

# REPORT DOCUMENTATION PAGE

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<b>13. SUPPLEMENTARY NOTES</b>					
<b>14. ABSTRACT</b>  The main objective of the project "Structural Foaming at the Nano-, Micro-, and Macro-Scales of Continuous Carbon Fiber Reinforced Polymer Matrix Composites," was to create fractal structures within each composite lamina and introduce scaled porosity. As this fractal structure was being created, it was observed that the structure imitated that of naturally occurring bird feathers. Consequently, the name given to this new generation of fractal structured composite materials with scaled porosity was deemed "Featherweight <sup>TM</sup> " Composites. The idea was to create lighter composites, and at the same time increase material strength. Through extensive characterization, two main interlayer enabling fractal systems were manufactured and characterized, first Carbon fiber – Epoxy Foam – CNTs, and second Carbon Fiber – Electrospun Nano-Fiber – CNTs, with both becoming transformed from the macro-, to micro-, and then nano-dimension. By focusing on low cost, the nano-materials utilized were not of the highest performance and quality. Nonetheless, with both interlayer systems, strength increased in several forms, such as fracture toughness (400%), stiffness (150%), flexural strength (80%), and tensile strength (20%), with percentages based on maximum values observed. At the same time, an experimental weight reduction of 10% was achieved. Finally, it is expected that if higher quality CNTs and matrix materials are used, the weight reduction can be higher, or even double that observed.					
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## Final Performance Report

Structural Foaming at the Nano-, Micro-, and Macro-Scales of Continuous Carbon Fiber Reinforced Polymer Matrix Composites

AFOSR Award Number:  
FA9550-09-1-0599

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## ACCOMPLISHMENTS

**Objective:** This research demonstrated the feasibility of introducing porosity at the macro-, micro- and nano-scales utilizing nanotechnology as the enabler in continuous carbon fiber reinforced polymer composites. The concept abbreviated as “MNM” has the potential of reducing conventional composite structural weight up to 40%.

**Approach:** Our approach was to work with conventional composite systems manufactured through the traditional prepreg and autoclave processing methodologies. However, based on our previous accomplishments with respect to understanding interlayer structures and introducing porosity in the epoxy matrix at the micro- and nano-scales, we were able to demonstrate the feasibility of our approach. Additionally, building on our work with honeycomb composite structures, where special papers and resins can be employed incorporating structural porosity at the nano-scale, we demonstrated feasibility at the macro-scale. Finally, in collaboration with our academic and industrial partners, we introduced porosity into the reinforcing carbon fibers, without sacrificing durability and manufacturability.

**Scientific Challenges:** Our major scientific challenge demonstrated that structural porosity at MNM scales could be introduced into the matrix, the carbon fiber reinforcement, and during prepreg lamination processing, without any major changes to properties and performance characteristics, while dramatically reducing overall weight (~40%).

## FINAL STATUS

In the following Table, the objectives of the proposed work are listed next to the progress and accomplishments.

No	Objective of Proposed Work	Status of Effort
1	Develop and characterize composites with a nano-foamed or micro-foamed interlayer structure	As a first step, we investigated foaming control into the matrix level with polyurethane foam reinforced with nano-clays of montmorillonite. (Accomplishment 1)
		Observed the pore surface reinforcement mechanism. (Accomplishment 1)
2	Investigate the effects of nano-cellular constructions both within the matrix interlayer and intralaminar areas, including fibers. Furthermore, investigate prepreg thickness and resin content effects on the thermomechanical performance of laminated sheet structures.	We investigated the potential of laboratory scale manufacturing hollow or nanoporous carbon fibers through CNT-polymer (Cel. Acetate) electrospinning. (Accomplishment 2)
		We improved material properties through the inclusion of aligned carbon nanotubes, as evaluated with our partners at MIT. (Accomplishment 6)
3	Characterize how the structure and properties of nano-foamed interlayer, micro-foamed interlayer, and nano cellular fiber based composites are affected by processing parameters and matrix backbone structure.	We explored and modeled hollow or nano-porous carbon fiber development during processing, degradation and electrospinning, in order to evaluate their structural contribution to the featherweight composites. (Accomplishment 2)

		The pressure influence during the curing process, separate from temperature, was investigated as a new cost efficient manufacturing technique, enhancing the cellular structures. (Accomplishment 3)
4	Determine how environmental effects through thermal and moisture cycling affect nano-foamed and micro-foamed / polymeric matrix composite (“PMC”) laminates by examining their dynamic properties.	Both thermoplastic and thermosetting matrices were tested under thermal fatigue and a moisture environment for their suitability as foaming host matrices. (Accomplishment 4)
5	Develop constitutive models for nano-foamed and micro-foamed PMC systems from single ply prepreg to multilayer laminated structures, accounting for anisotropy and viscoelasticity at different scales.	We investigated controlled nano-foaming at the matrix level. After the use of epoxy nano-foam reinforced with CNTs and carbon nano-fibers, the use of CNTs with large average diameters and low number of walls behaving as nano-pores within the matrix, was theoretically explored. (Accomplishment 5) Finally, we investigated and worked on the modeling of structural foaming materials. (Accomplishment 7)

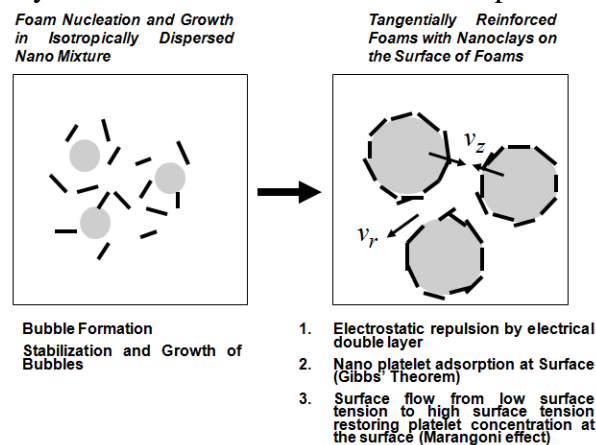
## DETAIL OF ACCOMPLISHMENTS

1) Based on our previous work, the research of carbon fiber reinforced composite materials with epoxy foams showed an improvement of mechanical and morphological properties, when compared with traditional ones. In general, from a macro, micro and nano point of view, the void content due to matrix foaming could be calculated from the equation:

$$\text{Void content} = \left\{ \frac{D_c - D_f}{D_c} \right\} \times 100$$

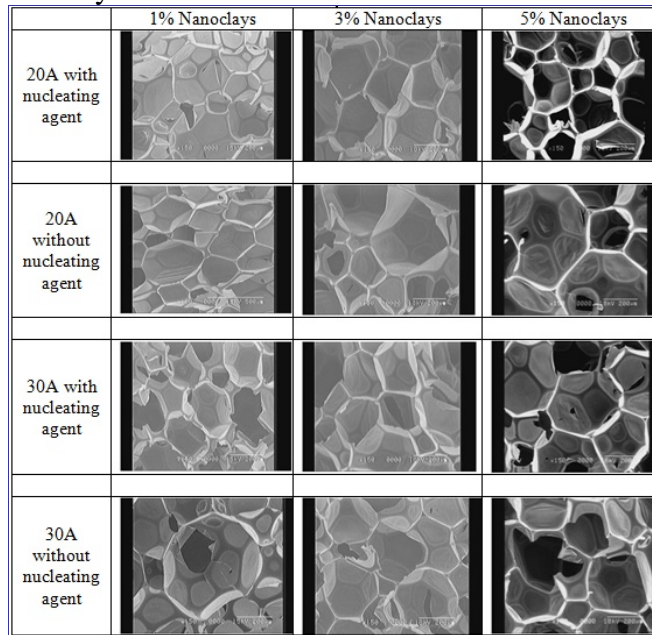
In this equation,  $D_c$  is the average density of reference samples and  $D_f$  is the average density of CFRP foam samples.

An effort was made to control void formation and size by adding nano-clays of montmorillonite in polyurethane foam with carbon prepreps. SEM observation was performed on polymer samples, layered with urethane, and reinforced with different contents of nano-clays. In addition, the potential control of cellular structures by internally introducing nano-additives on pore surfaces was investigated. Development of urethane foam layers reinforced by nano-clays of montmorillonite was a first step in this perspective.



**Figure 1: Tangentially Reinforced Nano-Foam Structures**

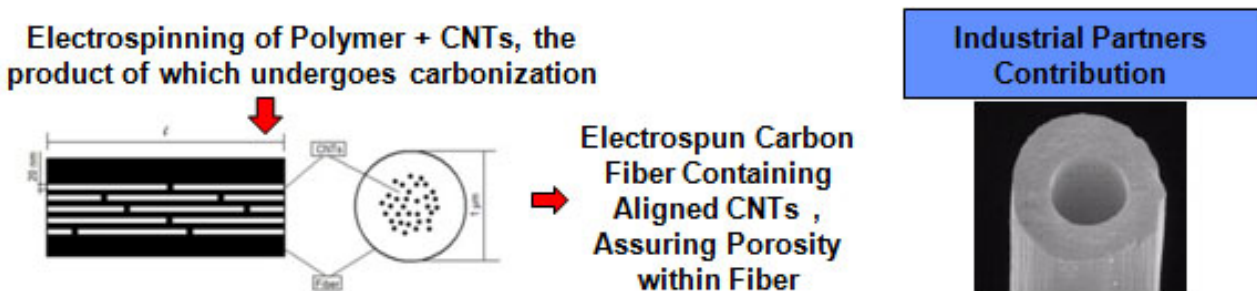
Furthermore, some SEM photos from the urethane layers reinforced with nano-clays of montmorillonite are presented below in different nano-clays contents.



**Figure 2: Urethane reinforced with Montmorillonite Nano-Clays in different fractions and with different nucleating agents**

The examination showed a general trend of greater quantity, smaller size, and more intensive reinforced pores, as weight content increased. From a size point of view, the nucleating agent did not really affect the two different systems, as there were no significant changes in pore size with respect to foam morphology, with and without nucleating agent. However, it was obvious that the nucleating agent assisted the pores in their accumulating more nano-clays on their surface.

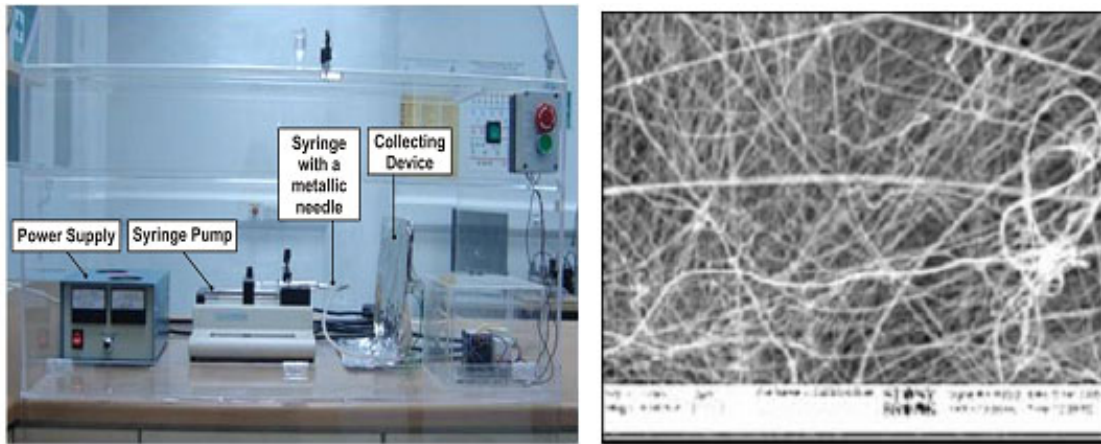
2) Second, another effort of our team focused on characterization of how aligned carbon nanotubes improved material properties. The possibility of including aligned CNTs in electrospun carbon fibers, and then carbonizing them, was investigated. Figure 3 shows a schematic of the fiber manufactured through electrospun CNTs.



**Figure 3: Carbon Fibers through Electrospun CNTs in polymer solution**

Figure 4 illustrates the basic electrospinning configuration that utilized a simple syringe-like apparatus. At the laboratory level, this apparatus consisted of three main devices: 1) a capillary tube (syringe) with a metallic needle of small diameter filled with the fluid solution or melt, 2) a high voltage power supply, and 3) a conductive collecting device. Both direct and alternating current (DC and AC) power supplies could be used for electrospinning, with DC being the most common.



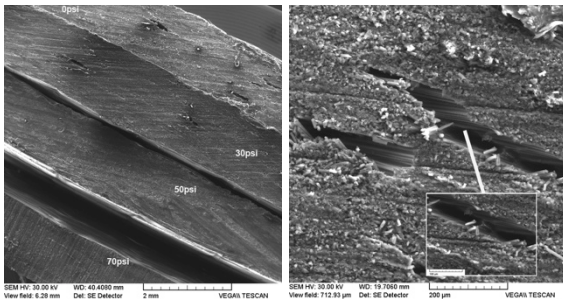


**Figure 4: Left: Electrosinning Configuration at a laboratory scale, Right: Nanofibers Produced through Electrosinning**

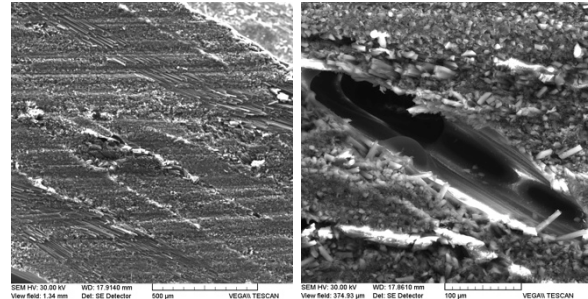
The electrospun carbon fibers due to their smaller diameter compared to normal carbon fibers are planned to be used in the fractal structures of featherweight composites.

**3)** Another area in which work was performed concerned manufacturing prepreg panels through pressure variation in a repair autoclave, and separating out pressure from temperature as a technique that could assist in void formation through pressure control.

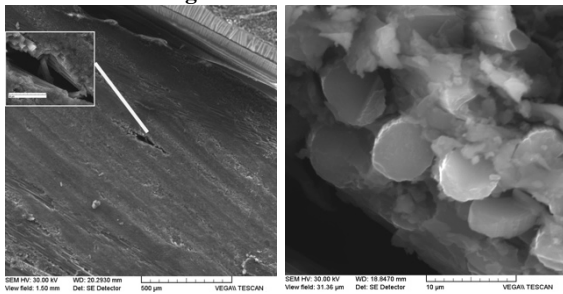
Evaluations of the results were made using Differential Scanning Calorimetry (results presented in prior documentation), Dynamic Mechanical Analysis, and Scanning Electron Microscopy. For conventional epoxy CFRPs manufactured in the repair autoclave with the same temperature profile, pressure was varied at 0, 30, 50 and 70 psi. Following are figures demonstrating the DMA and SEM results.



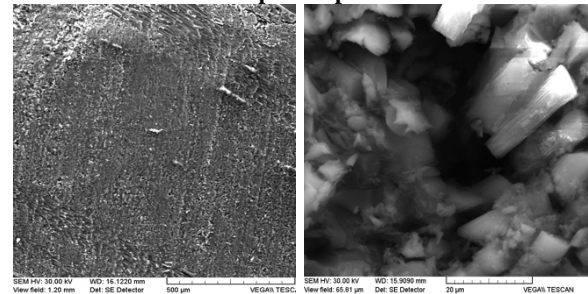
**Figure 5: left: All different samples manufactured in different pressures, right: Samples manufactured at 0 psi, showing increased void formation**



**Figure 6: left: The entire thickness cross section of a sample manufactured at 30 psi, right: Void also formed at 30psi sample**

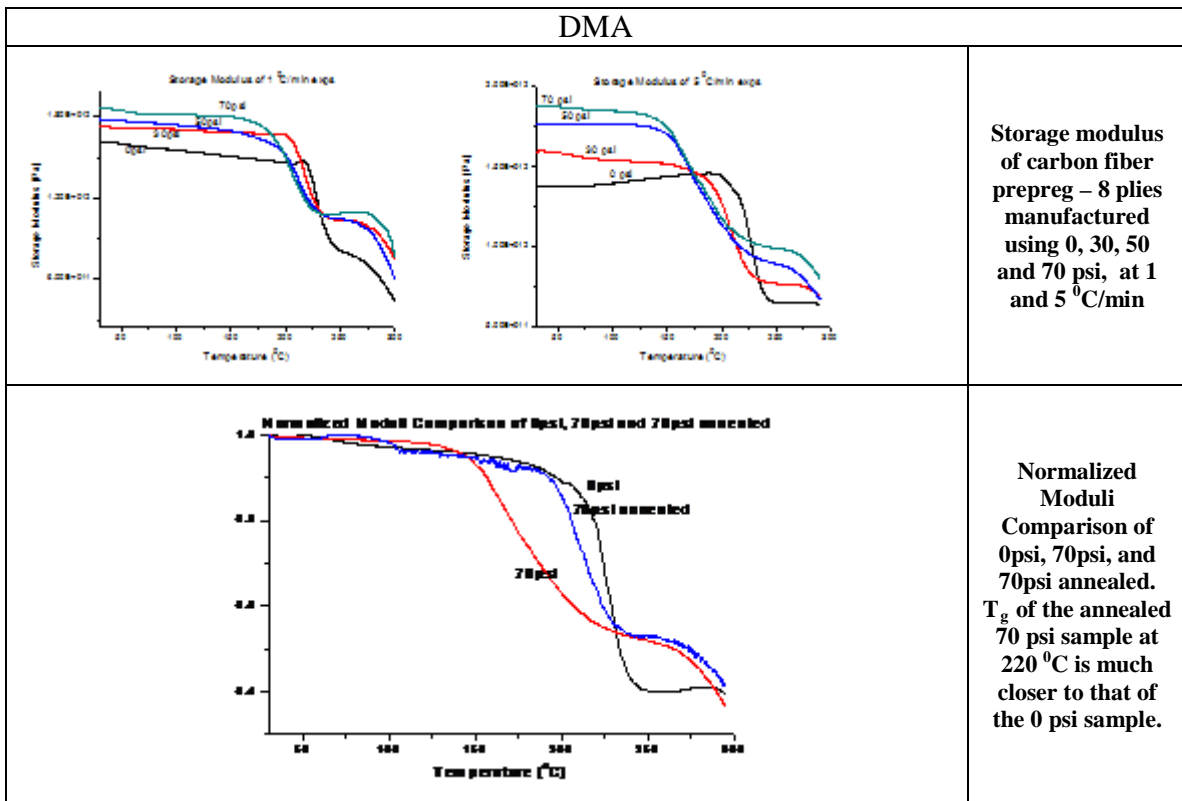


**Figure 7: left: The entire thickness cross section of a sample manufactured at 50 psi, right: Smaller voids also formed at 50 psi sample**



**Figure 8: left: The entire thickness cross section of a sample manufactured at 70 psi, right: Even smaller and less voids formed at 70 psi sample**

Scanning electron microscope photographs 5 through 8 clearly show void formation reduction in number and size as the manufacturing pressure increased. Scanning electron microscope observation was performed for samples from all pressures. The results verified that storage modulus increased as pressure increased, which also resulted in less void formation.



DMA verified SEM findings, as it was observed that there was a slight increase in modulus while manufacturing pressure increased. This was to be expected as the higher the pressure, the less the void formation within the composite. However, it was also observed that the glass transition temperature slightly decreased while the pressure increased. The glass transition reduction using a 1 °C/min heating rate was on the order of around 30 °C. ( $T_g = 206$  °C at 70 psi and  $T_g = 228$  °C at 0 psi). This difference expanded when the heating rate increased to 5 °C/min, to be on the order of around 50 °C ( $T_g = 180$  °C at 70 psi and  $T_g = 225$  °C at 0 psi). This finding can be explained from internal stresses that the higher pressure imports into the composite. Thus, the temperature increase relieved these stresses and the material softened, leading to an apparent decrease in the glass transition temperature. This was verified by annealing the 70 psi manufactured samples at 220 °C for an hour, and then performing DMA at a 5 °C / min heating rate. The  $T_g$  value increased from 180 °C to 214 °C, which was a value very close to that of the 0 psi manufactured sample.

4) Thermoplastic and Thermosetting matrix thermal and mechanical properties were illustrated through DSC and DMA results. Through DSC experiments, which were conducted on samples of all modes, we found that thermal shock and moisture adsorption at 60°C individually and combined, did not affect the crystallization and melting temperatures of the composite to a noticeable degree (Figure 9).

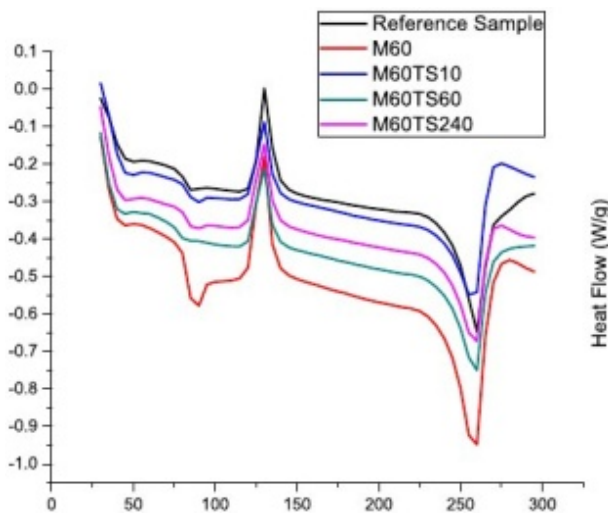


Figure 9: Moisturized below  $T_g$

On the other hand, the moisture adsorption at 90°C had a major impact on the crystallization of the composite. The large exothermic peak in Figure 9, implying crystallization, did not exist for these samples in Figure 10.

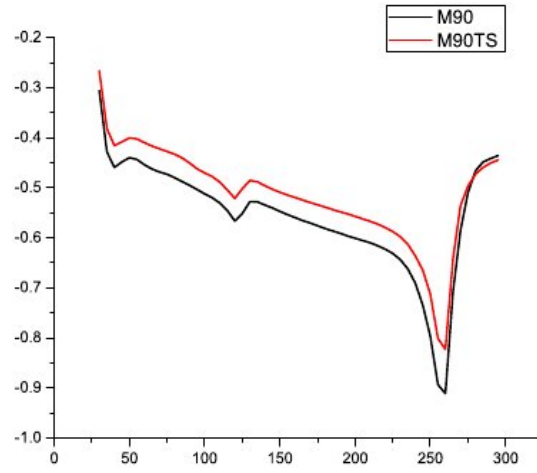


Figure 10: Moisturized slightly above  $T_g$

All samples were subjected to DMA testing, where the change in composite dynamic properties such as storage modulus ( $E'$ ) were measured. In the following Table (1) the storage modulus depending on the moisture absorption is shown. The thermal shock treatment alone did not result in any significant changes in storage modulus.

Table 1: Variation of storage modulus

Reference Sample	TS10	M60	M60TS10	M90	M90TS10
16,5 GPa	17G Pa	15,6 GPa	19 GPa	35,1 GPa	34,7 GPa

The crystallization that occurred in samples moisturized above  $T_g$ , resulted in a significant storage modulus increase, agreeing with DSC results.

5) Models were developed of control void formation and size, attained by the addition of nano-fibers during the autoclave process. Model CNTs of large diameter and low number of walls, utilized as nano-pores within the matrix, resulted in lower CNT reinforced matrix system densities. The nano-free volume within a CNT of any radius-number of walls combination can be found (Figure 11). The blue flat area corresponds to CNTs that cannot exist, as the radius is too small to fit the respective walls.

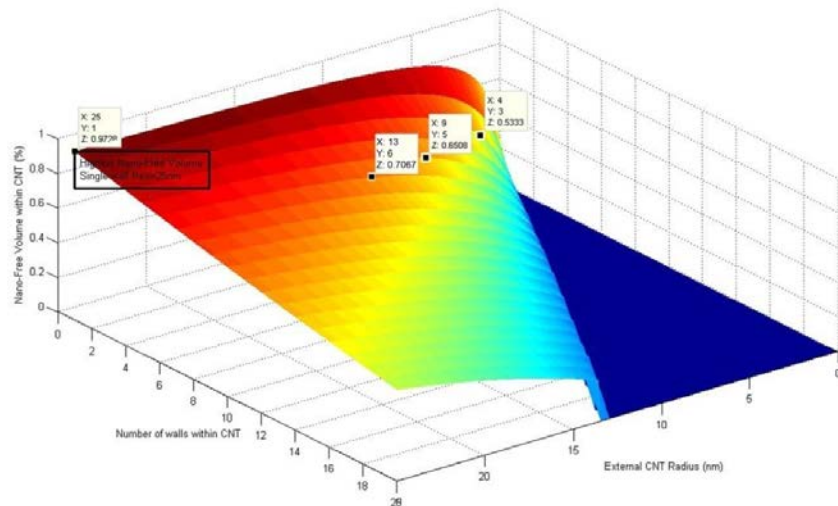


Figure 11: Nano-Free Volume within a CNT as a function of CNT External Radius and Number of Walls



Figure 12 shows polymer nanocomposite (PNC) density as a function of CNT volume fraction for different CNTs.

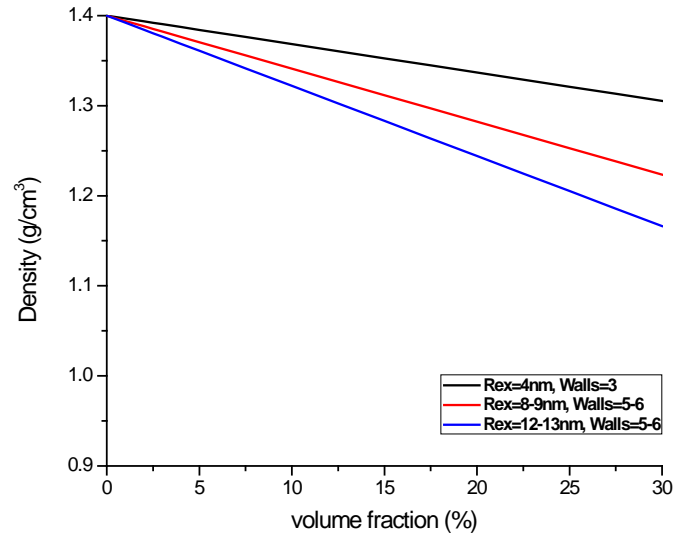


Figure 12: PNC Density as a function of CNT volume fraction for different CNTs

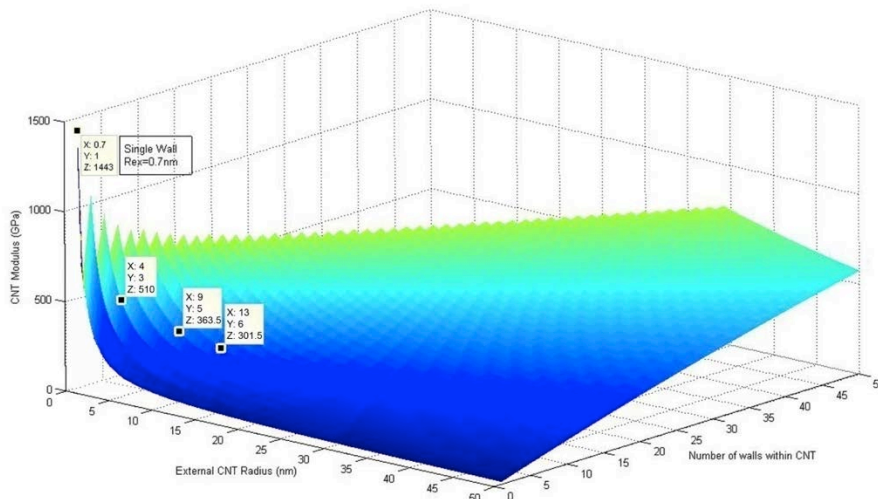
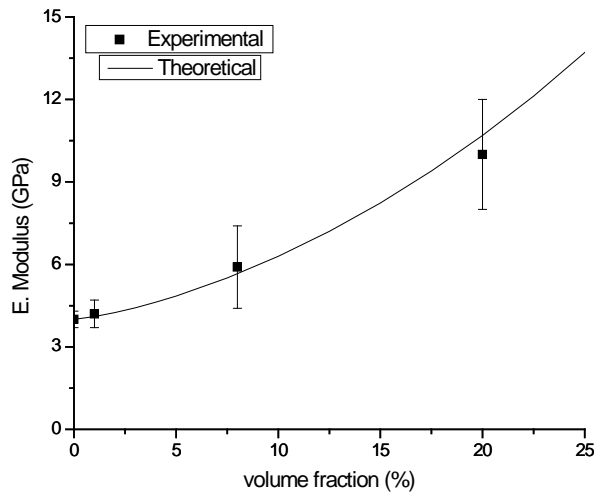
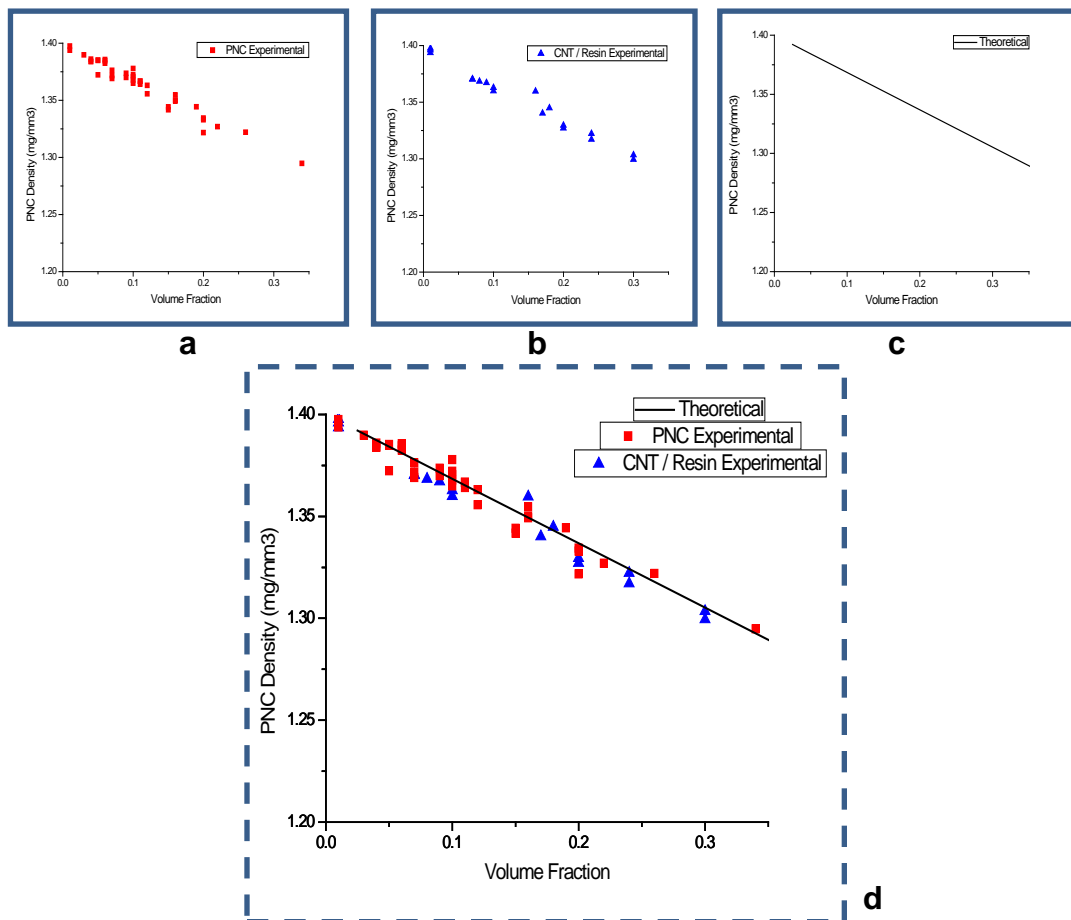


Figure 13: CNT Modulus as a function of CNT External Radius and Number of Walls

Figure 13 shows that the CNT modulus decreases as its radius increases, which is consistent with what mostly can be found in literature, and modulus increases as the CNT walls increase. Figures 14 and 15 present how the modulus of a PNC for CNT distribution of average diameter  $R_{ex}=4\text{nm}$ , and 3 Walls, as CNT volume fraction increases. The PNC modulus as a function of CNT volume fraction for aligned CNTs is plotted in Figure 16, to show the correlation of experimental and theoretical results.



**Figure 14: PNC Modulus for Aligned CNTs of  $R_{ex}=4nm$  and 3 Walls**



**Figure 15: PNC Density for Aligned CNTs of  $R_{ex}=4nm$  and 3 Walls (Different Experimental Approaches)**

Additionally, Figure 16 illustrates both theoretically and experimentally that the relation of modulus and density is not like in the classic materials, which means that the more CNT we put in the composite the higher the modulus but the less the density.

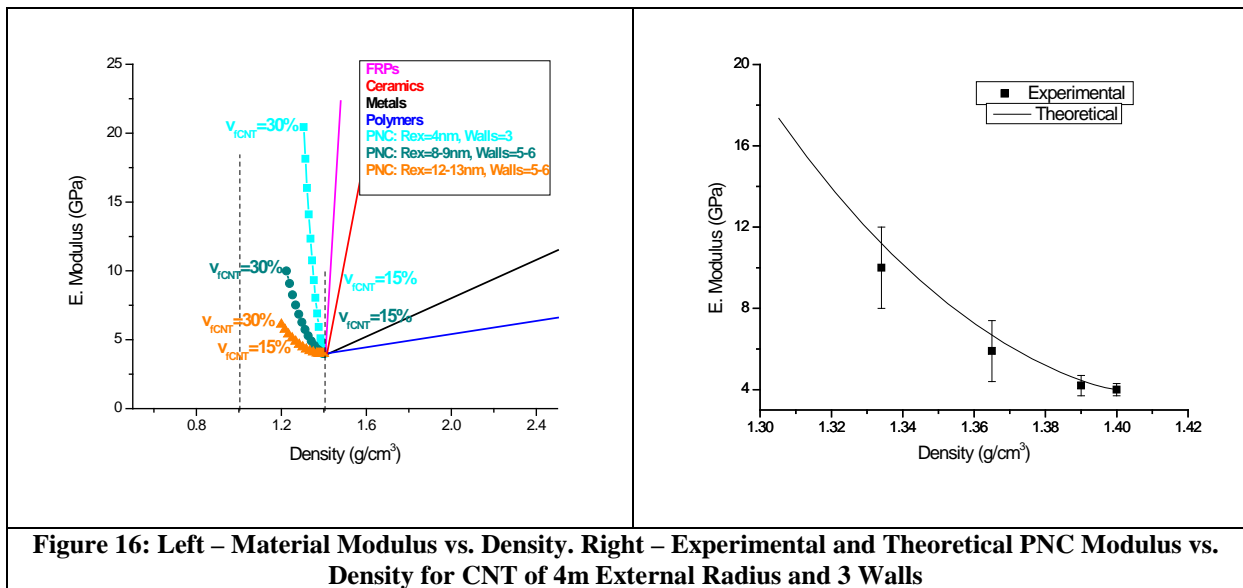


Figure 16: Left – Material Modulus vs. Density. Right – Experimental and Theoretical PNC Modulus vs. Density for CNT of 4m External Radius and 3 Walls

6) Material property improvement through the inclusion of aligned carbon nano-tubes was evaluated in-depth together with our partners at MIT. Significant progress has been made in the area of epoxy polymer nano-composites with CNTs in different volume fractions. The process followed for manufacturing and modulus experimental measurements are presented in Figures 17 and 18-19, respectively.

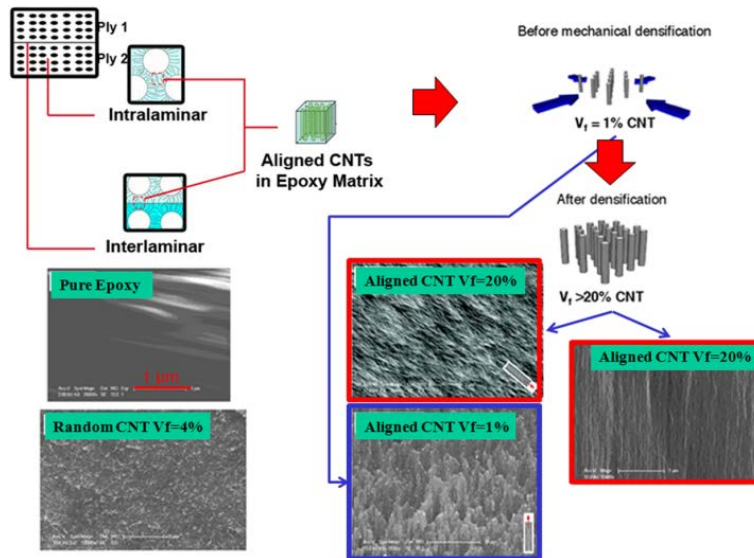


Figure 17: Polymer nano-composites manufactured with different CNT volume fractions

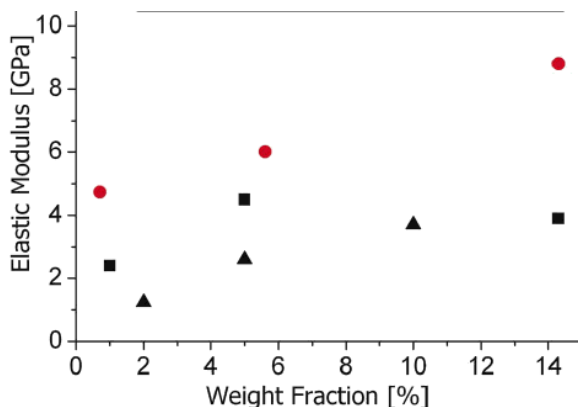
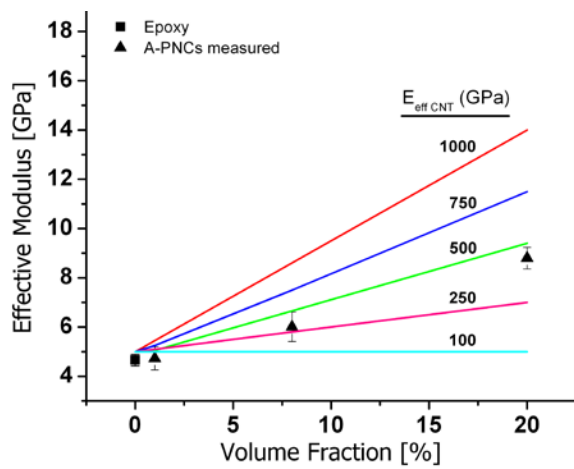


Figure 18: Aligned CNT Modulus vs. other investigations found in literature

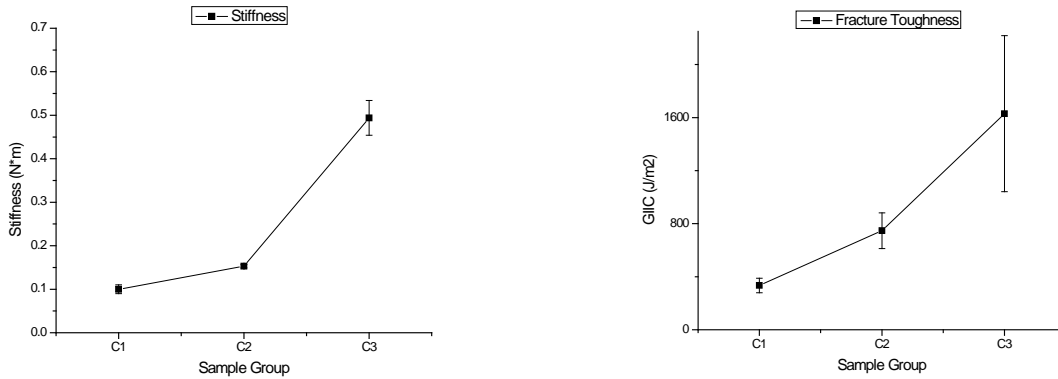
- Spheres: aligned PNCs
- Squares: randomly oriented CNTs in a thermosetting matrix
- Triangles: randomly oriented CNTs in a thermoplastic matrix



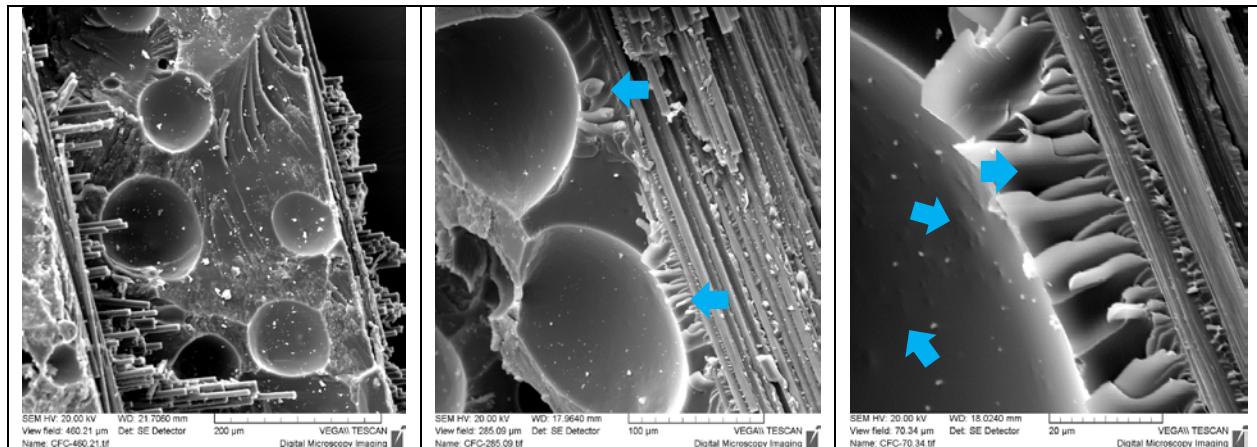
**Figure 19: CNT Fiber Modulus as Measured from Aligned CNT Nanocomposite Testing**

## 7) Material Model and Design

Through the above technologies, two different systems of nano-porous materials were formed to imitate feathers that can be found in nature, and create fractal structures between lamina, from macro to micro and nano dimensions. These model system materials consist of the wider group and concept of featherweight composites. The first model system included epoxy foam and CNTs in the interlayer. Foam and CNTs were gradually added to the material so there was a comparison with all kinds of specimens, and the contribution of both foam and CNTs could be evaluated (sample types: Control, CF + Foam, CF + Foam + CNTs). Figure 20 shows mechanical property improvements; Figure 21 demonstrates SEM observations; and weight savings are demonstrated in Table 2.

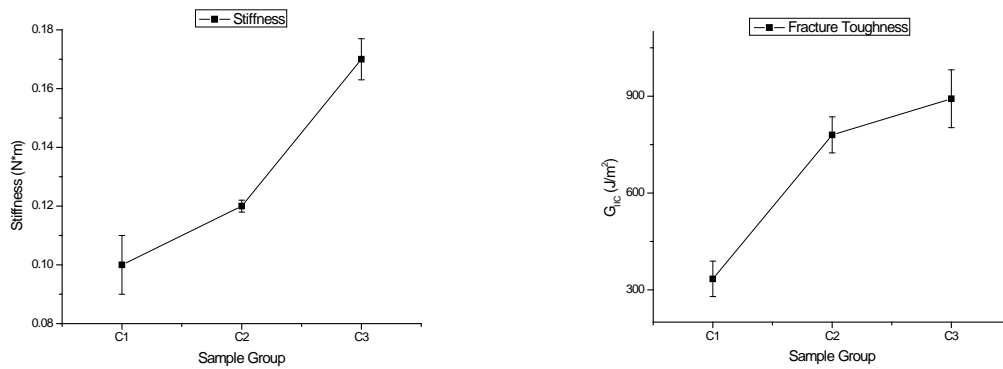


**Figure 20: Left: Stiffness Improvement of Epoxy Foamed Interlayer. Right: Mode II Fracture Toughness Improvement of Epoxy Foamed Interlayer**

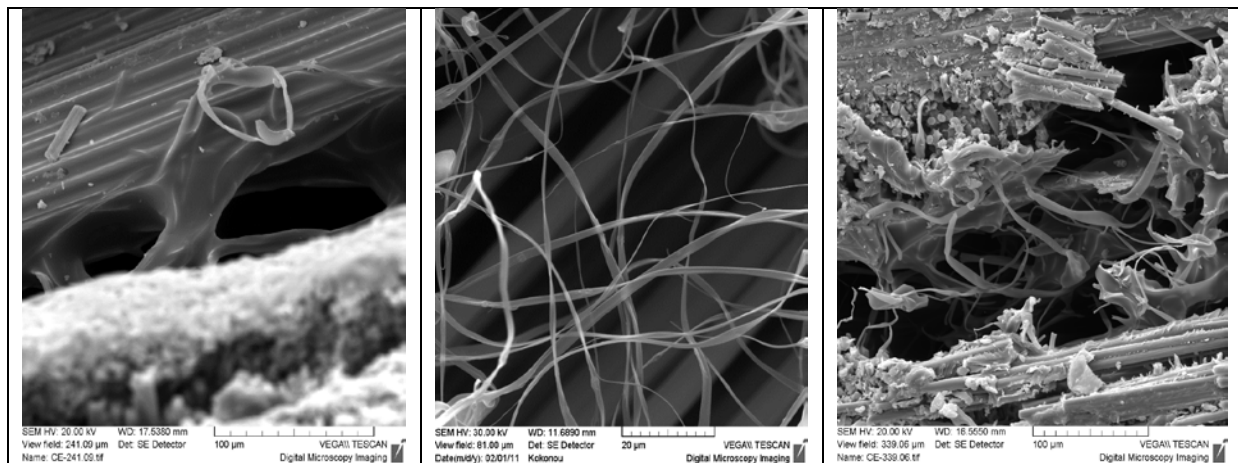


**Figure 21: SEM Observation**

Figures 22 and 23 demonstrate similar properties of another featherweight model, which has electrospun fibers with CNTs, rather than foam, in the interlayer.

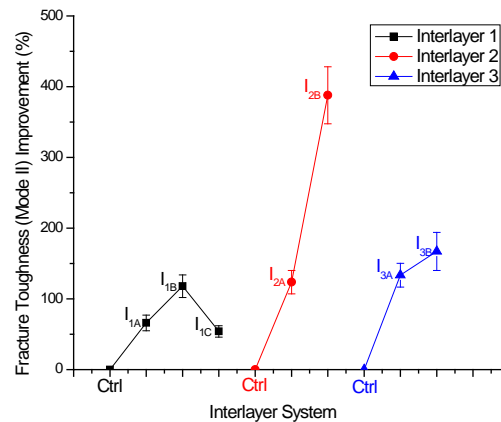


**Figure 22: Left: Stiffness Improvement of Electrospun Fiber Interlayer. Right: Mode II Fracture Toughness Improvement of Electrospun Fiber Interlayer**



**Figure 23: SEM Observations**

Finally, the total improvement of the new interlayer systems is presented (Figure 24), together with the weight reduction. The latter is not more than 9%, as heavy low cost CNTs were utilized. If cleaner, more expensive and therefore lighter CNTs were utilized, the weight savings could be much greater, depending on the CNT distribution.



**Figure 24: Mode II Fracture Toughness Improvement Comparison between the Three Different Interlayer Systems**



**Table 2: Average Densities of CFRP with All Interlayer Cases**

<b>Control CFRP Density (g/cm<sup>3</sup>)</b>		<b>1.69 ± 0.01</b>	
	<b>v<sub>f</sub>=25% (g/cm<sup>3</sup>)</b>	<b>v<sub>f</sub>=36% (g/cm<sup>3</sup>)</b>	<b>v<sub>f</sub>=44% (g/cm<sup>3</sup>)</b>
<b>Nylon Micro-Spherical Interlayer (1)</b>	1.66 ± 0.01	1.65 ± 0.011	1.645 ± 0.01
	<b>Epoxy Foam (g/cm<sup>3</sup>)</b>	<b>CNT Reinforced Epoxy Foam (g/cm<sup>3</sup>)</b>	
<b>Epoxy Foamed Interlayer (2)</b>	1.544 ± 0.034	1.561 ± 0.03	
	<b>Electrospun Fibers (g/cm<sup>3</sup>)</b>	<b>CNT Reinforced Electrospun Fibers (g/cm<sup>3</sup>)</b>	
<b>Electrospun Fibers Interlayer (3)</b>	1.6015 ± 0.02	1.615 ± 0.018	

### **NEW FINDINGS AND INVENTIONS**

During the final year of this effort, two disclosures were prepared for prospective Patent Applications together with Intellectual Ventures of Bellevue, Washington. These have been summarized in the Report of Inventions, DD Form 882. The Title of Inventions are:

- 1) “Homogeneous Nano-porous Carbon Fiber Reinforced Composite utilizing Carbon Nanotubes as the Nanoporosity”
- 2) “Carbon Fiber Reinforced Epoxy with CNT Reinforced Electrospun Carbon Nanofibers and CNTs Reinforcing the Interlayer”

The titles may be modified in the future at the prospective time of patent application.

### **CHANGES IN PROGRAM MANAGER**

During the Grant Period, the Program Manager changed from Dr. Charles Lee to Dr. Joycelyn Harrison.

### **PERSONNEL SUPPORTED**

1. P.I. involved in the research project:

Dr. James C. Seferis

2. Co-P.I. involved in the research project:

Dr. Chris N. Velisaris

3. Professionals

Prof. Charalambos Doumanidis

Prof. Nicholas Kanelopoulos

4. Graduate Student Interns

Dr. Vasileios Drakonakis

Mr. Aris Sfakianakis

5. In addition to the aforementioned personnel, the Massachusetts Institute of Technology, Nano-Engineered Composite aerospace Structures (NECST) was supported through subcontract.

Among the team members of our proposed work, Prof. Brian Wardle from MIT received tenure during the grant period. Moreover, Professor Doumanidis from University of Cyprus was on sabbatical with the Polymeric Composites Laboratory in 2010, assisting our effort in electrospinning, processing, and nano-technology, before accepting an IPA assignment as Program Director for the Nanomanufacturing Division, within the Division of Civil, Mechanical and Manufacturing Innovation (CMMI) of the National Science Foundation. Finally, Professor Kanelopoulos, Director of the Nano-pore Center of Democritus (National Center of Scientific Research) in Greece, joined our team while on sabbatical, significantly contributing to our research through matrix porosity development and analysis. Mr. Aris Sfakianakis worked with Professor Kanelopoulos in a joint program through Democritus, as well.

## **PUBLICATIONS**

- 1) Vasileios M. Drakonakis, Chris N. Velisaris, and James C. Seferis, Charalabos C. Doumanidis, CNT Reinforced Electrospun Nanofiber Interlayer System for a Featherweight Composite System, *Advances in Nanocarbons* (2012) Special issue of *Journal of Nanocomposites* (hindawi) – Submitted.
- 2) Vasileios M. Drakonakis, James C. Seferis, Charalabos C. Doumanidis, Curing Pressure Influence of Out-of-Autoclave Processing on Structural Composites for Commercial Aviation, *Modeling, Characterization and Processing of Advanced Composites* (2012), Special Issue (hindawi) – Submitted.
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## HONORS AND AWARDS

On June 15, 2005, Professor Seferis retired from the University of Washington and assumed an interim position as Research Professor at the Center for Composite Materials at the University of Delaware through December 31, 2006. He has reconstituted his laboratory, the Polymeric Composite Laboratory, first established at the University of Washington in collaboration with aerospace, chemical, and materials industries, in private facilities in Seattle, Washington. Professor Seferis and his team of full time professionals continue their research and development activities adding formally to their collaborative work with universities from the United States and abroad (Europe and Asia). He is still in demand for presenting his work, especially that funded by the AFOSR, of which he presented as keynote lecturer in meetings and colloquia during this and prior funding periods. In prior periods, these included both the 2007 and 2008 SAMPE Annual and Technical Conferences; a U.S.-Greece Bilateral Meeting; the International Conference on Structural Analysis of Advanced Materials (2007 and 2009); an MIT-Aeronautics Department Colloquium; the Japanese Conference on Composites and the Environment; CACRC workshops in Greece and Wichita (2007 and 2008); GloCal workshops in Seattle, Paris and Athens (2007 through 2009); and International Astronautical Conferences (2007 and 2008).

More recently, this and related work were presented at the International Conference on Structural Analysis of Advanced Materials 2009 in Tarbes, France, the American Society of Composites ASC-CASMA (2009) in Newark, Delaware, the International Astronautical Conference IAC09 in Daejeon, Korea (2009), the American Society of Composites ASC in Dayton, Ohio (2010), SAMPE in Seattle, Washington (2010), DURACOSYS in Patras, Greece (2010), The 18<sup>th</sup> International Conference on Composite Materials (ICCM) in Jeju Island, Korea (2011), and GloCal workshops in Seattle (2010 through 2012). We anticipate additional presentations in the future. Furthermore, this effort funded, in part, the Doctoral Dissertation of Dr. Vasileios Drakonakis at the University of Texas at Arlington entitled "CNT Reinforced Epoxy Foamed and Electrospun Nano-Fiber Interlayer Systems for Manufacturing Lighter and Stronger Featherweight<sup>TM</sup> Composites," under the direction of Professors James Seferis, Charalambos Doumanidis, and John Priest. Dr. Drakonakis completed the requirements for his degree in August 2012.