INTERLAMINAR REINFORCEMENT OF CARBON FIBER COMPOSITES USING ALIGNED CARBON NANOTUBES

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ABSTRACT

Aligned nanoscale fibers (carbon nanotubes, CNTs) are used to reinforce the interlaminar resin-rich region of aerospace-grade unidirectional carbon fiber laminates in an architecture termed "nanostitching". Nanostitching leads to a hybrid architecture where aligned CNTs (A-CNTs) are integrated at the interface of Carbon Fiber Reinforced Plastic (CFRP) plies. Here we manufacture nanostitched laminates and investigate the effect on laminate strength through *ex situ* and *in situ* mechanical testing. Short beam shear (SBS) and double edge notched tensile testing were conducted on both baseline and A-CNT reinforced laminates. No statistically significant change was found in SBS strength, however, a ~9% increase in tensile strength was observed in the double edge notched tensile tests. *In situ* tensile testing of the same double-edge notch configuration utilizing synchrotron radiation computed tomography (SRCT) showed little difference in damage accumulation between baseline and A-CNT reinforced laminates at 80% of failure stress, likely due to a general absence of delamination damage. Future work will focus on acquiring data at stress levels closer to ultimate failure in the double-edge notch configuration, testing configurations that exhibit delamination formation and growth in the damage progression, and improving preliminary modeling (results presented herein) to improve understanding of the different mechanisms at work.

1 INTRODUCTION

A fundamental limitation in advanced composite materials is the poor mechanical strength between ply interfaces. Different methodologies, such as stitching [1], z-pinning [2], and 3D weaving [3] have already been realized to provide through-thickness reinforcement. However, each of these improvements are limited by the simultaneous reductions of in-plane properties. In very recent years, nanoscale fillers, particularly vertically aligned carbon nanotubes (VACNTs), have become prime candidates to overcome this limitation in that they require very little filler content and thus would virtually cause no degradation to the laminate in-plane properties [4]–[15].

VACNTs are referred to within literature as A-CNTs, CNT forests, bundles, brushes and turfs. Herein, we will use these terms interchangeably. Previous research has demonstrated the benefits derived from nanoscale modification using A-CNTs as "nanostitches" (see Fig. 1) for both interlaminar fracture toughness [16] and substructural in-plane properties [17], but the underlying physics of the reinforcement mechanism still remain largely unknown.

In this work, we focus on short beam shear and double edge notched tensile strength testing, with the goal of understanding the strengthening and toughening mechanisms at the microscale originating from nanostitching.

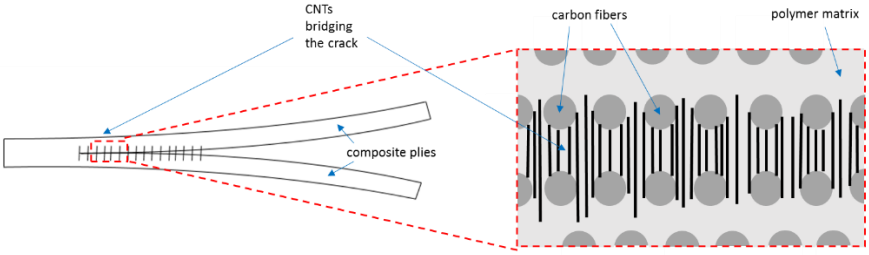

Figure 1: Illustration of nanostitch architecture

2 EXPERIMENTAL

In this section, the details about the fabrication of the nanostitched laminates are presented, followed by the detailed experimental procedure of strength tests: short beam shear strength and double edge notched tensile strength. Baseline and nanostitched specimens are machined from the same laminate.

2.1 A-CNT synthesis and laminate fabrication

The A-CNTs were grown in a 2" tube furnace (Lindberg/BlueM) by chemical vapor deposition (CVD) on a 3 cm × 4 cm Si wafer substrate. The growth time was set to be 55 s to produce ~20 µm forest. Further details can be found elsewhere [5,7].

The A-CNT forests were then manually transferred to the interlaminar region between aerospace-grade unidirectional (UD) AS4/8552 prepreg. The detailed process is described in Fig. 2. The next ply layup was continued until the entire layup was completed. Effectiveness of transfer was over 95% of wafer surface area.

The laminates were cured in an autoclave following the industry specifications (6 bar of total pressure at 1-3°C /min to 110 °C, hold for 1 h, heat again at 1-3°C to 180°C, hold for 2 h, cool down at 3-5 °C to 60°C and vent pressure, let cool to room temperature). Once the laminates were cured, the edges were trimmed and specimens were cut and polished to size for the different tests.

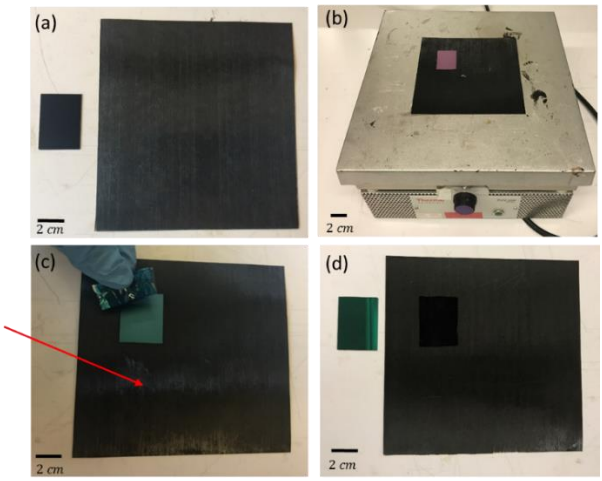

Figure 2: Transfer procedure of A-CNT forest onto prepreg. (a) A-CNT forest on a wafer (left) and a layer of prepreg (right). (b) Flip the wafer onto the prepreg and place it on a hot plate at 60 °C for 30 seconds. (c) Take prepreg off the hot plate and apply gentle pressure. (d) Empty wafer (left) and prepreg with transferred A-CNT (right).

2.2 Short beam shear testing

Following ASTM D2344 [19], the specimens were first cut with a diamond saw and then further polished in the following order: 500 grit sandpaper, 800 grit sandpaper, 1 µm Al_2O_3 suspension to remove the defects from bandsaw cutting and to meet the dimension specifications in the standard. The polished specimens were then subjected to a 3-point bending load. The test configuration is shown in Fig. 3. The test was performed on Zwick/Roell Z010 with a 10 kN load cell in displacement control. The specimen was loaded at 1 mm/min until failure (either greater than 30% load drop or travel exceeds the specimen thickness).

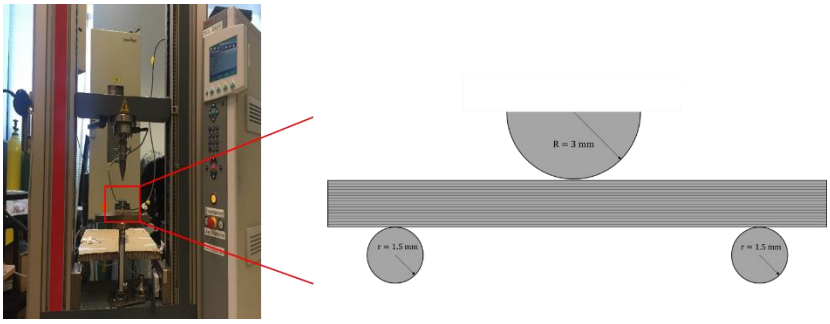

Figure 3: (Left) Zwick mechanical tester. (Right) Short beam shear specimen testing configuration.

2.3 Double edge notched tensile testing

The double edge notched tensile test is an in-plane strength test and the primary motivation for this test is to allow for *in situ* observation of damage progression using SRCT.

The specimens were machined with two 1.1 mm radius edge notches using a high-precision waterjet (Omax). Aluminum tabs were bonded to both ends of the specimen to allow load transfer in the *in situ* loading stage (Deben). The specimens were first tested *ex situ* using the Zwick/Roell Z010 with a 10 kN load cell and then *in situ* at ID19 beamline at European Synchrotron Radiation Facility (ESRF) in Grenoble, France. During *in situ* testing, the specimens were subjected to uniaxial tension with stresses ranging from 30% to 100% of the ultimate failure stress. SRCT scans were conducted at each load step. 2160 projections were taken for each scan and a 0.65 µm isotropic voxel size was achieved. The *in situ* loading stage and specimen configuration are shown in Fig. 4.

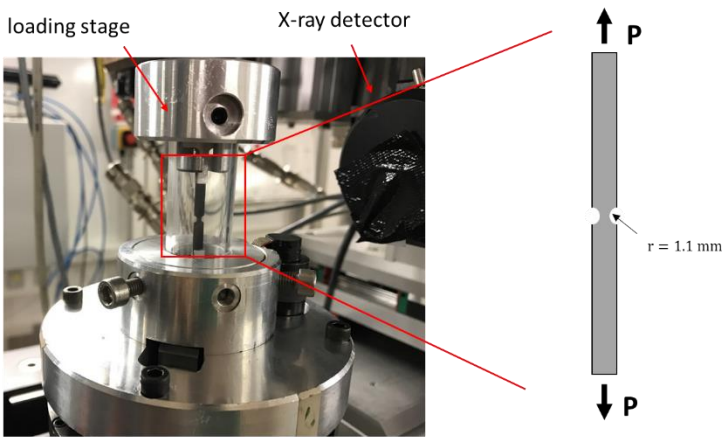

Figure 4: (Left) *In situ* loading stage. (Right) Double edge notched specimen configuration

3 RESULTS AND DISCUSSION

3.1 Short beam shear strength testing results

Prior work with a different material system (IM7/8552 UD prepreg) has shown ~8% increase in SBS

strength [20]. Here, no statistically significant improvement between baseline and nanostitched composites for AS4/8552 prepreg (Fig. 5) was observed. This suggests that the benefit of nanostitching varies one material system to another. Future work should investigate the optimum CNT parameters for short beam shear strength improvement for different material systems.

Numerical modelling was performed to help further understand the experimental results. Details of modelling can be found elsewhere [21]. It was found that for AS4/8552 prepreg laminates, the short beam shear strength does not increase proportionally to fracture toughness. In particular, a 5% increase in both mode I and mode II fracture toughness results in a ~4% increase in short beam shear strength, whereas a 15% increase in fracture toughness only results in a ~6% improvement in short beam shear strength.

Therefore, it is highly possible that even if we have a stronger and tougher interface from A-CNT reinforcement, the improvement in short beam shear strength can still be too small to be experimentally measurable (due to limited sample number and specimen variability).

A sensitivity analysis will also be carried out in the future, supplemented by experimental mode I and mode II fracture toughness characterization, to assist the further understanding of the effect of nanostitching on short beam shear strength.

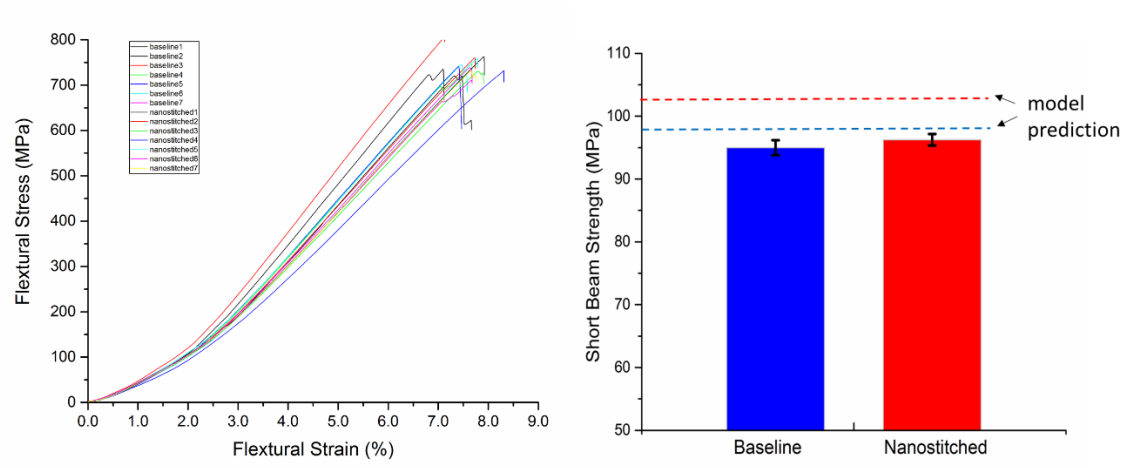

Figure 5: Short beam shear testing results

3.2 Double edge notched tensile testing results

The *ex situ* test was performed first to determine the ultimate tensile strength that will be used as the reference load in the *in situ* test. Fig. 6 shows that a ~ 9% improvement in tensile strength was observed for A-CNT reinforced specimens. To understand this improvement, SRCT *in situ* tensile testing was subsequently conducted.

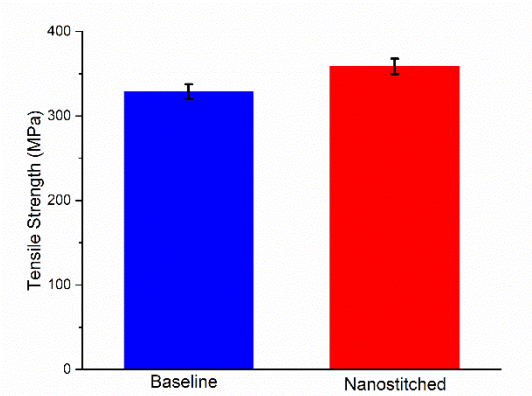

Figure 6: Double edge notched tensile testing results

The raw data generated from SRCT consists of a 3D grey-scale map of the region of interest of the sample, where grey values are correlated to the local material X-ray attenuation factor [10, 11]. By appropriately defining the threshold for grey values, different materials can be segmented. The segmentation was performed using commercial software (Avizo) and Fig. 7 shows the 3D reconstructed volume of the specimens at 80% of the ultimate tensile load. The cracks have been segmented and colored. Three major damage modes were observed: 0° ply splits, 45° and 90° transverse ply cracks. However, even at 80% of the failure load, there is no delamination present either in the baseline or nanostitched sample, which explains the lack of noticeable difference between the two.

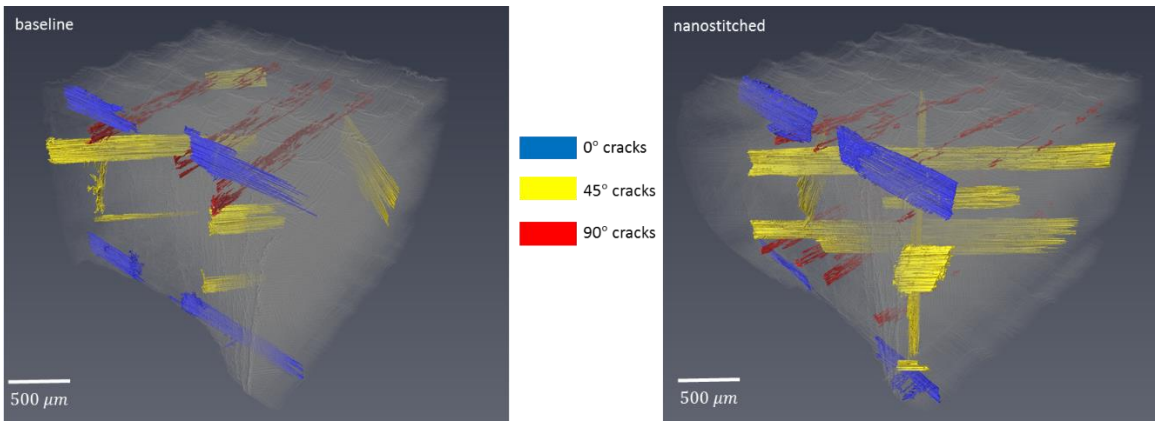

Figure 7: 3D segmented damage in specimens at 80% of the ultimate tensile load

Therefore, future work should focus on load steps that are close to failure (e.g., 95%, 98%, etc.) and are expected to have more delamination damage. More importantly, tests such as open hole compression or mode I should also be conducted to provide more information about the nanostitches at the interlaminar region.

4 CONCLUSIONS AND FUTURE WORK

Nanoengineered aerospace-grade composites were developed by nanostitching the plies together with A-CNTs. Although no short beam shear strength improvement was observed, tensile strength of double edge notched specimens was increased by ~9% for nanostitched laminates. The results presented here lead to clear immediate next steps. First, additional tests are warranted, such as mode I and mode II fracture toughness, and notched compression where interlaminar failure is more prominent. Second, more information should be extracted from the rich 3D synchrotron data to not only qualitatively evaluate, but also quantify the difference in damage modes, especially delamination damage between baseline and A-CNT reinforced composites. Quantitative damage assessments including crack opening displacement, crack tortuosity, and shear displacements will give stronger and clearer insights into damage suppression modalities due to CNTs. Lastly, a fundamental understanding of nano- to micro-mechanism A-CNT reinforcement needs to be elucidated via model-experiment correlation studies.

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