Effect of Carbon Nanofiber Z-Threads on Mode-I Delamination Toughness of Carbon Fiber Reinforced Plastic Laminates

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Abstract

Delamination is a major drawback of carbon fiber reinforced plastics (CFRPs). Studies have reported that carbon nanofibers (CNFs) can improve the delamination toughness of various FRPs. However, lack of CNF alignment control caused substantial uncertainty in the improvements. In this study, a novel CNFs z-threaded CFRP (ZT-CFRP), which utilized z-aligned CNFs as long-range reinforcement threading through the packed carbon fiber bed, was manufactured. The mode-I delamination toughness (G_{IC}) of the ZT-CFRPs was tested against both control CFRPs and unaligned CNF-modified CFRPs (UA-CFRPs). Through statistical comparison against control CFRPs, UA-CFRPs exhibited a relative change in mean G_{IC} and coefficient of variation of +13.99% and +116.35%, respectively, whereas the ZT-CFRPs of equivalent CNF concentration exhibited a relative change in mean G_{IC} and coefficient of variation of +28.93% and -12.33%, respectively. Accordingly, the CNF z-threads were found to play a positive role in toughening CFRPs, as supported by delamination experiments and microscopy analysis.

Keywords: A. Carbon fibres; A. Carbon nanotubes and nanofibres; B. Delamination; B. Fracture toughness

1. Introduction

1.1 Background and Previous Works

Given recent advances in composite manufacturing techniques, carbon fiber reinforced plastics (CFRPs) have become commonplace in a wide variety of engineering applications due to their high modulus-to-weight and strength-to-weight ratios. These traits make CFRPs particularly desirable to industries in which weight-savings is paramount, such as the aerospace and automotive industries. However, unlike traditional metals, CFRPs are anisotropic (i.e., the material properties are directionally-dependent). While CFRPs exhibit desirable material properties in the direction of carbon fiber reinforcement, they are severely limited in the through-thickness direction (i.e., z-direction), which lacks a reinforcing fiber network. Furthermore, CFRPs' z-directional mechanical strength is substantially weakened by the carbon fiber-resin matrix interface and the distinctly different mechanical properties between the rigid carbon fiber and relatively softer resin matrix. As CFRPs are typically used as laminated structures, the absence of effective z-directional reinforcement results in an inability to resist delamination when subjected to large out-of-plane mechanical loads.

Various methods have been studied to counteract delamination issues, however, the most common methods involve either fiber stitching [1-3] or nanoparticle reinforcements [4-13], which can be subdivided into interleaving and direct matrix dispersion approaches. Fiber stitching is costly and difficult for complex and large CFRP part manufacturing, thus its applications are constrained by part-size, complexity, and budget. Moreover, the stitching needles can break and interrupt the carbon fiber reinforcement, thereby reducing the CFRP's compressive strength in the x-y plane, particularly under hygrothermal conditions [3]. Therefore, the improvements in interlaminar fracture toughness provided by fiber stitching also cause the reduction in other critical material properties. Recently, nanoparticle reinforcements have also been used to provide significant increases in interlaminar fracture toughness. However, many of the reported results suffered from substantial increases in uncertainty [4, 5, 6, 11, 12].

An increasingly prevalent research approach in the field of nanoparticle reinforcement is the insertion of nano-reinforced resin layers (i.e., nanoparticle enhanced interlayers or interleaves) along the mid-plane of a FRP laminate, which enhances interlaminar strengths [4-7]. Lee et al. [4] achieved mode-I delamination toughness (G_{IC}) increases of over 30% using a CNT-laden nanowoven carbon tissue (NWCT) interlayer.

1 However, the coefficient of variation (i.e., CV, which is standard deviation divided by the mean) of the CNT-
2 NWCT was nearly nine-fold larger than the control CFRP. Arai et al. [5] increased G_{IC} by approximately 50%
3 using a CNF interlayer, yet their G_{IC} plots illustrate that the samples with the CNF interlayer exhibited
4 significantly larger standard deviations than the control sample. Garcia et al. [6] grew a dense z-aligned CNT
5 forest on a silicon substrate, transferred the CNT forest to a prepreg, and then joined two carbon fiber prepreg
6 layers to form a through-thickness aligned CNT forest interlayer (i.e., interleaf); this method provided G_{IC}
7 improvement of approximately 50% and 150% for AS4/8552 and IM7/977-3 commercial prepreps,
8 respectively, along the CNT forest interlayer as compared to the baseline CFRP laminates. Although CNTs
9 and CNFs in nanoparticle-reinforced interlayers have yielded positive results, the increase in thickness,
10 weight, and complexity makes interleaf implementation between every interlaminar plane of a FRP laminate
11 less practical for lightweight composite designs and manufacturing consistency. However, it should be noted
12 that selective interleaving, as elaborated in [6, 7], may still provide a potential method of circumventing these
13 inherent shortcomings by strategically reinforcing critical locations rather than the entire structure.

14 Another common nanoparticle reinforcement approach is to uniformly disperse nanoparticles in the
15 matrix material throughout the FRP. This direct matrix dispersion approach is fundamentally different from
16 the interleaving approach because its purpose is to homogeneously improve the matrix's mechanical
17 properties and also enhance the FRP's interlaminar toughness, which is typically considered a matrix-
18 sensitive property. However, many challenges exist in the dispersion process and the processes used to
19 incorporate the nano-resin into the FRP system. Despite the challenges, CNTs and CNFs have been reported
20 to enhance the interlaminar strength of glass fiber reinforced plastics (GFRPs) via uniform dispersion in the
21 matrix material without adding any interleaves between GFRP laminae. A generic approach is to infuse the
22 nano-resin into a preform of dry fibers via Liquid Composite Molding (LCM) processes. Sadeghian et al. [8]
23 successfully infused 1wt% CNF-modified polyester into GFRP laminates to improve interlaminar fracture
24 toughness by approximately 100%. In their study, a CNF/polyester matrix was infused into the preform of
25 random glass fiber mats using Vacuum Assisted Resin Transfer Molding (VARTM). This study also
26 elaborated on nanoparticle filtration issues, which occurred as the CNF/polyester mixture flowed through the
27 glass fiber preform. As the CNF length (approximately 50-500 μm) is much larger than the pore size within

the glass fiber bed (glass fiber diameter $\sim 20\ \mu\text{m}$, so the pore size is of the same order of magnitude), certain unaligned CNFs could be filtered by the glass fiber bed and gradually clog the flow channel, resulting in uneven CNF concentration and a visible color gradient in the GFRP. Fan et al. [9] infused MWCNT-modified epoxy into a preform of woven glass fiber mats via two different types of VARTM processes. The best improvement in the interlaminar shear strength (ILSS) was 33%, which was found in the 2wt% MWCNT-modified GFRP sample manufactured with injection and double vacuum assisted resin transfer molding (IDVARTM). The IDVARTM utilized a step-wisely controlled atmosphere to (i) reduce the atmosphere and permit more nano-resin being injected into the flow distribution medium layer under the vacuum bag and above the glass fiber preform and (ii) subsequently increase the atmosphere to compact the nano-resin filled flow distribution medium layer thus squeeze the nano-resin into the glass fiber preform that is under strong vacuum. Although IDVARTM can handle higher MWCNT concentration than traditional VARTM, IDVARTM was not able to create the preferable MWCNT alignment that traditional VARTM produced and IDVARTM provided a lesser increase in ILSS at 0.5wt% MWCNT concentration as well. The micrographs provided within their study indicated that the short CNTs (compared with the gap between two adjacent glass fibers in the pictures) had slightly preferred alignment along the flow direction while under a traditional VARTM process but not under IDVARTM. Accordingly, Fan et al. concluded that the slightly preferred MWCNT alignment contributed to the ILSS improvement in addition to MWCNT concentration.

Together, the accumulated literature suggests that lack of control over CNT/CNF alignment and filtration could induce inconsistent composite quality. Furthermore, since carbon fibers (diameter $\sim 5\ \mu\text{m}$) are much smaller than glass fibers (diameter $\sim 20\ \mu\text{m}$), the narrower gap between adjacent carbon fibers creates severe challenges in terms of CNF or CNT alignment and filtration control; these issues are compounded for CNF applications (compared with CNT applications), as CNFs are much longer (approximately $50\text{-}200\ \mu\text{m}$) and thicker (diameter $\sim 50\text{-}150\ \text{nm}$) than CNTs (length $\sim 5\text{-}20\ \mu\text{m}$; diameter $\sim 1\text{-}20\ \text{nm}$). As such, no significantly successful studies regarding the infusion of CNF-modified resin in CFRPs have ever been reported.

To avoid the aforementioned challenges, electrophoretic deposition of water-dispersed short CNTs (preferred length of $2\text{-}6\ \mu\text{m}$, shortened from original length of $10\text{-}20\ \mu\text{m}$ by nitric acid treatment) on woven carbon fabric, which was then dried and then stacked into a preform and infused with resin via VARTM

process, has been studied [10] and produced an interlaminar shear strength increase of approximately 30%. However, handling the deposited CNTs on dry carbon fiber fabric could be cumbersome. Wicks et al. [11] provided an alternative by growing CNTs in-situ on alumina fibers to create novel “fuzzy fiber reinforced plastics” or FFRPs, which yielded a 76% increase in mean G_{IC} and an approximately two-fold increase in the relative error (i.e., error/mean). Another FFRPs study by Wicks et al [12] illustrated the differences that CNT length and epoxy grade (aerospace grade and marine grade) played in regards to G_{IC} . For the aerospace epoxy, short CNTs actually reduced the G_{IC} by 50%, whereas the long CNTs increased the mean G_{IC} by 32%, while also increasing the relative error by nearly 20% w.r.t. control. For the marine epoxy, short CNTs and long CNTs increased the G_{IC} by 89% and 100%, respectively. However, short CNTs and long CNTs also increased the relative error by 12% and 78%, respectively. Due to the exposure to high temperatures and/or metal catalysts, this process is not compatible with carbon fabrics. Moreover, the uncertainty in the improvements may be a concern. As such, the FFRPs method still needs future improvements to make it more robust.

1.2 Research Rationale

This paper takes a notably different approach than the literature with respect to the nano-reinforcing strategy, manufacturing process, and the attempt to gain an understanding of the roles of CNF alignment in CFRPs. Based on insights from the aforementioned nano-resin VARTM literature [8], it is clear that CNFs can potentially benefit z-directional strengths. However, multiple studies have suggested [8, 9] that poor CNF alignment control and the aforementioned filtering effect could be responsible for reducing the effectiveness of the CNF reinforcement or even, in some cases, weakening the composites. Although the works by Garcia et al. [6] and Wicks et al. [11, 12] illustrated that both vertically-aligned CNTs in the interlayer and radially-aligned CNTs deposited on individual alumina fibers can improve interlaminar fracture toughness, none of the studies made a comparison against laminates with randomly distributed CNTs of the same ingredients. As such, none of these previous works provided any direct proof that improving CNF alignment and filtering issue could make the CFRP composite, of same ingredients, stronger. Nevertheless, it is reasonable to assume that one could maximize CNFs’ potential to reinforce the CFRP in the z-direction if one somehow aligns CNFs in the z-direction and reduces the chance of CNFs being filtered by the carbon fiber preform.

Another interesting fact not yet discussed nor utilized explicitly in previous works is the unique scale relationship between the dimensions of CNFs with respect to the carbon fiber's diameter and the gap between adjacent carbon fibers in a CFRP. Based on geometric argumentation, since the carbon fiber diameter is about 5-7 μm , each z-aligned CNF (length $\sim$ 50-200 μm) is theoretically long enough to thread through an array of carbon fibers and connect 7-100 carbon fibers. However, as carbon fibers are typically highly-packed in a high fiber volume fraction CFRP, one must also account for possible filtration. A long CNF being flow-transferred through the thickness of a highly packed carbon fiber bed will behave according to one or more of three distinct scenarios: (A) the CNF will thread through the highly packed carbon fiber bed in the preferred "zig-zag" or "wavy" path shown in Figure 1; (B) the CNF will entangle with other CNFs and become filtered by the highly packed carbon fiber bed; (C) the CNF will lose the preferred z-direction orientation and rotate in any direction within the x-y plane, which will lead to it being filtered by the highly packed carbon fiber bed. Note that scenarios B and C both increase the probability of additional CNFs being filtered during the flow-transfer process, as they clog open channels in the packed carbon fiber bed. Two studies [8, 13] contributed to additional understanding of the alignment and filtration process that occurs when a CNF or CNT is flow-transferred through a fiber bed. First, from the VARTM filtering experiments of infusing CNF/polyester mixture through random glass fiber mat preforms by Sadeghian et al [8], one can conclude that a thinner glass fiber preform will filter fewer CNFs due to reduced compounded probability that CNFs will aggregate or orientate within the x-y plane. Secondly, the microscopy flow experiments conducted by Fan and Advani [13] illustrated that resin flow around a single glass fiber tends to align MWCNTs along the glass fiber's circumference (similar to the streamline around a cylinder) and the resin flowing through a 100 nm-wide necking channel between two glass fibers tends to align MWCNTs in the direction of the channel. Additionally, they suggested that shear flow dominated the MWCNTs' orientation instead of Brownian motion. Furthermore, the study indicated that MWCNTs stayed in place once the resin stopped flowing.

By comparing the results from previous studies and trying to find a solution to address the aforementioned issues of using CNFs in CFRPs, Hsiao and Hickman [14] proposed a new multiscale FRP prepreg manufacturing method to incorporate z-aligned long carbon nanofibers dispersed in a B-stage resin into a thin sheet of dry carbon fiber fabric by utilizing an electric field-induced CNFs alignment control in the

B-stage resin followed by a non-isothermal unidirectional phase-changing CNF-resin flow transfer process. This paper will discuss the manufacturing of the interesting CNFs z-threaded CFRP (ZT-CFRP) samples by using the new multiscale FRP prepreg method [14] and the Out-of-Autoclave Vacuum-Bag-Only (OOA-VBO) curing process. It will also investigate the statistical effectiveness of using CNF z-threads (or z-aligned CNFs) in a CFRP laminate to improve the mode-I delamination toughness (G_{IC}). Due to the relatively long and moderately dense CNF z-threaded reinforcement, if the CNF z-threads are effective and have the zig-zag threading pattern as proposed in Figure 1, it is hypothesized that one could obtain significant and highly repeatable improvements in G_{IC} using a low concentration of CNFs. Additionally, as the CNFs are z-threading through carbon fiber arrays, it is hypothesized that there would not be resin-rich interleaving regions in the CFRP laminate. Therefore, any change in G_{IC} would not be due to interleaving effect (resin-rich zone to blunt the crack tip). Furthermore, it was hypothesized that unaligned long CNFs would reduce the delamination toughness or make it unreliable as some unaligned CNFs could act as defects (described in aforementioned scenarios B& C) that encourage crack propagation. Note that this hypothesis will help to validate the widely-accepted but not-yet-proven theory that unaligned CNFs or CNTs were responsible for inconsistent reinforcement in many of the aforementioned publications. This study presents the mode-I delamination experiments and the accompanying statistical analysis for CFRP samples constructed with CNF z-threads (ZT-CFRPs), unaligned CNFs (UA-CFRPs), and unmodified epoxy resin (control CFRPs).

2. Methods

2.1 Materials

In this study, three 16-ply CFRP laminates were constructed for each of the four following sample types: unmodified control CFRPs, CFRPs with 0.3wt% (measured as a percentage of matrix weight) unaligned CNFs (0.3wt% UA-CFRPs), CFRPs with 0.3wt% CNF z-threads (0.3wt% ZT-CFRPs), and CFRPs with 0.6wt% CNF z-threads (0.6wt% ZT-CFRPs). The carbon fiber system used in the CFRPs was a Torayca T300 plain-weave fabric (203 g/m² areal weight, 3K tow) acquired from Composite Fabrics of America. The polymer matrix was a two-part epoxy from Momentive that consisted of Epon 862 resin (a diglycidyl ether of bisphenol F) and an Epikure W catalyst (a non-MDA, aromatic amine curing agent containing diethylmethylenediamine). The CNFs were PR-24-XT-PS nanofibers from Pyrograf Products, Inc.,

which possess an average diameter of 100nm and lengths ranging from 50 μ m to 200 μ m [15]. To increase experimental efficiency, only the inner four plies of the UA-CFRPs and ZT-CFRPs contained CNFs; this is allowable since the delamination stress is concentrated on the centerline of the sample. A 60 μ m-thick polytetrafluoroethylene (PTFE) release film was inserted in the mid-plane of each CFRP to generate the starter crack [16].

2.2 Control Prepreg Manufacturing

For each required stack of control prepreg (6-ply stacks for UA-CFRPs and ZT-CFRPs, 8-ply stacks for unmodified CFRPs), a resin batch was produced by combining Epon 862 and Epikure W with a stoichiometric ratio of 100:26.5, mechanically mixing the solution for five minutes, and then degassing the mixture in a vacuum chamber at 80°C for one hour. A hand wet lay-up process was implemented to add the degassed resin to individual 254mm x 254mm sheets of woven carbon fabric. This process was repeated until the prepregs had been stacked to the appropriate number of plies and the prepreg stack was placed on a hot plate that had been preheated to 120°C. After one hour of heating, the matrix reached the desired degree of cure or “B-stage”, and the prepregs were removed from the hot plate and transferred to a -18°C freezer; the prepregs were sealed inside a layer of nylon bagging to prevent moisture absorption during storage. The desired B-stage allows the resin to remain solid at room temperature and flow at approximately 50°C; the amount of heating time required to achieve this B-stage was determined through trial-and-error.

2.3 UA-CFRP Prepreg Manufacturing

To form the uniformly dispersed CNF/epoxy nano-resin system (i.e., CNF/epoxy mixture), first, the CNFs were weighed and added to the appropriate amount of Epon 862, then combined using a magnetic stirrer. The mixture then underwent one hour of high-shear mixing by a Stir-Pak Laboratory Mixer operating at 900 rpm, followed by one hour of agitation in a QSONICA Q700 sonicator. A small sample taken from the resin was inspected under a microscope to ensure the aggregations were broken apart. Having disentangled the majority of CNF agglomerations, the designated amount of Epikure W was added to the solution, magnetically stirred for five minutes, and then degassed in a vacuum chamber for one hour at 80°C. The UA-CFRP prepregs followed the same hand wet lay-up process as the control prepregs, however, the B-stage UA-CFRP prepregs were packaged and stored at -18°C as individual plies instead of stack of plies.

2.4 Novel ZT-CFRP Prepreg Manufacturing

The CNF/epoxy mixture for the ZT-CFRP prepregs was first prepared using the same procedure as that used for the UA-CFRP prepregs. However, to obtain the novel ZT-CFRP prepreg, several additional steps are needed according to [14] and the research rationale discussed in Section 1.2. Figure 2 summarizes the process into four major steps as explained hereafter. (1) Introduce well-dispersed CNF/epoxy mixture into a thin sponge sheet to bear the nano-resin; note that the CNFs are randomly orientated at this step. (2) Use an electric field to align CNFs inside the thin sponge in the thickness direction and then rapidly cool the B-stage epoxy into solid-phase to lock the z-alignment of CNFs. (3) Apply a non-isothermal, phase-changing, unidirectional resin-transfer process to thread the z-aligned CNFs along with the resin flow into a preheated thin carbon fabric sheet. Note that during the non-isothermal phase-changing flow process, it is imperative to cool down and keep the majority of B-stage epoxy within the sponge solidified to lock the preferred CNF z-alignment. As such, the epoxy only starts to melt upon contact with the preheated carbon fabric, allowing all CNFs to start from a very well controlled z-aligned angle to thread through the carbon fiber array of the carbon fabric. After the pre-z-aligned CNFs enter the carbon fabric with a good entrance angle, the flow-induced alignment phenomena described in [13] will keep the CNFs flowing along the tangent of the circumference of each carbon fiber and the direction of flow channel formed by the gap of adjacent carbon fibers. In other words, this shear flow-induced alignment inside the packed carbon fiber bed will help the pre-z-aligned CNFs thread through the carbon fiber array inside the carbon fabric. To reduce the probability and negative impact of undesired CNF filtering effect caused by imperfectly-aligned CNFs, imperfectly dispersed CNFs (such as CNF aggregates), or blockages in the flow channels (i.e. scenarios B & C described in section 1.2), a thin carbon fabric sheet is preferred to a thick preform; thus, a thin prepreg is appropriate considering the nature of all involved physics. (4) After completing the CNF z-threading process, the carbon fabric sheet is cooled down to solidify the B-stage epoxy and obtain the ZT-CFRP prepreg.

The detailed process to implement the four major steps (illustrated in Figure 2) on the specific epoxy resin, CNFs, and carbon fabric system used in the mode-I delamination experiments are given as follows:

Step 1: After degassing, the prepared CNF/epoxy mixture was added via hand wet lay-up to a 254mm x 254mm sheet of nonwoven polyester breather material (henceforth referred to as the “sponge”) that had been

placed inside the confines of a parallel plate capacitor; prior to the lay-up process, the capacitor was preheated to 120°C on a hot plate and the top and bottom surfaces of the sponge were covered with a PTFE film.

Step 2: The resin-saturated sponge was then heated for 39 minutes before it was subjected to a 0.67 kV/mm electric field at 6 Hz for 60 seconds, which aligned the CNFs in the z-direction of the sponge sheet; the waveform was a DC pulse with a 50 percent duty cycle. The z-alignment was achieved because the electric field polarized the CNFs. This polarization created dipole moments at the opposing ends of the CNFs, which caused them to rotate and align lengthwise between the opposing plates of the capacitor. After completing the electric field induced CNF alignment process, the sponge saturated with B-stage resin containing z-aligned CNFs was rapidly cooled to 5°C to solidify the B-stage epoxy; the solidified B-stage epoxy ensured the CNFs remained in the preferred z-aligned orientation.

Step 3: Once the sponge had been thoroughly cooled, it was removed from the capacitor, centered between two layers of porous PTFE-coated fiberglass, and then placed atop a sheet of carbon fabric that was placed on top of a porous PTFE-coated fiberglass, a layer of polypropylene flow distribution media, and a supporting aluminum plate. A 254mm x 254mm caul plate was then placed on top of the sponge, the layers of peel ply were trimmed to the edges of the caul plate, and then the edges of the caul plate were constrained with a dam to ensure the unidirectional resin transfer occurred in the thickness direction. Two vacuum channels equipped with vacuum lines were then placed on top of the flow distribution media and sealant tape was placed on the edges of the aluminum mold to anchor a layer of vacuum bagging (see Fig. 2 for the setup). After vacuum (approximately 1 bar) was drawn on the samples, the mold was placed in an opened hot press that had the bottom plate preheated to 120°C (the top plate was not needed for this step so it was unheated and did not apply any compaction pressure to the mold). After ten minutes, the resin completed the transfer from the sponge into the carbon fabric and the resin had been heated long enough to achieve the same level of B-stage as the control CFRP and UA-CFRP prepregs. During this step, the individual as well as the integrated influences of (i) the one-sided heating and one-side cooling arrangement, (ii) the significant thermal conductivity difference between the resin system and the carbon fiber fabric, and (iii) the B-stage resin's ability to rapidly change between solid phase and liquid phase during the non-isothermal z-directional flow transfer process, were paramount to the success of the CNF z-threading through the carbon fiber array (i.e.,

packed carbon fiber bed). The CNF/epoxy mixture (largely composed of epoxy) has a relatively low thermal conductivity compared with compacted carbon fabric. This distinct thermal conductivity difference is helpful to maintain the majority of epoxy inside the sponge in solid phase and all the epoxy inside the carbon fabric in liquid phase, which maximizes the chance of z-threading CNFs through the carbon fabric.

Step 4: Upon completing the z-threading process, the ZT-CFRP prepreg was cooled down to room temperature and individually packaged and stored at -18°C according to the same procedure used for the UA-CFRP prepregs.

2.5 Laminate Sample Preparation

The control CFRP samples were stacked for curing in the following order: an 8-ply control CFRP prepreg, a PTFE film (used to form the precrack), and then another 8-ply control CFRP prepreg. The UA-CFRP samples had the following stacking sequence: a 6-ply control CFRP prepreg, two single-ply UA-CFRP prepregs, a PTFE film, another two single-ply UA-CFRP prepregs, and then a final 6-ply control CFRP prepreg. The ZT-CFRP samples used the same general stacking sequence as the UA-CFRPs.

After stacking, the prepregs were placed on a release agent-coated aluminum plate and then the prepregs were covered by a layer of peel ply and two layers of distribution media. Two vacuum channels equipped with high-temperature vacuum lines were placed on top of the distribution media and then sealant tape was placed on the edges of the aluminum mold to anchor a layer of vacuum bagging. After vacuum was drawn on the samples, the mold was placed in a hot press and the heated plates were closed until only a small gap remained between the top heating plate and the aluminum mold; this emulated an even-heating oven environment. The OOA-VBO cure cycle consisting of vacuum application, temperature, and timing used for curing these samples is shown in Table 1.

After demolding, the precrack tip location (i.e., the leading edge of the PTFE film) was marked on the face of CFRP to ensure the samples were cut orthogonal to the precrack edge. The targeted dimensions for the trimmed delamination testing samples were: 153mm length, 50mm initial precrack length, 25mm width, and approximately 3mm thick. Once cut, the samples were polished, the edges of the samples were coated with white acrylic paint, and then graduations were scribed in the paint using a fine-tipped razor blade.

2.6 Mode-I Delamination Testing

The mode-I interlaminar fracture toughness for each sample was evaluated using the double cantilever beam (DCB) method in accordance with ASTM D-5528 [16]. ASTM D-5528 was originally intended for testing unidirectional fiber systems; however, the standard also elaborates that woven fabrics can be used with the understanding that they tend to experience run-arrest phenomena as a result of resin rich pockets between the warps and wefts of the weave.

Prior to testing, the surfaces of two aluminum loading blocks and a small portion of the leading edge of each sample surface were sanded and then the blocks were bonded to the sample. After the adhesive cured, the samples were loaded into a MTS 810 universal testing machine equipped with a 50lb (~222N) load cell; the experimental testing setup is shown in Figure 3. Adhering to the standard testing procedure, the samples were loaded at a crosshead speed of 1mm/min until the crack had progressed 3-5mm from the precrack tip, then they were unloaded and the precrack tip location was marked on both sides of the sample. The samples were then reloaded at a crosshead speed of 1mm/min until the crack had progressed at least 50mm from the precrack tip. The crack location was tracked using a high-speed digital camera and the displacements were noted via the graduations on the sample's edge. The displacement data was combined with the load cell output to create load vs. displacement plots for each test.

The Modified Compliance Calibration (MCC) Method was used to interpret the interlaminar fracture toughness, G_{IC} , from the load vs. displacement data; this is shown in Eq. 1.

(1)

In Eq. 1, P is the applied load, C is the compliance ($C=\delta/P$), δ is the crosshead displacement, b is the sample width, h is the sample thickness, and α is an experimentally determined correction factor found by the least squares linear fit of the delamination length normalized by sample thickness (i.e., a/h) as a function of the cube root of compliance (i.e., $C^{1/3}$), wherein α is the slope of the linear plot of a/h vs $C^{1/3}$ [16].

2.7 Carbon Fiber Volume Fraction

To further explore the differences between the samples, the fiber volume fractions were determined by

$$vf = n * Aw \rho f * t \quad (2)$$

, where n is the number of plies, Aw is fiber areal weight (203 g/m²), ρf is fiber density (1.78 g/cm³), and t is laminate thickness measured for each sample. The carbon fiber volume fractions for the control CFRPs, 0.3wt% UA-CFRPs, 0.3wt% ZT-CFRPs, and 0.6wt% ZT-CFRPs were 53.41%, 52.14%, 52.65%, and 52.52%, respectively. The carbon fiber volume fractions of all samples are very close. Nevertheless, the slight differences could be due to either thickness measurement errors, manufacturing difference (such as B-stage resin control and prepreg resin content control), or difference in flow resistance while the excess resin was squeezed out from the CFRP prepreg during the OOA-VBO curing cycle. Theoretically, the presence of CNFs, either z-threaded or not, should add resistance against the resin flow in the carbon fiber bed and the compaction process during OOA-VBO curing. Further analysis regarding the compaction process, either using OOA-VBO or autoclave curing, could be the aim of future investigations.

2.8 Microscopic Characterization

The overall laminate structures and delaminated surface morphologies of the CFRP samples were characterized and compared using a Nikon Eclipse LV150 optical microscope (optical magnification range 100-3000x) equipped with an extended depth of focus (EDF) module. Additionally, a JEOL JSM-IT100 scanning electron microscope (SEM) was used to further explore the structure of the CNF z-threads network.

2.9 Laminate Structure Investigation

As described in previous sections, the CFRPs used in this study did not insert any interleaf film within their structures. Furthermore, it was hypothesized (Section 1.2) that no resin-rich layers similar to an interleaf were unintentionally formed during the CFRP manufacturing process; therefore, one will be able to directly relate the G_{IC} change to the role of CNFs instead of mixed roles between resin-rich interleaves and CNFs. Note that an interleaf is typically a thick thermoplastic film (approximately 100 μ m thick) that absorbs substantial plastic deformation at the crack tip during delamination, which blunts the crack tip to toughen a FRP laminate [7]. To verify the hypothesis that no interleaving layers exist in any of the CFRPs tested in this study, the as-produced layer-by-layer laminate structures were investigated using side-view optical micrographs (Figure 4) acquired from the upper half of the symmetric CFRP laminates right above the mid-plane region (annotated as region “2”) where the precrack was formed (annotated as region “3”). As the

laminates are composed of plain-weave carbon fabric, one can distinguish the interlaminar boundary between individual plies within the laminate by finding the regions (i.e., region “1”) where the warps from one ply merges with the wefts of the subsequent ply. If these laminates had resin-rich interleaves, either inserted or unintentionally generated during manufacturing, these warps and wefts in region “1” would never merge since they would be separated by a resin-rich interleaf layer as discussed in [7]. Upon examination of Figure 4, one can determine that there are no such resin-rich layers present between the plies of the CFRP laminates used in this study. Therefore, any observed differences in G_{IC} can be solely attributed to the role of CNFs in the tested CFRPs.

3. Results & Discussion

Figure 5 shows the load (P) vs. displacement (δ) plots for each of the four sample types and Figure 6 shows the G_{IC} (calculated by using Eq. 1) vs. delamination length (a). Table 2 presents a descriptive statistical summary comparing the performance of each respective sample type. The G_{IC} values in Table 2 were obtained by averaging all G_{IC} data, calculated by Eq. (1) for all P & δ data sets with the curve-fitted A_I , after peak load for each sample; the statistics for each laminate type were computed from the results. Further discussion based on Figure 5, Figure 6, and Table 2 will be given in the following sections.

3.1 Control CFRP

As shown in Figures 5a and 6a, the load vs. displacement plots and G_{IC} data for all the control CFRP specimens were consistent between individual tests. It is important to note that one of the control samples was manufactured with a longer initial crack length, which yielded a longer total delamination length than the other samples. However, this variation was accounted for in the compliance and GIC calculations; this is confirmed in Figure 6a, wherein the delamination length is longer, yet the GIC values are consistent with the other samples. In subsequent samples, this initial crack length was shortened to the targeted dimension to reduce the time required to run each test. In Figure 5a, the control CFRPs displayed the typical run-arrest phenomena of woven fiber systems. Despite this erratic behavior, the G_{IC} results in Table 2 reveal that the mean and CV for the control samples were 305.33 J/m² and 4.15%, respectively. Figure 7a displays an optical micrograph of the delamination surface of a control CFRP sample; the clean and smooth surface indicates that the delamination propagated through the matrix without interruption, leaving the carbon fibers relatively

undamaged. This is as expected for CFRP laminates constructed from plain-weave fabric, which, unlike unidirectional CFRPs, do not possess fibers bridging inside the crack length.

3.2 0.3wt% UA-CFRP

As shown in Figure 5b, the UA-CFRP specimens also experienced the run-arrest phenomena. Although the results in Table 2 show an approximate 13.99% increase in mean G_{IC} value (348.06 J/m^2), the unaligned CNF-reinforcement also added a relative increase in the CV by 116.35% w.r.t the control. The observed inconsistencies between individual sample tests occurred because of the lack of CNF alignment control, as hypothesized in Section 1.2. As the alignment of the CNF reinforcement is uncontrolled in the UA-CFRPs, it is possible that some CNFs provided local toughening to the laminate by pseudo-aligning in the through-thickness direction. However, some other CNFs that did not experience this pseudo z-alignment created nonhomogeneous defects between the carbon fibers in the form of CNF bundles (a non-uniform network of unaligned CNFs) or in-plane alignment. Unlike z-aligned CNFs, CNFs with in-plane alignment do not contribute their fiber strength to the interlaminar fracture toughness; this reduces local bonding strength in the z-direction, while providing unnecessary laminate reinforcement in the in-plane directions that are reinforced by the continuous carbon fabric. Figure 7b displays an optical micrograph of the delamination surface of one of the 0.3wt% UA-CFRPs. Like the control sample, the UA-CFRP delamination surface is also clean and smooth. As previously suggested, most CNFs are not z-aligned nor pseudo-z-aligned, therefore they become defects that encourage delamination; this phenomena explains the smooth fracture surface morphology. At the delamination surface, a CNF z-thread (i.e., z-aligned CNF) in the CFRP should have one root in the matrix, it should extrude from the root, and it should end with one broken tip as further explained in Figure 8. The local angle of a CNF z-thread may not be perfectly perpendicular to the delamination surface due to the zig-zag threading pattern through the highly packed fiber bed. Any CNF formation other than the “one-root and one-broken-tip” is not a z-thread. If one uses this “one-root and one-broken-tip” rule to examine the UA-CFRP delamination surface in Figure 7b, one can find that most of the observed CNFs are not z-threads since they either have two roots or are submersed in the matrix parallel to the delamination surface.

3.3 0.3wt% ZT-CFRP

As expected, Figure 5c confirms that the ZT-CFRPs experience the run-arrest phenomena in the load vs. displacement plots due to the plain-weave carbon fabric. However, in comparison to the control CFRPs and the 0.3wt% UA-CFRPs, the 0.3wt% ZT-CFRPs experienced less severe run-arrest events. Additionally, both Figures 5c (load vs. displacement) and 6c (G_{IC} vs. crack length) illustrate more consistent results between all 0.3wt% ZT-CFRP specimens. According to the data presented in Table 2, the mean G_{IC} value (393.67 J/m^2) increased by approximately 28.93% while reducing the CV by 12.33% w.r.t. the control. As such, in comparison to the control CFRPs, the ZT-CFRPs are significantly stronger and more reliable.

Comparing the UA-CFRP and ZT-CFRP results, as the testing setup and the CNF concentration were the same between the two types of CFRPs, the statistical improvements in the G_{IC} in terms of mean value and CV can be solely attributed to the role of controlled CNF alignment in the CFRP (i.e., CNF z-threads vs. unaligned CNFs). Furthermore, the increased repeatability between all 0.3wt% ZT-CFRP tests provides a more predictable fracture pattern, which could lead to better predictive models that help reduce the risk of catastrophic failures in products or systems that implement woven-fabric CFRPs.

Figure 7c displays an optical micrograph of the delamination surface of one of the 0.3wt% ZT-CFRP samples, which contains organized CNFs extending from beneath the carbon fiber tows. Additionally, other CNFs have been pulled out and/or fractured. The epoxy matrix under the carbon fibers was severely fractured due to the rooted CNF z-threads that extend from beneath the carbon fiber array. This observation illustrates the reason why the ZT-CFRP samples exhibited higher G_{IC} values: it takes additional energy to fracture and pull-out deeply-embedded, long (originally, CNF length is $50\text{-}200 \mu\text{m}$ [15]) carbon nanofibers threading through the array of carbon fibers and the matrix surrounding them. It is interesting to note that all of the micrographic features observed in Figure 7c can also be explained from the proposed schematic of CNF z-threading patterns shown in Figure 8 and Figure 1. In addition to optical microscopy, Figure 9 depicts a similar delamination surface of a 0.3wt% ZT-CFRP using scanning electron microscopy (SEM), which provides a higher resolution view than Figure 7c. As shown in the SEM micrograph, many of the fractured CNF z-threads protrude with very short length from the matrix material, which highlights two important ideas. First, this allows us to realize that many fractured CNF z-threads with extremely short pull-out lengths

are practically impossible to distinguish by optical microscopy alone, and, second, the short protruding length (i.e., CNF pull-out length) signifies a strong and effective bond (maybe also due to the mechanical interlock caused by the CNF's zig-zag threading pattern in the packed carbon fiber bed) between CNF and the epoxy in the novel ZT-CFRP laminate. Very complex fracture surface near the roots of broken CNFs indicate the CNF z-threads caused further complex fracture in the matrix when they were broken. Furthermore, the CNF z-threading observed in the optical and SEM micrographs illustrates that the new prepreg manufacturing process creates an organized network of CNF z-threads within the ZT-CFRPs; this observation explains the repeatability of results. One can compare Figure 7c, Figure 8, and Figure 9 and find consistent geometrical features of this novel ZT-CFRP laminate. By comparing the G_{IC} results and the microscopy analysis between the ZT-CFRPs and the UA-CFRPs with identical CNF concentration, one can conclude that controlled CNF alignment within the CFRP is critical to the delamination fracture toughness.

3.4 0.6wt% ZT-CFRP

As shown in Figure 6d, the 0.6wt% ZT-CFRPs were relatively consistent between tests in terms of G_{IC} value, yet Figure 5d displays rather erratic run-arrest cycles that are partially comparable to, but less severe than, those seen in the 0.3wt% UA-CFRPs. Despite doubling the CNF weight percentage, the mean G_{IC} value for the 0.6wt% ZT-CFRPs (371.54 J/m^2) fell below that of the 0.3wt% ZT-CFRPs (393.67 J/m^2). However, relative to the control CFRPs, the G_{IC} mean increased by 21.69% and the CV increased by 43.46%.

The disparity between the results for the 0.3wt% ZT-CFRPs and 0.6wt% ZT-CFRPs comes from issues with nanoreinforcement non-homogeneity. Although CNF aggregations are mostly broken apart with the combined high-shear mixing and sonication regime, as CNF concentration increases, the van der Waals forces between the nanofibers increase, creating a greater tendency for CNFs to aggregate. These aggregations diminish the performance of the alignment process, which inevitably results in laminates with inconsistent CNF alignment structures. Figure 7d highlights the extent of these CNF aggregations observed in the 0.6wt% ZT-CFRPs. Figure 10 further depicts the role CNF concentration plays in the CNF alignment control within a liquid epoxy throughout the electric field induced alignment process, which elaborates the reason the 0.3wt% ZT-CFRP had a more consistent CNF alignment structure. The orientation distribution function (ODF) method of nanoparticle alignment characterization described by Fan and Advani [13] was used to illustrate the

orientation state of the CNFs within the liquid epoxy during the electric field application process by utilizing the components of a second order orientation tensor in two dimensions to draw an ellipse [13] representing the CNF alignment. The orientation state ellipses generated by this method provide an indication in terms of both average CNF rotation angle and degree of alignment through the respective ellipse rotation and the size relation between the major and minor axes of the ellipse. For the latter, two extreme cases exist: if the ellipse is circular, the alignment is completely random, whereas, if the ellipse forms a line, the alignment is perfect.

As the alignment process reveals in Figures 10a, 10b, and 10c, (also, see video “[0.3wt% Alignment Video 5X Speed.mp4](#)”) the 0.3wt% sample achieved nearly perfect alignment in epoxy resin within 30 seconds of applying the electric field (i.e., the ellipse formed into a line that was rotated in the direction of the applied electric field), whereas the 0.6wt% sample (shown in Figure 10d, 10e, and 10f (also, see video “[0.6wt% Alignment Video 5X Speed.mp4](#)”) never surpassed a mediocre measure of alignment. The less effective electric field induced CNF z-alignment step (i.e., step 2 in Figure 2) subsequently caused less controlled z-threading process (i.e., step 3 in Figure 2) during the prepreg manufacturing, which explains the lack of increase in G_{IC} improvement at 0.6wt% CNF concentration. It is an aim of future works to improve the electric field alignment process to make it capable of aligning higher CNF concentrations.

4. Conclusions

To evaluate the effect of CNF z-threads on the mode-I delamination toughness of CFRPs, four sample types were constructed: control CFRPs, 0.3wt% UA-CFRPs, 0.3wt% ZT-CFRPs, and 0.6wt% ZT-CFRPs. The comparison among the control CFRPs, the 0.3wt% UA-CFRPs, and the 0.3wt% ZT-CFRPs proves that controlled CNF alignment in CFRPs has a significantly positive contribution to the mode-I delamination toughness G_{IC} (the mean and the CV) and further lessens the severity of the run-arrest events typically associated with CFRPs made of plain-weave carbon fabrics. However, comparisons between the 0.3wt% ZT-CFRPs and 0.6wt% ZT-CFRPs revealed a decreasing trend in G_{IC} improvement at the higher CNF concentration. This phenomenon was caused by nonhomogeneous CNF dispersion at higher CNF concentration, which led to CNF agglomerations that diminished the CNF alignment process. Further microscopy analysis shows completely different fracture morphologies of the four types of CFRPs. A comparison by integrating the G_{IC} data and microscopy of the 0.3wt% UA-CFRPs and 0.3wt% ZT-CFRPs

shows that CNF z-alignment can totally change the role of CNFs in a CFRP. For a high fiber volume fraction CFRP, the z-aligned CNFs thread through the packed carbon fiber bed in a zig-zag (or wavy) pattern and become z-threads in the CFRP, which helps create a complex and deep matrix fracture near the root of each broken CNF z-thread during the mode-I delamination process; therefore, more energy is consumed during the delamination. On the contrary, unaligned CNFs contribute to a smooth delamination surface and act as defects that encourage delamination.

This study illustrated that CNF z-threads can significantly improve the delamination performance of CFRPs. It also discussed the background issues to overcome, as well as the novel and detailed methodology used to manufacture CNF z-threaded CFRP prepreg. It is hopeful that further research into CNF dispersion and alignment improvement could produce ZT-CFRPs that utilize much higher CNF concentrations to provide dramatically higher mean G_{IC} values and very low coefficients of variation, which would be extremely beneficial to the industry as a whole from both peak performance and reliability perspectives.

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Fig. 1. Desired nanofiber z-threaded FRP presents the possibility of effective long-range reinforcement in the through-thickness direction (i.e., z-direction). Due to the long nanofiber length and zig-zag threading path, strong mechanical interlocks could be formed that yield highly effective z-direction reinforcement.

Fig. 2. Manufacturing of the CNF Z-threaded CFRP prepreg. Four majors steps are given as (1) saturate a thin sponge with B-stage CNF/resin mixture, (2) z-align CNFs in the sponge with electric field and solidify the B-stage resin to lock the CNF z-alignment, (3) apply non-isothermal, phase-changing, and z-directional resin transfer to thread the pre-z-aligned CNFs into a thin carbon fabric, (4) cool down the carbon fabric to solidify the B-stage resin and obtain CNFs z-threaded CFRP prepreg.

Fig. 3. Experimental test setup: (a) DCB fixture; (b) sample layout. Note that a_0 represents the initial crack length and L depicts the overall sample length.

Fig. 4. Optical micrographs of the upper-half of symmetric CFRP laminate layer-by-layer structures taken at 100x magnification: (A) control CFRP; (B) 0.3wt% UA-CFRP; (C) 0.3wt% ZT-CFRP; (D) 0.6wt% ZT-CFRP. In these micrographs, the provided annotations indicate: 1 = interlaminar areas where the warp & weft of successive plies within the laminate are joined; 2 = laminate mid-plane; 3 = gap formed by removing PTFE pre-crack insert. If these CFRP laminates had incorporated interleaves, there would be visible resin-rich region separating the regions where warp and weft met (i.e., region-1). As shown, none of the samples contained any resin rich interleaving regions.

Fig. 5. DCB testing experimental data for: (A) control CFRPs; (B) 0.3wt% UA-CFRPs; (C) 0.3wt% ZT-CFRPs; (D) 0.6wt% ZT-CFRPs. *Note that the data displayed here was captured during the reloading cycle.*

Fig. 6. DCB testing results using MCC approach for: (A) control CFRPs; (B) 0.3wt% UA-CFRPs; (C) 0.3wt% ZT-CFRPs; (D) 0.6wt% ZT-CFRPs.

Fig. 7. Optical micrographs of the post-fracture delamination surface morphology taken at 2000x magnification: (A) control CFRP; (B) 0.3wt% UA-CFRP; (C) 0.3wt% ZT-CFRP; (D) 0.6wt% ZT-CFRP. In these micrographs, the provided annotations indicate: 1 = CNFs rooted in matrix, 2 = CNF tips, 3 = CNF aggregation. A z-aligned or pseudo-z-aligned CNF should have a clear root in the matrix and a broken tip clearly exposed in air. In contrast to the one-root and one-tip formation, any CNF has either two roots or has a long-section submersed in the resin matrix parallel to the delamination surface is not z-aligned nor pseudo z-aligned.

Fig. 10. Optical micrographs of 0.67kV/mm electric field application (6Hz, 50% duty cycle) in uncatalyzed epoxy taken at 300x magnification of: (A) 0.3wt% CNFs at 0 seconds, (B) 30 seconds, and (C) 60 seconds; (D) 0.6wt% CNFs at 0 seconds, (E) 30 seconds, and (F) 60 seconds. The 0.3wt% sample was aligned by the electric field very effectively and one can also see some CNFs were straightened by the electric field from (B) to (C), which can also be shown by the change in the shape and rotation of the ODF ellipses. On the other hand, the 0.6wt% sample only achieved mediocre CNF alignment. (Note the videos for the two experiments can be found in “[0.3wt% Alignment Video 5X Speed.mp4](#)” and “[0.6wt% Alignment Video 5X Speed.mp4](#)” respectively)

Table 1
OOA-VBO cure cycle used for all laminates.

Time (min)	Temperature (°C)	Vacuum
60	23 (Room Temp)	Yes
30	45	Yes
120	120	Yes
240	180	No

Table 2
G_{IC} sample statistics used to highlight performance variations between sample types.

	Control CFRPs	0.3wt% UA-CFRPs	0.3wt% ZT-CFRPs	0.6wt% ZT-CFRPs
<i>Sample 1</i>	290.69	331.82	396.84	354.06
G_{IC} (J/m²) <i>Sample 2</i>	312.52	328.24	406.16	396.44
<i>Sample 3</i>	312.78	384.12	378.02	364.13
Mean, $\bar{x}$ (J/m²)	305.33	348.06	393.67	371.54
Standard Deviation, s (J/m²)	12.68	31.28	14.34	22.14
Coefficient of Variation, $s/\bar{x}$ (%)	4.15	8.99	3.64	5.96
Relative Mean G_{IC} Change (%)	-	+13.99	+28.93	+21.69
Relative G_{IC} Coefficient of Variation Change (%)	-	+116.35	-12.33	+43.46

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- Fig. 1.** Desired nanofiber z-threaded FRP presents the possibility of effective long-range reinforcement in the through-thickness direction (i.e., z-direction). Due to the long nanofiber length and zig-zag threading path, strong mechanical interlocks could be formed that yield highly effective z-direction reinforcement.
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- Fig. 5.** DCB testing experimental data for: (A) control CFRPs; (B) 0.3wt% UA-CFRPs; (C) 0.3wt% ZT-CFRPs; (D) 0.6wt% ZT-CFRPs. Note that the data displayed here was captured during the reloading cycle.
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Fig. 7. Optical micrographs of the post-fracture delamination surface morphology taken at 2000x magnification: (A) control CFRP; (B) 0.3wt% UA-CFRP; (C) 0.3wt% ZT-CFRP; (D) 0.6wt% ZT-CFRP. In these micrographs, the provided annotations indicate: 1 = CNFs rooted in matrix, 2 = CNF tips, 3 = CNF aggregation. A z-aligned or pseudo-z-aligned CNF should have a clear root in the matrix and a broken tip clearly exposed in air. In contrast to the one-root and one-tip formation, any CNF has either two roots or has a long-section submersed in the resin matrix parallel to the delamination surface is not z-aligned nor pseudo z-aligned.

Fig. 8. Proposed CNFs z-threading patterns through a CFRP help the understanding of possible morphology of CNF z-threads (i.e., z-aligned CNFs) in a delaminated CFRP laminate. The delamination surface is likely to occur at the necking gap between carbon fibers. The matrix (i.e., resin) near the delamination surface is likely to be shattered due to the interaction with the CNF z-threads under tension. Also, according to the geometry, any broken CNF z-thread in the delamination surface can only have one root in the matrix and one broken tip. A CNF z-thread can possibly be broken near the root or broken with the shattered matrix.

Fig. 9. SEM micrograph of 0.3wt% ZT-CFRP post-fracture delamination surface showing intact aligned CNFs. In this micrograph, the provided annotations indicate: 1 = CNFs rooted in matrix, 2 = CNF tips.

Fig. 10. Optical micrographs of 0.67kV/mm electric field application (6Hz, 50% duty cycle) in uncatalyzed epoxy taken at 300x magnification of: (A) 0.3wt% CNFs at 0 seconds, (B) 30 seconds, and (C) 60 seconds; (D) 0.6wt% CNFs at 0 seconds, (E) 30 seconds, and (F) 60 seconds. The same group of CNFs are highlighted and tracked with yellow squares for easy comparison. The 0.3wt% samples was aligned by the electric field very effectively and one can also see some CNFs were straightened by the electric field from (B) to (C), which can also be shown by the change in the shape and rotation of the ODF ellipses. On the other hand, the 0.6wt% sample only has mediocre CNF alignment results by the electric field. (Note the videos for the two experiments can be found in “0.3wt% Alignment Video 5X Speed.mp4” and “0.6wt% Alignment Video 5X Speed.mp4” respectively)

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