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| <b>REPORT DOCUMENTATION PAGE</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                |             |                       |                                   | <i>Form Approved<br/>OMB No. 0704-0188</i>      |                                                  |
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| <b>4. TITLE AND SUBTITLE</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |             |                       |                                   | <b>5a. CONTRACT NUMBER</b>                      |                                                  |
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| <b>6. AUTHOR(S)</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |             |                       |                                   | <b>5d. PROJECT NUMBER</b>                       |                                                  |
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| a. REPORT                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | b. ABSTRACT | c. THIS PAGE          |                                   |                                                 | <b>19b. TELEPHONE NUMBER (Include area code)</b> |

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**17. LIMITATION OF ABSTRACT.** This block must be completed to assign a distribution limitation to the abstract. Enter UU (Unclassified Unlimited) or SAR (Same as Report). An entry in this block is necessary if the abstract is to be limited.

**Title: Nanostitched Composites with Improved Interlaminar and Intralaminar Strengths for Advanced Airframes in Sea-based Aviation**

**Grant/Contract Number:** ONR Grant N00014-13-1-0213

**Performer:** Massachusetts Institute of Technology (MIT)

**PI:** Prof. Brian L. Wardle, MIT Bldg. 33-408, 77 Massachusetts Avenue, Cambridge, MA 02139.  
[wardle@mit.edu](mailto:wardle@mit.edu), 617-252-1539

**Organization:** necslab, Dept. of Aeronautics and Astronautics, MIT

**Abstract:**

The research objectives are twofold: (1) identify dominant mechanisms in polymer matrix composites reinforced by aligned carbon nanotubes (CNTs) at ply interfaces, focusing on strength effects of nanostitched fiber reinforced plastic (FRP) systems of interest to the Navy, and (2) build the experimental and engineering expertise to understand and then utilize this mechanistic understanding. These objectives were accomplished through an *ex situ*, and preliminary *in situ*, experimental program utilizing optical microscopy, scanning electron microscopy (SEM), and 3D X-ray computed tomography (CT) for visualization of damage progression. Highlights of findings include new nano manufacturing insights for process-structure relations in such systems, increased (~8%) interlaminar static shear strength and observation of fatigue enhancement of 300% increase in lifetime, as well as ~7% increase in in-plane tensile strength. Improvements are linked to stronger and tougher interfaces – *ex situ* observations indicate desirable intralaminar cracking vs. interlaminar failure when CNTs are incorporated. *In situ* failure testing utilizing synchrotron computed tomography is still being analyzed, but will give stronger and clearer insights into damage suppression modalities and alteration of the damage progression vector due to the CNTs. The program in this grant has been continued via a Bridge proposal and complemented by a DURIP award to provide an enhanced local ability to perform *in situ* hi-resolution microCT damage testing under a variety of loading conditions. With more rapid turnaround of ideas to findings, the DURIP and Bridge programs will allow us to innovate in areas such as quantitative damage assessment in composites and imaging nanostructures. The current program has benefited from complementary programs at MIT and from within the ONR Sea-based Aviation broader portfolio, especially in the areas of modeling, as well as through working with NAVAIR for guidance on several topics including experimental testing and materials baselines; the Bridge program is expected to benefit similarly.

Combined with other work in Wardle's group, such as recent 3D tomographic nano-scale characterization with NIST, this will lead to the first process-structure-property relations spanning nano-scale to macroscopic properties. The combined impact of this program will be new understanding for designing manufactureable 3D nano-reinforced advanced composites for Navy aerostructure and other applications.

**Summary/Overview:**

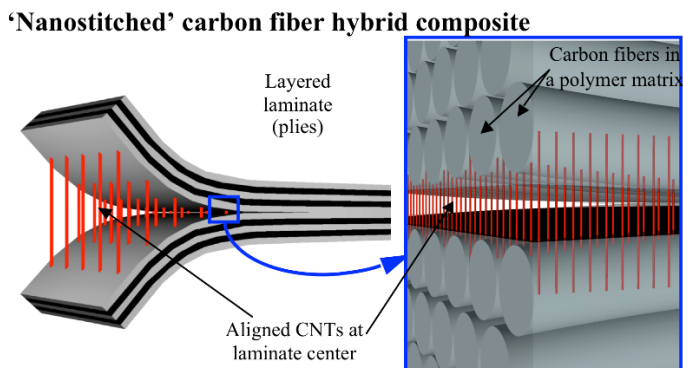
Please see the abstract above for a full summary. Note that the results presented below represent in-process work continuing through the Bridge grant. Several journal articles representing completed work are in preparation, but all require further analysis of CT data that has recently been acquired. This work was sponsored by the Office of Naval Research, ONR, under grant/contract number N00014-13-1-0213. The views and conclusions contained herein are those of the authors and should not be interpreted as necessarily representing the official policies or endorsements, either expressed or implied, of the Office of Naval Research, or the U.S. government.

### **Introduction:**

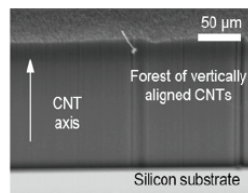
This project focuses on the use of aligned nanoscale fibers (carbon nanotubes, CNTs) to reinforce the interface of polymer matrix composites (PMCs) in an architecture termed 'nanostitching'. Nanostitching leads to a hybrid architecture (see Fig. 1) where aligned CNTs (A-CNTs) are integrated (see Fig. 2) at the interface of Fiber Reinforced Plastic (FRP) plies, such as Carbon Fiber and Glass Fiber Reinforced Plastics (CFRP and GFRP). The current work focuses on aerospace-grade unidirectional (UD) CFRP laminates of interest to the Navy.

### **Background:**

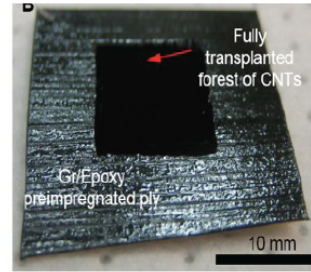
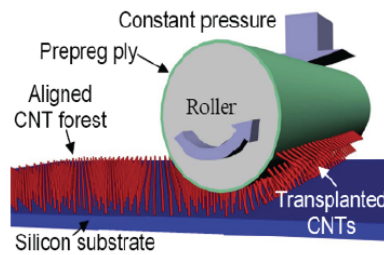
Approach and Research Description: The nanostitch approach can work with off-the-shelf (unmodified) systems and has been integrated into existing composite manufacturing, and is thought to be versatile with regard to fiber and polymer type, i.e., Navy materials and manufacturing processes can be targeted immediately. Toughness enhancement has been measured and modeled, and more recently in-plane strength properties have been shown to be significantly increased (10-40% depending on metric) suggesting that such material improvements can yield significantly advantaged designs due to the compatibility of the nanostitch with prepreg materials including CFRP. Activities and Accomplishments appear in the next section.



**Figure 1.** Nanostitch architecture: Aligned CNTs stitch together composite ply interfaces but do not disturb the microfiber morphology (nor do the CNTs increase the interlaminar region thickness).



- 1) Grow aligned CNTs on hi-temp. substrate
- 2) Transplant CNTs to composite at low (room) temp.
- 3) Process the composite



**Figure 2.** Nanostitch manufacturing overview and a picture of an exemplary unidirectional CFRP system (lower right corner).

### **Results/Findings and Conclusions:**

Here we detail four key results/findings of A-CNTs in composites from the current program.

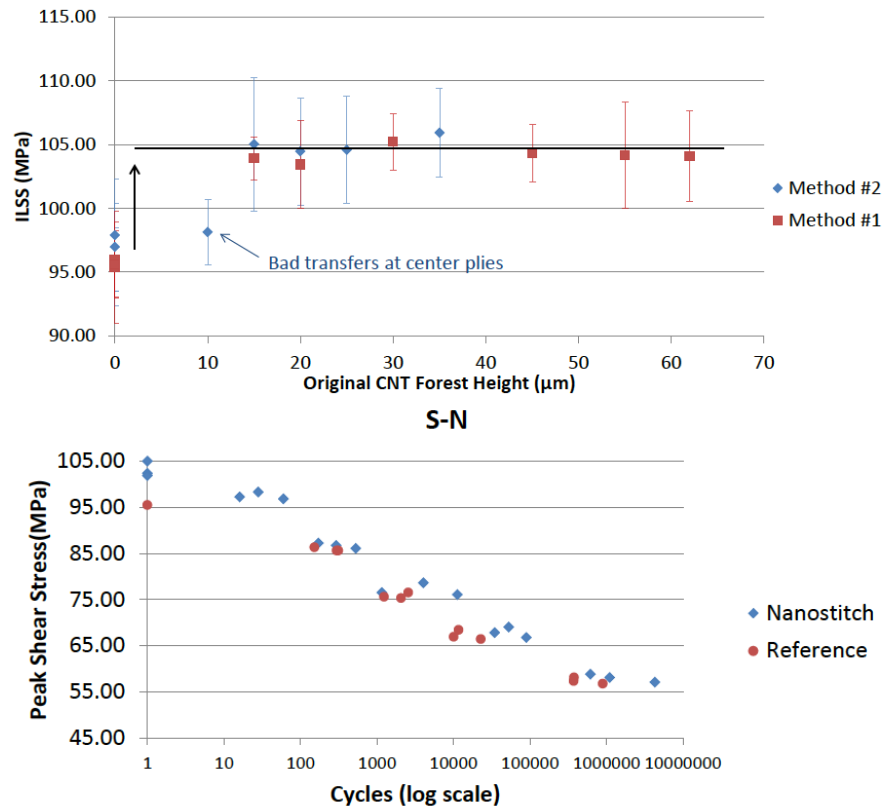
#### **1. Nanostitch height control**

Significant synthesis and manufacturing advances over the initial nanostitch work (see Fig. 2) made this new work possible. Among the morphological factors controlling the nanostitch reinforcement effectiveness, the A-CNT height is primary, and the transfer method used as well as the A-CNT as-grown height are the governing factors. Our current ONR program research has established  $\pm 2$   $\mu\text{m}$  control of height, enabling a study of nanostitch height and transfer method on the resulting nanostitch morphology inside the laminates (process-structure relation), and their effect on short beam shear strength (SBS) response (structure-property relation). We have tested a range of forest heights (5-65 $\mu\text{m}$ ) for unidirectional (UD) IM7/8852 aerospace carbon fiber prepreg in a quasi-isotropic layup. In-depth *ex situ* optical and SEM inspections of the nanostitched interlaminar regions, and static SBS testing under ASTM D2344, established that an as-grown A-CNT forest height of 12.5  $\mu\text{m}$  in conjunction with a preferred transfer process (of two considered) as the best A-CNT nanostitch manufacturing parameters resulting in a nearly 9% improvement in SBS.

#### **2. Interlaminar strength and fatigue improvement for UD IM7/8852 prepreg**

Results from the current ONR program have resulted in a >9% improvement in interlaminar shear strength (ILSS) via ASTM D2344 short beam shear testing across different methods of manufacture and irrespective of forest height (5-65 $\mu\text{m}$ ) for unidirectional (UD) IM7/8852 aerospace carbon fiber prepreg in a quasi-isotropic layup. From optical and scanning electron microscopy (SEM) imaging, cracks in nanostitched samples frequently propagate outside of the interlaminar regions, i.e., in the intralaminar regions. This is in contrast to the reference samples, which appear to propagate

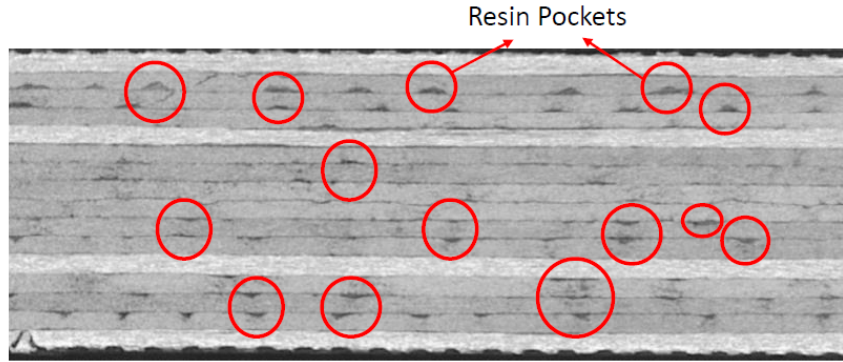
mostly along interfaces near the sample center as in a traditional composite. We have also completed ILSS fatigues testing that show a 3X improvement in lifetime across all load levels. These results (see Fig. 3) suggest that the A-CNTs can sufficiently reinforce the interlaminar region to drive crack propagation elsewhere.



**Figure 3.** Nanostitched composite vs. baseline summary data for static (top) and fatigue (bottom) ILSS testing of IM7/8552 aerospace carbon fiber prepreg laminates.

### 3. Process-structure relation highlights prepreg issue

Inspecting the nanostitched interlaminar region in the current ONR research program has revealed a defect in the tow-to-tow junctions in the IM7/8552 UD prepreg material (see Fig. 4) received from the manufacturer. As the CNT nanostitch is transferred on the prepreg it adopts the fiber topography and the effect of the fiber tow defect is translated into an inhomogeneous interface with large resin-rich (pure polymer) regions. This was not observed in the AS4/8552 UD prepreg to date and transition to the AS4/8552 prepreg was agreed with ONR and NAVAIR. Current work has confirmed that there is no tow-to-tow defect observed with the AS4/8552 system. Static SBS strength tests gave a ~4% increase in SBS. However, we have manufactured the 16 ply quasi-isotropic UD AS4/8552 nanostitched laminate with 12.5 μm tall A-CNT forests based on the IM7/8552 'optimal' CNT height. This height was found to be insufficient in the AS4/8552 system as the nanostitch does not entirely fill the interface.



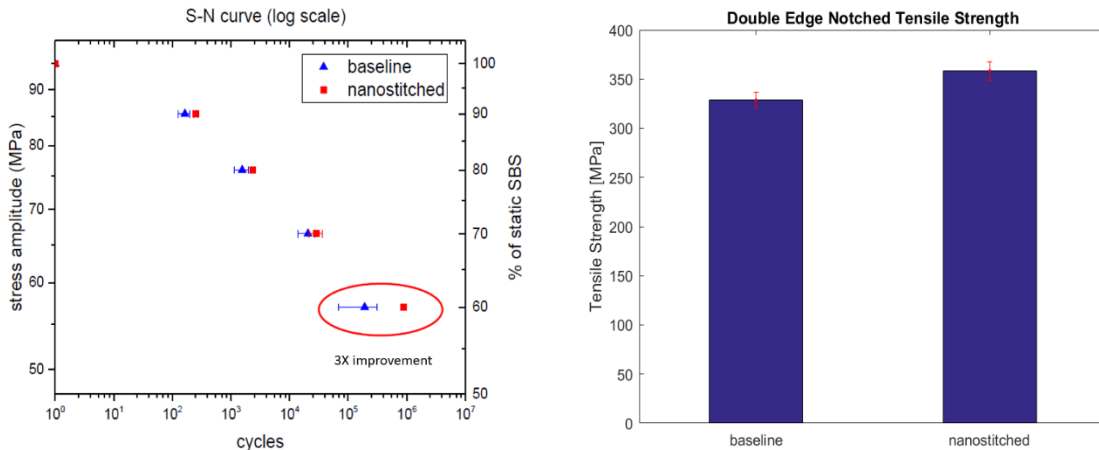
**Figure 4.** Optical image of the cross section of quasi-isotropic UD IM7/8552 laminate revealing tow-to-tow junction defects (resin pockets).

#### **4. Interlaminar and in-plane reinforcement for UD AS4/8552 prepreg**

Static ILSS (via SBS) for 20  $\mu\text{m}$  A-CNTs and fatigue ILSS testing have been completed for the newly-selected UD AS4/8852 prepreg with high-cycle fatigue life increased by 300%. Optical images of high-cycle fatigue samples revealed more desirable intralaminar cracks for the nanostitched samples. Double edge notched tensile testing also been completed and resulted in a nearly 7% increase in tensile strength (see Fig. 5). Both tests indicated stronger and tougher interfaces when CNTs are incorporated in traditional composites.

Furthermore, 180 A-CNT forests were grown to manufacture samples for Mode I and Mode I fatigue (ASTM D5528 and ASTM D6115) testing as well through spring 2017. The collection of tests explores the fundamental material parameters of interlaminar fracture toughness,  $G_{Ic}$  and  $G_{IIc}$  for both static and fatigue testing and will be utilized in predictive damage modeling via another collaboration.

Finally, *ex situ* and *in situ* scans of baseline and nanostitched laminates via synchrotron computed tomography have been acquired in Nov. 2016. This data is still being analyzed, but will give stronger and clearer insights into damage suppression modalities and alteration of the damage progression vector due to the A-CNTs. These planned work will be continued via the new bridge proposal.



**Figure 5.** Nanostitched composite vs. baseline summary data for static fatigue ILSS (left) and double edge notched tension testing (right) of UD AS4/8552 aerospace carbon fiber prepreg laminates.

### Transitions and Impacts:

Numerous transitions and impacts have been identified:

- Finite element analysis (FEA) of the nanostitched architecture is being supported through MIT's NanoEngineered aerospace Composite STructures (NECST) aerospace industry Consortium working particularly with NECST Member ANSYS and via collaborations with Prof. Pedro Camanho's group
- Nanocomposite data has been shared, and more is planned, with Prof. Aditi Chattopadhyay at Arizona State University to help populate multi-scale modeling of these nanostitched materials
- Parallel laminate-level strength testing supported by industry continues as part of the NECST Consortium
- With Navy permission, and Dr. Anisur Rahman (NAVAIR) and other DoD representatives attending, results from this program are shared with industry twice yearly at MIT's aerospace industry NECST Consortium meetings
- Ongoing collaborations with Dr. Seth Kessler at Metis Design Corp. (MDC) explore multifunctional aspects of aligned-CNT nanocomposites and nanoengineered composites, including a successful ice protection system (IPS) for the Navy (NAVAIR). That work is progressing to full-scale integration testing and the technology (co-developed by MIT/Wardle and MDC/Kessler was recently licensed to United Technologies Aerospace Systems (UTAS) to make it available for DoD and commercial applications
- Ongoing collaborations on imaging of A-CNT nanocomposites (similar to the nanostitched interlaminar region) utilizing 3D tomographic transmission electron microscopy (TEM) has been completed working with the National Institute for Standards and Technology (NIST), with the potential to look at the A-CNT interfaces directly in future work

### Plans and Recommendations for Future Work:



The program in this grant is currently being continued via a Bridge grant and complemented by a DURIP award to provide an enhanced local ability to perform *in situ* hi-resolution  $\mu$ CT damage testing under a variety of loading conditions. The Bridge proposal tasks pick up directly from the current work and are comprised of two Major Tasks in each of the Government Fiscal Years (GFYs) 1 and 2 of the bridge program. The tasks will continue to utilize *ex situ* testing and characterization using Harvard's existing low-resolution  $\mu$ CT, with the balance of the work shifting to *in situ* testing and characterization utilizing high-resolution  $\mu$ CT on the DURIP tool in mid 2017. The future testing is significantly informed by our recent in-plane strength testing results and discussion within the current program with NAVAIR's Drs. Anisur Rahman and Diane E. Hoyns, including the Composite Testing Recommendations memo for the current project. Transition and Transition Opportunities will be developed as in the original proposal.

#### **Appendix (Data and Charts):**

See figures above.

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### **Metrics:**

Collaborations/Navy Interactions were discussed above. Personnel Supported include several undergraduate researchers, a graduate student in AeroAstro, and now a PhD student in Mechanical Engineering at MIT, as well as assistance from a Postdoc on occasion. No current Patents or Awards. Talks/Presentations are summarized below and in addition 3 journal articles are in process based on the conference papers given below:

- Presentations
  - June 2015, Keynote, "Hierarchical Nanoengineered Advanced Composite Materials", NanoTech 2015, Washington DC.
  - June 2016, Keynote, "A Nanoengineers's Perspective on EHS Topics in Emerging Advanced Nanoengineered Hierarchical Materials", 2016 International Nanotoxicology Congress, Boston, MA.
  - Oct. 2016, Lecture, "Bulk Nanoengineered Structural Materials: Focus on Mechanical and Multifunctional Properties," MIT 10.585 Engineering Nanotechnology, Cambridge, MA.
- Publications
  - "Interlaminar Shear Strength Investigation of Aligned Carbon Nanotube-Reinforced Prepreg Composite Interfaces", Diana Lewis and Brian L. Wardle, 56th AIAA/ASME/ASCE/AHS/ASC Structures, Structural Dynamics, and Materials Conference, AIAA Science and Technology Forum, 2015.
  - "Damage Modeling of Thin-ply Nano-reinforced Composite Laminates," Carolina Furtado, Xinchun Ni, Estelle Cohen, Albertino Arteiro, Brian L. Wardle, and Pedro P. Camanho, accepted to *21<sup>st</sup> International Conference on Composite Materials (ICCM)*, Xi'an, China, Aug. 20-25, 2017.

- “Interlaminar Reinforcement of Carbon Fiber Composites Using Aligned Carbon Nanotubes,” Xinchun Ni, Estelle Cohen, Carolina Furtado, Albertino Arteiro, Gabriel Valdes, Travis Hank, Nathan Fritz, Reed Kopp, Gregor Borstnar, Mark N. Mavrogordato, S.M. Spearing, Pedro P. Camanho and Brian L. Wardle, accepted to 21<sup>st</sup> *International Conference on Composite Materials (ICCM)*, Xi'an, China, Aug. 20-25, 2017.
- “Synergetic Effects of Thin Ply and Nanostitching Studied by Synchrotron Radiation Computed Tomography,” Estelle Kalfon-Cohen, Reed Kopp , Xinchun Ni, Nathan Fritz, Albertino Arteiro, Gregor Borstnar, Mark N. Mavrogordato, S.M. Spearing, Pedro P. Camanho, and Brian L. Wardle, accepted to 21<sup>st</sup> *International Conference on Composite Materials (ICCM)*, Xi'an, China, Aug. 20-25, 2017.