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A new light transmission method to evaluate the through thickness fibre alignment in transparent resin

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ABSTRACT

This work presents a new method to analyse vertical alignment of short fibres inside transparent resin layers using in situ measuring of light transmission through the layer. In this method, a light source is placed on one side of the composite layer and on the opposite side, a phototransistor measures the transmitted light intensity. A feature of the alignment process causes increased light transmission with increased fibre alignment. Using this technique, alignment of short nickel coated carbon fibres inside epoxy resin layers under magnetic field were evaluated and the effect of fibre content, field strength, fibre length and resin thickness on the alignment quality were characterised. An important finding is that the ratio of fibre length and resin thickness showcases a limit to the alignment degree. Finally, an analytical model is introduced which aims to predict average fibre angles based on the sample configuration, showcasing good agreement with experimental results.

1. Introduction

Fibre reinforced polymer (FRP) composite materials have excellent strength and stiffness to weight ratio in comparison with metallic materials. Industries specially in the transport sector strive to replace their legacy metallic components with those made from composite materials [1]. In the aerospace industry, continuous carbon fibre/epoxy resin laminates are most popular material of choice, once arranged in a multi-directional stack, provide very good in-plane mechanical properties [2]. However, the lack of through thickness reinforcement of laminated composites results in poor through thickness properties, with the inter-laminar strength being the major weakness of laminated composite materials. For this reason, delamination of the laminate layers often become the dominant failure mechanism in FRP composite laminates. Improving the through thickness reinforcement of these materials is an ongoing field of research, with many approaches from interleaving [3], Z-pinning [4,5] to 3D weaving [6] have been proposed.

One approach to through thickness reinforcement is the use of short carbon fibres in composites which is useful to generate the necessary improvements in fracture toughness, especially if the fibres are aligned normal to the crack growth direction. This alignment could also provide additional electrical and thermal property advantages [7,8]. Through thickness alignment of the short fibres could be utilized to manufacture very thin composite films with oriented thermal and electrical properties. Embedment of these thin films between composite laminates could be used to makes the composite laminate increase its vertical thermal and electric conductivity and act as a through thickness reinforcement. Despite these benefits, through thickness alignment of fibres in thin resin film is very complex and more challenging in comparison with alignment in bulk resin systems [9]. Film thickness relative to fibre length is an important feature that can contribute to restrict fibre mobility for alignment.

Different methods have been demonstrated to change the orientation of short fibres suspended inside liquid resin through the application of a suitable force. The force could be either mechanical such as ultrasonic vibrations [10], resin flow induced orientation [11], or through application of an external field such as electrical [12,13] and magnetic fields [14,15].

Applying an electrical field to a liquid resin/fibre mixture, induces a dipole torque in the conductive short fibres which causes them to rotate to the lowest energy position which is in the field direction [16,17], This technique is applicable with both AC and DC currents, however, despite its simplicity, high electrical voltage (100–200 V/cm) are needed which can limit this procedure for application in large scale production [9].

Alignment using magnetic field is an alternative efficient approach thus attracting interest of several researchers [18]. In this method, the short fibres (e.g. carbon fibres) coated with a thin layer of ferromagnetic material (e.g. Nickel) allows a magnetic field to impose magnetic torque

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to the fibres, which rotates and moves them to the lowest energy position, being the longest dimension of the fibre aligned parallel to the magnetic field direction. The torque magnitude depends on multiple parameters which include fibre shape anisotropy, its angle with the field vector and ferromagnetic properties of fibre. This torque can be theoretically calculated using specifically developed analytical models [15,19]. For alignment in epoxy resin, this driving torque is opposed by momentum of drag force of viscous resin, consequently an equilibrium is achieved which determines the final angle of the fibre.

Alignment of the fibres using magnetic field has two useful advantages in comparison to the other methods. First advantage is its contactless nature which provides ability to manipulate the fibres freely in a 3D configuration. Second advantage is the accessibility of low-cost magnetic sources (e.g. permanent magnet) which create strong enough magnetic field to conduct the alignment procedure [20]. For even more accurate control of the magnetic field, electromagnets may be utilised.

The evaluation of fibre alignment has predominantly been conducted using analysis of the final cured sample cross section. One of the first research works in this field is by Yamashita et al. [21] which experimentally evaluated the orientation of nickel coated carbon fibres using magnetic fields inside bulk resin. It was shown there to be an upper limit to the critical fibre volume fraction above which alignment using magnetic field is not achievable. Maya et al. [20] used confocal microscopic images and numerical analysis to evaluate and quantify the alignment of nickel coated carbon fibre inside epoxy resin at different magnetic fields, which have been created using neodymium permanent magnets. The use of microscopic images to evaluate fibres alignment has been reported by several other researchers as well [22,23].

Evaluation of fibre alignment using microscopic images has some weaknesses which can affect the results and procedure. Other than the challenging and time-consuming nature of analysis, the most important problem of this method is reduction of a 3D tilted fibre to a 2D image, which will significantly affect the results as none of out of plane tilts can be detected. The use of two perpendicular images (cross sections) may improve the results but significant errors can result through the machining and polishing process. Nevertheless, none of these methods provide a way to assess the alignment rate. With in situ measurement of the fibre alignment with magnetic field, a great insight can be gained on the behaviour of various parameters to the alignment quality. Also, there are no research conducted on the alignment behaviour of short fibres inside thin resin films.

In this research, a new experimental method has been developed which allows for in-situ monitoring of fibres alignment. This method based on the light transition through a transparent resin which allows for measurement of the transmitted light intensity as a function of fibre position, angle and time. This new method is relatively simple to conduct, which can provide useful data to understand the alignment rate and level based on different parameters. In this paper the alignment degree of short ferromagnetic fibres of varying length are evaluated inside resins of different thicknesses. The analysed data is then used to create an analytical model based on shadow projection of fibres, to estimate the average fibre angles achieved during the alignment process. The proposed approach can then be used to predict the alignment quality relative to several important parameters such as resin thickness to fibre length ratio, magnetic field strength and fibre content.

2. Fibre alignment characterisation

2.1. Materials and sample preparation

Short nickel coated carbon fibres are selected as the reinforcing phase (Marktek Inc. Company). The nominal length of the fibres is 0.1, 0.5 and 1 mm (diameter of the fibres is ~6.5 μm) and the weight fraction of the coating nickel is 40%. Araldite LY1564 epoxy and XB3403 hardener (Huntsman Corp.) with weight ratio of 100:36 was used as the matrix. This resin system has low initial viscosity (~200 mPa S) at room temperature with long pot life (above 15 h), making it suitable for this investigation.

Epoxy and hardener were added to a pot with the appropriate ratios and mixed uniformly by hand. Then dry short carbon fibres were added to the mixture and stirred manually for ~2 min until full immersion of the fibres. Due to the fibre agglomeration and entanglement, the pot was then placed inside an ultrasonic bath in controlled room temperature for 5 min to achieve homogenous distribution in the resin mixture. The prepared resin was then gently poured to transparent glass Petri dishes to the required thickness by controlling sample weight. Due to very low viscosity of the resin and the low thickness of the resin sample, any bubbles inside resin (which mostly were created during fibre dispersion) escape immediately after pouring to the petri dishes therefore degassing was not necessary.

2.2. Alignment measurement

Principle of the developed method for alignment measurement is based on the light transmission through the sample, illustrated in Fig. 1a. The sample is placed in a fixture which has an LED light source directly below and a phototransistor above. The magnetic field is produced using two 100 mm square permanent ferrite magnets with 25 mm thickness. The field strength is controlled by changing the distance between the magnetic surfaces as shown in Fig. 1b. The magnetic field at the midpoint of the two square magnets is completely vertical (confirmed with theoretical calculations from Camacho and Sosa [24]) which results in exact conditions necessary to achieve full through thickness alignment of the fibres. The position of the plates inside the fixture are carefully controlled to achieve the exact magnetic field intensity at the level of the resin mixture. The magnetic field intensity was measured using a handheld Tesla meter.

Once the sample is placed in position, the phototransistor collects continuous measurement of the received light intensity through the petri-dish and resin mixture. As the fibres begin to align in the thickness direction (Y axis), their projected cross section becomes smaller, which results in higher light intensity measured.

With this process the transmitted light vs. time curves can be collected and plotted for different conditions. Typical light intensity plot is shown in Fig. 2. The initial condition of the test is first calibrated by adjusting the light intensity to a constant value called reference light (RL).

In this study, the reference light is set as 6000 LUX and calibrated with petri dish filled with neat resin to the desired resin thickness. This means the light intensity is adjusted to 6000 LUX for each resin thickness before each test batch. The entire test is conducted inside an evenly lit room with no change in external light source throughout each test. Once calibrated, the neat resin sample is replaced with the resin fibre mixture sample which immediately initiates the fibre alignment process. This calibration process effectively normalises the data collected without needing to consider light refraction through the petri dish and the resin mixture.

To note, this proposed alignment measurement method is best suited for low thickness resins. Since a larger resin mixture would mean very high quantity of fibres in the light path direction that would effectively block most of light passage, even when fibres are relatively aligned. Through trial and error, the authors have found that to improve the measured data quality, the reference light intensity should not be extremely high or low. With either too high or low RL the sensitivity to the transmitted light become too small to detect the relative changes reliably.

2.3. Test procedure

Overall, two set of tests have been conducted with first set to compare the effect of magnetic field strength, fibre content and fibre length on the alignment quality and the second set to compare the resin

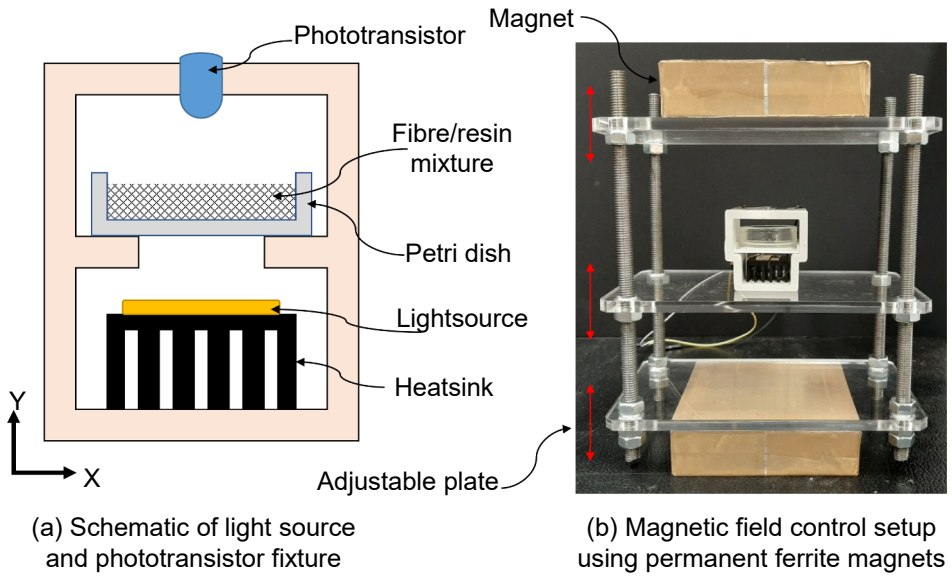

Fig. 1. Fibre alignment measurement test fixture. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

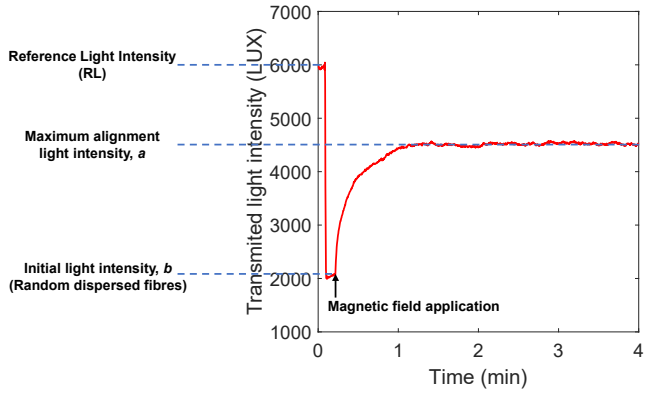

Fig. 2. A typical light transmission plot with key features highlighted. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

thickness influence.

The first test set will characterise the alignment of 0.5 mm and 1 mm fibres at four different fibre content weight fractions (W_f) 0.5 wt%, 1.0 wt%, 1.5 wt% and 2.0 wt% in four different magnetic field strength 20, 40, 60 and 80 mT with resin thickness of 2.5 mm. Each test condition was repeated three times, in total, 96 tests have been conducted.

The second test set will measure the alignment quality of samples with W_f of 1 wt% of 0.1, 0.5 and 1 mm fibres in resin thickness range between 0.5 mm up to 6 mm.

To conduct quantitative comparison of the measured alignment quality, an alignment degree (α) is defined as:

$$\alpha = \frac{a-b}{RL-b} \tag{1}$$

where b is the initial light intensity equivalent to the fibres in random distribution and *RL* is reference light intensity which fixed at a constant 6000 LUX in this study. For each sample tested, there exists an upper plateau in the maximum transmitted light intensity that is reached. As can be seen in an example from Fig. 2 this is defined as the maximum alignment plateau, a which often occurs quickly after few minutes or may take as long as few hours to reach. To achieve a consistent and non-subjective approach to measure this plateau, an exponential function Eq. (2) is fitted to the non-linear portion of the curve initiating at the point of magnetic field application.

$$lightintensity = a-(a-n_1)e^{n_2 t^{n_3}} \tag{2}$$

Using the nonlinear least-squares solver in MATLAB toolbox the parameters a, n_1, n_2 and n_3 are fitted. The fitted value of *a* is thus equivalent to the maximum light intensity at infinite time and is subsequently used in Eq. (1).

Alignment degree (α) shows the ratio of the increase in the detected light intensity to the reference light intensity (RL). The inclusion of *b* in this equation normalises the effect of initial position/dispersion of the fibres, this is important as different starting conditions of the fibres before alignment were found to have significant influence on the results for the same sample configuration.

2.4. Results and discussions

2.4.1. Magnetic field strength and fibre volume fraction

In Fig. 3, the results of light intensity vs alignment time curves for fibre length of 1 mm are shown. For all the fibre content, the increase in magnetic field strength results in higher light transmission which equates to more transparency in the resin because of increased vertical fibre alignment.

This is attributed to the higher magnetic force and torque which is induced to the fibres at higher field strength. For all conditions, the curves reach a plateau after only a few minutes and only a few cases it takes longer (>60 mins) to reach the plateau. Once the maximum light intensity has been reached, continued application of the magnetic field does not improve the alignment.

What is quite clear from the results is the increase in fibre content causes significant decrease in the overall alignment. Even at highest magnetic intensity of 80mT, the maximum alignment degree achieved for 2.0 wt% fibre content is 0.24 ± 0.1. The reason for this is due to the fibres interlocking, effectively getting tangled, which prevent free movement and rotation of fibres. Increased fibre content may appear to reduce the alignment rate (time to reach plateau). This behaviour again indicates the higher interlocked fibres, gradually and partially separate from to align. These tests highlight that gravity and resin drag forces are not the only forces which the magnetic force need to overcome to achieve high alignment degree. It is clear the level of fibre dispersion prior to the alignment has strong influence on the overall alignment degree.

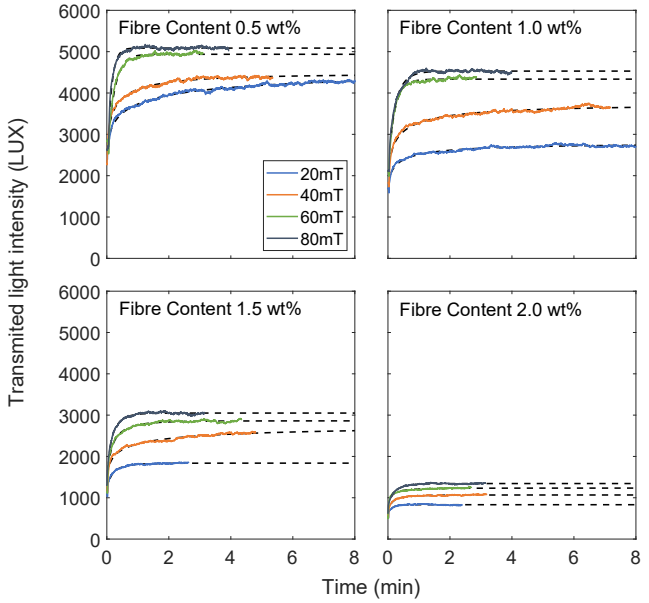

Fig. 3. Light transmission curves for alignment of 1 mm fibres inside 2.5 mm thick resin with change in magnetic field intensity and fibre content. Dashed lines are fitted exponential function (Eq. (2)). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

It should be considered that due to very high aspect ratio of the fibres (~150), the fully aligned fibres would occupy very negligible cross section of sample in the thickness direction.

The alignment profile for 0.5 mm fibre length is shown in Fig. 4. The results follow similar trend to the 1 mm fibres length tests with two significant differences. The first difference is the lower maximum transmitted light intensity of the 0.5 mm fibres than those for 1 mm fibres. This indicates that magnetic momentum is proportional to the fibre length and longer fibres can achieve better alignment (even though longer fibres may possess higher drag force). Also, the total number of fibres for 0.5 mm will be double those of 1 mm fibres when using same weight percentage, which will increase the interlocking of the shorter fibres. The second significant difference is the alignment rate, which is lower than 1 mm fibres, since more samples (especially in samples with high fibre content) take over 8 mins to reach the plateau. Again, this can be attributed to the higher number of fibres and interlocking which reduce fibre mobility and hinder alignment quality. This can be an issue for resin system with short pot-life or when the manufacturing processing speed needs to be high. To note, although, duration of some of the tests is short, the plateau was calculated using the fitted function (Eq. (2)) as explained in Section 2.3.

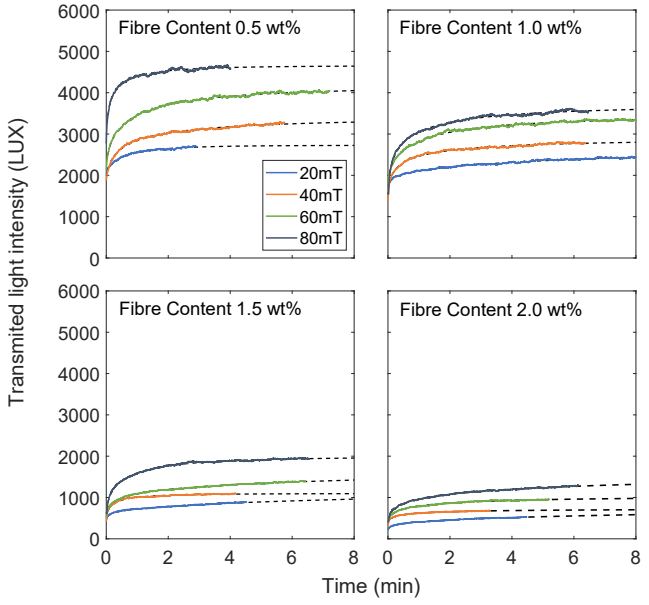

Fig. 4. Light transmission curves for alignment of 0.5 mm fibres inside 2.5 mm thick resin with change in magnetic field intensity and fibre content. Dashed lines are fitted exponential function (Eq. (2)). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

From all the tests conducted, four samples were selected to conduct cross section microscopy to evaluate the alignment degree qualitatively. These samples were kept inside the alignment fixture at room temperature for over 48 h to allow for resin to completely cure. The chosen samples were 20 mT, 2 wt% fibre content and 80mT, 1 wt% fibre content for both 0.5 mm and 1 mm fibres lengths.

Fig. 5a shows cross sectional image of 0.5 mm fibres with the conditions producing lowest alignment quality. As can be seen, there are many fibres that appear to be vertically aligned in 20 mT field strength, however there are many more fibres still interlocked. The high fibre concentration (2.0 wt%) and low field strength is not able to overcome the entanglements. The increase of magnetic field to 80mT and fibre content reduction to 1 wt% seen in Fig. 5b show cases a well aligned sample with only few evidence of non-vertical fibres.

Fig. 5c show the alignment of 1 mm fibres at 20 mT with 2 wt% fibre content and 80 mT, 1 wt% fibre content samples respectively. The alignment quality is very similar to those of 0.5 mm samples.

The cross-section images also confirm the adequate initial mixture quality with no agglomeration or voids present.

When fillers inside a matrix become exposed to strong magnetic field for long durations, they will become aligned but also may coalesce into a chained network [17,19]. In all the conducted tests in this study, there were no visual indication of this chained network. In this study, the sample thickness to fibre length ratio is not high enough to allow parallel fibres to coalesce and make longitudinal chains. However, in a different experiment it was observed cases of a very strong and non-uniform magnetic field can cause in-plane fibre migrations and make dense fibre islands in a wide resin layer disturbing the uniformity of the fibre distribution. This behaviour was not evident in the small samples and relatively low magnetic field strengths used in this paper.

Using Eq. (1) the alignment degree (α) for all the test samples have been calculated and presented in Fig. 6. It is clear for 2 wt% fibre content, for both fibre lengths at low magnetic field strength, there is negligible alignment with average alignment degree of <0.1. For 0.5 mm fibres, at low magnetic field strength of 20 mT, the highest alignment degree, even for 0.5 wt% samples are ~0.2. At the highest end, the best alignment was achieved for 1 mm fibre length at 0.5 wt% fibre content with 80mT magnetic field strength with alignment degree of 0.76 ± 0.015 (Fig. 5d). For the same condition with 0.5 mm fibres, the highest alignment degree achieved was 0.61 ± 0.032 (Fig. 5b). Looking at the alignment quality from Fig. 5b we can see that even at alignment degree of 0.61 we have majority of the fibres appearing to be vertical in orientation. Therefore, we can make a qualitative assessment that achieving an alignment degree of >0.6 can be considered good alignment quality.

Results in Fig. 6 show large scatter which can be attributed to fibre dispersion homogeneity right before application of magnetic field. This dispersion inhomogeneity increases when the number of fibres is low or for longer fibre lengths. Tighter scatter may be achieved if the initial mixing conditions is improved and larger sample area for light transmission measurement is used.

2.4.2. Resin thickness

To investigate the effect of resin thickness on the alignment degree,

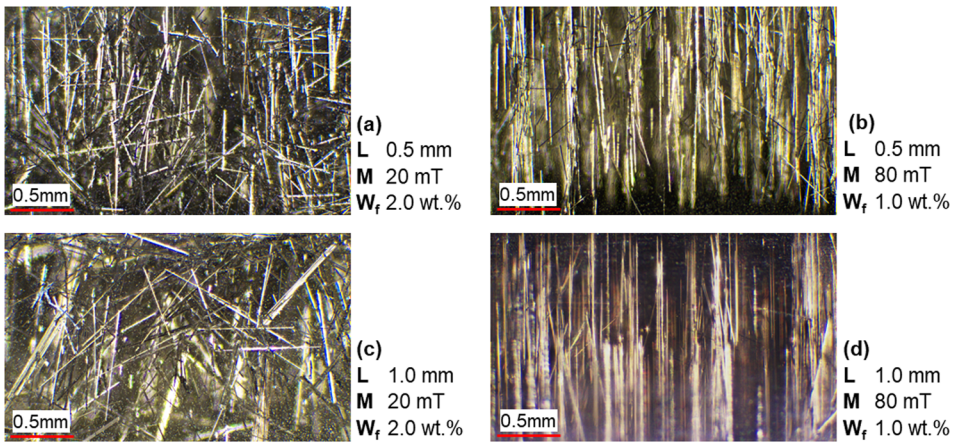

Fig. 5. Cross section microscopy of samples at four selected alignment conditions inside 2.5 mm resin thickness. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

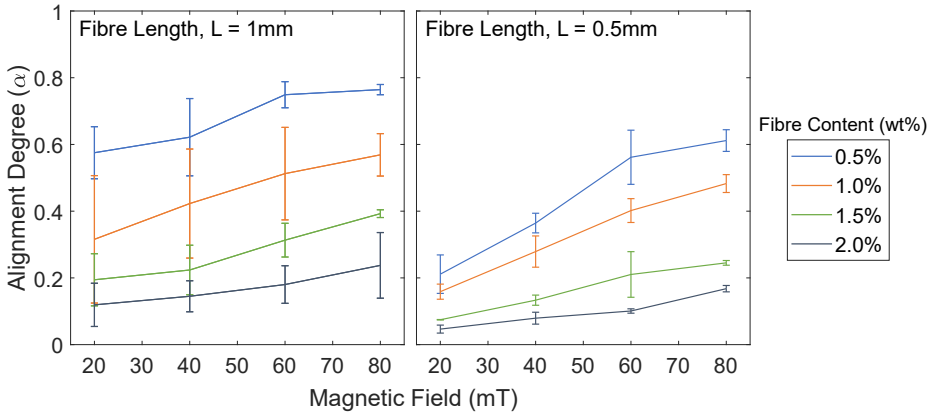

Fig. 6. Alignment degree (α) for 0.5 mm and 1 mm fibres in different conditions inside 2.5 mm resin thickness. (Error bars indicate 1sd in the scatter). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

three different fibre lengths of 0.1 mm, 0.5 mm and 1 mm were tested at fixed magnetic field strength of 80mT and the fibre content of 1 wt%, see Fig. 7. For 1 mm fibre length at equal resin thickness of 1 mm, the alignment degree is negligible at <0.1. Visual inspection of the samples verified this with the fibres not appearing any different to the original random state. The increase of the resin thickness up to 4 mm, improves the alignment degree achieving an average constant alignment degree of ~0.7 with relatively larger scatter.

The same trend is seen for 0.5 mm fibres showing negligible alignment for resin thickness of equal size. It is clear, as the fibres try to rotate, the resin free surfaces halt the fibres free movement. Only at sample thickness above 2.0 mm do we start to see maximum alignment degree which remain on average constant at ~0.4. The same behaviour was expected for 0.1 mm fibres but due to the limitations of epoxy surface tension on a glass petri dish, it was not possible to make resin thickness smaller than 0.5 mm. The alignment degree for 0.1 mm fibre

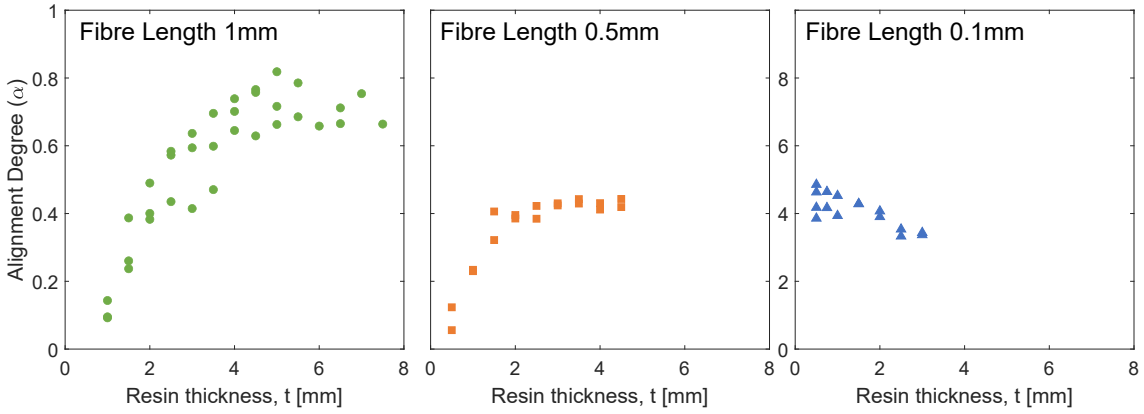

Fig. 7. The effect of resin thickness on the alignment degree (α) of 1.0, 0.5 and 0.1 mm fibres lengths with 1 wt% fibre concentration and 80mT magnetic field strength. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

lengths remain relatively constant around 0.3–0.4. The slight and gradual reduction with increase of thickness is not due to lower alignment of the fibres, but due the fact that larger resin thickness accommodates more fibres in thickness direction resulting in more instance of transmitted light being blocked or scattered.

The lack of alignment of fibres in low sample thicknesses can be attributed phenomenon of higher force needed to overcome the surface tensions near the free surface of the fluid. So, according to the newton law for fluids, higher shear stress is required to make a relative movement for the fibre and separate it from the free surface. It appears, the fibres stick to the surface and a very high force (proportional to the resin viscosity and reversely proportional to the fibre/surface distance) is needed to move it. To reduce the effect, the surface tension and viscosity of the resin will need to be reduced, either through increased temperature or use of surfactant.

In Fig. 8 the data is replotted with the resin thickness normalised against fibre length. The trend suggests that above normalised thickness of ~5 the alignment quality remains constant. Meaning to achieve maximum alignment degree, the resin thickness needs to be 5 times larger than the fibre length. For 0.1 mm fibre length this equates minimum resin thickness of 0.5 mm and for 0.5 mm fibre length, minimum resin thickness of 2.5 mm to achieve maximum alignment degree. For a thin film application, the resins of 0.5 mm or thicker may no longer considered to be thin film. Thereby to achieve improved alignment degree in thin film form, further investigation will need to explore ways to improve the alignment process.

3. Analytical model of alignment angle

The alignment degree (α) presented is the measure of the transmitted light intensity through the samples. It is necessary to find a relation between the alignment degree (α) and the average fibre angles. In this section a theoretical model is developed based on the fibre projected area (i.e. fibre shadows), probability of the fibre overlap and overall change in this during the convergence of the alignment process to predict the average fibre angles. Two key assumptions are:

- The light is completely parallel and vertical to the sample surface (any refraction/reflection is considered negligible).
- The intensity of light detected by phototransistor is proportional to the area of the resin that is transparent and not obstructed by the fibres

Consider a sample with 1 × 1 mm^2 surface area and thickness of equal to the fibre length (Fig. 9). In this instance the fibres are initially vertical then rotate to a given angle.

The projected area of a fibre (A) and a small change of that (dA) due to the fibre small rotation (dθ) can be described using Eqs. (3) and (4).

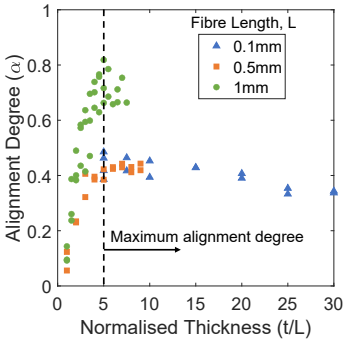

Fig. 8. Alignment degree (α) for normalised thickness (resin thickness/fibre length) for 0.1, 0.5 and 1 mm fibres with 1 wt% fibre content and 80mT magnetic field strength. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

Where L is the fibre length; d is the fibre diameter and θ is the average fibre angle.

$$A = \left(\frac{\pi}{4}\right)\cdot d^2\cdot sin\theta + d\cdot L\cdot cos\theta \tag{3}$$

$$dA = \left(\frac{\pi}{4}\cdot d^2\cdot cos\theta - d\cdot L\cdot sin\theta\right)d\theta \tag{4}$$

The projected shadow of a single fibre at angle θ can be calculated with the integration of Eq. (4) with θ in the range of 90° (fully vertical) to θ with 0° being fully horizontal which becomes:

$$Shadowofasinglefibre : A_1 = \int_{90}^{\theta}\left(\frac{\pi}{4}\cdot d^2\cdot(1+cos\theta) - d\cdot L\cdot sin\theta\right)\cdot d\theta \tag{5}$$

For a single fibre, calculation of the shadow area is trivial, however, if we consider all the fibre quantities, n together, the situation will be more complicated due to the overlapping fibre shadows varying fibre angles. To solve this problem, an average angle is considered for all the fibres and a probability factor P(θ) is introduced which is the ratio of transparent area to total area:

$$p(\theta) = \frac{transparentarea}{totalarea} = \frac{1 - ShadowArea}{1} = \frac{1 - A_T(\theta)}{1} \tag{6}$$

The probability factor, P(θ) is ~1 when all fibres are fully vertical (θ = 90°) and reduces with decreasing θ. This means as fibres become more aligned, they have higher probability of covering the transparent area than overlapping with the shadows of other fibres. According to this factor, any dθ change in a fibre angle which leads to dA change in the projected area, will result in P(θ)·dA change in the transparent area of the sample. For samples with multiple overlapping fibres, Eq. (5) is multiplied by total number of fibres, n and the probability factor, P(θ):

$$Shadowofmultiplefibres(A_T) = \int_{90}^{\theta} n\cdot\left(\frac{\pi}{4}\cdot d^2\cdot(1+cos\theta) - d\cdot L\cdot sin\theta\right)\cdot P(\theta)\cdot d\theta \tag{7}$$

For higher fibre content or resin thicknesses, the factor reduces because of increased shadow area. The effect of fibre content, angle and resin thickness on the probability factor value is shown in Fig. 10.

By embedding Eq. (6) into Eq. (7), the shadow of all fibres together could be calculated implicitly:

$$A_T(\theta) = \int_{90}^{\theta} n\cdot\left(\frac{\pi}{4}\cdot d^2\cdot(1+cos\theta) - d\cdot L\cdot sin\theta\right)\cdot(1 - A_T(\theta))\cdot d\theta \tag{8}$$

Number of fibres in the 1 mm^2 area of the sample with thickness of T and fibre weight fraction of W_f is defined according to:

$$n = \frac{4\cdot w_f}{\pi\cdot d^2\cdot(2 - w_f)}\cdot\frac{T}{L} \tag{9}$$

Thereby, embedding Eq. (9) in Eq. (8) will result in:

$$A_T(\theta) = \int_{90}^{\theta}\frac{4\cdot w_f}{\pi\cdot d^2\cdot(2 - w_f)}\cdot\frac{T}{L}\cdot\left(\frac{\pi}{4}\cdot d^2\cdot(1+cos\theta) - d\cdot L\cdot sin\theta)\right)\cdot(1 - A_T(\theta))\cdot d\theta \tag{10}$$

For a sample 1 mm^2 when the average angle of fibres is θ, the transparent area will be:

$$Transparent\ area\ (a) = 1 - \int_{90}^{\theta}\frac{4\cdot w_f}{\pi\cdot d^2\cdot(2 - w_f)}\cdot\frac{T}{L}\cdot\left(\frac{\pi}{4}\cdot d^2\cdot(1+\cos\theta) - d\cdot L\cdot\sin\theta\right)\cdot(1 - A_T(\theta))\cdot d\theta \tag{11}$$

The ratio of calculated transparent area at any angle (θ) (which could be conducted numerically for given variables) to total area (which is unity) is equivalent to the intensity of transmitted light through the sample to the reference light intensity.

In the experimental tests, the alignment degree (α) was introduced

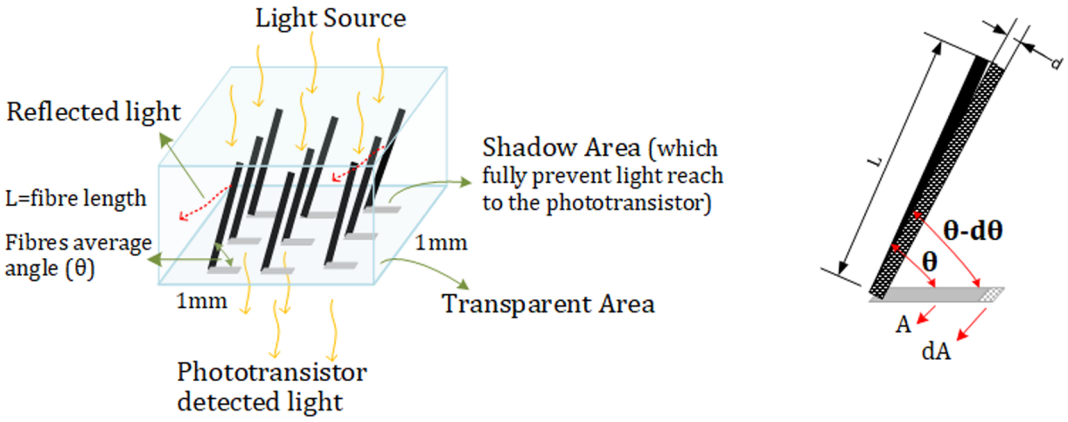

Fig. 9. Illustration of the fibre alignment inside resin (left) and the projected area of a single fibre or shadow area (right). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

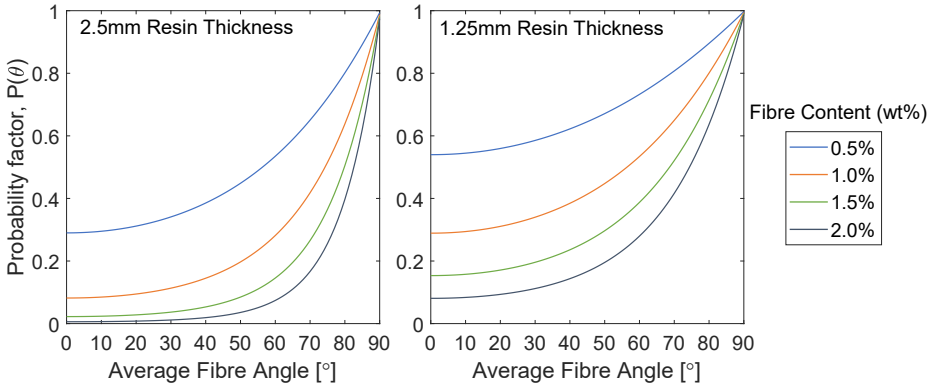

Fig. 10. Effect of average fibre angle on the probability factor P(θ) for different resin thicknesses and fibre contents. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

which defines the fibre alignment based on the initial distribution and position and the reference light intensity. As was shown in the previous section, the initial condition of the fibres had a strong influence on the final reference light intensity. This change was due to various reasons including the initial distribution and fibres settling to different levels before alignment initiation. For example; when the resin/fibre system gets mixed and dispersed just before the magnetic field application, it was found the fibres show improved alignment compared to those where the sample had chance to settle.

To include the initial condition in the theoretical model an assumption is made that all the fibres are randomly and homogenously distributed in the 3D space before the alignment. This means that the fibres have average angle of 45° which results in different transparent area ratio for each fibre weight fraction. The initial transparent area ratio, b' are calculated numerically and given in Table 1.

By rewriting Eq. (1) in the form of Eq. (12) where the reference light intensity is normalised to 1, b' is the unaligned fibres transparent area ratio (initial conditions given in Table 1), a' is the transparent area.

$$\alpha = \frac{a' - b'}{1 - b'} \tag{12}$$

Table 1
Initial condition of the transparent area 1 mm fibre length.

Fibre content (wt.%)	0.5%	1.0%	1.5%	2.0%
Initial transparent area (b') for 2.5 mm sample thickness	0.41	0.17	0.066	0.026
Initial transparent area (b') for 1.25 mm sample thickness	0.64	0.41	0.26	0.17

With Eqs. (11) and (12) the average fibre angle relation to the experimentally measured alignment degree, α is numerically calculated and presented in Fig. 11 for 1.25 mm and 2.5 mm resin thicknesses. Only one fibre length is shown in Fig. 11, given that the difference between 1 mm and 0.5 mm fibres was <1% which can be neglected.

3.1. Model verification

To verify the theoretical model prediction of the average alignment angle, the samples in Fig. 5 were analysed using image processing toolbox of MATLAB to extract the average fibre angles visible in the images. Although these microscopic images are useful for qualitative analysis, it should be considered that angles measured in the microscopic images will have an error since they are 2D projection of true 3D orientation. This error is illustrated in Fig. 12 where the out of plane tilt, γ is clearly lost in the projected microscopic images. To correct for this, the relation between true fibre angle (θ) and projected angle of microscopic images (β) is derived to be:

$$\beta = Arctan\left(\frac{L \cdot sin(\theta)}{L \cdot cos(\theta) \cdot cos(\gamma)}\right) = Arctan\left(\frac{tan(\theta)}{cos(\gamma)}\right) \tag{13}$$

By assuming a uniform tilt of fibres in different directions, the relation for all of the fibres could be averaged according to:

$$\beta = Arctan\left(\sqrt{2} \cdot tan(\theta)\right) \tag{14}$$

As it can be seen in Eq. (14), projected angles are always bigger than true angles with the difference between these two angles for different alignment is given in Fig. 12.

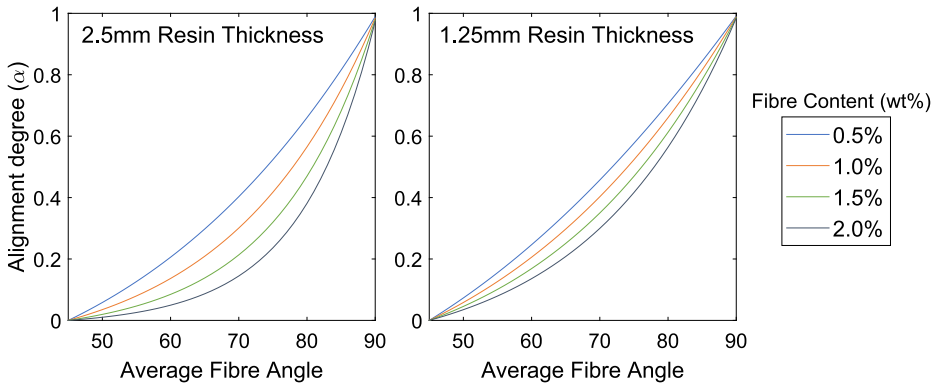

Fig. 11. Theoretical model relating alignment degree (α) to the average fibre angle for different resin thicknesses and fibre contents. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

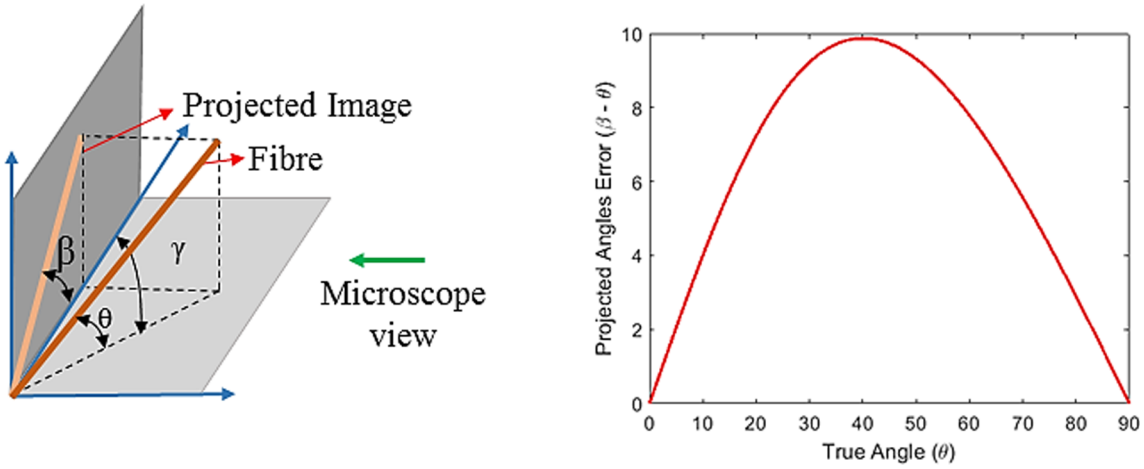

Fig. 12. Relation between projection angle of microscopic images and true angle of fibres. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

For the four sample cross section images given in Fig. 5, the average of projected angles is measured using image processing toolbox of MATLAB. From each image over 100 fibres are detected with each of their projected angles directly measured and averaged. The average projected angles are then normalised using Eq. (14). Using the average experimental alignment degree α, the corresponding theoretical angle using Fig. 11 were also calculated. The results are presented in Table 2.

As can be seen the developed theoretical model estimates the average angle of fibres accurately for well aligned samples, however, the accuracy reduces significantly when the alignments quality is low. This can be attributed to three main reasons, first, uncertainty in alignment degree value which has high variation at low quality alignments. Second, uncertainty in the image processing procedure where detection of the fibres angle is not ideal since only a minority of the fibres are visible. Third, assuming an average angle for all of the fibres in theoretical model, which works well for highly aligned conditions but results in increased errors in for low alignment or randomly dispersed fibres. Nevertheless, with maximum error of 8%, the proposed model and analysis procedure is in good agreement.

Table 2
Comparison of the average angle from experimental results against theoretical model (angles in degrees).

Sample	1 mm, 80 mT, 1 wt%	0.5 mm, 80 mT, 1 wt%	1 mm, 20 mT, 2 wt%	0.5 mm, 20 mT, 2 wt%
Projected average angle (experiment)	84	82	66	62
True average angle	81	79	58	53
Theoretical model average angle	80	77	66	59
Error	−1%	−2%	8%	6%

4. Discussions and conclusion

In this investigation a new method based on the light transmission is developed for in situ monitoring of the alignment of short fibres inside a transparent resin system. A new theoretical model is developed to help evaluate the results by equating the alignment degree to average fibre angle inside the mixture. The presented procedure works well allowing the alignment degree and rate to be measured for different conditions of fibre content, fibre lengths, resin thicknesses and magnetic field intensity.

Overall, it was found for the alignment at the beginning of the process to be very fast, with most of alignment occurring within few seconds of introducing the magnetic field. A plateau is often reached after few minutes. Across all conditions increase of magnetic field strength was shown to produce increased alignment degree. A key limiting feature to alignment quality was found to be the fibre content weight percentage. Samples with 2 wt% fibre content showed very limited alignment whilst maximum alignment degree were reached with 0.5 wt% samples. An interesting phenomenon observed was the change in the maximum alignment degree for samples of same fibre content but different fibre lengths. Decreasing the fibre length reduced alignment degree across all fibre content weight percentages. The reason for this is due to the higher

number of shorter fibres causing more chances of entanglements thereby reducing alignment quality.

Resin thickness has a significant effect on the fibre alignment. The overall alignment was found to be negligible when the resin thickness is equal to the fibre length. This is because fibre rotations being limited at the free surface of the resin according to the newton's fluid mechanics law. In order to achieve maximum alignment degree, the resin thickness must be at least 5 times the fibres length, which is one of the new findings of this study.

The proposed theoretical model based on the fibre shadow projection method, allows for the conversion of the alignment degree to the average fibre angle. Verification of this method showed good agreement with experimentally measured fibre angles and demonstrates this approach to be effective for measuring in-situ average short fibre angles from light transmission.

In the future studies, continuous production of thin through thickness aligned and B-staged resin layers will be investigated. Achieving high through thickness fibre alignment in low resin thickness to fibre length ratio will be a key challenge and worth investing methods to achieve improvement to this.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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