

ACTIVE COMPOSITE RIGIDIZATION USING TEMPERATURE-CONTROLLED RESISTIVE HEATING

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ABSTRACT

An active approach for initiating rigidization in carbon-fiber reinforced polymer (CFRP) composites unites electrical resistivity to mechanical stiffening. Temperature control is implemented in efforts to reduce the curing time of the composite and to increase control of the curing process. Specifically, proportional-integral (PI) feedback control of internal resistive (Joule) heating establishes an electrically-controlled, thermally-activated material. Precise temperature tracking (less than 1°C error at steady state) was achieved for controlled sample heating tests. The rigidization of these materials was quantified and compared for different curing profiles by measuring the increase in bending stiffness as well as verifying resin cure completion with DSC. Small samples of carbon-fiber tow (Toho Besfight G40-800 (12k)) coated with toughened epoxy Unyte powder resin (Hydrosize Technologies) were successfully rigidized (15 – 20 times stiffer than the flexible, uncured material) and fully-cured through a controlled heating routine in only 24 minutes and required less than 0.1 W-hr/cm of electrical energy. Experimental studies on how the prescribed curing profiles (including curing temperature and time) affect matrix consolidation and resin curing were performed and methods for reducing the curing time and energy were investigated.

INTRODUCTION

The use of inflatable and rigidizable structures in solar arrays and other space structures has the potential to drastically reduce the weight, volume, and cost of payloads. Inflatable components consist of ultra-lightweight, flexible materials that enable compact packaging prior to launch. They require techniques for controlling structural shape and stiffness once rigidized on orbit. In order to ensure on-orbit survivability, inflatable structure materials must be impervious to the environmental conditions in space—such as ionizing radiation, UV and particle radiation, atomic oxygen, and possible impacts from space debris and meteoroids.¹ Further, they must also provide stable operation over a useful storage and mission life.

Rigidizable materials and methods for causing structural stiffening of inflatable space structures

have been addressed in many ways.²⁻⁴ Thermal curing, passive cooling, UV curing, strain-hardening, inflation gas reaction, foam inflation, and solvent evaporation are examples of both passive and active stiffening. In general, three types of materials are typically used for rigidization methods: Aluminum laminates, constructed from layers of polymeric (i.e. Kapton or Mylar) and aluminum films, are rigidized when slight over-inflation of the laminate stresses the aluminum layer to its work-hardening limit.^{3, 5, 6} Plastic deformation in the aluminum eliminates wrinkles in the laminate and increases its mechanical properties. Thermoplastic materials soften when heated above their glass transition temperatures and harden when cooled, allowing them to be used in passive cooling rigidization.⁷ In contrast, a thermoset is “a polymer that can be caused to undergo cross-linking to produce a network polymer.”⁸ When heated above its cure onset temperature, cross-linking (curing) results in a networked structure that has higher rigidity, dimensional stability, and resistance to heat and chemicals.

Allowing the material to passively rigidize in the space environment does have its advantages. These techniques require little input, eliminating complex control strategies and bulky power supplies. On the other hand, because these methods initiate by exposure to a required environment, minimal input can translate into minimal control. Structures rigidized passively can also exhibit weak spots caused from uneven consolidation and may require many hours to achieve complete rigidization.² This deficiency leaves the structure in a period of vulnerability, all the while requiring a supply inflation pressure.

Active methods, such as using embedded resistive heaters,² and passive techniques, for example UV solar curing,^{9, 10} can be used to trigger the input (heat)-output (cured, consolidated polymer matrix) relationship inherent in thermosets. Researchers at ILC Dover used embedded resistive heating elements to cause material consolidation on a thermosetting resin that cured at 120°C for 45 minutes.² Recent work by Naskar and Edie,¹¹ focused on the active consolidation of a carbon-fiber tow coated in ULTEM (GE) resin. Instead of using embedded heating elements, resistive (Joule) heating was performed by passing electric current through the re-

sistive/conductive carbon fibers in order to heat the adjacent polymer resin. Their work validated internal resistive heating by showing that the ULTEM resin, when heated to 380°C, underwent rigidization. However, their resistive heating process lacked the ability to precisely control material temperature and the power requirements were excessive (300W) due to the large cure temperature required.

This study investigates the use of internal resistive heating to induce matrix consolidation and curing in thermoset-coated carbon fiber tows for the application of rigidizing flexible, inflatable spacecraft. Feedback-temperature control is implemented to provide control over the consolidation process such that the rigidized composites can be evaluated with respect to heating parameters such as curing temperature and time. The rigidization was quantified in terms of the increase in composite bending stiffness as well the completion of the resin cure. Methods for shortening the rigidization process and reducing the electrical energy consumed are traced to curing profile parameters.

MATERIALS

The composite material used for this study is a PAN-based carbon-fiber tow coated with a thermosetting polymer resin. Toho Besfight G40-800 (12k), a high tensile strength tow, provides the underlying tensile strength of the composite and is the medium to which the polymer matrix adheres. The adjacent thermoset resin forms cross-linking bonds (high-branched polymer networks) when the polymer is heated to a temperature above its cure onset temperature. Using the inherent electrical resistivity present in the carbon-fiber tow, electric current passing through the material results in Joule heating, which increases the temperature of the composite. When the temperature reaches the cure onset temperature, the thermoset polymer forms a highly-branched network of cross-linking covalent bonds.⁸ Embedding the carbon fibers in a more compliant, yet tougher resin matrix allows for transverse loads imparted onto the composite element to be transmitted through shear stress along the length of the fibers.¹² By controlling when this strengthening occurs and in prescribing how long it takes, internal resistive heating actively induces matrix consolidation for rigidization purposes.

Two thermosetting resins, U-Nyte Set 201A and 201B epoxy binders, are employed for consolidation testing via resistive heating. Both are novel thermosetting resins developed by Hydrosize, Inc. and exhibit low cure-onset temperatures in the range

from 100°C to 150°C. These materials feature high glass transition temperatures, thermal stability, high cohesive and adhesive properties, and solvent resistance.¹³ The first resin, U-Nyte Set 201A contains 97.2 wt% bisphenol A toughened epoxy, 2.6 wt% dicyandiamide, and 0.2 wt% Amicure AMI-2 (a cure accelerator). U-Nyte Set 201A lacks the Amicure curing agent, which postpones the onset of curing, and was developed in order to improve rheological properties (i.e. lower viscosity in the melt) for better processing. Recall that work by researchers at Clemson¹¹ used ULTEM poly(etherimide) (PEI) thermosetting resin developed at GE Plastics. Comparatively, ULTEM resin offers many of the same advantages (high T_g , excellent thermal stability, and chemical resistance¹⁴) as the U-Nyte Set but requires a much higher curing temperature (380°C). Since rigidization through internal resistive heating is an active technique, power is required to induce the material consolidation. Higher cure temperatures demand larger amounts of power and possibly longer curing times.

Resin Characterization

Differential scanning calorimetry was performed in the Department of Chemistry at Virginia Tech to experimentally measure the glass transition and cure temperatures for each U-Nyte Set resin. In DSC, the heat released (exothermic) or absorbed (endothermic) by a material is measured as the sample is heated through a defined temperature range. This type of thermal analysis provided a method for verifying the curing behavior of the U-Nyte Set 201B as stated by Hydrosize and also obtaining similar data for the second (U-Nyte Set 201A) resin. More importantly, successful rigidization curing profiles can be designed from knowledge of the thermal behavior of each resin.

The thermal analysis obtained through DSC provides information about key thermal events in the heating of the resins. Though similar in shape, the temperatures at which melting and especially curing are different. First, the U-Nyte Set 201B resin experiences melting as the temperature increases from 70-90°C, with an endothermic peak located at 75°C. Cure onset for this material occurs at 100°C and the curing exothermic peaks at 150°C. The U-Nyte Set 201A resin, which lacks the additional curing agent (Amicure), experienced a much later cure onset temperature of 167°C. Separate rheology tests on this resin indicate that with an increased temperature window prior to cure onset, a minimum viscosity of 14 Pa·s was measured at 170°C. Compared with the U-Nyte Set 201B resin, which did not flow well

and ultimately cured, the 201A resin trades a lower cure temperature for better rheological properties. Namely, the ability to melt, flow, and wet the fibers adequately prior to cure onset increases the strength and effectiveness of the rigidized composite.¹² In this light, it is desired to develop a resistive heating method that can be used to prescribe and maintain the necessary temperature profiles required for effective consolidation and resin curing.

Table 1. Summarized U-Nyte Set Resin Properties

Type	Temperatures (°C)			Advantages	Disadvantages
	Melting	Cure Onset	Peak Curing Exotherm		
201A	50-60	167	197	Lower melt viscosity Lower curing temperature	Higher curing temperatures Does not "flow" well in the melt
201B	60-75	100	150		

Sample Preparation

The carbon fiber tow was coated with the resin using a dry powder "prepreg" system (DPPS) developed at Virginia Tech. In this procedure, uncoated tow is pulled from a spool and run through a tow-spreader at 15 ft/sec. This pneumatic device blows a focused stream of air across the fibers causing them to "spread." The resin (in powder form) was then hand-sprinkled onto the spread fiber tow and the coated tow is then passed through a convection oven for 20 seconds at 175°C in order to better adhere the resin particles to the fibers. Lastly, the coated tow is then re-spooled for later use. It was determined that the mass fraction of the resin of the coated material ranged from 40 – 60%. The typical sample used in this study consisted of a 15 – 20 cm length of the resin-coated fiber tow.

EXPERIMENTAL METHODS

Prior to small-scale testing, multiple experiments were performed in order to establish methods for prescribing a desired heating schedule as well as analyze the consolidated material. First, Joule heating as a method for causing matrix consolidation and possible cure was evaluated with a set of simple resistive heating tests. Developing an accurate, robust heating method was crucial in being able to actively prescribe full-scale rigidization. The experimental setup used to apply closed-loop resistive heating to this material is then discussed. Next, the curing profile selection for both U-Nyte Set 201A and 201B-coated carbon fiber tows are presented and mechanical stiffness testing with additional DSC analysis are

used to evaluate samples hardened by internal resistive heating. Lastly, the increased strength of the rigidized boom is quantified.

Open-loop Resistive Heating

To verify that Joule heating could be used to cause sufficient matrix heating, a simple open-loop heating experiment was performed on a carbon fiber tow coated with U-Nyte Set 101 resin. This particular resin, unlike the U-Nyte Set 201 epoxy resins, did not contain a curing agent, making it incapable of developing a cross-linked structure. However, this test was designed to measure whether the heated samples were stiffer (due to consolidation effects only) than the original state.

For this experiment, a 16.5 cm coated sample was strung between two posts and its initial vertical deflection was marked. Then, a 149g mass was hung from the midpoint of the sample and the resulting deflection was recorded. Following resistive heating, the undeflected and deflected (using the same mass) positions were again noted. It was desired to see that the vertical deflection under load was less for stiffer, post-heated samples. Two different samples were heated in an open-loop fashion using a Xantrex XHR 300V-3.5A DC Power Supply to generate the voltage. The applied voltage (14–15V for 3 minutes) was controlled manually during the heating process and temperatures were not measured.

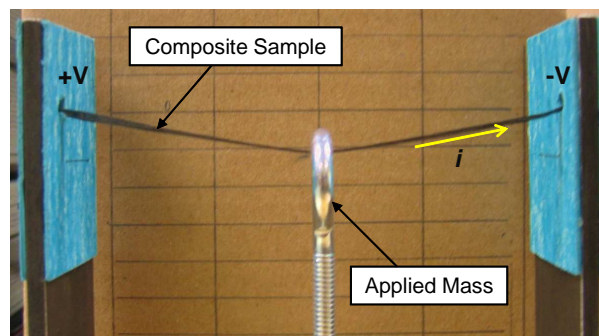


Figure 1. Experimental fixture for measuring the stiffening effect of CFRP composite using resistive heating.

Temperature-Controlled Resistive Heating

Feedback control, based on a PID control algorithm was selected to administer control over the resistive heating process. This method, which relies on comparing a measured variable (i.e. temperature) to a desired value, eliminates the need for precise system modeling and can even minimize the effects of external disturbances. It has been previously shown

that feedback temperature control allows for tailored curing schedules to be prescribed for composite consolidation.^{15, 16} Again, this study employs the use of a tuned proportional-integral (PI) controller to provide a robust and accurate heating method.

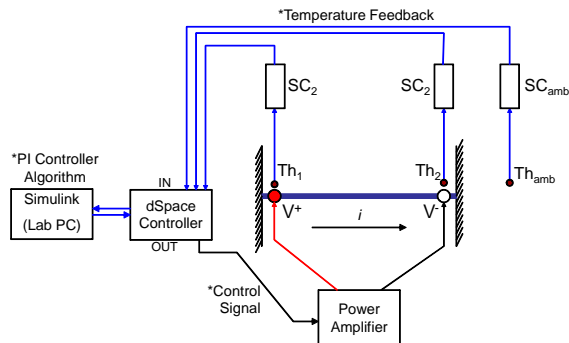


Figure 2. Block diagram of the experimental setup used for feedback temperature control during heating.

The process of establishing temperature-controlled resistive heating began by fixing a 15 – 20-cm (6 – 8-inch) long sample of resin-coated tow at end. Omega J-type (Iron-Constantan) thermocouples (36-ga.) placed at two separate locations along the sample measured temperature. An additional thermocouple was also used to record the ambient air temperature. Three signal conditioners (Omega #CCT-22-0/400C) with ranges of 0 – 400°C provided cold-junction reference points for each thermocouple and produced 0 – 10V voltage signals proportional to each measured temperature. These voltage signals were then input into dSpace and converted into temperature values in Simulink. A proportional-integral (PI) control algorithm compared the maximum of the measured temperatures (in efforts prevent overheating the sample) with the desired temperature and produced a voltage signal designed to minimize this difference. This voltage (generated in Simulink/dSpace and amplified by a Xantrex XHR 300V-3.5A DC Power Supply/Amplifier) was applied across the length of the sample, resulting in current flow through the material. The temperature change due to heating from this current signal was then measured in subsequent sampling events. The described process repeated each time a new sample temperature was taken (a sampling rate of 5 Hz was used for this study).

Curing Profiles for Effective Consolidation

The goal for rigidization is to transform the material from an initially flexible state to a rigid one. In

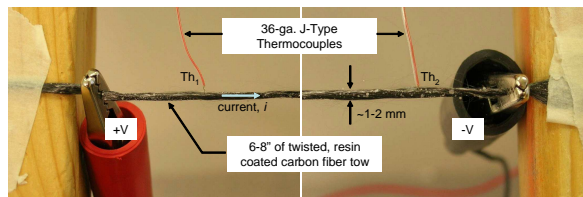


Figure 3. Samples were fixed on each end and alligator clips were used to send electric current through the material. Note that only the ends of the entire sample are shown.

effective composite rigidization, the resin particles consolidate around the reinforcing fibers creating a continuous resin matrix. Loads imparted onto cured composites are directed through the polymer and transferred to fibers along their axial direction.¹² The curing profiles were designed using the results of the thermal analysis of both resins (Table 1) and were selected so as to induce resin particle softening and consolidation prior to the gel point.¹⁶

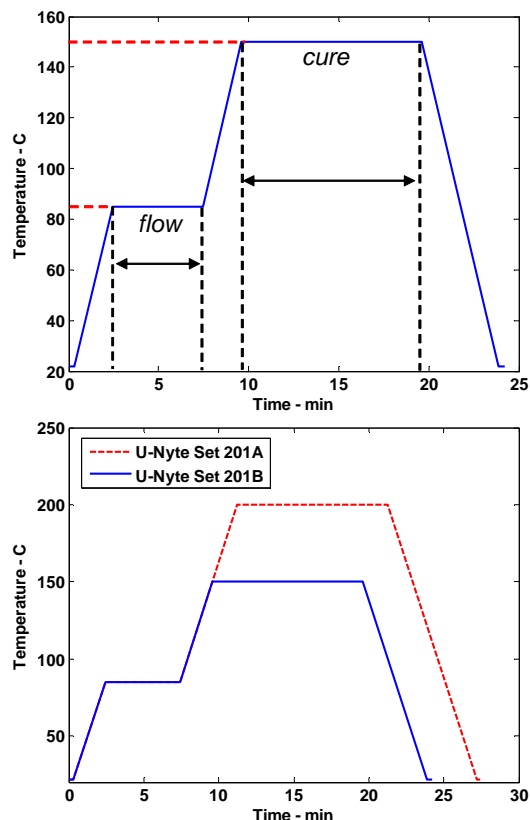


Figure 4. Typical curing profile (top) and specific profiles designed from DSC data on the U-Nyte Set resins (bottom).

As a starting point, curing temperatures of 200°C (for 201A resin) and 150°C (for 201B resin) were chosen by selecting the peak curing exotherm loca-

tions from the DSC results. Intermediate *flow* regions of 85°C for 5 minutes (located between the melting and cure onset temperatures for both resins) were also incorporated in order to facilitate resin consolidation. Previous experiments with these materials demonstrated that if the cure temperature was achieved too quickly, the resin particles would cure before consolidating. Samples cured in this manner were inherently weaker as the cured resin particles could not transfer load effectively to the reinforcing fibers. A constant heating (and cooling) rate of 30°C/min was selected for increasing (and decreasing) sample temperature through the test. A representative curing profile as well as the actual heating schedules prescribed to the two types of samples in this study are illustrated in Figure 4.

Rigidized Material Evaluation

An instrumented bending strength test fixture was designed to quantify the increase in strength caused by resistive heating. In this setup, a sample fixed at each end was deflected at its midpoint. A 100g load cell (Transducer Techniques GSO-100) measured the deflection force while a laser vibrometer (Polytec OFV 303 Sensor and OFV 3001 Controller) focused on the load cell detected the deflection (Figure 5).

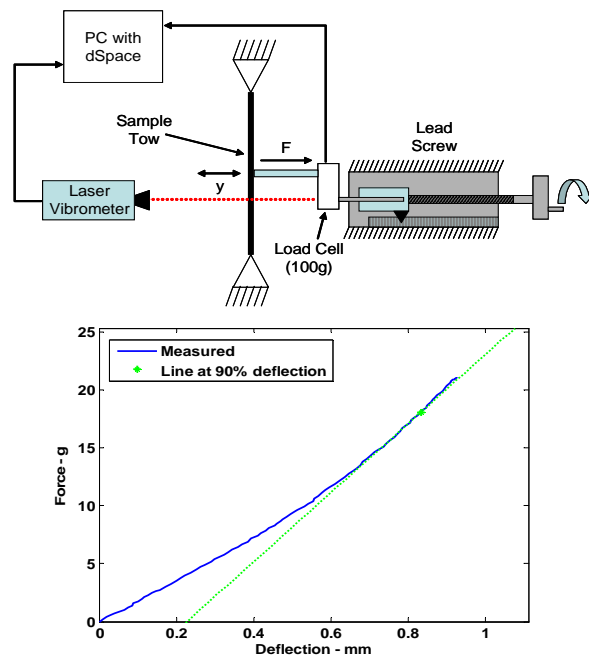


Figure 5. The test fixture for measuring sample bending strength (top) and typical force-deflection data (bottom) for determining stiffness.

The stiffness of a given sample was then measured as the instantaneous slope of the force versus deflec-

tion curve at 90% of maximum deflection. It was noticed that the force-deflection relationship was not entirely linear, possibly due to loading effects and local material deformation. The relationship did become more linear at higher deflections, and so the stiffness was measured as the slope in this region. Stress and strain were not used in this method as inconsistencies in the materials promoted imprecise calculations. Instead, slope values from force and deflection data were compared for all samples tested in this manner.

Lastly, samples hardened through resistive heating were subjected to DSC testing in order to verify that the resin cured. For fully-cured samples, the resins should exhibit a cured glass transition temperature (Hydrosize states that this occurs near 110°C) but no cure exotherm. Quantitative cure completion was not evaluated as these tests were performed on composite samples that included both fiber and resin. Without knowing how much of each component is present, the total amount of heat released during a cure exotherm can not be compared with that of initially uncured material. Instead, this DSC testing was merely used to categorize the samples as either partially or fully cured.

RESULTS

This study aimed to develop and use temperature-controlled resistive heating to actively consolidate thermoset-coated carbon fiber composites. The experimental results of the aforementioned tests are presented in order. A more-detailed discussion of the findings follows this section.

Open-loop Heating

From the first experiment described, the vertical displacement due to mass loading was measured before and after resistive heating. The displacement values were estimated with the help of a background scale placed behind the fixed samples. Incremented with a 1.27 cm vertical grid, the displacement values measured were accurate to about 0.32 cm. Prior to heating, the sample experienced a midpoint deflection of 1.59 cm under loading. This “string-like” deflection created a sharp point in the deflection profile of the sample (Figure 1). The post-heated sample deflected only 1.27 cm, a 0.32 cm decrease compared to the unheated material sample. This procedure was repeated a second time on a new sample and the post-heated sample again deflected less, marking a slight increase in the bending stiffness of the material.

Temperature-Controlled Resistive Heating

Feedback temperature control allows for a desired temperature curing profile to be prescribed to the rigidizable materials in focus. Previous work¹⁵ found that experimental tuning of the PI controller results in minimal temperature overshoot and steady-state accuracy. A representative heating cycle and tracking signal (measured material temperature) is shown.

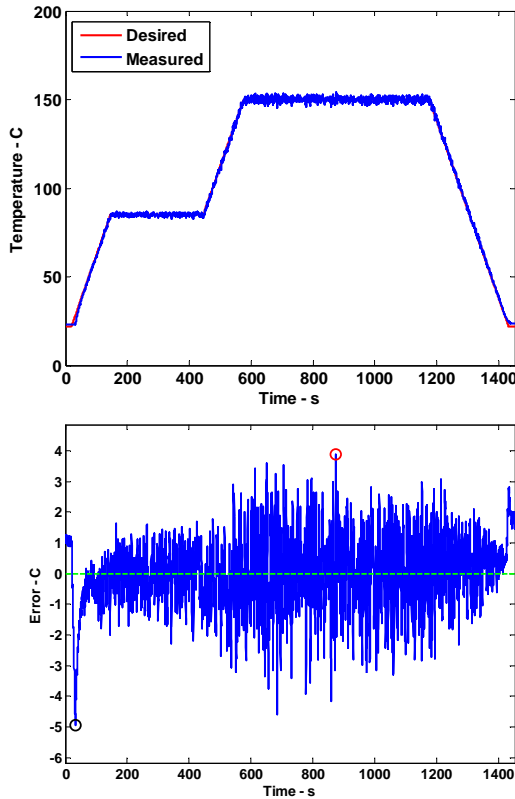


Figure 6. Feedback temperature control, using a tuned PI controller, provided accurate temperature matching to an arbitrary temperature profile.

Throughout the heating schedule in Figure 6, accurate temperature tracking (1.1°C RMS or 3.3% error) was maintained. The curing profile (temperature versus time) chosen for this case was arbitrary, though it demonstrates that feedback temperature control allows resistive heating to provide desired curing profiles of any shape. Primarily, the ability to minimize temperature overshoot ensures that this process can heat samples to a desired temperature without overstepping the mark. In cases where sub-cure onset temperatures are required, this measure prevents unwanted resin curing. Overheating the sample, and risking degradation at extreme temperatures, is also eliminated.

Prescribing Active Rigidization

This method of rigidization was first tested on carbon fiber tow samples coated with U-Nyte Set 201A and 201B, respectively. The idea was to implement curing profiles designed from previous thermal analysis (DSC) data on these resins in order to demonstrate resin curing and matrix consolidation through resistive heating. As a starting point, curing temperatures of 200°C (for 201A resin) and 150°C (for 201B resin) were chosen by selecting the peak curing exotherm locations from the DSC results. Intermediate *flow* regions of 85°C for 5 minutes (located between the melting and cure onset temperatures for both resins) were also incorporated in order to facilitate resin consolidation.

The resistive heating process produced a visible change in the appearance of the coated tow. In regions subjected to resistive heating (i.e., in the current flow path), the initially-discrete resin particles melted, consolidating around the carbon fibers. A comparison image of the pre-heated and post-heated composite material is shown.

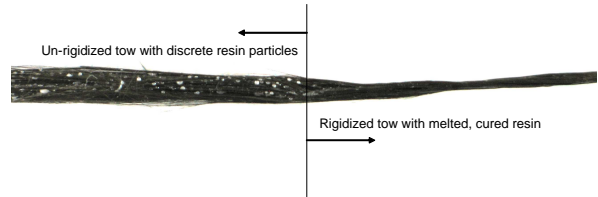


Figure 7. Incorporating a *flow* region into the temperature profile caused the U-Nyte Set resin particles to consolidate fully during their curing cycles.

Mechanical stiffness values and DSC analysis for the two resin types demonstrate that both resistive heating schedules caused matrix consolidation (Figure 7) and resin curing (Figure 8). The cured samples were 14 – 21 times stiffer than the flexible, uncured material and exhibited no additional cure exotherms during the DSC testing. The two heating routines required roughly 1.60 and 1.10 W-hr of electrical energy (during 27 and 24 minutes of heating, respectively) for the carbon tows coated with 201A and 201B resins, respectively. On a length basis, these materials require 0.08 – 0.11 W-hr/cm of rigidizable materials for the tested schedules. Peak electrical power ranged from 5 – 8 W during these tests.

DISCUSSION

In order to achieve rigidization, both consolidation and curing of the polymer matrix are required.¹⁶

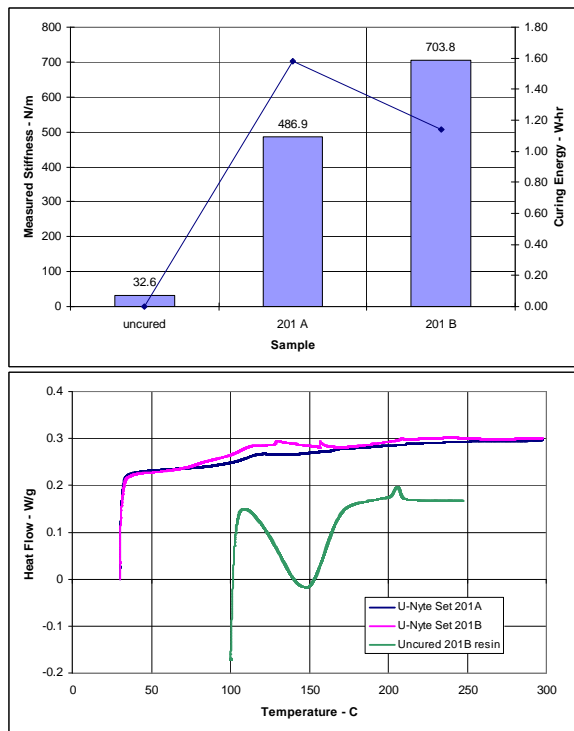


Figure 8. Measured mechanical stiffness (top) and cure completion via DSC analysis (bottom) for U-Nyte Set 201A and 201B composite samples.

The work presented here strives to actively initiate and control the rigidization of carbon fiber tows coated with a thermosetting resin.

Resistive Heating Validation

The first round of testing verifies that the heat generated through resistive heating can be used to transform discrete polymer particles into a continuous matrix phase. The effect of this transformation was seen as an increase in the bending stiffness of composite samples fixed on each end and subjected to vertical loads at their midpoints. Quantifying the increase in stiffness posed an interesting question of how to model these material specimens: Are they beams in bending or strings in tension? While both methods were explored, string theory was chosen to represent these materials, as their deflected shapes (Figure 1) lacked the continuous, cubic-like curvature required in beam theory. Specifically, string theory treats the samples as linear, elastic materials that possess no resistance to bending and whose measured vertical deflections result from stretching in the string. The results of the string theory calculations showed that polymer consolidation alone (without cross-linking) caused a 2–3 times increase in the restorative force (resistance to bending) of the material.

Table 2. Calculated restorative forces for both samples before and after heating.

Sample	Material Condition	Measured Deflection, δ_s (cm)	Deflection Angle, α ($^\circ$)	Stretched Length, L_s (cm)	Linear Strain, ϵ (cm/cm)	Calculated Tension, F		Change in F_s (%)
						N	N	
1	pre-heat	1.59	10.8	8.46	0.0181	3.89	215.06	
1	post-heat	1.27	8.7	8.40	0.0116	4.84	416.03	93.45
2	pre-heat	1.75	11.9	8.49	0.0219	3.55	162.13	
2	post-heat	1.27	8.7	8.40	0.0116	4.84	416.03	156.60

Once-heated, these materials possessed noticeable bending stiffness (especially in samples that have curable resin). The dilemma in post-processing this data was that a rigidization process is designed to convert an initially flexible, “string-like” material into a rigid, “beam-like” composite. String theory applied well to the pre-cured sample, though may not accurately depict model samples with noticeable and measurable bending stiffness. These evaluations fall short in predicting the precise amount of stiffness increase to be expected through rigidization. The lack of a curing agent in these samples also prevents the effects of cross-linking from being witnessed. Conversely, this test did successfully demonstrate that the heat generated within the fibers through Joule heating is sufficient for heating the adjacent matrix. Both samples showed an increase in stiffness over their original states due to consolidation and the disappearance of discrete resin particles during heating confirmed the polymer transformation.

Prescribed Consolidation and Curing

The temperature-controlled resistive heating tests on samples containing either U-Nyte Set 201A or 201B resin demonstrated significant stiffening and complete resin curing. Though samples coated in the U-Nyte Set 201B measured larger stiffness values, composites containing this resin are not necessarily stronger. This resin was finely ground with mortar and pestle prior to fiber coating in order to increase its melting capability before curing. A smaller particle size, which increases how well the resin adheres to the fiber tow during the pre-pegging process, and a heavier resin distribution have probably influenced the rigidity of these specific samples.

This test also illustrated that the U-Nyte Set 201A resin requires more energy to achieve a full cure. Though it benefits from better rheological (flow) properties in the melt, the lack of Amicure curing agent shifted its cure onset temperature from 100°C to 150°C. Higher curing temperatures result in more curing energy. The two resins that were tested are not equal, and their individual attributes necessitate different curing profiles. The primary strength of this study was to demonstrate that resistive heating can be used to cause full matrix consolidation and complete curing for effective composite rigidization.

Comparing to previous work using an ULTEM resin,¹¹ which required more than 340W of peak power, these samples were fully cured and rigidized with the application of only 5 – 8W of peak power and 1.10 – 1.60W-hr of total electrical energy. Though the amount of material and test configuration were not equal, the reduction in curing temperature from 380°C for the ULTEM to 150°C for the U-Nyte Set is listed as a major factor in reducing the energy supplied to the material.

Intelligent Design of a Cure Schedule

Additional work has been performed in designing the curing schedule so as to reduce the required amount of energy and shorten the total process time while still achieving substantial stiffening and complete resin curing.¹⁶ The curing schedules used for the tests in this paper were based on thermal analysis of the resins to identify key thermal events. In differential scanning calorimetry (DSC), the sample temperature is increased at a constant rate (typically 5°C/min or 10°C/min) and the heat absorbed or released by the sample is measured. As a result, this test alone does not give information on the rate of curing or total time required to achieve full curing at a given temperature. The results of the study on both U-Nyte Set 201A and 201B (Figure 8) confirmed that a dwell time at the curing temperature of 10 minutes assured complete curing. However, does this requirement change at different curing temperatures and is this the minimal amount of time required to produce fully-cured samples?

Two studies, one in which the curing temperature was varied and one where the curing time (dwell time at the curing temperature) was varied, outline the relationship between temperature and time. The results of this work,¹⁶ illustrated the requirements in temperature to achieve both consolidation and curing and demonstrated that while consolidation occurs when the polymer softens and flows at temperatures above its glass transition temperature, curing only occurs when the material is then taken to a temperature above cure onset. Further, if the polymer temperature is increased to cure onset too quickly, the particles cure without consolidating. Samples subjected to such heating routines were fully-cured according to DSC tests, but lacked the strength that is gained through successful matrix consolidation.

In Figure 9, both the amount of energy and mechanical stiffness increased with cure temperature. This trend was expected as the energy supplied should increase for higher required temperatures. Also, the stiffness increases as the polymer

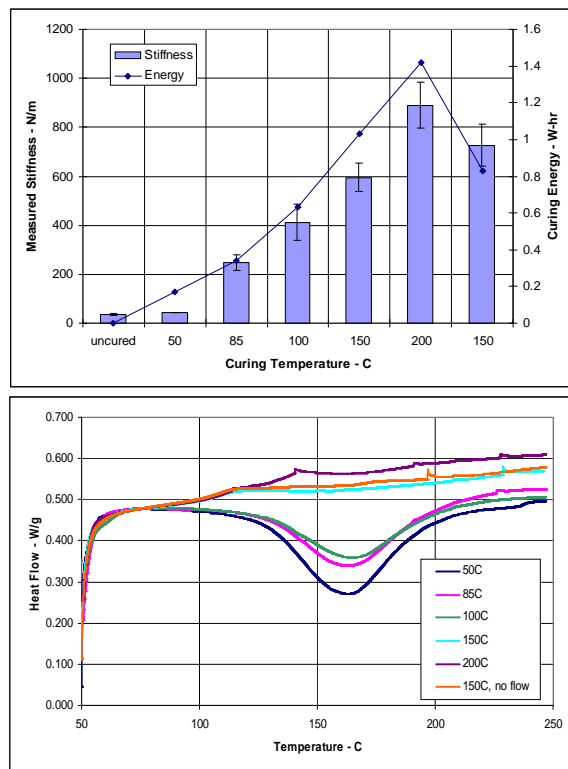


Figure 9. Stiffness and energy data (top) and DSC plots (bottom) for samples cured at various curing temperatures.

first consolidates (for temperatures above 75°C) and then begins to cure at 100°C. Samples cured for 10 minutes at temperatures lower than 150°C exhibited incomplete curing. The presence of uncured or partially-cured material in thermosetting materials (which have limited shelf-lives prior to curing⁸) after resistive heating may subject the composites to chemical and physical instabilities over time.

Curing time must also be considered when designing a curing schedule as the rate of polymerization increases with increasing temperature.⁸ The previous test demonstrated that fully-cured resin resulted from 10 minutes of curing at 150°C, while samples cured at only 100°C resulted in partial curing. By allowing the samples to cure longer at a temperature of 100°C, cure completion is expected. Conversely, a cure temperature of 150°C may permit shorter curing times and still produce complete curing. An experiment in which the curing time was decreased from 10 minutes down to 30 seconds illustrated this concept.

While samples cured for 1 minute or longer produced significant increases in stiffness, only samples that were cured for 5 minutes or longer fully cross-linked. The DSC plot demonstrates that the cure

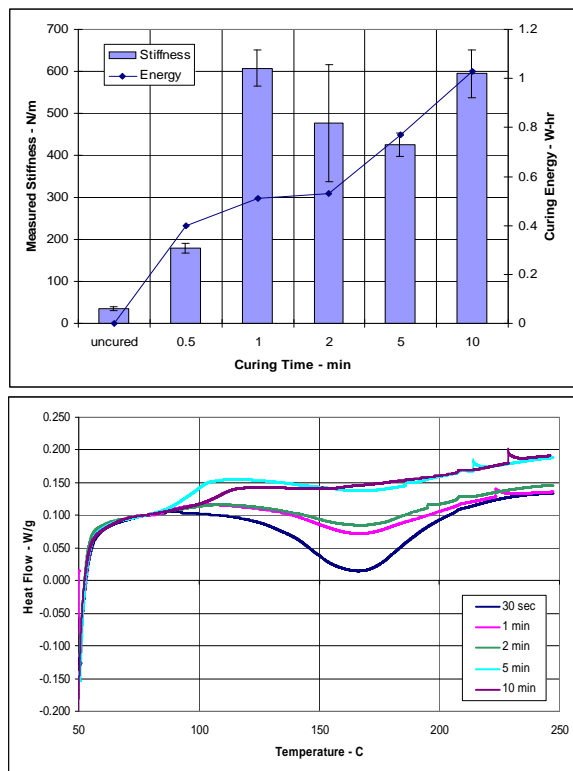


Figure 10. Measured stiffness (top) and DSC plots (bottom) for samples cured at 150°C for different lengths of time.

exotherm present in the first three samples becomes more shallow as curing time increases. Compared to the base curing profile, which included 10 minutes at 150°C, this test asserts that curing the sample for half the time (5 minutes) produces roughly the same amount of stiffening and a fully-cured resin matrix. By cutting the curing time in half, this process has been shortened and the total electrical energy was reduced from 1.0W-hr to 0.8W-hr, a 20% savings in both time and energy.

This discussion is meant to clarify that prescribing rigidization through resistive heating allows for the curing schedule to be intelligently chosen. Variations in the curing temperature as well as curing time affect both the mechanical stiffening as well as the long-term stability of the polymer composite. Further, these routines should be selected in order to minimize the cost of rigidization—both in the amount of energy to be supplied as well as the time that it takes to transform the material from a vulnerable state to one that is permanently robust.

CONCLUSION

The consolidation and curing of thermoset resin-coated carbon fiber tows was achieved through feed-

back temperature-controlled, internal resistive heating. A proportional-integral (PI) control strategy was used to monitor and correct differences between a desired curing temperature profile and a measured material temperature. The effective rigidization of carbon fiber tows coated with one of two U-Nyte Set 201 epoxy resins was shown to cause significant increases in the bending stiffness (15 – 20 times) and complete resin curing. The novelty of applying temperature control to resistive heating of CFRP materials was further complimented by the advantages of using a low-cure temperature epoxy resin such as U-Nyte Set 201B. Heating schedule variables such as temperature and time were examined experimentally for fine-tuning the curing schedule to reduce energy requirements and shorten the process time. The results of this work highlight the feasibility of using resistive heating for rigidizing inflatable space structures. However, issues of controlling temperature across large sections of CFRP material presents different challenges and would require higher electrical currents. The high energy demands likely to cause heating in a real structure and the additional equipment needed for on-orbit processing provide additional reasons to explore passive rigidization techniques.

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