DEVELOPMENT OF AN ABLATIVE 3D QUARTZ / CYANATE ESTER COMPOSITE FOR THE ORION SPACECRAFT COMPRESSION PAD

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ABSTRACT

With the successful flight test of the Orion Multipurpose Crew Vehicle last December, NASA is well on its way to sending humans into deep space with the ultimate goal of putting astronauts on Mars in the 2030s. Orion will receive some upgrades for its next launch in 2018 including a newly developed 3D quartz / cyanate ester composite material for the compression pad. Multiple pad locations in the heat shield serve as a part of the mechanism for holding the Crew and Service Modules together during most mission phases prior to separation followed by Earth re-entry of the Crew Module. Thus the compression pad must survive a structural and aerothermal loads, and protect the adjacent structure and heat shield materials from over-heating.

This paper describes the approach used for developing the new 3D composite, including continuous 3D weaving on an automated loom followed by resin transfer molding. Mechanical, thermal, arc jet, and stress relaxation testing of the 3D composite are also described.

1. INTRODUCTION & OVERVIEW

The 3-Dimensional Multifunctional Ablative Thermal Protection System (3D-MAT) Project was a 3-year effort to develop a 3D Woven Thermal Protection System (TPS) solution to meet the structural and thermal needs of the Orion Exploration Mission (EM) compression pad. The project was a collaborative effort between Space Technology Mission Directorate's Game Changing Division and the Orion Multi-Purpose Crew Vehicle (MPCV) Program.

1.1 Orion Compression Pad Background

The compression pads on the Orion Exploration Flight Test (EFT-1) vehicle, which launched on December 5, 2014, were part of the retention and release (R&R) mechanism that held the Crew Module (CM) and Service Module (SM) together during launch and mission phases prior to separation and Earth re-entry. Figure 1a depicts the 3 components of Orion, and Figure 1b shows a photograph of the Crew Module prior to being attached to the Service Module. The compression pads are part of the heat shield and also transfer structural load between the two modules and thus have multiple functions and requirements. A drawing of the EFT-1 R&R mechanism is shown in Figure 2a while Figure 2b shows a photograph of the EFT-1 compression pad and explosive bolt during installation. The explosive bolt tensioned between the CM and SM pre-loads the compression pad in compression and shear. Additional structural loads are incurred during launch/ascent, and during the pyroshock separation event separating the two modules. Finally, the exposed compression pads experience significant aerothermal loads during atmospheric re-entry with heat fluxes of up to one thousand watts per square centimeter and surface temperatures of two thousand degrees Celsius expected.

Figure 1a. Components of Orion MPCV.

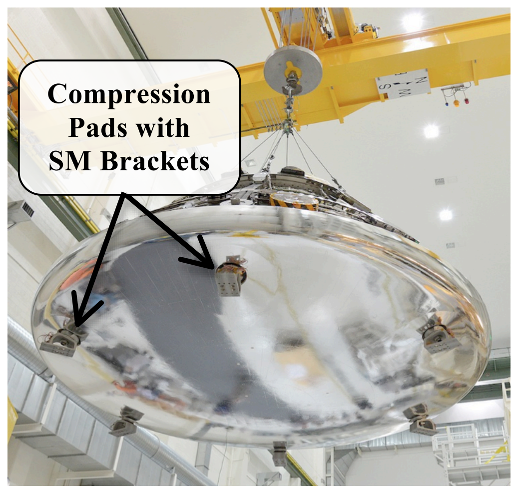

Figure 1b. Orion EFT-1 Crew Module being prepared for attachment to the Service Module.

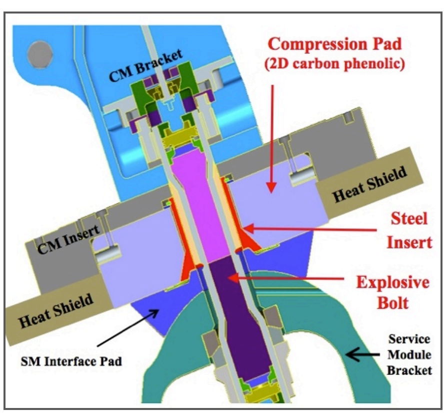

Figure 2a. Drawing of Crew/Service module retention and release mechanism.

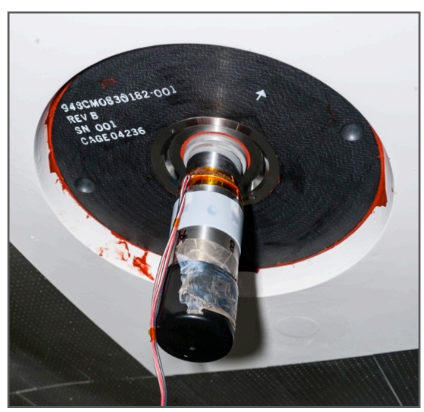

Figure 2b. EFT-1 carbon phenolic compression pad with steel insert and explosive bolt.

1.2 Compression Materials Challenge

The initial design envisioned for the EFT-1 compression pad utilized an all-composite carbon phenolic (MX4926N) pad similar to that shown in Figure 3 to transfer the compression and shear loads between crew and service modules, with an explosive bolt to tie the modules together until separation. Laminated carbon phenolic (MX4926N) with Space Shuttle solid rocket motor nozzle liner heritage[1] was selected to leverage the large pre-existing large property database (especially A-basis allowable strengths). However, structural analysis predicted interlaminar shear loads that exceeded the allowable for carbon phenolic, and thus the all-composite pad design would not close using carbon phenolic. To remedy this, a steel insert was incorporated into the pad (Figure 2a) in order to transfer the shear loads. The steel significantly enhanced the structural robustness of the compression pad, but also greatly increased the thermal conductivity of the pad system and thus the heat transfer into the adjacent structure and heat shield materials.

The carbon phenolic/steel compression pad system solution worked well for the Earth-orbit EFT-1 mission where the entry heating was relatively low. However, all subsequent Exploration Missions (EM) to more distant solar system locations would experience significantly higher re-entry velocity and heating upon return. The EFT-1 design could not meet the thermal protection requirements for EM-1, specifically the maximum temperatures allowed for the underlying heat shield structure and the adjacent Avcoat acreage TPS material bond line.

The Orion program was interested in pursuing a 3D composite material solution for EM compression pads. A survey of available materials found no currently produced 3D composite material that could meet the size, structural and thermal requirements of the EM compression pad. However, two of the authors working on a project with other NASA and industry collaborators to produce and assess a variety of 3D woven TPS materials[2,3,4,5] decided to explore a fast-track tailored woven TPS solution that could suit the Orion compression pad needs.

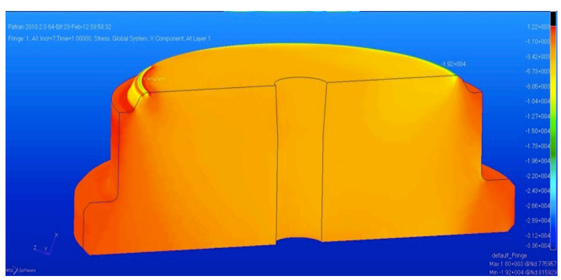

Figure 3. Cross-section view of a finite element model of carbon phenolic compression pad "top hat" design under load. This design was predicted to fail due to poor interlaminar strength.

1.3 Woven Thermal Protection System Project

In 2011 a small group of researchers[i] at NASA Ames Research Center began assessing the possibility of using 3D weaving, both with and without resin infusion, to make spacecraft heat shields. The Woven Thermal Protection System (WTPS) effort started with seed funding from an Ames Center Innovation Fund and was followed by an award from NASA's Office of Chief Technologist in response to a competed[ii] effort for high-payoff high-risk areas of need for NASA

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[i] WTPS NASA researchers included Ellerby, D.; Feldman, J., Stackpoole, M.; Venkatapathy, E. and later included partners at Bally Ribbon Mills: Wilkinson, C; Bryn, L.

[ii] NASA Office of Chief Technologist Broad Area Announcement

technology advancement. The effort produced many laboratory-scale materials from a variety of fiber and resin compositions including dry woven, partial resin densification, and full resin densification over a larger range of densities as shown in Figure 4.

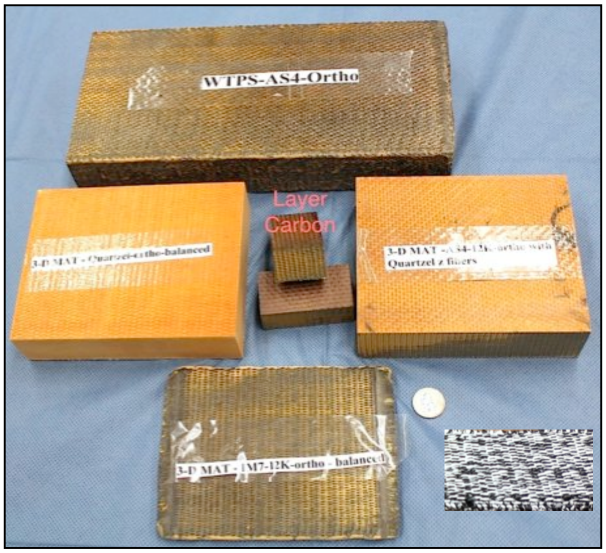

Figure 4a. Photo of various Woven TPS prototype materials.

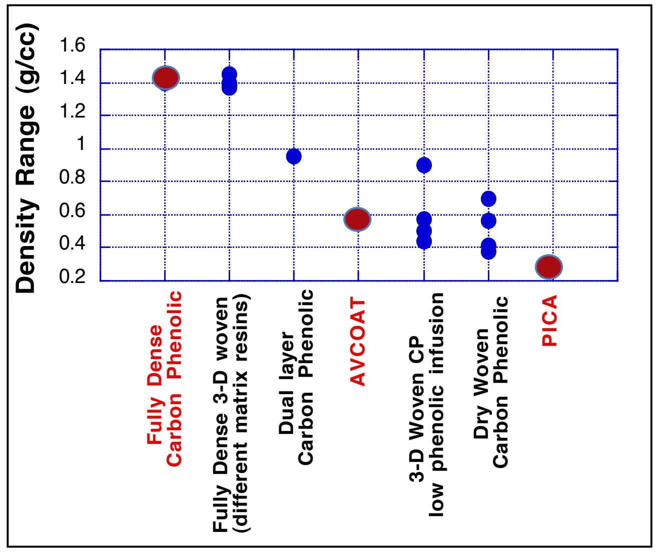

Figure 4b. Density range of Woven TPS materials (blue dots) compared to heritage TPS.

1.4 3D Multifunctional Ablative Thermal Protection System (3DMAT) Project

The 3D-MAT project was initiated in mid-2012 as a collaboration between Orion and the Space Technology Mission Directorate's Game Changing Division to tailor a WTPS solution for the Orion compression pad and develop it to Technology Readiness Level (TRL) 4 prior to transferring the technology to Orion for further development through EM-1 flight. The Orion prime contractor, Lockheed Martin, participated in the project and became the recipients of the technology upon transfer at the end of the project. Key requirements for the compression pad are listed in Table 1.

Table 1. Driving Requirements for Orion Compression Pad Material

Type	Requirement
Thermal	Compression pad to heat-shield/carrier-structure interface shall maintain positive margin against the 500 °F maximum bondline temperature throughout the mission
Structural	Compression pad shall carry compression moment & shear loads
Structural	Tension tie shall maintain preload with losses due to creep & joint relaxation
Structural/ Thermal	Compression pad shall thermally function after exposure to the separation bolt pyro-shock event
Size	Pad material shall be manufacturable to at least 3 in. thickness by 10 in. diameter

The project was split into two phases with Phase 1 focused on establishing viability of a woven TPS solution before the larger investment in Phase 2 to scale up and develop the material.

Phase 1 of 3D-MAT spanned 6 months and consisted of assessing feasibility of tailoring a WTPS solution by producing several prototype materials (Figure 4a), obtaining key material properties, and conducting thermal and structural analyses. Phase 1 concluded with the selection of a high fiber volume fraction 3D orthogonal woven quartz architecture based on preliminary thermal and structural data and analyses. An assessment matrix for the prototype WTPS and heritage materials evaluated during Phase 1 is shown in Table 2.

Table 2. Phase 1 Material Evaluations

Material	Z Fiber Loading	Thermal Performance	Structural Performance
3D orthogonal quartz + Phenolic OR Cyanate Ester OR Polyimide	33 %	Good	Good
3D ortho. carbon with quartz in Z + phenolic	16 %	TBD	TBD
3D Layer-to-Layer carbon + phenolic	5-10 %	Poor	Poor
3D orthogonal carbon + phenolic	16 %	Fails	Fair
3D orthogonal carbon + phenolic	33 %	Fails	TBD
2D carbon phenolic (no steel insert)	0 %	Poor	Fails
2D carbon phenolic (steel insert)	0 %	Fails	Poor

Phase 2 lasted 2 ½ years and consisted of weave scale-up, resin selection, full-scale billet manufacture and a suite of testing and development activities undertaken to advance the TRL to 4 for the compression pad application. Activities included mechanical, thermal and other physical property testing, arc jet testing, stress relaxation testing, and thermal/ablation response model development. The progression of development is depicted in Figure 5. Pyroshock testing, which is perceived as very low risk, is planned as part of the Orion development after tech transfer and will raise the TRL to 5.

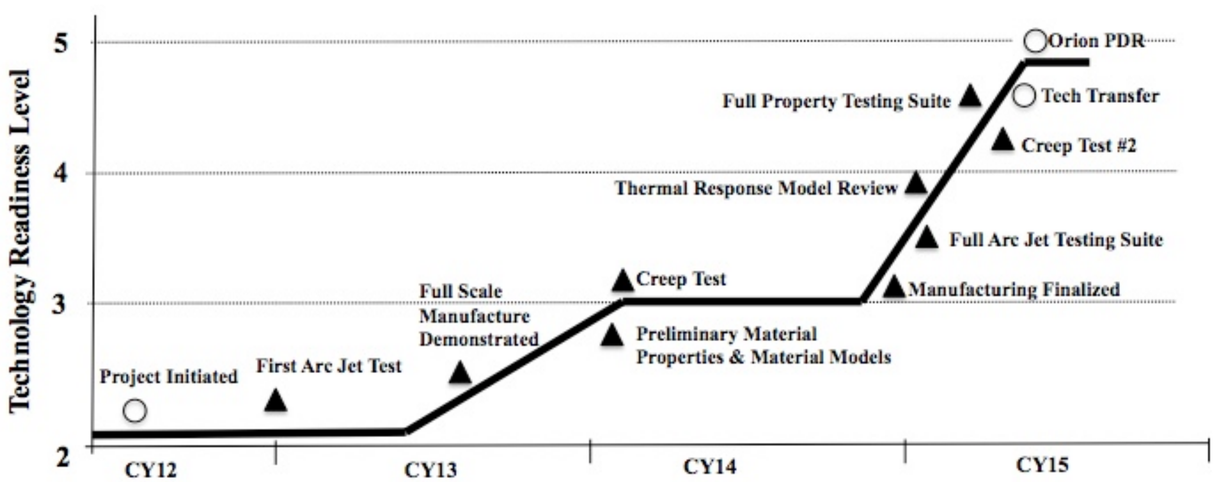

Figure 5. 3D-MAT development pathway.

2. COMPOSITE MANUFACTURE

This section describes the manufacturing development for 3DMAT material. Part fabrication consists of 3 separate steps including 1) weaving, 2) resin infusion, and 3) final machining as shown in Figure 6.

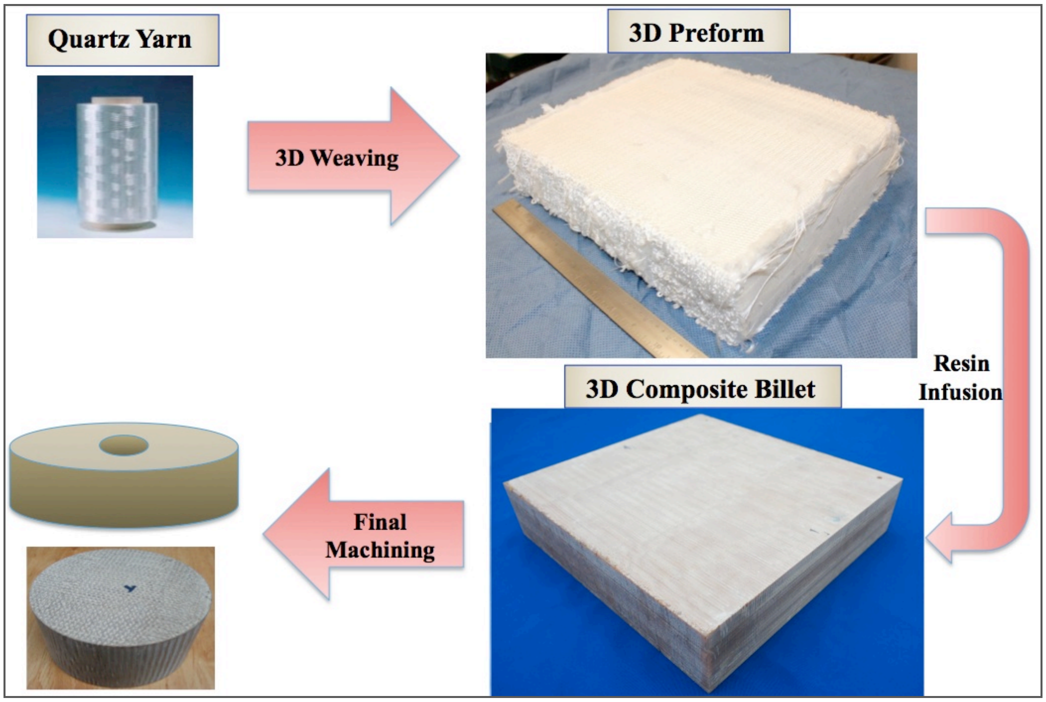

Figure 6. 3DMAT manufacturing steps.

2.1 3D Weaving

2.1.1 Weave Architecture

The high fiber volume 3D orthogonal weave architecture was chosen based on its ability to provide superior structural robustness compared with layer-to-layer or angle interlock 3D weave types, which would tend to limit thermal conductivity at the cost of strength.[6] Quartz fiber, whose thermal conductivity is one fifth that of carbon fiber, was selected in order to manage heat transfer from the outer mold line to the inner mold line. A balanced weave with a third of the fibers running in each of the X/Y/Z directions was designed, which offers the benefit of simplifying structural testing and analysis. Weaving was performed on a jacquard loom using Saint Gobain's Quartzel® fiber.

2.1.2 Weaving Scale-Up

Bally Ribbon Mills had prior experience weaving 5 cm (2 in.) thick 3D orthogonal quartz material; however, this was done at 15 cm (6 in.) width and with different fiber loadings (Z much less than 1/3). In order to weave the 3D-MAT design at the full 30.5 cm (12 in.) width by 7.6 cm (3 in.) thickness, a new jacquard harness, providing individual warp yarn control, was designed and integrated into the loom. The resulting full-scale woven material (Figure 7) was cut at 33 cm (13 in.) lengths and the areal weight, fiber volume, fiber loading, and dimensional tolerances were measured with Bally Ribbon Mills developed procedures in order to meet NASA specified requirements.[7]

Figure 7. 3DMAT quartz preform weaving at Bally Ribbon Mills.

2.2 Resin Infusion

2.2.1 Resin System Screening

Three resin systems were evaluated by infusion of 3D quartz preforms and subsequent mechanical testing and arc jet testing at Orion EM-1 relevant conditions. The resin systems included: phenolic, which has significant heritage use in TPS, a low-viscosity RTM cyanate ester, which was the most readily processed resin, and a high glass transition temperature (T_g) polyimide resin, which offered the best high-temperature structural performance.

Phenolic resin was favored due to its heritage use in TPS[8] and was infused into a 3D quartz preform via resin transfer molding (RTM). Due to the nature of the phenolic resin system, it is not possible to achieve full densification with a single infusion-cure cycle, and the resulting billet consisted of 13 % porosity. The relatively low Z-compression strength[iii] of 62 MPa (9.0 ksi) for the quartz / phenolic billet is attributed to the high porosity. While it is possible to further densify the billet with additional resin infusion and cure cycles, this approach was not pursued.

The high T_g polyimide resin PETI-330 was infused into a 3D quartz preform via RTM to produce a 3D woven quartz / polyimide (WQPI) billet for testing and evaluation. In the first infusion run, the quartz preform was inadvertently over-compressed causing a distortion of the through-thickness (Z) fibers. The team believed this could be remedied; however, WQPI was not pursued further. The high temperature structural performance of polyimide was beneficial, but not crucial for the compression pad. The lower availability and higher cost of PETI-330 compared to the other resin systems put it at a disadvantage, and the high temperature processing of polyimide also made pursuit of WQPI less desirable. With the lowest porosity of the three quartz composites initially produced, WQPI had the highest Z-compression strength[iii] of 390 MPa (57 ksi).

[iii] Average of three measurements using ASTM D-695 with 0.5 x 0.5 x 1.0 in. prisms

The low-viscosity EX-1510 RTM cyanate ester resin system from Tencate was readily used for RTM at room temperature, making 3D woven quartz / cyanate ester (WQCE) manufacture the most straightforward of the three systems screened. The WQCE material had very good Z-compression strength[iii] at 340 MPa (49 ksi). All three quartz composites performed well in terms of recession and backface temperature during preliminary arc jet testing at two conditions: 750 W/cm^2 heat flux, 30 kPa pressure, 45 second duration, and 400 W/cm^2 heat flux, 35 kPa pressure, 65 second duration. Based on the available mechanical test data, arc jet performance, and particularly the manufacturability of the three composites, WQCE was selected for further manufacture development and testing in Phase 2 of the project. From this point the two acronyms 3D-MAT and WQCE will be used interchangeably to refer to the material selected and developed for the Orion compression pad.

2.2.2 Resin Transfer Molding

A variety of infusion techniques were evaluated using the EX-1510 cyanate ester resin system including vacuum bag approaches such as vacuum assisted RTM and pressure assisted RTM. Ultimately, the most successful and reliable process used a hard tooling RTM approach developed by San Diego Composites, yielding 3D-MAT billets with a bulk density of 1.79 g/cm^3 and porosity less than 0.5 %. The infusion vessel is shown in Figure 8. A total of 24 quartz / cyanate ester billets were produced over 20 months of the 3D-MAT project, with process refinements primarily designed to minimize porosity. These billets were used to support a battery of tests planned to raise the TRL for use as compression pad on Orion Exploration Missions.

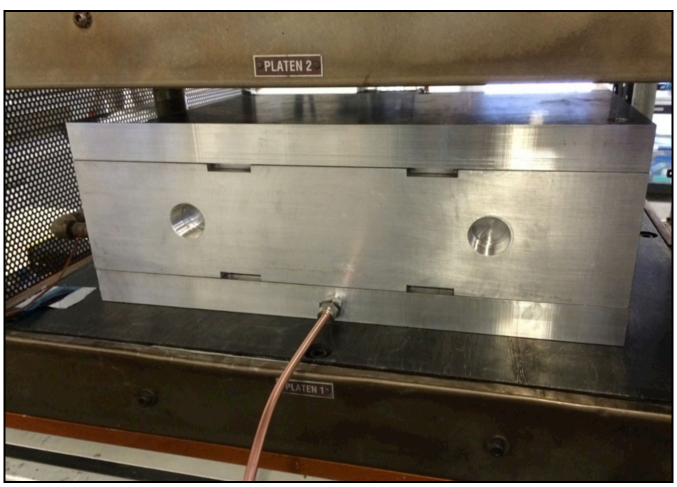

Figure 8a. 3DMAT RTM Vessel at San Diego Composites.

Figure 8b. Vessel installed a hydraulic press for cure.

3. DEVELOPMENT TESTING & MODELING

A suite of development tests was performed in order to raise the technology readiness of 3D-MAT material to level 4/5 prior to fully transferring the technology to Orion for further development through EM-1 flight, scheduled for 2018. This section discusses the development activities, which included material property characterization, aerothermal arc jet testing, thermal response model development and stress relaxation testing.

3.1 Material Property Testing

An extensive set of material properties was characterized to enable thermal response modeling and thermal structural analysis. The testing was conducted in two phases during manufacture process development. The first phase of testing provided preliminary properties to support initial design and analysis of the Orion EM-1 compression pad and used early WQCE material with higher porosity compared to the final process (~3 % open porosity compared to < 0.5 % for the final manufacturing process). The material property test matrix is shown in Figure 9, which represents the testing conducted throughout the Phase 2 development effort, split nearly evenly between early WQCE and finalized WQCE material. The majority of the testing was conducted at Southern Research Institute, with NASA Langley Research Center, NASA Johnson Space Center, NASA Ames Research Center and Galbraith Laboratories performing a portion of the work. Some of the key results will be highlighted, including compressive testing, tensile testing, and thermal conductivity.

	Property	Measured Temp (°C)	Measured Temp (°F)	Orienta-tion	grand total
Thermal Model	Thermal Conductivity - virgin	21 – 290	73 – 550	Z	4
		"	"	X	2
		"	"	Y	2
	Thermal Conductivity - char	10 – 1530	50 – 2800	Z	6
	Specific Heat Capacity-virgin	10 – 290	50 – 550	-	3
	Specific Heat Capacity-char	120 – 290	250 – 2500	-	3
	Thermal expansion-virgin	-100 - 1530	-150 – 2800	X	4
		"	"	Y	4
		"	"	Z	2
	Elemental Composition - virgin	n/a	n/a	n/a	6
	Elemental Composition - char	n/a	n/a	n/a	6
	Heat of Combustion - virgin	n/a	n/a	n/a	3
	Heat of Combustion - char	n/a	n/a	n/a	3
	TGA / Char Yield - virgin	23 – 1010	73 – 1850	n/a	6
	Emissivity - char	23	70		6
Structural	Tensile strength, strain, modulus	23	75	Z	13
		260	500	Z	2
		23	75	X	9
		23	75	Y	9
	Compressive strength, strain, modulus	23	75	Z	20
		260	500	Z	3
		23	75	X	26
		260	500	X	4
		23	75	Y	18
		260	500	Y	5
		23	75	45° bias	58
		260	500	45° bias	2
	Shear strength, strain, modulus	23	75	XY	14
		260	500	XY	3
		23	75	XZ	17
		260	500	XZ	3
		23	75	YZ	17
		260	500	YZ	3
	Poisson's Ratio	23	75	XY	6
		23	75	YX	6
		23	75	ZX	6
	Porosity	n/a			20

Figure 9. 3DMAT material property test matrix.

3.1.1 Compressive Testing

Compressive testing was performed according to ASTM D-695 using a 1.27 by 1.27 by 2.54 cm (0.50 by 0.50 by 1.00 inch) right prism. Stress vs. strain curves for the on-axis (X, Y, Z) and bias (45° XY) orientations at room temperature are shown in Figure 10a. The on-axis tests show linear high stiffness response until a yield-like "failure onset" in the 60-75 ksi range, at which point additional loading at low stiffness occurs until ultimate failure.

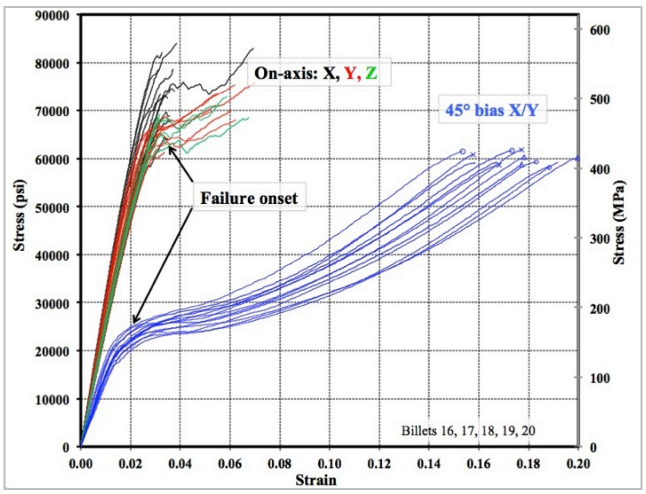

Figure 10a. Compressive stress-strain curves for on-axis and bias specimens.

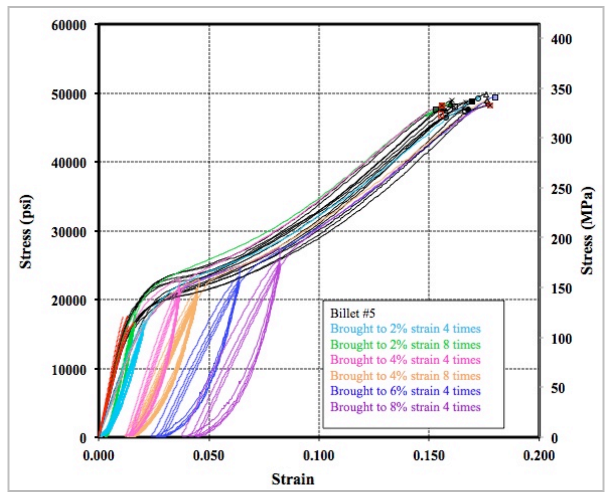

Figure 10b. Cyclic compressive response for bias specimens.

For the bias samples, the fibers are not being loaded along their axis but rather are being scissored, and thus the modulus is lower. Loading at a moderate stiffness is followed by a yield-like "failure onset" at relatively low stress levels of 20 – 25 ksi, with additional loading up to about 60 ksi prior to ultimate failure. In order to understand the nature of the "failure onset" knee in the bias specimen curve, several samples were cyclically loaded to various points past the knee, unloaded, and finally loaded to failure. The results of the cyclic testing are shown in Figure 10b. While specimens do have a permanent set once compressed beyond the "failure onset" point, subsequent cycles show repeatable loading/unloading curves, and the ultimate strength is unaffected by the loading/unloading cycles. This phenomenon demonstrates the robustness of the 3DMAT material. Note that the cyclic testing shown in Figure 10b was performed on an early billet of 3DMAT (WQCE-5), which consisted of 3 % porosity, and thus has lower ultimate strength compared to the final 3DMAT billets, which consisted of less than 0.5 % porosity).

3.1.2 Comparison to 2D Carbon Phenolic (MX4926N)

Material property characterization of WQCE demonstrated the enhanced mechanical performance of the 3D-MAT material relative to 2D laminates such as carbon phenolic. Additionally, the use of lower thermal conductivity quartz fibers significantly improves the thermal performance for this compression pad material compared to the EFT-1 composite. A comparison of key properties for 3D-MAT and MX4926N is provided in Table 3. Notably, the Z-reinforcement results in an interlaminar tensile strength that is 9 times that of the laminate. With a system thermal conductivity that is about two thirds that of carbon phenolic, the insulation capability of 3D-MAT is also significantly superior to the EFT-1 pad material.

Table 3. Property Comparison for 3D-MAT and Carbon Phenolic

Property	**3D-MAT vs. 2D Carbon Phenolic (MX4926N)**
Compression Strength (ultimate):	
On-axis	140%
45° axis	135%
Tensile Strength (ultimate):	
Interlayer (Z)	900%
In-plane (X/Y)	245%
Thermal Conductivity:	
Interlayer (Z)	75%
In-plane (X/Y)	55%

3.2 Arc Jet Testing

Arc jet testing was performed at a variety of conditions relevant to the EM-1 entry envelope in the Ames TP3 facility. Test objectives were: 1) characterize the material's aerothermal performance including recession and in-depth thermal response at a range of EM-1 relevant conditions, and 2) support thermal/ablation response model development. A 10.2 cm (4.0 in) diameter model with in-depth thermocouples was used, as shown in Figure 11. Some of the arc jet models received an extra post-cure cycle in order to assess whether it affected performance; no significant difference was discerned. The extended post-cure models are indicated with a "PC" designation after the model ID number in Table 4.

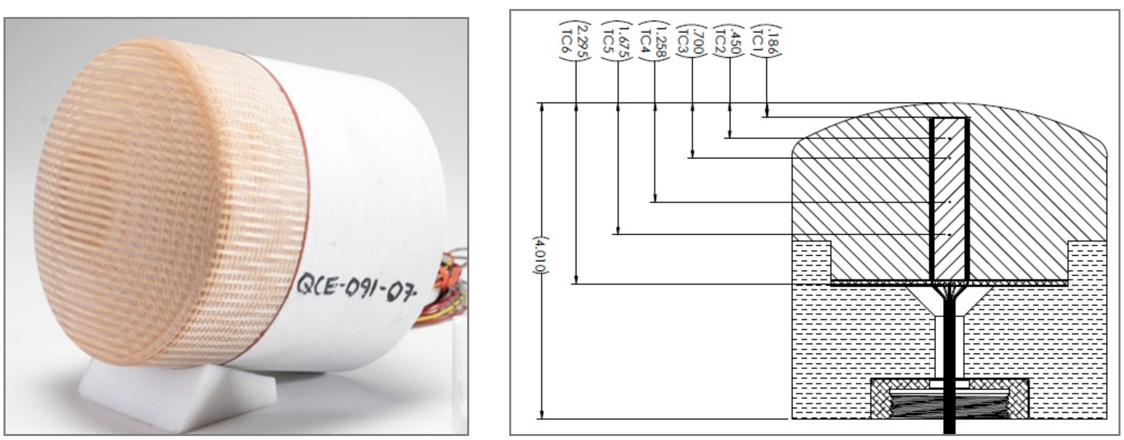

Figure 11. Photo and drawing of arc jet model with in-depth thermocouple instrumentation

A summary of conditions and test results is provided in Table 4. The model surface temperatures were measured during the test using optical pyrometers. Recession was determined by coordinate measurement before and after arc jet exposure. Under the more severe conditions (> 400 W/cm^2), quartz fiber melt flow was observed during the test via video, consistent with the peak surface temperatures of 2200 °C and greater. A small amount of recession was confirmed with post-test measurements. For the lower test conditons (< 250 W/cm^2), no fiber melt was observed in the test video, consistent with the measured peak surface temperatures below 1800 °C, and very small amount of model growth was measured after the test. Photos of the models during and after the arc jet test are shown in Figure 12.

Table 4. Arc Jet Test Conditions and Model Measurements

Condition	Heat Flux (W/cm^2)	Pressure (kPa)	Model ID Number	Time (s)	Max Surface Temp (°C)	Recession (mm)
1	660	35.4	04 05-PC	60 60	2400 2300	3.0 2.0
2	585	55.0	01	120	2350	4.1
3	465	16.0	06 011-PC	100 100	2200 2250	3.5 1.3
4	215	16.2	07 08 12-PC	150 100 150	1680 1650 1720	-0.6 -0.6 -1.9
5	205	55.1	02 03	120 80	1700 1750	-0.4 -0.1
6	140	3.3	09 10-PC	200 200	1550 1600	-0.5 -0.7

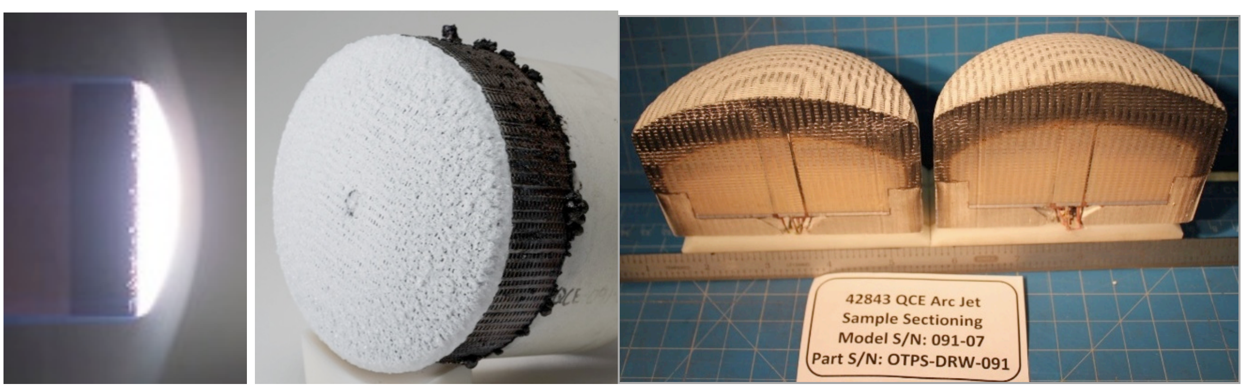

Figure 12**.** Arc jet models during test (205 W/cm^2), after test (585 W/cm^2), and cross-sectioned post-test (215 W/cm^2).

3.3 Thermal Response Model Development

A physics-based thermal/ablation response model[9] was developed using the physical properties of 3D-MAT and was used to prediect in-depth temperature response during arc jet testing. The model is a critical part of maturing 3D-MAT for use as the Orion EM-1 compression pad and will be used to thermally size the thickness of 3D-MAT required to protect the adjascent materials on the heat sheild. Comparisons of the predicted and measured in-depth temperature response for the 585 W/cm^2 and 205 W/cm^2 conditions are shown in Figure 13.

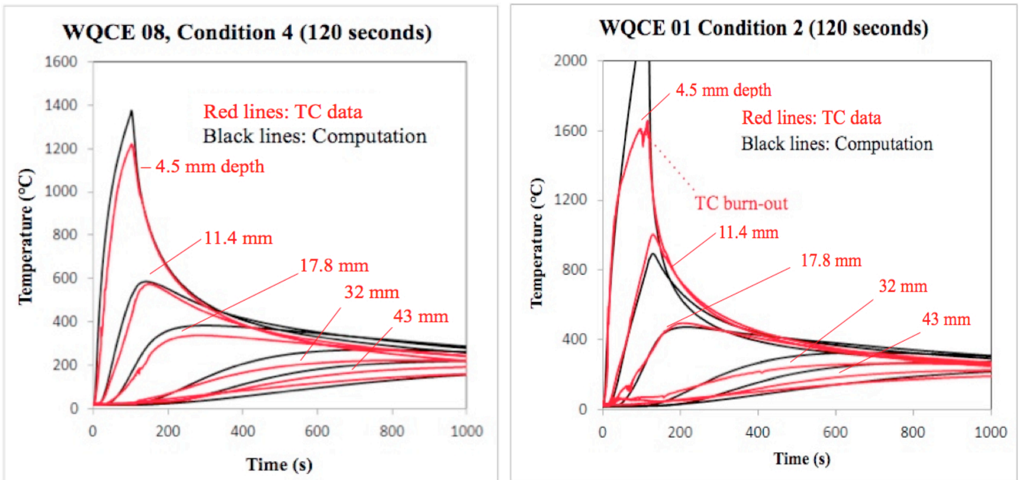

Figure 13. In-depth temperature response measurement (red) and prediction (black) for the 215 W/cm^2 (left) and 585 W/cm^2 (right) arc jet tests.

3.4 Stress Relaxation Testing

During vehicle assembly the compression pad is preloaded via torqueing of the bolt shown in Figure 2. This preload must be maintained over several months with minimal loss, which can occur due to relaxation of the bolt/pad/structure assembly. In order to assess the amount of stress relaxation that might occur due to creep in the 3DMAT material, a stress relaxation test was performed at NASA Langley Research Center using cylindrical specimens 10.2 cm (4.0 in.) in diameter by 6.6 cm (2.6 in.) thick compressed between two steel plates, as shown in Figure 14. A total load of 240 kN (54,000 lbs) was introduced using six instrumented fasteners. After a week, the fasteners were re-torqued. Two WQCE specimens and one MX4926N specimen were tested with this methodology.

The first WQCE test specimen was obtained from an early 3DMAT billet that contained 3 % porosity, and underwent stress relaxation testing for nearly a year. The second WQCE specimen came from a billet using the final process and had less than 0.5 % porosity. This specimen is still undergoing stress relaxation testing at the time of writing; the first two months of data are presented. Figure 15 shows the total load over time, including re-torque. The percent load reduction over time after re-torque is also shown in Figure 15. All three tests exhibit small load reductions, with material creep, rig relaxation and fastener response drift all potential contributors to the response curves.

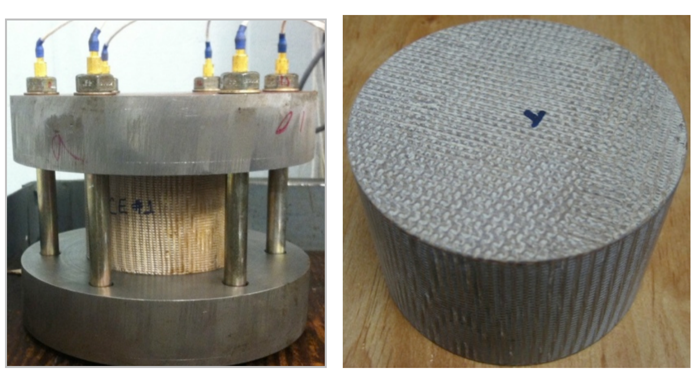

Figure 14. Stress relaxation test setup and test specimen.

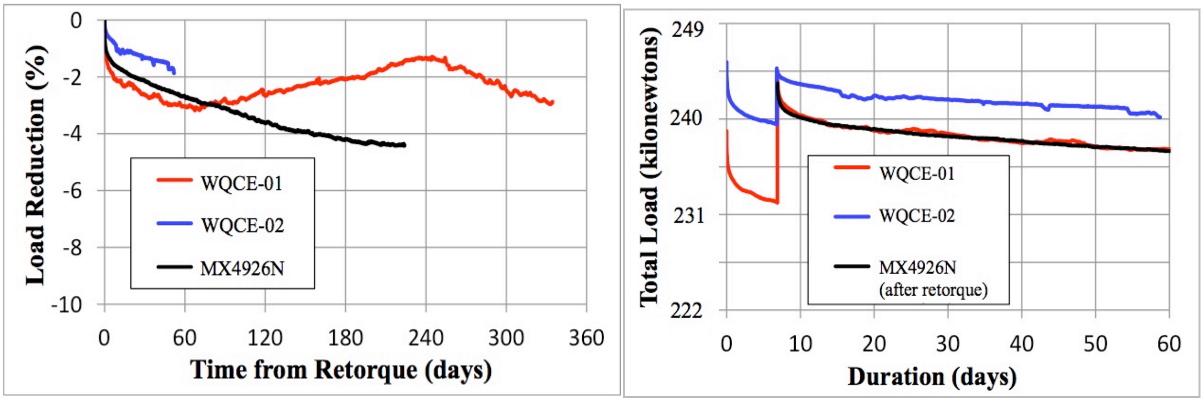

Figure 15. Load reduction (left) and total load (right) as a function of time.

4. CONCLUSIONS

A new 3D woven quartz / cyanate ester composite has been developed to TRL 4/5 to meet the needs of Orion Exploration Mission compression pads. The 3D-MAT material derives structural robustness from the high fiber volume fraction 3D orthogonal weave and full resin densification (< 0.5 % porosity) at the large scale of 30 cm width by 7.6 cm thick and maintains a relatively low thermal conductivity via use of quartz fibers. This combination of properties is well suited for thermal protection system materials that also have substantial structural requirements. The successful execution of TRL 4/5 testing and development activities resulted in the successful transfer of 3D-MAT's enabling technology to Orion's prime contractor Lockheed Martin for further development through the EM-1 flight, scheduled for launch in 2018. Billet production for EM-1 development and flight hardware is well underway at the time of writing this paper.

New weaving and infusion technologies were developed in order to produce the 3D-MAT material. Continuous, automated 3D weaving of a preform of this type and at this scale has never before been demonstrated, to the knowledge of the authors. Likewise, the full densification of large 3D preforms was also not well established, and the RTM process developed herein is unique and enabling for the WQCE material. These technologies were facilitated by the investment of NASA's Space Technology Mission Directorate and the Orion Multipurpose Crew Vehicle Program.

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