# SANDWICH COMPOSITE, SYNTACTIC FOAM CORE BASED, APPLICATION FOR SPACE STRUCTURES

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## ABSTRACT

The current Solid Rocket Booster (SRB) launch vehicle has several metal based components that require a Thermal Protective System (TPS) be applied to the exterior surface to ensure its structural integrity and to protect the interior hardware from aerodynamic heating. TPS materials have distinct disadvantages associated with their use. One disadvantage to the application of TPS is that it can act as a debris source to the Space Shuttle Orbiter during flight and it also adds weight to the system without directly contributing any structural strength. One of the specific areas examined under this program was to replace a metal/TPS system with polymer based composites. A polymer matrix based sandwich composite was developed which had both structural and insulative properties to meet the high aerodynamic structural and heating load survival requirements. The SRB Nose Cap was selected as a candidate for this application. The sandwich system being qualified for this application is a carbon/epoxy outer and inner skin with a high strength-low thermal conductivity syntactic foam core.

KEY WORDS: Composites, High Temperature Mechanical Testing, Aerothermal Testing

## 1. INTRODUCTION

The current Space Shuttle Solid Rocket Booster (SRB) Nose Cap is a non-recovered, Thermal Protection System (TPS) coated, metallic structure. United Technologies USBI and Marshall Space Flight Center (MSFC) initiated a Shuttle Upgrades Project to develop a composite nose cap as a replacement article for the SRB. Composite materials offer a strength-to-weight advantage over metallic structures. Additionally, the TPS, which can be a debris hazard and adds weight without contributing strength, can be eliminated because of the thermal capabilities of a composite sandwich structure.

The CNC design is composed of a sandwich construction. Two facesheets of Hexcel's AGP370-8H/3501-6 (AS4/3501-6) graphite/epoxy encapsulate a core of 3M SC350G syntactic foam. An aluminum mesh covers the outer skin for lightning protection. A significant amount of mechanical and thermal property testing was performed on the material used in the SRB Composite Nose Cap (CNC). The testing performed was based on requirements set by the MSFC Materials, Processes, and Manufacturing Department, and the Structures, Mechanics, and Thermal Department, and USBI. The testing performed at MSFC provided A-basis allowables for stress analyses. Thermal testing provided accurate properties for modeling and aerothermal test verification.

## 2. MECHANICAL PROPERTY TESTING

Mechanical testing was performed on three lots of AS4/3501-6 and SC350G for material characterization. Allowables were generated from the test results per the Material Characterization Test Plan[1]. Testing was performed on the graphite/epoxy lamina, bare core, and the complete sandwich. The lamina level test specimens were moisture conditioned to simulate potential moisture absorption due to environmental exposure at Kennedy Space Center. Heating ramp rates were also controlled to better simulate the heating rate of the SRB CNC during ascent. The sandwich test specimens were tested with more gradual heating rates and longer hold times to insure thermal equilibrium.

**2.1 Test Specimen Conditioning** Hypalon is currently qualified for use as a moisture barrier on SRB insulation. Hypalon was initially to be used as a moisture barrier on the SRB CNC. As a verification of the effectiveness of Hypalon as a moisture barrier, a series of moisture conditioning tests were performed on the AS4/3501-6 material. The test series moisture conditioned three sets of samples: coated graphite/epoxy, bare graphite/epoxy, and Hypalon film. The specimens were conditioned at 71° C (160° F) and 93% RH. Using the rule of mixtures the weight gain of the Hypalon film was subtracted from the weight gain of the Hypalon coated specimens. This yielded the estimated weight gain of the composite underneath the Hypalon. The bare and coated composite specimens exhibited similar weight gains (Figure 1). Thus, Hypalon was determined to be ineffective as a moisture barrier on low absorptivity materials such as graphite/epoxy.

Due to the ineffectiveness of Hypalon as a moisture barrier, material properties were developed in the "wet" state. MIL-HDBK-17 and ASTM D 5229 recommend that a specimen be moisture conditioned to an equilibrium state before it is considered "saturated". Equilibrium is defined by the above standards as a change in weight less than 0.01% in a 7 day period (24 hours if diffusivity is known). There are no standard moisture conditioning methods. Data generated on 3501-6 in the MIL-HDBK-17 was conditioned for 30 days at 60° C (140° F) 95% RH. Hercules generated wet properties on AS4/3501-6 by boiling in water for 24 hours. Fiberite immersed AS4/934 in 74° C (165° F) water for 7 days. Based on the data generated on the nose cap material, none of these methods provided sufficient time to reach equilibrium.

In addition to weight gain, the glass transition temperature of the moisturized specimens was also evaluated. Unconditioned "ambient" samples had Tg values of 197° C (386° F) and 193° C (380° F) obtained from DMA and DSC thermal analysis equipment. Specimens were conditioned at 49° C (120° F), 71° C (160° F), and 82° C (180° F) at 93% RH. The Tg and weight gains of the specimens were evaluated at regular intervals. Conditioning at 82° C indicated a substantial drop in Tg during the first week of conditioning. The Tg began to plateau after the second week of conditioning (Figure 2). The Tg versus percent weight gain is plotted in Figure 3.

The absorption of water into a graphite/epoxy material follows Fick's second law. The moisture equilibrium content is primarily dependent on percent relative humidity. A change in temperature increases the diffusivity of the material, but has little effect on the equilibrium.[2] Thus, an increase in temperature at constant humidity level only decreases the time required to reach equilibrium. The diffusivity was calculated at both 49° C and 71° C for 93% RH. Using the Arhenius relationship diffusivity was estimated for 82° C (180° F) and 29° C (85° F). Now moisture gain due to six months exposure at 29° C and 93% RH (simulated beach exposure) could be estimated. The corresponding time required at 82° C to reach the same moisture level was approximated (Figure 4).

Assuming two sided diffusion on a 7 ply laminate, six month exposure at 29° C 93% RH yields a 0.7% weight gain. One-sided absorption yields 0.3% weight gain. To obtain a 0.7% weight gain at 82° C a six-ply laminate requires 5 days. Eight and ten ply laminates require conditioning for 9 and 14 days respectively. A ten day soak should result in moisture levels of 0.9%, 0.75%, and 0.6% weight gain for 6, 8, and 10 ply laminates respectively. This is above the expected moisture gain in a seven-ply laminate on the beach when exposed to one-sided absorption. Thus, a 10-day soak of the test specimens at 82° C (180° F) 93% RH should conservatively represent a six-month beach exposure. Subsequent conditioning at 82° C 93% RH indicated the estimated weight gains were reasonably accurate.

**2.2 Lamina Level Testing** Four lamina level tests were identified to establish the mechanical properties of the SRB CNC. Tensile tests determined the ultimate tensile stress, modulus, and Poisson's ratio. In-plane shear tests determined the in-plane shear stress and shear modulus. Compression tests determined the compressive stress and modulus. While, double notch shear tests determined interlaminar shear stress. Properties were obtained at 23° C (75°F), 116°C (240°F), 177°C (350°F), 249°C (480°F), and 316°C (600°F). Residual properties at 177°C were also obtained after a 316°C cycle. The 316°C properties are representative of the maximum temperature the outer skin of the nose cap will experience during flight. The 177°C properties (post 316°C cycle) are representative of the maximum load case of the nose cap during ejection and parachute deployment. These lamina mechanical properties will be used in the stress model of the sandwich structure. Additional tests were performed on sandwich samples as a verification of the model.

Elevated temperature testing on graphite/epoxy materials is typically performed with a moderate heating rate and a minimum hold of five minutes once the test specimen reaches test temperature. This hold verifies temperature equilibrium, reduces thermal gradients, and reduces the effects of thermal expansion during the test. However, the Hexcel 3501-6 epoxy begins to rapidly degrade at temperatures well above the cure temperature of 191°C (375°F). Since the nose cap only experiences a short duration temperature spike, properties were desired with minimal hold time and rapid heat rate. The flight temperature profile was therefore used as a guide for heating rates. Rapid heating was also desired for testing in the hot/wet condition, as a prolonged dwell time would facilitate moisture loss.

Following the flight thermal profile complicated testing. The rapid heating rate of 5.5°C (10°F) per second could not be obtained in a furnace. Thus, alternate heating methods were required. Two banks of quartz lamp infrared heaters were obtained for tensile (ASTM D 3039) and in-plane shear tests (ASTM D 3518). Through use of a Dimension controller different heating profiles were programmed and run. A two inch gage length can be held within 1% of the set point temperature. This system is capable of heating tensile coupons to 1090°C (2000°F) and will ultimately find future applications at MSFC. Strain was determined with high temperature extensometers and was digitally recorded in a MTS Testworks software program.

Compression and interlaminar shear testing also presented difficulties in testing when high heating rates are required. To facilitate heating, the ASTM D695 dog bone was chosen as the compression test coupon. A modified fixture was fabricated which supports both ends to prevent brooming. Nikrothal strip, a high resistance metal, was used as a contact resistance heater. High DC current was passed through nikrothal strip, which was in contact with both sides of the specimen. The current is controlled with a Hewlet Packard power supply. By controlling the current level the temperature was controlled. This heating method was also applied to the double notch shear (ASTM D 3846) test method.

Testing was also performed on the bare core. Tensile, compression, and shear tests were all performed at 23° C (75°F), 177°C (350°F), and 249°C (480°F). Core properties were used in conjunction with the graphite/epoxy lamina properties to model the sandwich properties. Subsequent sandwich tests verified the model.

**2.3 Sandwich Testing** Mechanical Testing was performed on the sandwich structure as a verification of processing as well as the stress model. The sandwich was composed of an outer facesheet of AS4/3501-6, a core of eight plys of SC350G syntactic foam, and an inner facesheet of AS4/3501-6. The inner and outer facesheets were each a seven ply laminate with an orientation of (0/90/45/0/-45/90/0). The sandwich panels were processed with the same cure conditions as the lamina. Sandwich samples were tested at room temperature and 177°C. Sandwich tests included edgewise tension and compression, open-hole tension and compression, flatwise tension, compression after impact, and double and single lap shear bearing tests.

A full-scale prototype nose cap was fabricated for process verification and structural testing. Tensile and compression specimens, as well as Hot Gas test specimens were machined from the prototype nose cap (P1). Due to the curvature of P1, mechanical test specimens were only obtained for the longitudinal direction. P1 mechanical test results were similar to flat panel test results.

# 3. THERMAL CHARACTERIZATION

The thermal characterization of the materials used in the sandwich is subdivided into three interrelated categories: thermal property testing, aerothermal testing, and computational modeling. The measured properties are used in the modeling. The aerothermal testing simulates design loads and evaluates the resilience of the sandwich panels under these loads. Three of the aerothermal panels used interstitial thermocouples and an IR measured surface temperature to calibrate the computational model.

**3.1 Thermal Property Testing** Thermal conductivity, specific heat, and density were measured for the graphite/epoxy, the syntactic core, pure epoxy, and the sandwich configuration. Emissivity and absorptivity were measured for the sandwich configuration only. The conductivity was measured by comparative rod analysis (per ASTM E 1225). The specific heat was measured by adiabatic calorimetry. And the density was measured by the gravimetric method[3]. These data were taken for three lots of materials, with two samples per lot. Specific heats were measured over a range of -18° C (0° F) to 260°C (500° F), thermal conductivities from -18° C (0° F) to 371° C (700° F), and densities at ambient temperatures. Emissivity was measured per ASTM E 408, Method A, and the absorptance per ASTM E 903-96. Radiation property data were measured for each panel prior to aerothermal testing, and were taken at ambient temperature. All thermal property data are summarized in Figures 5 and 6.

**3.2 Thermal Modeling** The model includes six layers, as shown in Figure 7. The outermost layer is pure epoxy. Moving inward, the next layer is a pure epoxy-aluminum mesh combination, followed by seven plies of graphite/epoxy, eight layers of syntactic core, another seven plies of graphite-epoxy, and a thin layer of pure epoxy. Although paint on the outer surface is in the design, it was not included in thermal modeling. Model calibration was performed on a 1-D transient computational thermal model. The time-dependent measured IR surface temperature from a calibration panel was imposed as a thermal boundary condition on the 1-D model. In addition, backside radiant and convective cooling are taken into account.

The interstitial thermocouples on the calibration panels are located in the following locations: (1) underneath the outermost graphite/epoxy ply, (2) at the interface of the outer graphite-epoxy laminate and the syntactic core, (3) at the center of the core, (4) at the interface of the core and the inner graphite-epoxy laminate, and (5) just inside of the outermost graphite-epoxy ply. These locations are also shown in Figure 7. The exact locations of each thermocouple, as well as layer thicknesses, were determined via an analysis of X-rays of the panel at various through-thickness locations. Examples of the comparison of measured with predicted temperatures are given in Figure 8. The agreement is generally with ± 11° C (20° F), indicating a successful calibration. The modeling techniques were then extended into a 3-D model of the nose cap, including the pilot and drogue parachutes.

**3.3 Aerothermal Testing** Aerothermal testing, apart from the calibration testing previously mentioned, can be separated into the following categories: delamination and general response, paint coating compatibility, capability tests, combined environments, beach exposure, and curved panel testing. The delamination and general response testing is self-explanatory, and used unpainted panels. Capability tests were run until extensive delamination occurred. Paint compatibility tests examined the adherence of the paint to the panels. Combined environment panels were exposed to a simulated lightning strike before aerothermal testing. This testing included both painted and unpainted panels. Beach exposure panels were placed at the beach at Kennedy Space Center for six months. Control panels were maintained at Marshall Space Flight Center for six months. This testing included both painted and unpainted panels. All of these panels were flat 30.48 cm x 48.26 cm (12"x19") panels. Curved panels were cuts of the spherical shell shape of the beanie or the conical shell shape of the lower part of the nose cap, with a shadow of 30.48 cm x 48.26 cm. These panels were tested bare. In general, the environment used was a simulation of the design flight environment (the stagnation point), with modifications depending on the needs of the test. Panels were tested wet and dry, and three lots of materials were used.

## 4. TEST RESULTS

All of the lamina level tests were performed within 8 hours of conditioning at 82°C (180°F) 93% RH. The samples were weighed prior to testing to verify adequate weight gains due to moisture absorption. The samples were heated as discussed above and load was applied as the sample reached the desired temperature. The moisture had little effect on the samples tested at room temperature. However, the shear and compression samples tested at elevated temperatures demonstrated a reduction in stress. The samples that followed the flight thermal profile (heated to 316°C and cooled to 177°C) demonstrated an increase in strength compared to samples tested at 177°C. This increase in strength was attributed to moisture loss in the specimen during the brief temperature spike. Table 1 summarizes the test results. The flight profile samples are represented with an asterisk (177°C *).

Core material and sandwich samples were not moisture conditioned. The sandwich tests were performed to verify modeling and fabrication and not used to develop allowables. Sandwich tests performed on flat panels and the prototype nose cap correlated well. As expected edgewise compression strength demonstrated a large decrease in strength at 177°C. Compression tests performed on material after aerothermal testing and after beach exposure exhibited the same strength as the control panels. Compression after impact was performed after impacts of 0, 14, 27, and 41 Joules (0, 10, 20, 30 ft-lbs). The residual compressive strength appears to plateau after 27 J of impact energy. The core and sandwich test results are also available in Table 1.

All of the aerothermal panels passed the acceptance criteria, except the bare beach exposure and associated control panels. These panels delaminated on the outermost ply prematurely for lots 1 and 3. The beach exposure and control panels tested the same for all three lots, indicating that the cause of the failures was either material or process related and not due to the beach exposure. The cause of the premature delamination has not been identified at this point.

Aerothermal testing with thermocouples incorporated within the test specimen was successfully demonstrated. There was good agreement between the measured and predicted temperatures on the aerothermal calibration panels (Figure 8). The thermal model calibration was successful, and the majority of the aerothermal testing was successful, lending to the feasibility of the design.

## 5. CONCLUSIONS

Material characterization for the composite nose cap was successfully completed. Advances in test capabilities were made at MSFC. Aerothermal tests were successfully performed with embedded thermocouples. These tests showed that the AS4/3501-6 material could meet the design requirements after brief exposure to high temperature. Mechanical property tests were performed following the temperature-time profile of the nose cap. The diffusivity of the AS4/3501-6 was determined so moisture gain at a given temperature and humidity could accurately be predicted. A conservative moisture conditioning procedure was performed so that the test specimens were adequately "wet". By following the temperature-time profile of the nose cap more representative test data were obtained. Upon completion of the test program, A-basis allowables were generated for stress modeling. Subsequent laminate level tests on the sandwich material provided additional data for model verification.

## 6. References


1. Composite Nose Cap Material Characterization Test Plan, 10PLN-0140, MSFC-PEC Operations, June 1999.
2. MIL-HDBK-17-1E Chapter 6.3
3. T. R. Barnett and H.S. Starrett, Thermal Conductivity, Specific Heat, and Density Evaluation of the Composite Nose Cap Materials, Report SRI-ENG-99-039-9655-I-F, Southern Research Institute, Birmingham, Alabama, October 1999.

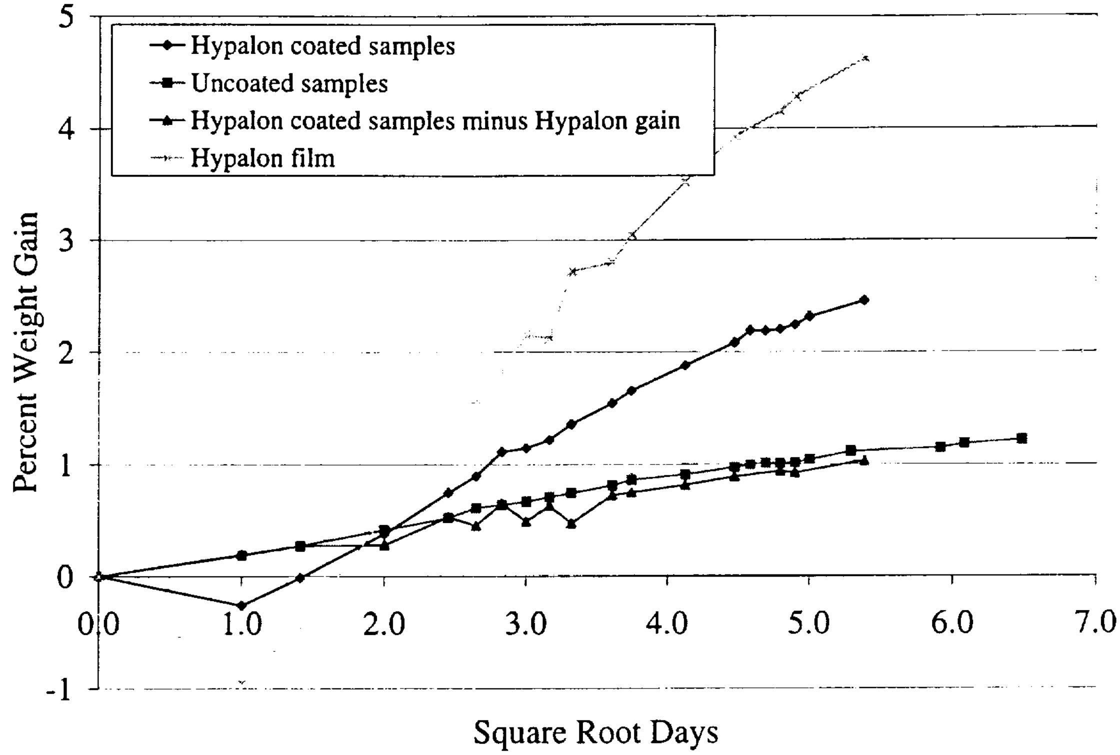


Figure 1. Moisture Gain in Coated and Uncoated AS4/3501-6 at 71°C and 93%RH

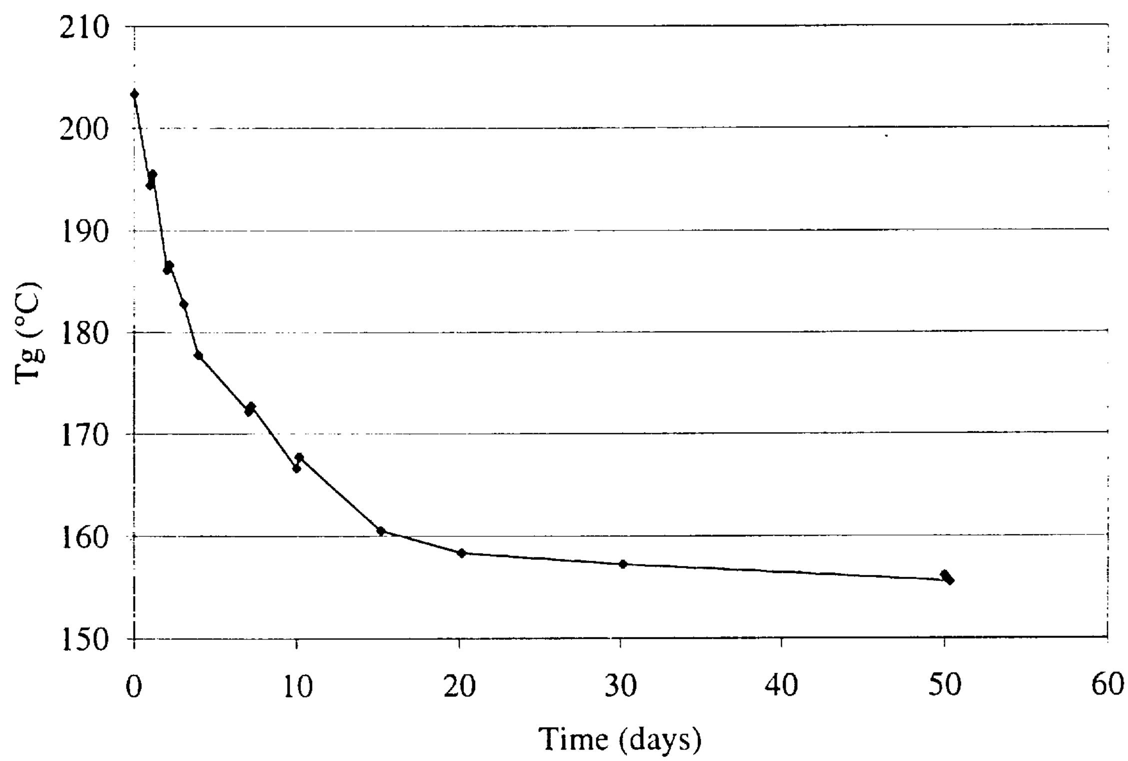


Figure 2. Glass Transition Temperature of AS4/3501-6 Versus Exposure Time at 82°C and 93%RH

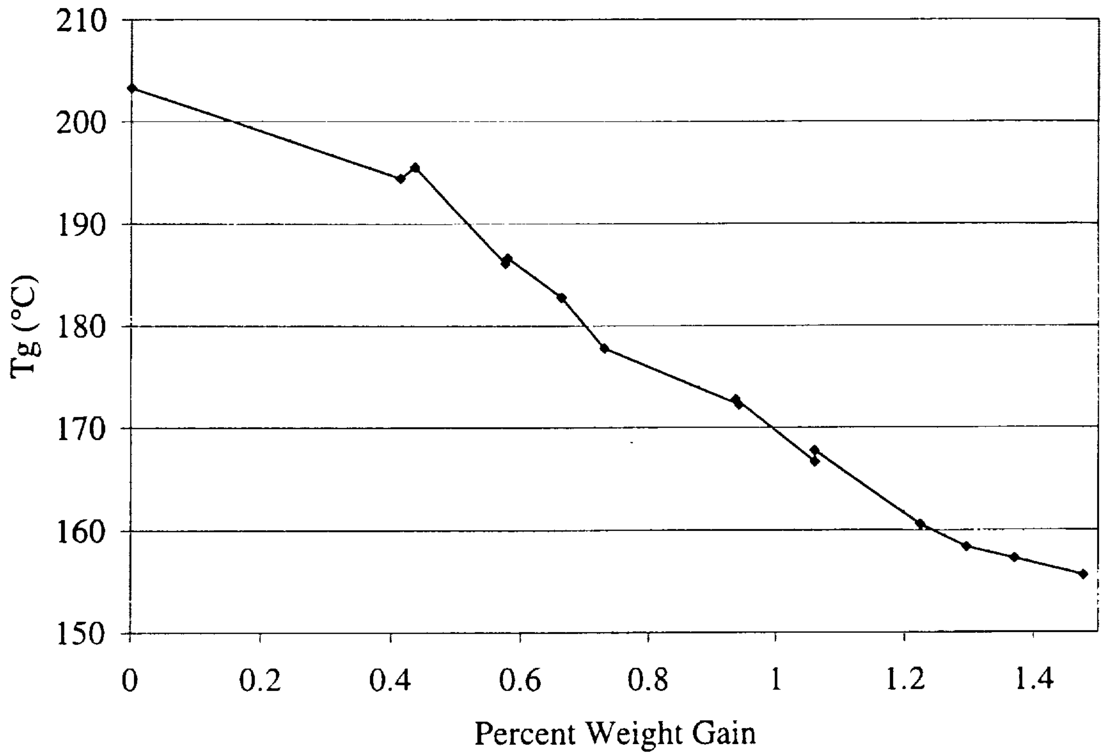


Figure 3. Glass Transition Temperature Versus Percent Weight Gain of AS4/3501-6

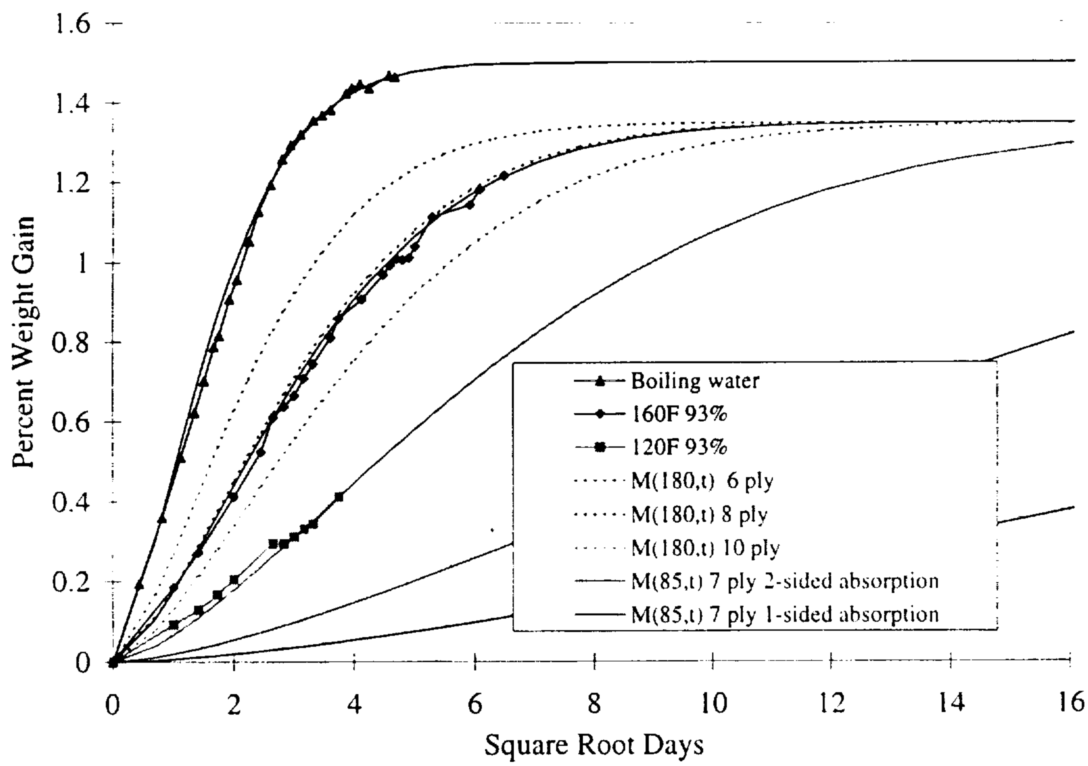


Figure 4. Moisture Absorption of AS4/3601-6 at 93% RH and Various Temperatures

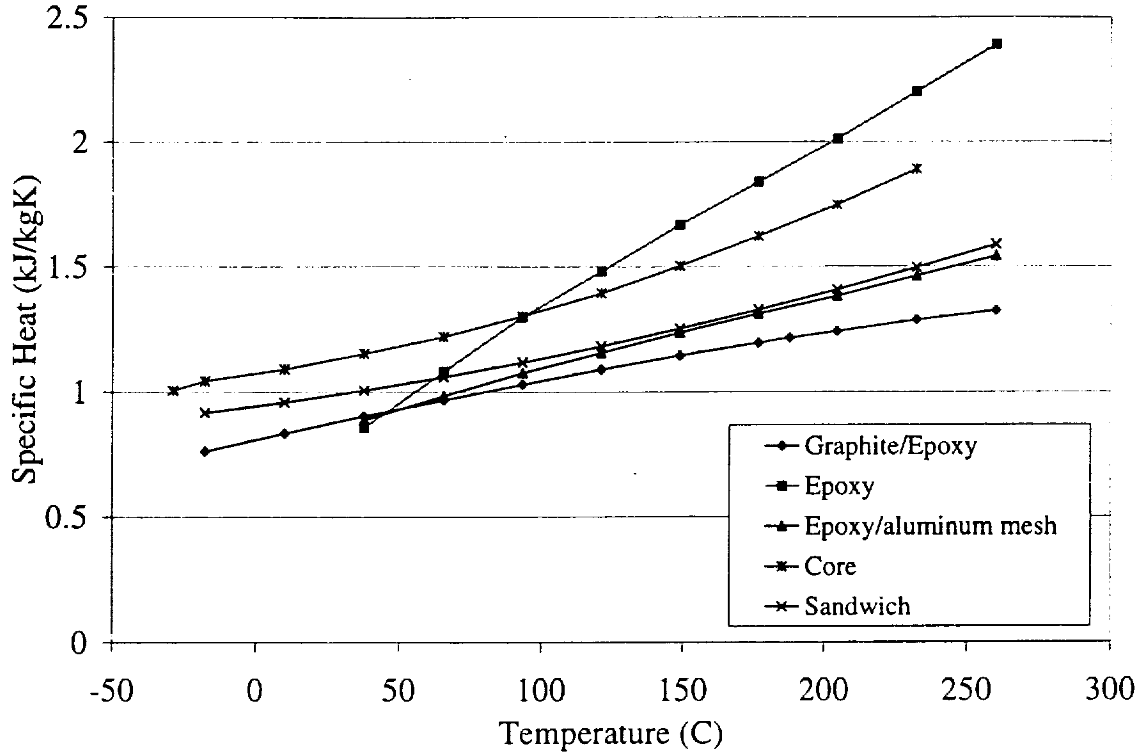


Figure 5. Specific Heat of CNC Component Materials

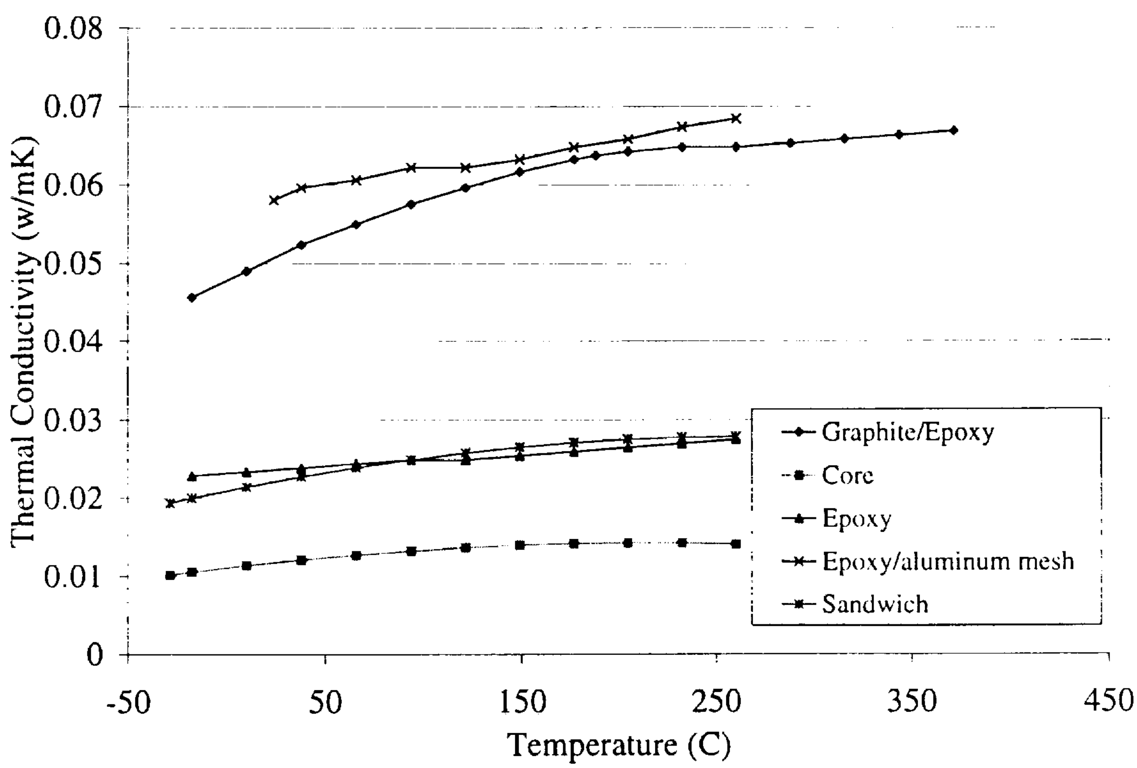


Figure 6. Thermal Conductivity of CNC Component Materials.

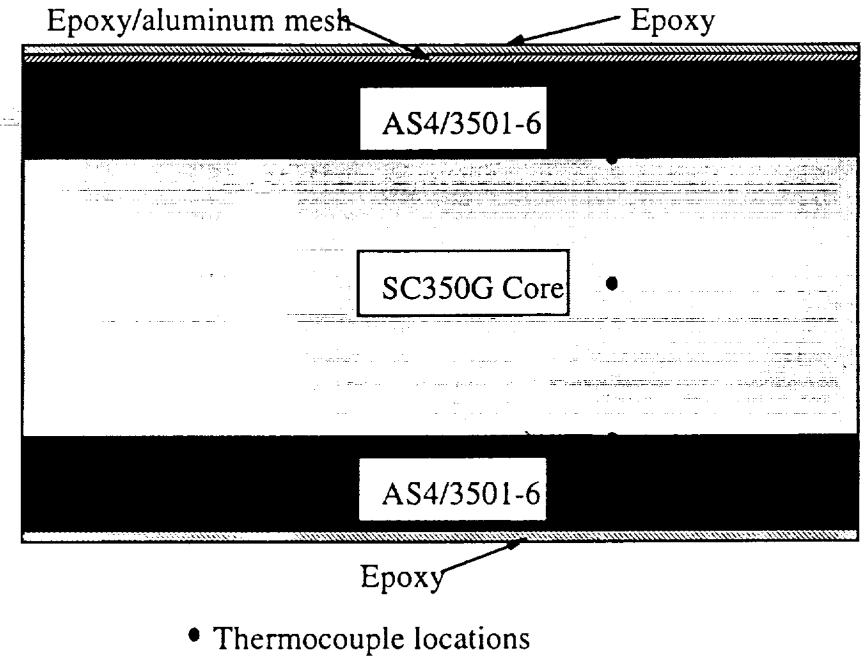


Figure 7. Thermocouple locations on Aerothermal Calibration Panel

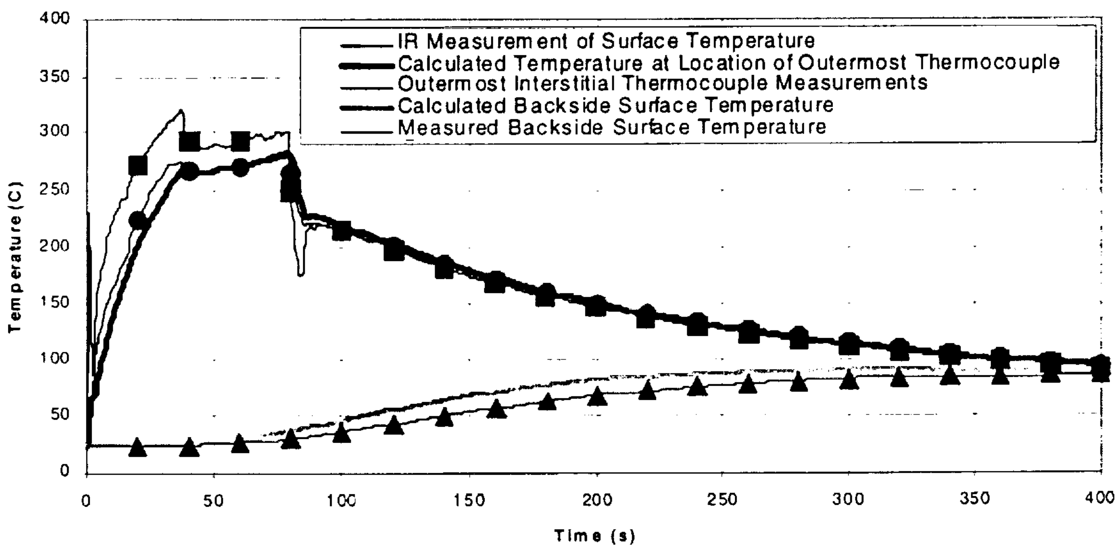


Figure 8. Measured and Predicted Temperatures on Aerothermal Calibration Panel

| Lamina Level Tests (AS4/3501-6) | | 75°F | 240°F | 350°F* | 550°F | 350°F | 480°F |
|---|---|---|---|---|---|---|---|
| Orientation | Property | Average | Average | Average | Average | Average | Average |
| Warp | Tensile Stress (MPa) | 769.2 | 762.4 | 647.7 | 516.0 | 693.0 | 514.6 |
| Fill | Tensile Stress (MPa) | 721.2 | 709.0 | 552.8 | 395.2 | 582.1 | 494.4 |
| Warp | Tensile Modulus (GPa) | 67.6 | 59.1 | 49.7 | 34.7 | 50.8 | 37.9 |
| Fill | Tensile Modulus (GPa) | 66.6 | 54.3 | 43.7 | 29.4 | 49.6 | 45.7 |
| 45 | In-Plane Shear (MPa) | 91.7 | 53.8 | 21.7 | 5.5 | 12.2 | 6.4 |
| 45 | Shear Modulus (MPa) | 5037 | 3223 | 780 | 331 | 425 | 304 |
| Warp | Poisson's ratio | 0.06 | | | | | |
| Fill | Poisson's ratio | 0.07 | | | | | |

* Flight Profile

| Lamina Level Tests (AS4/3501-6) | | 75°F | 240°F | 350°F* | 600°F | 350°F | 480°F |
|---|---|---|---|---|---|---|---|
| Orientation | Property | Average | Average | Average | Average | Average | Average |
| Warp | Compr. Stress (MPa) | 571.3 | 268.5 | 156.1 | 28.7 | 86.3 | 35.6 |
| Fill | Compr. Stress (MPa) | 567.3 | 281.9 | 156.8 | 29.7 | 80.1 | 39.4 |
| Warp | Compr. Modulus (GPa) | 59.7 | 53.9 | 36.2 | 12.1 | 44.6 | 20.9 |
| Fill | Compr. Modulus (GPa) | 60.3 | 55.2 | 31.5 | 12.6 | 42.0 | 19.7 |
| Warp | Interlaminar Shear (MPa) | 48.5 | 27.6 | 11.0 | 1.3 | 6.6 | 2.3 |

| Core Tests (SC350G) | | 75°F | 350°F | 480°F |
|---|---|---|---|---|
| Orientation | Property | Average | Average | Average |
| x | Tensile Stress (MPa) | 25 | 17 | 8 |
| x | Tensile Modulus (MPa) | 3 | 2 | 1 |
| z | Flatwise Compr. (MPa) | 68 | 41 | 13 |
| zx | Shear Punch (MPa) | 31 | 20 | 5 |

| | | | | Prototype 1 | |
|---|---|---|---|---|---|
| Sandwich Tensile Tests | | 75°F | 350°F | 75°F | 350°F |
| Orientation | Property | Average | Average | Average | Average |
| 0 | Tensile Load (kN/m) | 2995 | 2696 | 2901 | 1925 |
| 45 | Tensile Load (kN/m) | 2004 | 1639 | | |
| z | Flatwise Tension (MPa) | 14.9 | 6.2 | | |
| 0 | Open-Hole (NASA 1092) (kN/m) | 3789 | 3894 | | |

| | | | | Prototype 1 | |
|---|---|---|---|---|---|
| Sandwich Compression Tests | | 75°F | 350°F | 75°F | 350°F |
| Orientation | Property | Average | Average | Average | Average |
| 0 | Compr. Load (kN/m) | 2970 | 1820 | 2656 | 1827 |
| 45 | Compr. Load (kN/m) | 2603 | 1484 | | |
| 0 | Post IHGF Comp (kN/m) | 2597 | 1679 | | |
| 0 | Post beach Comp (kN/m) | 2966 | 1576 | | |
| 0 | Open-Hole (0.25" hole) (kN/m) | 1858 | 1168 | | |

| Double Lap Shear (Bearing) | | 75°F | 350°F | 480°F |
|---|---|---|---|---|
| Orientation | Property | Average | Average | Average |
| 0 | Ultimate Bearing (kN) | 29.0 | 16.1 | 9.3 |
| 45 | Ultimate Bearing (kN) | 28.6 | 15.6 | 9.3 |

| Compression after Impact | | 75°F | 350°F |
|---|---|---|---|
| Orientation | Property | Average | Average |
| 0 | Load (kN) 0 J | 305 | 142 |
| 0 | Load (kN) 14 J | 185 | 129 |
| 0 | Load (kN) 27 J | 147 | 105 |
| 0 | Load (kN) 41 J | 143 | 110 |

Table 1. Mechanical Property Test Results