NONDESTRUCTIVE, NONCONTACT QUANTIFICATION OF CARBON FIBER ALIGNMENT AND ORIENTATION BY HIGH-SPEED MICROWAVE ELLIPSOMETRY

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ABSTRACT

Novel short-fiber composites facilitate the manufacture of tailorable feedstock for small formed parts. In these composites, the alignment and orientation of the short fibers must be controlled to achieve the desired composite properties. While there are several processing variables that can be correlated to fiber alignment and orientation, there is a need for a fast, nondestructive, noncontact measurement technique to quantify local alignment and orientation in real time. Such a technique would enable real-time control of processing variables, resulting in higher quality composites. Here, we propose high-speed microwave ellipsometry as such a technique. To evaluate our approach, we measured five short-fiber composites samples made from a four-layer stack of carbon-fiber mats. These samples included one known control sample and four blind samples that were unknown at the time of testing. The four blind samples were known to be either a control, a sample with all layers rotated by 5°, a sample with a single unknown layer rotated by 5°, or a sample with a single unknown layer rotated by 15°. In this paper, we present our results demonstrating the effectiveness of this technique and discuss a path for real-time, large-scale imaging of fiber alignment and orientation.

1. INTRODUCTION

To realize commercial tailorable feedstock, materials engineers need to produce highly aligned layers of fibers and they need to control the orientation of each layer in the ply feedstock [1]. This need implies that there are at least two process variables of interest: fiber alignment [2] and fiber orientation [3].

In this paper, alignment is a measurand that quantifies the average alignment of each carbon fiber in a layer relative to each other. A relative alignment with a value of one means that all the carbon fibers in a layer are parallel to one and another. A relative alignment with a value of zero means that all the carbon fibers are uniformly distributed by angle relative to each other. Likewise, we define orientation as the orientation of a layer relative to the direction of the electric field in the measurement (Fig. 1). A relative orientation is an angle that describes which direction the carbon fibers in all the layers point to relative to some electric field orientation. When multiple layers form a ply, the alignment and orientation values from each layer combine.

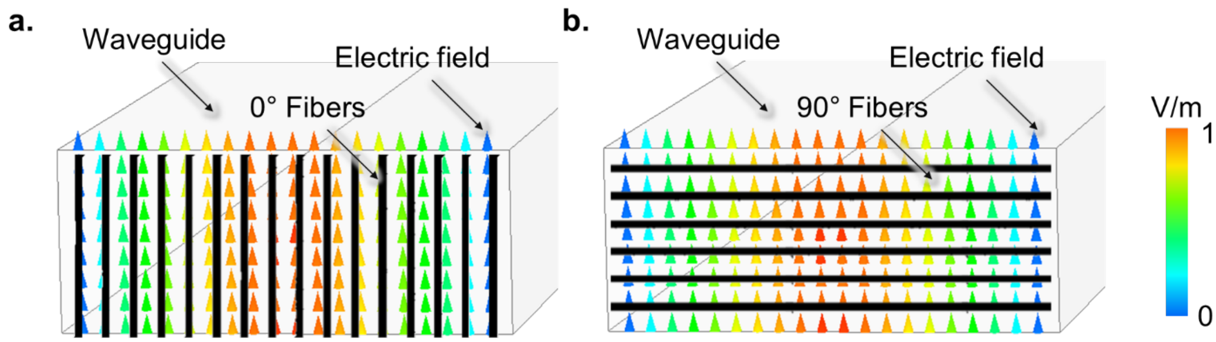

Figure 1. A schematic of how carbon fibers (black lines) can be oriented relative to an electric field (colored arrows) in waveguide. (a) A parallel orientation, which is defined as 0°. (b) A perpendicular orientation, which is defined as 90°. The color bar indicates the strength of the electric field in V/m.

There are several ways to measure alignment and orientation [4]. For example, imaging the ply on a light table is a common technique. In our experience, this technique also works on multilayer samples. To get spatial information, an image can be segmented. This optical technique can be fast, leveraging advances in real-time image processing (*e.g.*, OpenCV). This optical technique is also transmission-based. As such, it does not work for samples that cannot pass light, cannot differentiate between layers, and it is difficult to implement for three dimensional samples. Yet another technique uses eddy current sensors [5]. This eddy current approach has some trade-offs, which include operating in the near-field with a small working distance and requiring the composite to have a conductivity within a specific range [4].

Microwave ellipsometry is an alternative approach (Fig. 1) [3]. This technique uses the relative reflected power of a microwave signal measured as a function of the angle between the carbon fibers and the polarized incident microwave radiation [6]–[8]. When the electric field is parallel to the carbon fibers, the electric field moves charge along the length of the fiber. The current flow in this case is much the same as it would be in a conducting sheet, resulting in a combination of reflections and absorption of the electrical signal. When the electrical field is perpendicular to the carbon fibers, the electric field cannot move charge in the fiber as far, and instead charges must capacitively couple from fiber to fiber through the host matrix in the ply. In this case, the ply acts more like an insulator that a conductor, and there is a substantially higher amount of transmission through the ply in addition to some reflection and absorption. The resultant relative reflected power versus multiple angles creates a squared-ellipse model on a polar graph, which can then be related to the alignment and orientation. The microwave ellipsometry approach can work on thick samples, it is comparatively easy to implement in three dimensions, it can work in the far field, and it can potentially work with a wide range of conductivities.

In what follows, we demonstrate microwave ellipsometry on short-fiber plies. In section 2, we introduce the theory behind microwave ellipsometry (2.1) and show our rudimentary experimental setup (2.2). Next, we discuss our blind sample test (2.3). In this test, each ply had three layers that were aligned, and a middle layer was misaligned by some nominal amount. We apply our step-by-step procedure and show our results on the blind-sample test. Finally, we provide an outlook for scanning microwave ellipsometry based on its strengths and elaborate on the drawbacks of this technique as implemented here.

2. EXPERIMENTATION

2.1 Theory

Microwave ellipsometry uses the relative reflected power, which is measured with a vector network analyzer (VNA). The VNA measures a relative reflection coefficient (Γ), which is related to the reference impedance of the port (Z_r) and the impedance of the sample (Z_{sample}). The resultant reflection coefficient is given as,

$$\Gamma = \frac{\mathrm{Z_r} - \mathrm{Z_{sample}}}{\mathrm{Z_r} + \mathrm{Z_{sample}}}.$$

Error! Bookmark not defined.(1)

The relative reflected power is simply the square of the reflection coefficient. After we measured (1), we found that the general form of a squared ellipse modeled the relative reflected power as,

$$\mathrm{P} = \Gamma^2 = \left(\frac{\mathrm{a} \cdot \mathrm{b}}{\sqrt{\mathrm{a}^2 \cos^2(\theta - \theta_o) + \mathrm{b}^2 \sin^2(\theta - \theta_o)}}\right)^2,$$

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where a is the minor axis, b is the major axis, and the orientation is described by θ_o. From (2), we define the orientation as θ_o, and the alignment as,

$$\chi_{ab} = 1 - \frac{a}{b}$$

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It is valuable to consider the limits of (3). For a highly aligned very conducting sample when the electrical field is aligned with b then b is close to one, and a is close to zero. Any nonzero value of a is then due to electrical conduction through the matrix of the ply, capacitive coupling between fibers and any reflection from the isotropic substrate material, which would tend to be the same for any angle. In this case, the value of χ_{ab} in (3) is close to 1. Conversely, for a sample that has highly conducting fibers that are uniformly distributed throughout the layer at random angles, the value of χ_{ab} in (3) is close to zero, since a is similar in value to b. This former case represents a layer (or ply) that is aligned, and the latter case represents a layer (or ply) that is not aligned. At this point, we can only say that for weakly a conducting matrix an alignment closer to one indicates a more aligned sample. Control samples with known alignments could calibrate this alignment value, allowing us to quantify alignments across different processes. That said, we assert that simple variations in the alignment value suffice for process control.

We note that (2) was true for our measurement configuration, where the backing plane consisted of a dielectric and a metal plate. In later experiments not shown here, we found that a Cassini oval was a better model of relative reflected power as a function of angle. In this case, we used a microwave absorber in lieu of the dielectric spacer. Either approach works, but the latter case the signal-to-noise was bigger. Figure 2 shows the squared-ellipse function as the blue solid line, and the minor and major axes are labeled with the orientation.

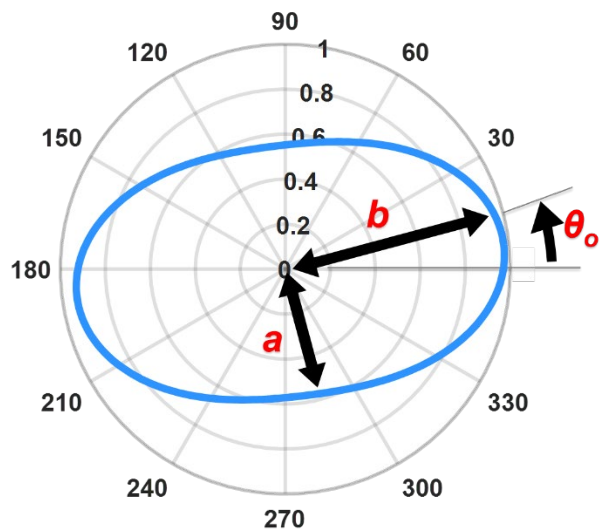

Figure 2. A squared ellipse that models the relative reflected power as a function of angle, where the angle is defined as the angle between the electric field and the sample-under-test. The blue solid line is the squared-ellipse fit, θ_o is the orientation, a is the minor axis, and b is the major axis.

2.2 Experimental setup

Our experimental setup used a rotation stage, a foam dielectric spacer, a WR-42 rectangular waveguide flange, a translation stage, and a vector network analyzer (VNA). We connected the VNA to the waveguide flange with a coaxial cable and contacted the waveguide flange to the material-under-test via a micromanipulator. After positioning the flange over the axis of rotation, we rotated the material-under-test to different fixed angles. At each angle, we measured Γ (Eq. 1) with the VNA at 18 GHz.

We show a schematic of the measurement setup in Fig. 3a and a photograph of the measurement setup in Fig. 3b. In the next section, we provide a detailed step-by-step experimental procedure.

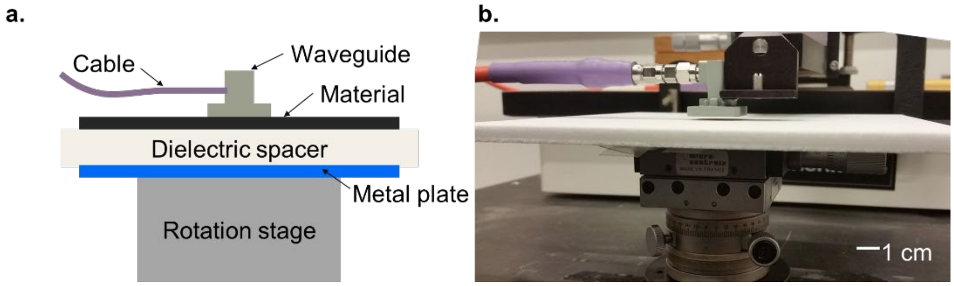

Figure 3. The microwave ellipsometry measurement setup. (a) A schematic of the measurement setup. (b) A photograph of the measurement setup.

2.2.1 Step-by-step procedure

We developed a step-by-step procedure to show how our microwave ellipsometry measurement works in practice.

1. Perform a 1-port coaxial calibration at the end of the cable that connects to the waveguide flange.
2. Perform a waveguide calibration at the end of the waveguide flange.
3. Level the waveguide flange with a reference flat (Fig. 4a).
4. Validate that the waveguide flange is at the center of the axis of rotation.
5. Perform a waveguide calibration at the end of the waveguide flange.
6. Place a sample on the dielectric spacer.
7. Align the sample to the edge of graph paper affixed to the dielectric spacer (Fig. 4b).
8. Rotate the sample (Fig. 4c).
9. Measure Γ.
10. Repeat step 6 through step 9 for the chosen number of angles between 0° and 360°.
11. Flip sample and repeat step 10 for the other side of the sample-under-test.
12. Square the measured values to compute the relative reflected power.
13. Fit the data from step 9 to a squared-ellipse model.

14. Compute the alignment and orientation.

15. Move to a different position on the sample and repeat step 1 through step 8.

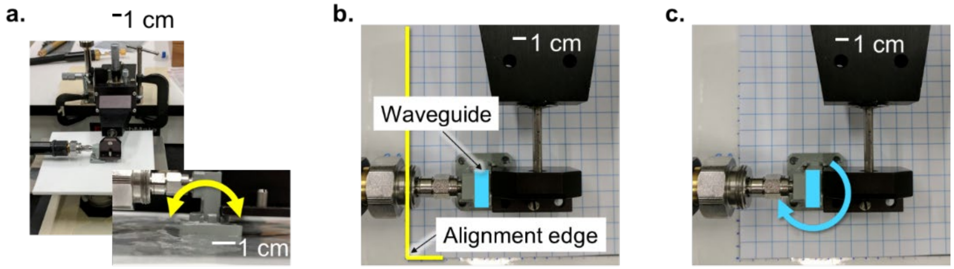

Figure 4. Selected steps from the step-by-step procedure. (a) Level the waveguide flange with a reference flat. (b) Align edge of the sample to the edge of graph paper that is affixed to the dielectric spacer with polyimide tape. (c) Rotate the sample.

2.3 Blind samples

To test our measurement procedure, we obtained four blind samples and a control sample (Fig. 5). The four blind samples had an unknown sample orientation and alignment at the time of measurement. Each sample consisted of four layers of aligned short carbon fibers. For an individual layer, we used IM7* carbon fiber that had been chopped to an average length of approximately (5.0 ± 0.1) mm. The microstructure of samples made from the alignment process is discussed in detail in elsewhere.

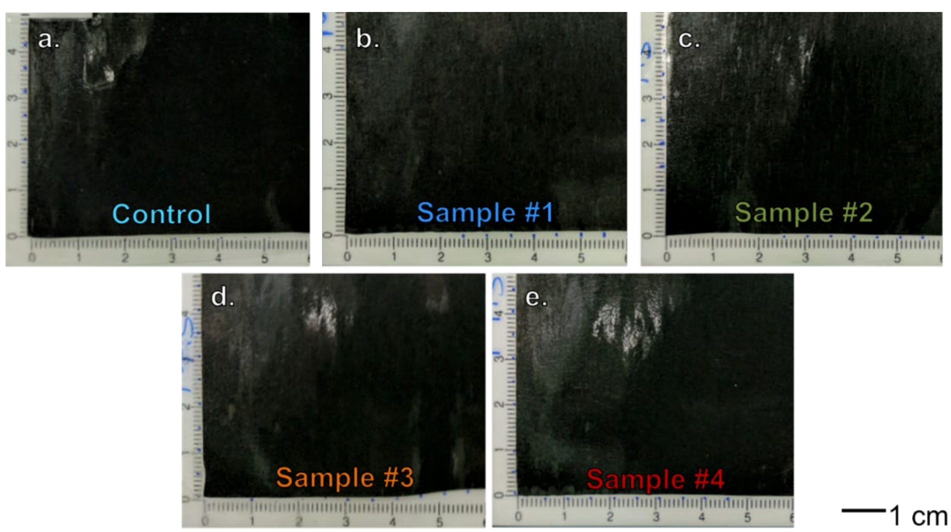

Figure 5. Short carbon fiber samples used to test the microwave ellipsometry measurement technique. (a) Control sample with each layer to minimize the light transmitted through the ply. (b) Sample #1 was identical to the control. (c) Sample #2 had all the layers rotated by 5°. (d) Sample #3 had the second of four layers rotated by 5°. (e) Sample #4 had the second of four layers rotated by 15°. At the time of measurement, these alignments were unknown.

After completing the fabrication of an individual layer, we fabricated the ply. To make the ply, we aligned each layer on a light table, controlling the layer-to-layer alignment with a protractor. Once we aligned the fibers, we impregnated and consolidated the ply with a thermoplastic polymer (polyetherimide – Ultem 1000*) in autoclave for 4 hours at 2×10^6 Pa and 335 °C. For the control sample, we aligned each layer to minimize the light transmitted through the ply. For the blind samples, Sample #1 was identical to the control, Sample #2 had all the layers rotated by 5°, Sample #3 had the second of four layers rotated by 5°, and Sample #4 had the second of four layers rotated by 15°. Figure 5 shows the control and blind samples. Each sample was approximately 6 cm×5 cm.

2.3.1 Test cases

Before we measured the blind samples, we measured two controls: no sample, and a laminated aluminum sheet. We took the 'no sample' measurement by placing the waveguide flange in direct contact with the dielectric spacer. We took the 'aluminum' measurement by placing the waveguide flange in direct contact with a laminated aluminum sheet placed on top of the dielectric spacer. We expected that the 'no sample' case would look like a circle on the microwave ellipsometry graph with a smaller radius (Fig. 6a), because the signal propagated and was absorbed or radiated out the sides of the dielectric spacer before a fraction of the power was reflected into the waveguide flange. We expected that the 'aluminum' case would also look like a circle on the microwave ellipsometry graph with a radius close to one (Fig. 6b), because the signal was immediately reflected into the waveguide flange and more of the signal was reflected. In contrast with these isotropic samples, Sample #2 (Fig. 6c) shows a rotated squared-ellipse, which is consistent with an aligned carbon fiber sample with a 5° orientation.

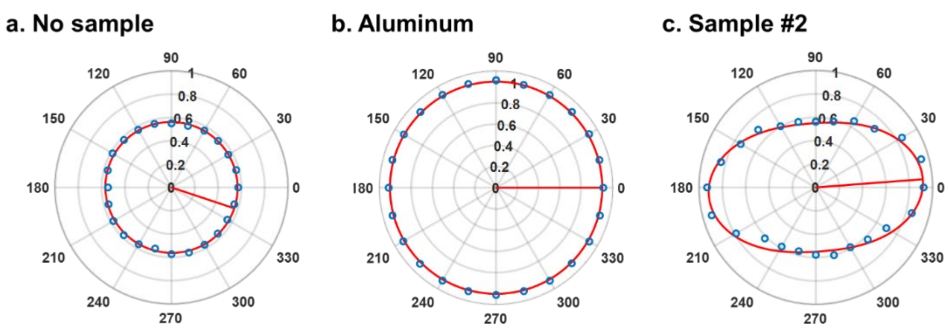

Figure 6. Microwave ellipsometry data at 18 GHz for no sample, an aluminum sheet, and Sample #2. (a) 'no sample' case measurements (blue circles) appears as a circle, has a radius less than one, and fits an isotropic model (red circle). (b) Aluminum case measurements (blue circles) appears as a circle, has a radius close to one, and fits an isotropic model (red circle). (c) Sample #3 measurements (blue circles) appears as an ellipse, has an orientation close to 5°, and fits an anisotropic model (red circle).

3. RESULTS

After validating our measurements with the test cases in Section 2.3.1, we measured all the blind samples, and the control sample. We then fit the microwave ellipsometry data to the model in (2), extracting the orientation (θ_o) and alignment (χ_{ab}). We computed uncertainty in the orientation (θ_o) and alignment (χ_{ab}) by propagating the error via a numeric Jacobian incorporating the measurement uncertainty and fit residuals. Unfortunately, the error in the orientation included the uncertainty of placing the sample on the dielectric spacer by hand, which added in quadrature to approximately $\pm$ 1°. This contribution to the uncertainty was very large compared to the other sources of error. Table I shows a summary of our results.

Table I. Blind samples results for orientation (θ_o) and alignment for (χ_{ab}). Uncertainties are rounded up.

Name	Description	Orientation, θ_o[°]		Alignment, χ_{ab} [a.u.]	
		Side A	Side B	Side A	Side B
Control	Nominally aligned	0.5 ± 1.2	NA	0.37 ± 0.01	NA
Sample #1	Nominally aligned	0.4 ± 1.1	-0.2 ± 1.0	0.36 ± 0.01	0.37 ± 0.01
Sample #2	All layers rotated by 5°	5.4 ± 1.2	-5.3 ± 1.3	0.40 ± 0.01	0.40 ± 0.01
Sample #3	One middle layer rotated by 5°	1.0 ± 1.1	-0.1 ± 1.0	0.38 ± 0.01	0.37 ± 0.01
Sample #4	One middle layer rotated by 15°	4.6 ± 1.1	-3.3 ± 1.4	0.27 ± 0.01	0.26 ± 0.01

3.1 Orientation measured by microwave ellipsometry

From Fig. 7, we determined that Sample #1 (blue) was identical to the control (light blue). Based on the orientation data, we also determined that Sample #2 (green) had all layers rotated by 5°, and Sample #4 (red) had one middle layer rotated by 15°. If the error in the orientation had been less than $\pm$1°, we could have also concluded that Sample #3 (orange) had had one middle layer rotated by 5°. Taking a quarter of 5°, we obtained a value of 1.25, which is consistent with the value obtained for Sample #3 Side A. Likewise for Sample #4, a quarter of 15° is also consistent with the value obtained for Sample #4. For Sample #3 (orange), we speculate that layer rotated by 5° was closer to Side A. For Sample #4 (red), we speculate that layer rotated by 5° was also closer to Side A.

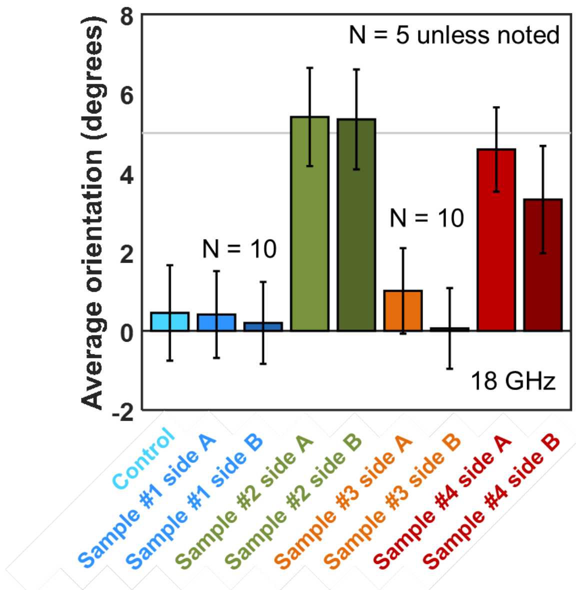

Figure 7. Absolute values for the average orientation for control and four blind samples. Side A and Side B corresponded to the top-facing and bottom-facing sides, respectively. The number of measurements on each sample was N = 5 for the control, Sample #2, and Sample #4. For Sample #1 and Sample #3, the number of measurements on each sample was N = 10. The measurement frequency was 18 GHz.

3.2 Average alignment measured by microwave ellipsometry

From Fig. 8, it appears that Sample #2 had the highest alignment value compared to the other blind samples in the test. Sample # 4 had the lowest alignment value compared to the other blind samples in the test. Based on Fig. 7, we expected that Sample #4 should have the lowest alignment, because this sample had a middle layer rotated by 15°. This sample shows that orientation and alignment become convolved when measuring multiple layers. From the perspective of the electric field (Fig. 1), the carbon fibers in this sample appeared more uniformly distributed. The other samples had similar alignment values compared to the uncertainty. We observed that Sample #3, which has a middle layer rotated by 5°, has a comparable alignment value to the control and Sample #1, which is identical to the control.

Simulations (not shown) suggest that microwave absorber in lieu of a metallic plate changes the squared-ellipse model into Cassini oval model, which improves signal-to-noise. This experimental modification is intended for future work. Even in this preliminary configuration the uncertainty in the alignment values from waveguide ellipsometry is small compared to the variance of the alignment value over the nominally aligned samples (Sample #1, Sample #2). This fact is important to note because it implies that alignment as measured by waveguide ellipsometry can inform process control.

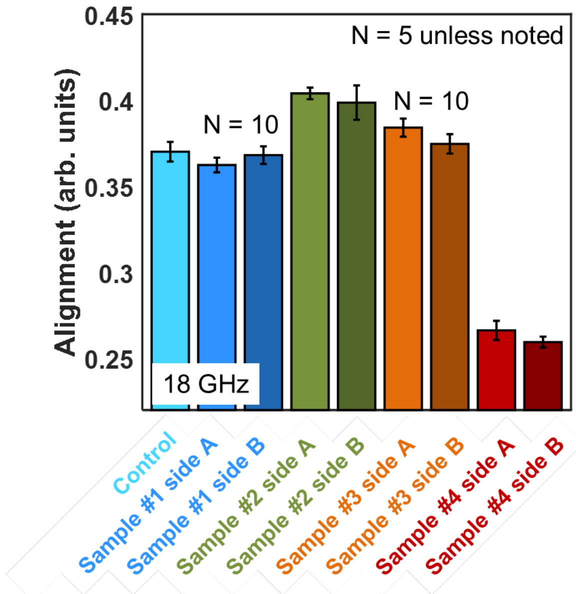

Figure 8. Absolute values for the average alignment for control and four blind samples. Side A and Side B corresponded to the top-facing and bottom-facing sides, respectively. The number of measurements on each sample was N = 5 for the control, Sample #2, and Sample #4. For Sample #1 and Sample #3, the number of measurements on each sample was N = 10. The measurement frequency was 18 GHz.

4. CONCLUSIONS

In this paper, we demonstrated quantitative measurements of alignment and orientation of short fiber composites by microwave ellipsometry. Next-generation processing techniques need in-line quality assurance tools for real-time feedback of processing variables, and formed parts require *in situ* characterization as the material is laid up to improve control. We showed that by acquiring the relative reflected power as a function of angle at 18 GHz we were able to quantify the alignment and orientation, demonstrating a new technique that can be used for in-line process control and for formed parts.

To test our approach, we measured four samples whose nominal misalignment was unknown at the time of test. After we applied the microwave ellipsometry technique, we successfully identified the alignment of each sample. By measuring both sides of each sample, we were able to speculate about which side of the sample had the nominally misaligned layer. We applied this approach to carbon fiber composites. Future work will integrate robotics to create three dimensional images of alignment and orientations. Additional efforts include the development of real-time scanning systems. Finally, we developed an uncertainty analysis and concluded that the uncertainties in the initial alignment of the sample relative to the waveguide flange was the dominant source of error.

We summarize by stating that carbon fiber alignment and orientation of multilayer composites can be quantified by microwave ellipsometry.

5. ACKNOWLEDGMENTS

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6. REFERENCES

[1] S. W. Yurgartis, "Measurement of small angle fiber misalignments in continuous fiber composites," *Compos. Sci. Technol.*, vol. 30, no. 4, pp. 279–293, Jan. 1987.

[2] A. Anwer and H. E. Naguib, "Multi-functional flexible carbon fiber composites with controlled fiber alignment using additive manufacturing," *Addit. Manuf.*, vol. 22, pp. 360–367, Aug. 2018.

[3] Z. Li, A. Haigh, C. Soutis, and A. Gibson, "Principles and Applications of Microwave Testing for Woven and Non-Woven Carbon Fibre-Reinforced Polymer Composites: a Topical Review," *Appl. Compos. Mater.*, vol. 25, no. 4, pp. 965–982, Aug. 2018.

[4] "Review on quality assurance along the CFRP value chain – Non-destructive testing of fabrics, preforms and CFRP by HF radio wave techniques - ScienceDirect." [Online]. Available: https://www.sciencedirect.com/science/article/pii/S1359836815001419. [Accessed: 01-Feb-2019].

[5] G. Bardl *et al.*, "Automated detection of yarn orientation in 3D-draped carbon fiber fabrics and preforms from eddy current data," *Compos. Part B Eng.*, vol. 96, pp. 312–324, Jul. 2016.

[6] F. Gambou, B. Bayard, and G. Noyel, "Characterization of material anisotropy using microwave ellipsometry," *Microw. Opt. Technol. Lett.*, vol. 53, no. 9, pp. 1996–1998, 2011.

[7] F. Sagnard, F. Bentabet, and C. Vignat, "In situ measurements of the complex permittivity of materials using reflection ellipsometry in the microwave band: theory (part I)," *IEEE Trans. Instrum. Meas.*, vol. 54, no. 3, pp. 1266–1273, Jun. 2005.

[8] F. Sagnard, F. Bentabet, and C. Vignat, "In situ measurements of the complex permittivity of materials using reflection ellipsometry in the microwave band: experiments (Part II)," *IEEE Trans. Instrum. Meas.*, vol. 54, no. 3, pp. 1274–1282, Jun. 2005.