ADVANCED COMPOSITE MATERIALS TECHNOLOGY FOR ROTORCRAFT

Andrew Makeev*, University of Texas at Arlington, Arlington, Texas, USA
Charles Bakis and Eric Strauch, Penn State University, University Park, PA, USA
Mark Chris, Bell Helicopter Textron, Fort Worth, TX, USA
Peter Holemans and Gina Miller, Boeing Rotorcraft Systems, Ridley Park, PA, USA
Don Spencer, Kaman Helicopters, Bloomfield, CT, USA
Nicolas Patz, Patz Materials & Technologies, Benicia, CA, USA

Abstract

Composite materials are increasingly used in rotorcraft structures to reduce weight and improve efficiency. The rotorcraft industry is constantly in need of higher-performance materials that offer improved mechanical strength and stiffness at a lower weight. In polymer-matrix composite structures, matrix-dominated failures impose severe limitations on structural performance. The objective of this work is to advance composite material technologies for rotorcraft through the use of nanoadditives to improve structural efficiency. Technical challenges and potential solutions for improving matrix-dominated performance of prepreg composites through nanoparticle reinforcement, are discussed. In particular, a promising technology for improving compression and interlaminar strength and fatigue performance, is identified. The advanced materials technology is based on high weight content loading of approximately 100-nm diameter nanosilica particles in low-viscosity resins. Such technology resulted in compression strength improvement for intermediate-modulus carbon-fiber/epoxy-matrix 250° F curing prepreg composites as recently demonstrated by 3M. This work not only supports the initial findings of 3M regarding the improvement of compression strength performance but also demonstrates improved interlaminar material properties including fatigue performance, and expands the material design space. Fatigue performance is critical to rotorcraft dynamic components as they are subject to extreme oscillatory flight loads that can result in material fatigue failures.

1. INTRODUCTION[1]

Fiber-reinforced composite materials are increasingly used in rotorcraft primary structures. The industry is constantly in need of higher-performance materials that offer improved structural performance at a lower weight. Rotorcraft dynamic components are among the most challenging composite applications as they are subject to extreme flight loads that are oscillatory in nature and cause material to fail in fatigue. In polymer-matrix composites, matrix-dominated failures such as delamination and low fiber-direction compressive strength compared to the fiber-direction tensile strength, impose significant limitations on structural performance and longevity characteristics.

In fiber-reinforced polymer composites, the polymer matrix and the matrix-fiber interface are much weaker than the fibers. The incorporation of nano-sized reinforcement in the matrix may improve the recognized weaknesses of composites, such as interlaminar and compressive strengths. However, the implementation of such advanced materials in rotorcraft has been limited by conflicting information in the literature on the best approach for the enhancement of matrix-dominated properties, the lack of suitable material property data, and unproven repeatability and manufacturability at the structural scale. Therefore, a need exists to develop a knowledge base that characterizes advanced material technologies in conjunction with the manufacturing methods employed to process and post-process the advanced materials, establish process controls, and finally demonstrate the feasibility of the advanced materials, with the emphasis on fatigue life and life-cycle costs, versus conventional materials for rotorcraft applications.

The Vertical Lift Consortium, which represents a collaboration of U.S. Government, rotorcraft industry, and academia to develop and transition innovative vertical lift technologies, recently started the Advanced Materials Technology (AMT) Program with a goal to advance material technologies for the improvement of rotorcraft material strength and fatigue behavior. Specific objectives included (a) screen state-of-the-art material technologies; (b) select the most promising materials for improved matrix-dominated performance and acceptable processing and handling qualities; and (c) develop a database of material properties for use in structural design. Thus, a knowledge base

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* Corresponding author, e-mail: makeev@uta.edu.

providing a foundation for the insertion of advanced materials in rotorcraft applications, is being developed.

The AMT program is a multi-year collaborative effort of U.S. rotorcraft Original Equipment Manufacturers (OEMs) including Bell Helicopter, Boeing Rotorcraft, Sikorsky Aircraft, Kaman Helicopters, and research labs of academic institutions including University of Texas Arlington (UTA) and Pennsylvania State University (PSU). The AMT program also engaged commercial manufacturers of pre-impregnated fiber reinforced polymer composites (prepreg) and offered a unique opportunity for the rotorcraft OEMs and the material manufacturers to work together towards the development of material solutions that improve structural strength and fatigue behavior.

The AMT program started with a screening effort which identified the most promising candidate technologies for improvement of interlaminar fatigue performance and compressive properties. Such improvement was compared to the prepregs (carbon and glass fibers) currently used in rotorcraft structures. Not only structural performance but also manufacturability has been considered. For example, success criteria included improvement of mechanical properties of 350° F curing systems relative to a legacy baseline 350° F cure toughened epoxy system (Hexcel 8552 [1]) without deteriorating processing and handling qualities such as viscosity, tack and working life. Currently, several rotor system applications use 250° F curing resins due to the relatively high viscosity of 350° F curing toughened epoxy systems such as 8552 [1]. Success criteria also included the improvement of mechanical performance of 250° F curing systems relative to legacy baseline 250° F cure toughened epoxy systems such as Cytec 381 [2] and E773 [3], at a lower viscosity compared to 8552.

Among many candidates for improving compressive and interlaminar properties of composite materials are nanosilica-loaded matrices. Nanosilica (~100-nm diameter silica particles) is cost-effective; enables high loading (more than 40% weight content in the resin) with minimum impact on viscosity; and can be uniformly dispersible through surface chemistry technology (functional groups) [4, 5]. In 2009, 3M launched 3M™ Matrix Resin 3831, a 36% nanosilica weight content 250° F curing epoxy resin system designed for use in composite prepreg manufacturing processes [5]. Initial implementation of this resin in the sporting goods market has produced carbon-fiber composite fishing rods with 60-90% increased compression-dominated bending failure loads [5].

3M disclosed the material morphology as well physical and mechanical properties of their resin systems subject to various nanosilica loadings. Also, mechanical properties of carbon/epoxy nanosilica prepregs were published [4, 5]. Unidirectional prepreg tape for each of the resin systems studied in Refs [4, 5] was produced by Patz Materials and Technologies (Benicia, CA) using TR50S carbon fiber (Grafil Inc., Sacramento, CA). In this work, the material characterization is expanded to carbon-fiber and glass-fiber prepregs applicable to rotorcraft structures. In particular, compression and interlaminar material properties including fatigue curves are determined and compared to existing production rotorcraft prepreg material systems. It is worth noting that the previous studies employed standard testing and never questioned their suitability for measuring true material properties. For example, the ASTM D 2344 standard short-beam shear (SBS) test method [6] used in Ref [5] does not capture the interlaminar shear (ILS) strength and modulus material properties. Also, ILS fatigue characterization of carbon-fiber and glass-fiber epoxy nanosilica prepreg composites has not been accomplished before. Furthermore, assessment of the interlaminar tensile (ILT) material properties, including strength, modulus, and S-N curves, has not been attempted in the previous studies. This work not only supports the initial findings of 3M regarding the improvement of compression performance of intermediate-modulus carbon-fiber/epoxy-matrix 250° F curing prepreg composites but also expands the material design space to glass-fiber prepregs, and clarifies the interlaminar properties of nanosilica prepreg composites including fatigue behavior.

2. MATERIALS

As noted in the previous section, nanosilica-loaded prepreg composites are considered in this work. References [4, 5] provide the details of the material structure for 250° F curing carbon/epoxy nanosilica prepregs as well as their matrix-dominated properties measured using standard test methods. As listed in Refs [4, 5], matrix stiffness can be a primary variable affecting composite compression strength in the fiber direction because fiber microbuckling – a major compression failure mechanism – depends on the amount of support provided by the matrix to the fibers. Incorporation of hard particles into polymers increases their modulus and can increase fracture resistance [4, 5, 7]. Micron-scale inorganic fillers have been used to modify cured resin properties, but when processed into fiber-reinforced composite structures, these large particles are filtered out by the reinforcing fibers. Another undesirable effect of conventional micron-size fillers is increased resin viscosity before curing, which can compromise composite processing qualities [4, 5]. 3M attempted to achieve the desirable resin modulus and laminate compression strength improvements through the incorporation of smaller, nano-sized amorphous silica particles into thermoset-matrix resins [4, 5].

In 2009, Patz Materials and Technologies (PMT) began working with 3M to address specific applications where 3M's nanosilica technology could

yield significant benefit for composite structures. PMT would formulate the applicable thermosetting polymer technology for the specific application and 3M would apply the nanosilica to that polymer. The key to the performance of the nanosilica in each polymer system is 3M's ability to tailor the surface chemistry for the nanosilica particles. For each formulated product that PMT supplied, 3M would develop the appropriate surface chemistry for the particles. This allows the nanosilica particles to become an integral part of the polymer system, and thus the desired particle attributes could be realized in the composite structure. This also allows the particles to flow freely with the polymer system thus only minimally increasing the viscosity of the formulated product. The non-agglomerated compatibilized nanosilica can be evenly dispersed throughout the composite structure without filtration by the fiber array.

The highly compatible nature of the functionalized particles enables epoxy resins with levels of nanosilica exceeding 50% resin weight content. The nanosilica particles form non-aggregated dispersions at all loading levels. Figure 1 shows a representative 250° F cured carbon/epoxy composite laminate with 36% weight content of approximately 100-nm diameter functionalized silica particles evenly dispersed between 7-μm diameter carbon fibers [4, 5].

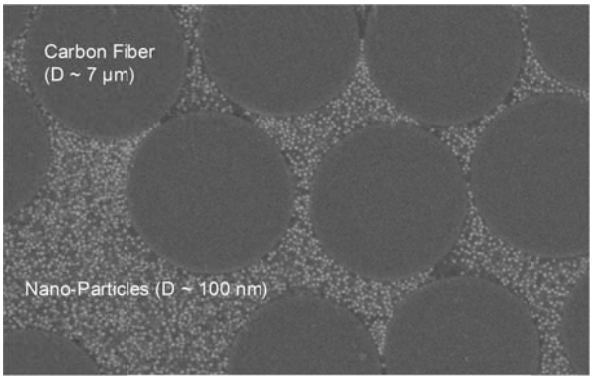

Figure 1. SEM image of a carbon/epoxy laminate cross section demonstrating even dispersion and lack of conglomeration for the nanosilica particles [4, 5]

The composite materials studied by 3M included unidirectional intermediate-modulus TR50S carbon-fiber prepreg tapes produced by PMT. The prepreg tapes were made using 250° F curing epoxy blend with 48% weight content of approximately 100-nm diameter silica, diluted to 15%, 25%, 35%, and 45% weights. Control prepreg containing no silica was also made. All prepreg tapes were 12 in (30.48 cm) wide. The areal weight of carbon fibers was 145 g/m^2. A 0.0051 in (0.129 mm) nominal cured ply thickness was listed [4, 5]. The fiber volume fraction was approximately 60%. References [4, 5] report the details of the curing cycle as well as properties of the resin systems.

The AMT program also engaged PMT to produce prepregs applicable to rotorcraft structures. Intermediate-modulus carbon and high-strength glass fibers were selected for the initial evaluation: IM7 and IM8 carbon (Hexcel, Stamford, CT) and S2-glass (AGY, Aiken, SC). Glass fibers were made with several types of commercial sizings: 463 and 933 sizings were used in this work. The reason for the different types of sizing was that unlike carbon fibers, the functionality of sizing for glass fibers provides not only handling qualities but also chemical bond between the fibers and the resin. PMT made unidirectional prepreg tape with two general types of epoxy resin (250°F and 350°F cure). The areal weight of the carbon and glass fibers were approximately 145 and 295 g/m^2, respectively, while nominal ply thicknesses were 0.0056 and 0.0090 in., respectively. Based on this information and specific gravities of 1.780 for IM7 carbon and 2.475 for S2-glass, the calculated nominal fiber volume fractions for the carbon-fiber and glass-fiber composites were approximately 57% and 52%, respectively. In addition to the control prepregs containing no nanoadditives, prepreg tapes with up to 40% resin weight content of nanosilica (approximately 100-nm diameter) were produced. It is worth noting that densities of the composites with 40% nanosilica, characterized in this work, were comparable to the corresponding legacy composites. The nanosilica prepregs which PMT produced for the AMT program had either 20% or 40% nanosilica resin weight content. Notations PMT(20%NS) and PMT(40%NS) are used to indicate the appropriate nanosilica weight content in the resin.

It is noteworthy that 250° F curing IM7/PMT and IM8/PMT carbon prepregs are made by the same prepregger (PMT) using the same resin and nanofiller types, and also using intermediate-modulus carbon fibers with similar properties to TR50S carbon fibers employed in the 3M studies [4, 5]. Therefore such prepregs are expected to exhibit similar behavior. In the following sections, fiber compression, ILS, and ILT material properties pertinent to rotorcraft applications are compared to the best 250° F curing prepregs (such as Cytec 381 [2]) currently used in composite rotor structures whenever possible. As the subject of measuring true interlaminar material properties is still evolving and the AMT work is in progress (e.g. ILS S-N curves are not available for IM7/381 yet), this work also presents the relevant properties of 250° F curing glass prepregs (S2/381 ILS S-N curve has been determined.) All material properties in this work correspond to the room-temperature ambient (RTA) test condition (70-72°F, 40-60%RH).

In addition to the 250° F curing prepregs, this work includes the ILS fatigue performance of 350° F curing carbon prepregs. Hexcel 8552 [1] has become a benchmark in 350° F curing carbon prepregs in rotorcraft structures and there is significant data

available in literature. Recent publications document the development of experimental techniques for measuring accurate ILS and ILT material properties, including modulus, strength, and S-N curves, and the application of such techniques to IM7/8552 prepreg [8 – 13]. In this particular situation, not only nanosilica-loaded prepregs but also carbon nanotube (CNT) and graphene additives are considered due to their recent publicity as potential solutions for improving interlaminar properties. PMT manufactured 350° F curing IM7-carbon prepregs with 0.5% CNT, 0.8% graphene nanoplatelets (GR), and a mixture of 40% nanosilica and 0.8% graphene (40%NS + 0.8%GR) by resin weight. A uniform dispersion of CNT and GR in the PMT resin was challenging at higher weight content as the CNT length and the GR in-plane dimensions are not nm but microns. Also, CNT and GR are known to increase resin viscosity at higher weight content.

Experimental data generated in this work are presented in a limited fashion as the AMT program effort to generate reliable material performance characteristics, is in progress, and the availability of data appropriate for publication is limited. However, such limited information indicates potential benefits of the advanced materials technology to emerging rotorcraft platforms and prompts more extensive material qualification.

3. FIBER COMPRESSION

In Refs [4, 5], fiber-direction compression strength of the 250° F curing TR50S-carbon/epoxy nanosilica prepreg composites was assessed based on SACMA SRM 1R-94 [14] RTA testing of tabbed unidirectional 12-ply laminates. Nine specimens were tested for each material configuration. Table I lists compression strength data corresponding to the different nanosilica weight contents in the resin.

Table I. Fiber-direction compression strength of 250°F curing carbon prepreg composites [4, 5]

Silica (wt%)	Strength (ksi)	FV (%)
0	258	62.5
15	267	61.2
25	274	60.3
35	276	60.3
45	287	59.2

The apparent strength changed by 11.2% at 45 wt% nanosilica loading. After the strength values were normalized to a 60% fiber volume (FV), the change from the unfilled to the most highly filled material became 17.4%.

In this work, in-plane fiber compression performance of 250° F curing PMT prepregs was evaluated using the ASTM D6641 combined loading compression (CLC) test method [15] with a 50/50 (50% 0-deg. plies and 50% 90-deg. plies) cross-ply laminate. Use of a cross-ply laminate rather than a unidirectionally reinforced laminate reduces the maximum load applied to the specimen and the CLC fixture splits the load path between face shear and end loading, thereby avoiding premature failure at either the grip entry region or the end of the specimen without the need for tabs. However, the use of a cross-ply specimen requires that classical laminated plate theory [16] be used to back-calculate the stress in the 0-deg. plies at failure. While it is recognized that variations in matrix modulus will affect the relationship between stress at the laminate level and 0-deg. ply level, back-out factors for the 0-deg. stress were held constant throughout this investigation, based on typical unidirectional ply properties: 1.6 for glass/epoxy and 1.9 for carbon/epoxy. Multiplying the laminate stress by these back-out factors provides the stress in the fiber direction of the 0-deg. plies. The fiber-direction compressive strength of a ply determined using a 50/50 cross-ply specimen is considered to be representative of the *in situ* compressive strength of a unidirectional ply in a wide range of practical laminate configurations used in aircraft structures [17].

Based on ply and laminate properties estimated at the start of this investigation, a 0.5 in. specimen unsupported length, and other specimen design recommendations in ASTM D6641, a $[90/0]_{4s}$ laminate was selected to achieve 0-deg. ply failure prior to Euler buckling for both the carbon/epoxy and glass/epoxy laminates. The thicknesses of the glass and carbon fiber laminates were approximately 0.144 and 0.091 in., respectively. Uniaxial strain gages of $1/16^{th}$ in. grid length (Measurements Group CEA-06-062UW-350) were bonded to both sides of the specimen to record mean and bending strains in the loading direction. Strains and load were recorded throughout the compression tests using a digital data acquisition system. Five to six replicate tests were run for each type of material.

The CLC test results for 0-deg. ply compressive ultimate strength are normalized to the fiber-direction compressive strength of 250° F curing IM7/381 production prepreg composite, measured based on the SACMA testing of unidirectional laminate, which is 215 ksi (COV 3%) [2]. The CLC cross-ply laminate modulus results are normalized to the calculated cross-ply modulus of IM7/381 (11.4 Msi). These results are plotted in Figure 2. The coefficient of variation was typically less than 5% for the strength and modulus data. Such results are consistent with Refs [4, 5]. An additional set of data for the IM7/PMT(40%NS) cross-ply laminate, obtained using the SACMA test method, shows a higher 0-deg.

compressive strength than the same laminate tested using the CLC test method.

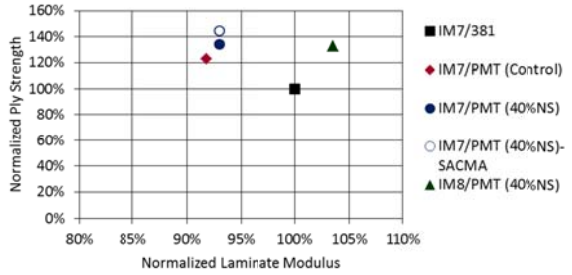

Figure 2. Compression results for 250° F curing carbon composites with and without 40% nanosilica

All the PMT systems exceed the baseline strength – with or without the nanofiller. The use of 40% nanosilica improves the strength the most. Only the IM8 composite (with 40% nanosilica) exceeds the modulus of the baseline system. The additional set of data for IM7 fiber and 40% nanosilica, obtained using the SACMA test method, shows slightly higher strength than similar material tested using the CLC test method. Cross-ply laminates were used in both test methods. The RTA strength improvement over the baseline, in this case, was as high as 45%.

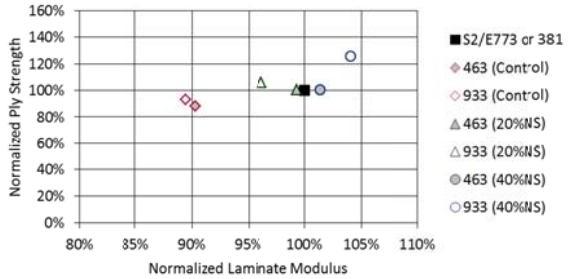

Figure 3. Compression results for 250° F curing S2-glass composites with and without nanosilica (notation for PMT prepregs starts with a number corresponding to AGY sizing)

Next, the fiber-direction compressive properties are compared for the 250° F curing glass/epoxy systems. Figure 3 shows the compression modulus and strength data normalized to the SACMA test results for unidirectional S2/E773 which exhibits similar compressive properties to S2/381 tape (185 ksi) [2, 3]. The CLC compression modulus data are normalized to a calculated cross-ply modulus of 4.45 Msi for S2/E773 and S2/381. The coefficient of variation for the strength and modulus data was less than 5%.

The S2/PMT prepreg with 933 sizing with 40% nanosilica performs the best of all the PMT systems, exceeding the baseline in terms of strength (almost 30% fiber-direction compressive strength increase) and modulus. The 933 sizing is always slightly stronger than the 463 sizing at any nanosilica concentration investigated. Overall, the 250° F curing glass and carbon PMT prepregs exhibit similar trend with the 40% nanosilica materials performing the best of all the PMT systems shown in this comparison.

4. INTERLAMINAR SHEAR (ILS)

In Ref [5], short-beam shear (SBS) strength of the 250° F curing TR50S-carbon/epoxy nanosilica prepreg composites was assessed based on ASTM D 2344 [6] RTA testing of unidirectional 24-ply laminates. Samples of 10 SBS specimens were tested, each measuring 0.25 x 0.75 in. (2t x 6t, t=thickness). A span of 4t (0.5 in.) was used. Table II lists the SBS strength data corresponding to the different nanosilica weight content in the resin system.

Table II. ASTM D 2344 SBS strength of 250°F curing carbon composites [5]

Silica (wt%)	SBS Strength (ksi)
0	13.5
15	14.9
25	15.5
35	16.8
45	17.3

ASTM D 2344 SBS strength improved with increasing silica content. An increase of 27% was measured at the highest nanosilica concentration [5].

It is known that ASTM D 2344 test method does not capture ILS material strength and cannot measure ILS modulus [6]. In this work, ILS strength and modulus material properties were measured using the modified SBS test methodology recently developed at UTA [8, 10, 11]. The modified SBS test method is applicable to fatigue loading conditions [10]. Reference [11] is the first publication aimed at initiating the standardization process for the new method.

A few details specific to the test configuration used in this work must be mentioned. The unidirectional SBS coupons to measure the ILS material properties are approximately 0.25-in. thick and wide; and 1.75-in. long. The span is 1.2 in. The loading nose diameter is modified from the ASTM D 2344 0.25-in. to 4 in. for carbon/epoxy and to 2 in. for glass/epoxy SBS coupons to avoid compressive damage at the loading nose under static and fatigue loading. Figure 4 shows the SBS test setup [10, 11] used in this work.

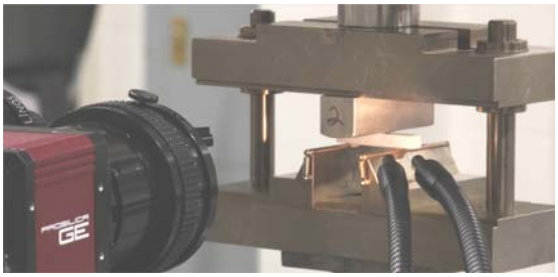

Figure 4. Modified SBS test setup for measuring ILS modulus, strength, and fatigue performance [10, 11]

Five to six specimens were tested to determine ILS strength and modulus for each material configuration. The digital image correlation (DIC) full-field deformation measurement technique was used to capture the surface strain components. DIC based strain measurement was combined with simple geometric stress approximation for measuring ILS modulus [8, 10, 11]. Linear elastic modulus approximation corresponding to the slope of ILS engineering stress-strain curves between 1,000 and 6,000 με was used in this work. It is worth noting that ILS stress-strain curves for unidirectional carbon and glass prepreg composites become highly nonlinear at 1% engineering shear strain [8 – 11]. The ILS modulus values were 0.709 Msi (COV 2.38%) for IM7/381 and 0.567 Msi (COV 4.60%) for S2/381 unidirectional tape.

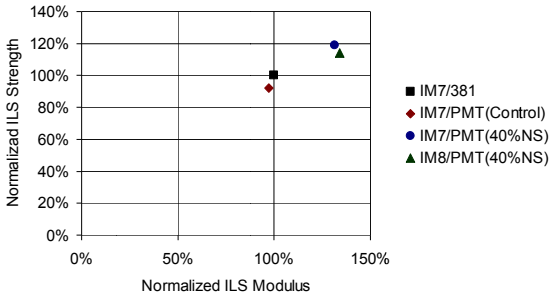

Figure 5. ILS strength and modulus results for 250° F curing carbon composites with and without nanosilica

The modified SBS method typically results in lower values of ILS strength compared to results from standard SBS tests provided by the prepreg manufacturers. For example, the ILS strength approximation for IM7/381 unidirectional tape composite is 12.0 ksi (COV 1.77%) while Cytec lists 13.3 ksi (COV 3.1%) apparent SBS strength [2]. Similarly, the ILS strength approximation for S2/381 unidirectional tape composite is 10.6 ksi (COV 2.02%) while Cytec lists 12.3 ksi (COV 4.1%) apparent SBS strength [2]. The same 3/4*(Applied Force)/(Cross Section Area) expression was used to calculate shear stress values in both cases.

Figure 5 shows the ILS modulus and strength test data for 250° F curing carbon prepreg composites, normalized to the IM7/381 baseline. PMT prepregs with 40% nanosilica exhibit up to 30% higher ILS strength compared to the control prepreg composite; and up to 20% higher ILS strength compared to the IM7/381 production prepreg composite. The strength increase compared to the control prepreg is consistent with the 3M findings [5].

Next, ILS S-N curves are determined for the 250° F curing carbon PMT prepreg composites with and without nanosilica. The S-N curves are generated based on constant load amplitude unidirectional SBS fatigue tests run at 0.1 load ratio and 10 Hz frequency. The custom SBS test configuration ensures a consistent ILS failure mode [10].

The SBS coupons were tested in a uniaxial servohydraulic load frame with 5.5 kip (25 kN) load cell capacity. The tests were conducted at the RTA condition and an infrared thermometer was used to monitor coupon temperature. No heating of coupons was observed. Figure 6 shows the ILS S-N curves for the 250° F curing carbon composites.

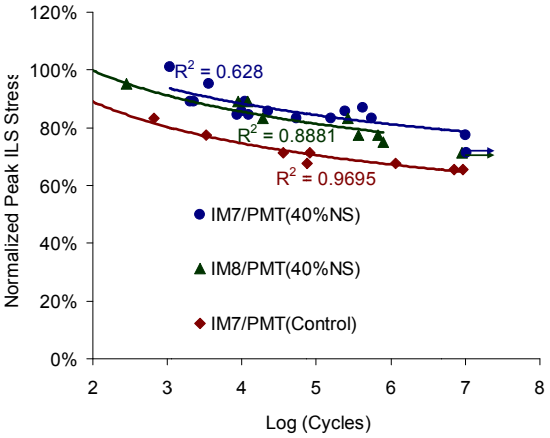

Figure 6. ILS S-N curves for 250° F curing carbon composites with and without nanosilica

To generate the ILS S-N curves, the ratio of the peak shear stress and the baseline (IM7/381) ILS strength was plotted against the log of the number of cycles to failure. The failure was defined as the onset of a visually-detectable delamination. Power law S-N curves were determined based on linear regression. The 10 million cycle runouts, indicated with arrows in Figure 8, were not included in the trend approximation.

ILS fatigue characteristics for the 250° F curing carbon PMT prepreg composites exhibit similar improvement as the ILS strength properties. The subject of measuring true interlaminar material properties is still evolving and a true ILS S-N curve is not yet available for IM7/381. Based on the trend information, the PMT

prepreg composites with 40% nanosilica show more than a factor of ten increase in fatigue life compared to the control laminate. The ILS fatigue data for IM7/PMT(40%NS) show larger scatter compared to the other composites. About half of the IM7/PMT(40%NS) SBS fatigue coupons were mistakenly tested with a 2-in diameter loading nose instead of the 4-in diameter. Compression damage was detected in some of the carbon SBS coupons fatigue tested with the 2-in diameter loading nose.

The 250° F curing S2-glass prepreg composites with nanosilica and the appropriate fiber sizing also show significant improvement in the ILS characteristics. Figure 7 shows the ILS modulus and strength test data normalized to the S2/E773 baseline composite with a 10.0 ksi (COV 2.19%) ILS strength and a 0.609 Msi (COV 1.98%) ILS modulus.

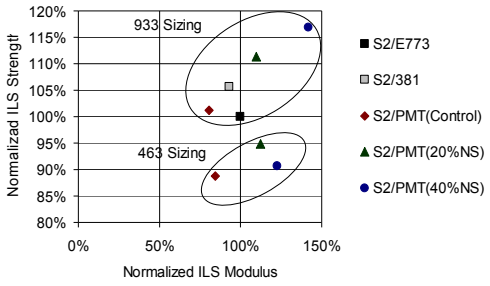

Figure 7. ILS strength and modulus results for 250° F curing S2-glass composites

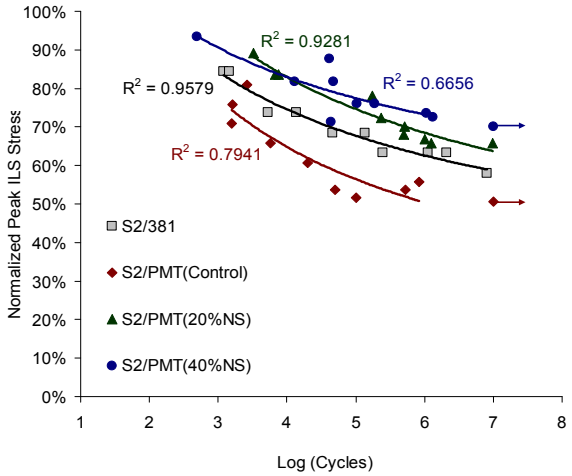

Figure 8. ILS S-N curves for 250° F curing S2-glass composites with and without nanosilica

Figure 8 shows the ILS fatigue data with the peak stress values normalized to the ILS strength of the S2/E773 composite. S2/PMT had 933 sizing based on the best ILS strength behavior. ILS fatigue data for the S2/381 composite slightly outperformed S2/E773 (not included in the figure).

Similar to the carbon ILS fatigue data, the S-N curves were generated based on constant load amplitude unidirectional SBS fatigue tests run at 0.1 load ratio and 10 Hz frequency. The loading nose diameter was two inches. All SBS coupons exhibited shear delamination failure. The tests were conducted at the RTA condition and the infrared thermometer was used to monitor coupon lateral surface temperature. No appreciable increase of the surface temperature was detected. Figure 8 shows that nanosilica improves ILS fatigue performance of 250° F curing S2-glass PMT prepreg composites with 933 sizing compared to the legacy system.

The final sets of unidirectional SBS test data represent ILS material properties of 350°F curing carbon/epoxy composite systems. Figure 9 shows the ILS modulus and strength test data normalized to the IM7/8552 baseline composite with a 16.0 ksi (COV 2.70%) ILS strength and a 0.742 Msi (COV 2.26%) ILS modulus. The IM7/8552 SBS coupons were not manufactured (cured and machined) by the same laboratory that manufactured the IM7/PMT laminates.

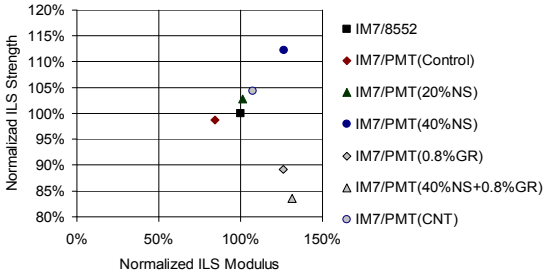

Figure 9. ILS strength and modulus results for 350° F curing carbon composites

The IM7/PMT prepreg composite with 40% nanosilica shows a 12% higher ILS strength compared to the control material and a 14% higher ILS strength compared to the IM7/8552 system. It is worth noting that IM7/PMT(40%NS) also outperformed the prepreg composites with CNT and graphene. In fact, adding only 0.8% graphene to the resin with 40% nanosilica reduced the ILT strength by 26% compared to the single 40% nanosilica in the resin. ILS strength values for the composites with 0.8% graphene only and with 0.8% graphene and 40% nanosilica were too low to support their fatigue performance evaluation.

Figure 10 compares ILS fatigue data for 350°F curing carbon composites selected based on their ILS strength behavior. Constant load amplitude unidirectional SBS fatigue tests were run at 0.1 load ratio and 10 Hz frequency. The peak stress values were normalized with respect to the mean ILS strength value for the IM7/8552 composite to plot the S-N data. All SBS coupons exhibited shear delamination failure.

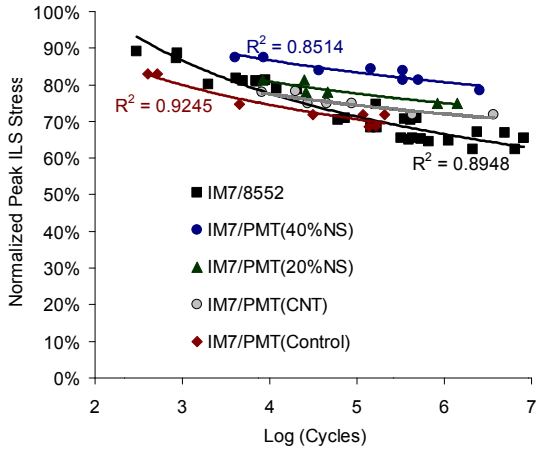

Figure 10. ILS S-N curves for 350° F curing carbon composites with and without nanofillers

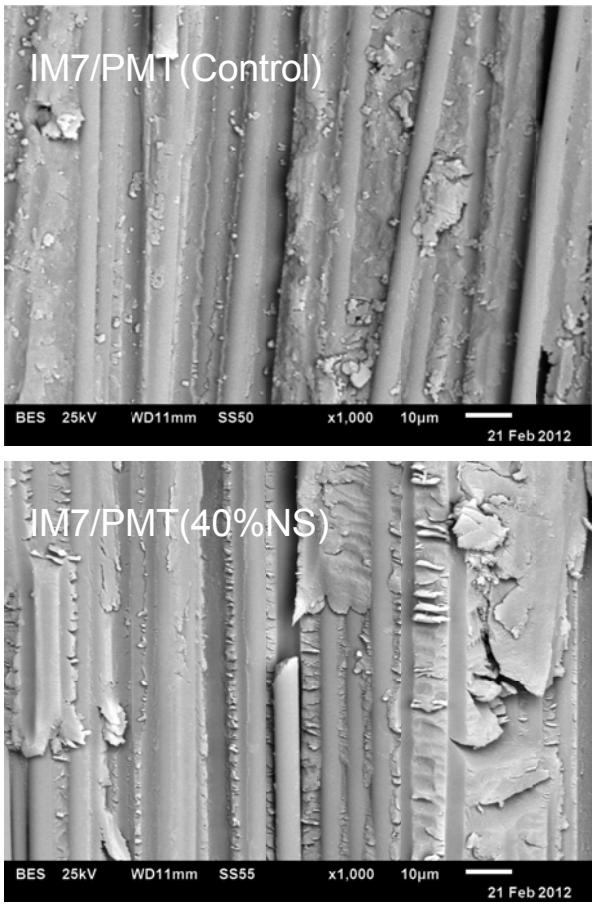

Figure 11. SEM images of ILS failure surfaces of 350°F curing carbon prepreg composites (1,000 x)

IM7/PMT(40%NS) showed significant improvement in ILS fatigue performance compared to the control material and the legacy system.

Figures 11 and 12 compare SEM images of ILS fatigue failure surfaces of carbon composites with and without nanosilica.

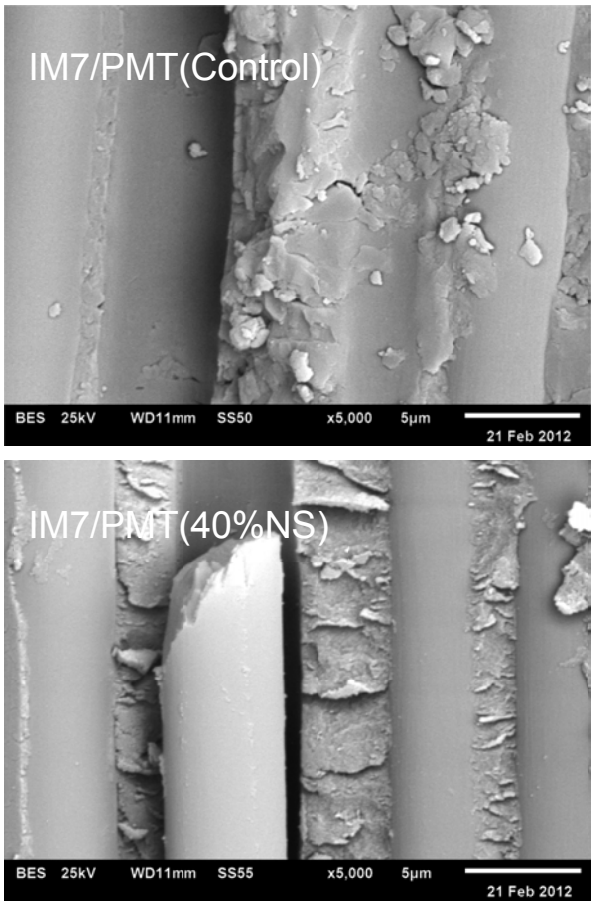

Figure 12. SEM images of ILS failure surfaces of 350°F curing carbon prepreg composites (5,000 x)

The nanosilica-loaded resin shows a rougher failure surface (i.e., shear hackles) compared to the resin without nanosilica, confirming a different shear failure mechanism.

5. INTERLAMINAR TENSION (ILT)

ASTM D 6415 curved-beam (CB) test method [18] was utilized in this work for measuring ILT strength. Samples of five to six 0.25 – 0.26 in. thick and 1 in. wide unidirectional CB coupons tested per ASTM D 6415 specifications to determine ILT strength. A CB laminate was laid on a male tool (an angle bracket), and a matching female tool (another angle-bracket) was placed on top of the laminate and cured in an autoclave. The outer corner of the male bracket had approximately 0.25 in. radius and the inner corner of the female bracket had approximately 0.5 in. radius. These two brackets formed the inner and outer surfaces and corners of the curved-beam laminate.

After the laminate was cured, it was cut with a diamond saw into CB coupons.

Figure 13 shows the ASTM D6415 CB test setup and a typical tensile delamination failure. All static specimens were tested at the standard 0.05 in/min displacement rate, and exhibited similar behavior. The failure mode was tensile delamination starting in the CB specimen radius area, typically at about two thirds of the thickness inward from the outer radius of the bend, corresponding to the maximum ILT stress location, and quickly propagating through the flanges. The DIC technique was used in conjunction with the ASTM D6415 stress calculation to determine ILT modulus [12].

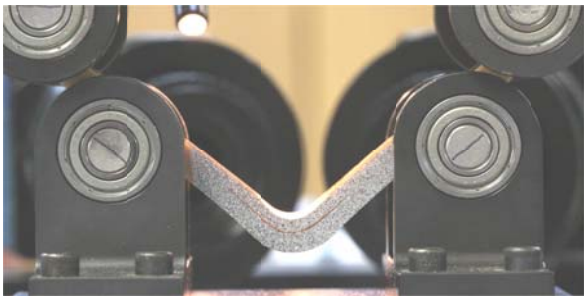

Figure 13. ASTM D 6415 test setup and tensile delamination failure of a unidirectional CB specimen

Figure 14 shows normalized CB strength and ILT modulus data for the 250° F curing carbon composites, normalized to a 6.5 ksi (COV 15.1%) CB strength and a 1.77 ksi (COV 4.15%) ILT modulus of the baseline IM7/381 composite. The 40% nanosilica-loaded prepreg composites show significant improvement in the mean CB strength over the control prepreg, in this case, as high as 62%.

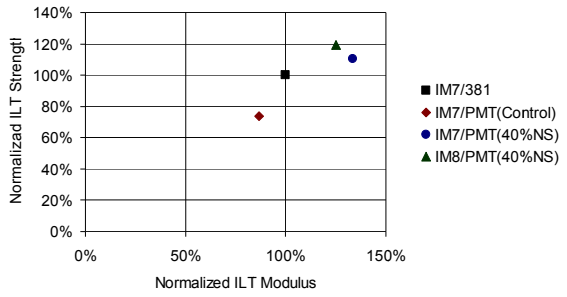

Figure 14. ILT strength and modulus results for 250° F curing carbon composites

However, the coefficient of variation (COV) of the CB strength data was much higher compared to the COV of the fiber compression and the ILS properties. The COV was 17.6% for IM7/PMT(Control); 8.41% for IM7/PMT(40%NS); and 4.31% for IM8/PMT(40%NS) prepreg composites. The COV of the ILT modulus data was 6.66% for IM7/PMT(Control); 5.57% for IM7/PMT(40%NS); and 4.0% for IM8/PMT(40%NS) material systems.

Although the mean ILT strength improvement for the nanosilica-loaded carbon composites over the baseline IM7/381 is 10% for the IM7/PMT(40%NS) and 20% for the IM8/PMT(40%NS), large scatter in the CB strength data for the baseline and the control prepreg composites is worth noting. The large scatter is consistent with recent assessment of the effects of porosity detects in IM7/8552 (350° F curing prepreg composite) unidirectional CB test specimens. Much larger samples of the IM7/8552 CB specimens were tested and the COV in the ASTM D 6415 CB strength was as high as 26.5% [12]. Large scatter was also encountered in the S-N fatigue data generated based on the ASTM D 6415 ILT stress approximation [13].

References [12, 13] showed that such a large scatter in the IM7/8552 CB test results was due to porosity defects. Although the porosity volume content in the IM7/8552 CB articles was extremely low (less than 0.12%), small individual voids present in the radius area reduced ILT strength and caused large scatter. A method for accurate ILT strength calculation based on three-dimensional computed tomography measurement of the critical defects coupled with finite element stress analysis was developed [12, 13]. The new method was able to capture the effects of porosity defects in the IM7/8552 CB specimens on their ILT strength and fatigue behavior. The ILT strength corresponding to the pristine (porosity-free) condition was remarkably higher (62%) compared to the ASTM D 6415 AVG CB strength approximation for IM7/8552 specimens. It has been determined that scatter in the ASTM D 6415 CB strength approximation reflects the manufacturing quality to produce the CB laminates as much as ILT material properties.

It is worth noting that although scatter in the ASTM D 6415 CB strength of the IM7/PMT(40%NS) and IM8/PMT(40%NS) was relatively low, larger sample sizes than five to six coupons tested in this work are required for a reliable assessment of the susceptibility to manufacturing defects including porosity.

Figure 15 shows CB fatigue data for 250°F curing carbon composites. Constant load amplitude unidirectional CB fatigue tests were run at 0.1 load ratio and 5 Hz frequency. The peak ASTM D 6415 approximate stress values were normalized with respect to the mean CB strength value for the IM7/381 composite to plot the S-N data. All CB fatigue coupons exhibited tensile delamination failure.

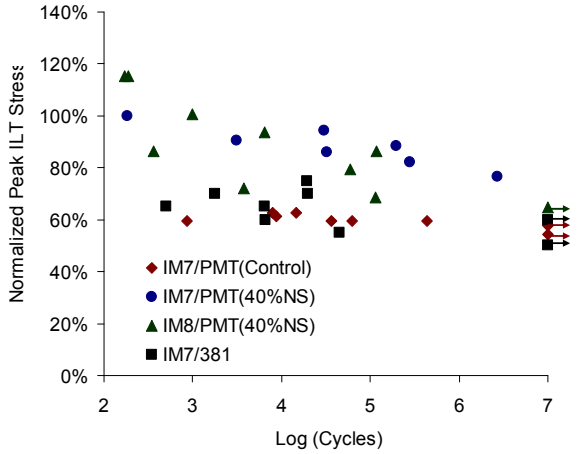

Figure 15. ILT fatigue data for 250° F curing carbon composites with and without nanosilica

Large scatter in the ILT fatigue test data is evident. IM7/PMT(Control) and the IM7/381 CB coupons had similar ASTM D 6415 stress values at 1,000 cycles to failure and 10,000,000 cycle runouts. It does not necessarily mean that S-N curve is "flat" as a higher stress level might result in the same trend if large number of coupons is tested. IM8/PMT(40%NS) also had similar stress levels at much different lifetimes, from thousands of cycles to a runout in some cases. Due to limited recourses, the CB fatigue sample sizes were 10 coupons – too small for any reliable assessment of the S-N fatigue curves. And scatter in the CB fatigue data for glass composites was even worse than for the carbon composites characterized in this work.

A more thorough follow up assessment of the manufacturing defects and their effects on the ILT strength and fatigue performance is required as suggested in [12, 13]. Fidelity of the non-destructive inspection needed to characterize the critical defects becomes extremely important. The susceptibility of the CB radius area to delamination limits the nondestructive inspections to the radius area and makes such specimens strong candidates to study the effects of manufacturing defects.

6. CONCLUDING REMARKS

This work shows that incorporating nano-sized silica reinforcement in the matrix may improve the well known weaknesses of carbon-fiber and glass-fiber thermoset-matrix prepreg composites, including compressive strength in the fiber direction as well as interlaminar strength and fatigue performance characteristics. The implementation of such advanced materials technology in rotorcraft has been limited by conflicting information in the literature, the lack of reliable material property data, and unproven repeatability and manufacturability at the structural scale. A need exists to collaboratively develop a knowledge base that characterizes advanced material technologies in conjunction with the manufacturing methods employed to process and post-process the advanced materials, establish process controls, and finally demonstrate the feasibility of the advanced materials, with the emphasis on fatigue life and life-cycle costs, versus legacy composite materials for rotorcraft applications.

Test results indicate that prepreg composites with 40% nanosilica weight content in the matrix demonstrate improved fiber-direction compressive strength and the interlaminar strength and fatigue performance, and maintain comparable density and lower resin viscosity compared to the legacy systems. As high as 45% improvement in compressive strength; 20% improvement in the interlaminar strength and more than a factor of 10 increase in fatigue life are demonstrated. Functionalized nanosilica particles are cost-effective and well-integrated in the resins used in this work. Small diameter (100 nm) of the nanosilica particles, compared to the fibers, enables uniform dispersion in the composite. On the other hand, micron-scale length of CNT and in-plane dimensions of graphene platelets cause filtration of such fillers by the fibers, and result in poor interlaminar performance of prepreg composite material systems.

Physical mechanisms governing the improvement in matrix-dominated performance must be further investigated. For example, nanosilica increases ILS and ILT stiffness of the composite system. It is expected that increased matrix-dominated stiffness provides better support to the fibers and therefore it might improve compressive strength in the fiber direction. But matrix stiffness is far from being the only characteristic driving fiber compression strength. Bond of the fibers to the resin and the nanosilica to the resin must also be strong. Appropriate sizing is critical to enabling good chemical bond of the fibers to the resin in the glass-fiber prepreg composites. Also, the SEM assessment showing a rougher ILS failure surface in the nanosilica-loaded resin compared to the resin without nanosilica, confirms different shear failure mechanism yet to be understood. Better understanding of the failure mechanisms is required for engineering optimum material reinforcement.

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REFERENCES

[1] Hexcel Corporation. 2012. HexPly 8552 Epoxy Matrix, Product Data, available online at http://www.hexcel.com/Resources/DataSheets/Prepreg-Data-Sheets/8552_us.pdf, last accessed on July 30, 2013.

[2] Cytec. 2012. CYCOM 381 Epoxy Prepreg, Technical Data Sheet, available online at http://www.cemselectorguide.com/pdf/CYCOM_381_032012.pdf, last accessed on July 30, 2013.

[3] Cytec. 2011. CYCOM E773 Epoxy Prepreg, Technical Data Sheet, available online at http://www.cemselectorguide.com/pdf/CYCOM_E773_032112.pdf, last accessed on July 30, 2013.

[4] Hackett, S.C., Nelson, J.M., Hine, A.M., Sedgwick, P., Lowe, R.H., Goetz, D.P., Schultz, W.J. 2010. The Effect of Nanosilica Concentration on the Enhancement of Epoxy Matrix Resins for Prepreg Composites, SAMPE, available online at http://multimedia.3m.com/mws/mediawebserver?mwsId=tttttvW9lEgUmy7VpzAVpy7_2XW62EW9iXut2Xut2tttttt--&fn=Prepreg_WhitePaper704.pdf, last accessed on July 30, 2013.

[5] Hackett, S.C., Nelson, J.M., Hine, A.M., Sedgwick, P., Lowe, R.H., Goetz, D.P., Schultz, W.J. 2010. Improved Carbon Fiber Composite Compression Strength and Shear Stiffness through Matrix Modification with Nanosilica, *American Society for Composites Twenty-Fifth Technical Conference*, 2010.

[6] American Society for Testing and Materials. 2006. Standard Test Method for Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates. ASTM Standard D 2344/D 2344M, ASTM International.

[7] Kinloch, A.J., and R.J. Young. 1983. *Fracture Behaviour of Polymers*. Elsevier Applied Science Publishers Ltd.

[8] Makeev, A., He, Y., Carpentier, P., Shonkwiler, B. 2012. A Method for Measurement of Multiple Constitutive Properties for Composite Materials. *Composites: Part A*, 43 (12): 2199–2210.

[9] He, Y., Makeev, A., Shonkwiler, B. 2012. Characterization of Nonlinear Shear Properties for Composite Materials Using Digital Image Correlation and Finite Element Analysis, *Composites Science and Technology*, 73 (2012): 64–71.

[10] Makeev, A. 2013. Interlaminar Shear Fatigue Behavior of Glass/Epoxy and Carbon/Epoxy Composites, *Composites Science and Technology*, 80 (2013): 93–100.

[11] Makeev, A., He, Y., Short-Beam Shear Method for Assessment of Stress-Strain Curves for Fiber-Reinforced Polymer-Matrix Composite Materials, Accepted for Publication, *Strain*, 2013.

[12] Makeev, A., Nikishkov, Y., Seon, G., Armanios, E. 2013. Methods for Assessing Interlaminar Tensile Properties in Composite Materials, *Proceedings of American Society for Composites 28th Technical Conference*, University Park, Pennsylvania.

[13] Makeev, A., Nikishkov, Y., Seon, G., Armanios, E. 2013. Effects of Defects of Interlaminar Performance of Composites, *Proceedings of the 39th European Rotorcraft Forum*, Moscow, Russia.

[14] SACMA SRM 1R-94, "Test Method for Compressive Properties of Orientated Fiber-Resin Composites," Suppliers of Advanced Composite Materials Association, Arlington, VA, 1994.

[15] ASTM D6641-09, Standard Test Method for Compressive Properties of Polymer Matrix Composite Materials Using a Combined Loading Compression (CLC) Test Fixture, ASTM International, West Conshohocken, PA, 2009.

[16] Jones, R.M., *Mechanics of Composite Materials*, 2nd Edition, Taylor & Francis, New York, NY, 1998.

[17] Adams, D. F., and Welsh, J. S. 1997. The Wyoming Combined Loading Compression (CLC) Test Method, *Journal of Composites Technology and Research*, 19 (3): 123-133.

[18] American Society for Testing and Materials. 2006. Standard Test Method for Measuring the Curved Beam Strength of a Fiber-Reinforced Polymer-Matrix Composite. ASTM Standard D 6415/D 6415M, ASTM International.