	/	/	%	MPa	MPa	MPa	MPa	MPa
MD	/	52.08 ± 1.28	0.00 # 0.00	84.00 ± 4.27	0.142 ± 0.007	39.65 ± 1.81	3839 ± 111	0.48	0.56	3.44 ± 0.34	118.65 ± 4.18	85.56	95.29	9662 ± 363	2463
	45	50.09 ± 0.93	11.54 ± 0.19	111.90 ± 5.91	0.180 ± 0.009	50.24 ± 3.03	4960 ± 230	0.48	0.58	3.83 ± 0.13	145.84 ± 1.75	99.03	121.80	11665 ± 82	3013
	75	49.23 ± 0.50	11.83 ± 0.09	106.51 ± 6.61	0.175 ± 0.011	54.40 ± 2.31	5056 ± 123	0.47	0.56	3.64 ± 0.47	152.70 ± 6.76	96.21	113.98	12979 ± 379	3014
	105	47.28 ± 1.27	11.93 ± 0.10	109.82 ± 7.30	0.180 ± 0.012	55.68 ± 1.57	5217 ± 174	0.47	0.57	4.90 ± 0.28	150.94 ± 5.26	97.75	123.89	11025 ± 154	3088
	105	33.27 ± 1.01	11.91 ± 0.48	101.44 ± 5.19	0.201 ± 0.010	45.12 ± 2.19	4618 ± 182	0.45	0.54	3.56 ± 0.13	156.28 ± 3.88	93.52	126.04	12571 ± 180	2739
CD	45	50.09 ± 0.93	5.84 ± 0.08	50.88 ± 2.13	0.082 ± 0.003	18.48 ± 0.35	2245 ± 40	0.49	0.59	6.83 ± 1.68	45.49 ± 1.47	69.26	77.39	3702 ± 502	1708
	75	49.23 ± 0.50	5.51 ± 0.12	45.56 ± 2.72	0.075 ± 0.004	17.24 ± 0.47	2131 ± 31	0.50	0.58	4.58 ± 0.66	60.46 ± 1.29	66.39	62.53	5920 ± 204	1658
	105	47.28 ± 1.27	5.18 ± 0.08	42.37 ± 2.95	0.070 ± 0.005	14.62 ± 0.37	1958 ± 114	0.50	0.55	4.38 ± 0.88	54.25 ± 1.61	64.89	57.23	6537 ± 2028	1571
	105	33.27 ± 1.01	3.21 ± 0.22	23.86 ± 2.20	0.047 ± 0.004	8.09 ± 0.17	1100 ± 27	0.45	0.57	1.80 ± 0.17	55.53 ± 4.12	58.47	35.14	5437 ± 316	1593

Modeling

The tensile strength and the elastic modulus are modeled using the rule of mixture (ROM). In addition, the tensile strength is modeled using the Kröling model. For the strength values of the matrix, a breaking stress of 87 MPa and an elongation at break of 10.3 % were assumed [**7**]. The tensile strength of the fibers is determined in the ZeroSpan measurement and corresponds to the breaking force in relation to the cross-sectional area. The fiber content, in turn, was determined using equation (2).

As shown in **Fig. 5(a)** and **Fig. 5(b)**, the models are only reasonably capable of modeling the composite strength as well as the elastic modulus. In the main fiber direction, the strength parameters are underestimated. The Kröling model describes the tensile strength better than the ROM model, but the values are also too low. In contrast, both models overestimate the tensile strength transverse to the fiber direction, with the exception of the composite made of approx. 30 g/m^2 paper. Modeling the elastic modulus with ROM, the modeled values are far below the experimentally determined ones. The measured E-modulus is three to four times larger than the modeled E-modulus. This might be due to the fact that modeling was carried out with the elastic modulus of the paper and not of the paper fiber. In addition, it must be mentioned that paper fibers as well as their strength properties are also not homogeneous, as ROM assumes. Consequently, all models must be improved or other models must be used to describe these paper composites. Possible model improvements would be to take into account the non-homogeneous property distribution of the fibers, the consideration of the fiber elastic modulus or the use of other paper and matrix properties.

Comparison

Kröling [**7**] formed composites from laboratory sheets and oriented paper from the Dynamic Sheet Former. The sheets tended to have a high basis weight, which meant that the fiber content was only between 20 and 40 %. Kröling achieved the highest tensile strength of 142 MPa with the composite with the highest fiber orientation of the original paper. The laboratory sheets used by Kröling have a basis weight of 160 g/m^2 and the composites formed from them have a fiber content of 39,1 % and a tensile strength of about 101 MPa. In their studies, Prambauer *et. al.* [**3**] obtained the highest strength values for composites made from copy paper with a basis weight of 80 g/m^2. They used polypropylene grafted with maleic anhydride. For this specific composite, the tensile strength was 88 MPa and the elastic modulus was 6.8 GPa, based on a fiber content of about 40 %. Zadorecki *et. al.* [**18**] prepared composites of kraft paper and polyester that had a fiber volume fraction of 32 %. Tensile strengths of 98.0 MPa and an elastic modulus of 8.1 GPa were measured for these composites. The strengths of Kröling, Prambauer *et. al.* and Zadorecki *et. al.* are lower than the comparable composite strengths studied in this paper. This may be due to the lower fiber content, the different matrix, the different paper, and especially the different degree in fiber orientation.

The strength characteristics are comparatively lower than for carbon or glass fiber reinforced composites, but advantages of the paper composites are the low density as well as the low paper price.

CONCLUSION

In this work, composites were prepared from isotropic as well as oriented laboratory papers and epoxy resin. It was shown that the paper as well as composite strength properties in the main fiber direction is greater in the oriented papers/composites than in the isotropic papers/ composites. By changing the wire jet velocity difference during production, the degree of fiber orientation can be adjusted, but this is not significantly seen in the measurement results. However, the composites made here from highly oriented paper have higher strength characteristics than comparable paper composites made from isotropic as well as oriented paper from other works. It should be noted that the composites differ in terms of the resin used, the raw paper as well as the fiber content. An important advantage of paper composites is their sustainability. Natural fibers store CO_2 during growth. They are recyclable and may be safely thermally recycled. In the long term, it is likely possible to replace the epoxy resin with a bio-based solution as well. Another advantage is the low density of the paper composites as well as the very low raw paper price compared to carbon or glass fiber reinforced composites, making paper composites economically attractive. Paper composites can be used, for example, for automotive casings, interior solutions in ships or mobile homes, in lightweight construction applications, as well as in the building sector.

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CONFLICTS OF INTEREST

There are no conflicts to declare.

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SUPPORTING INFORMATION

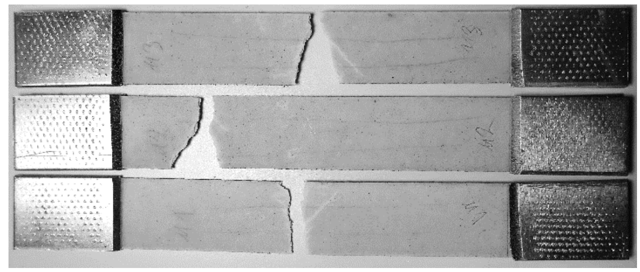

Fig. 7. Image of composites after mechanical testing (main fiber-direction).

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GRAPHICAL ABSTRACT

Paper composites from oriented and isotropic paper as well as epoxy resin are generated. The influence of fiber orientation in the paper on composite strength is investigated. The results indicate an increase of tensile strength with increasing fiber orientation.