

# FLEXIBLE THERMAL PROTECTION SYSTEM DEVELOPMENT FOR HYPERSONIC INFLATABLE AERODYNAMIC DECELERATORS

9<sup>TH</sup> INTERNATIONAL PLANETARY PROBE WORKSHOP  
16-22 JUNE 2012, TOULOUSE

Joseph A. Del Corso<sup>(1)</sup>, Walter E. Bruce, III<sup>(1)</sup>, Stephen J. Hughes<sup>(1)</sup>, John A. Dec<sup>(1)</sup>, Marc D. Rezin<sup>(2)</sup>, Mary Ann B. Meador<sup>(3)</sup>, Haiquan Guo<sup>(4)</sup>, Douglas G. Fletcher<sup>(5)</sup>, Anthony M. Calomino<sup>(1)</sup>, and F. McNeil Cheatwood<sup>(1)</sup>

<sup>(1)</sup>NASA Langley Research Center, MS 431 Hampton VA 23681, USA, *Joseph.A.DelCorso@NASA.Gov*

<sup>(2)</sup>NASA Ames Research Center, Moffett Field, CA 94035, USA, *Marc.D.Rezin@NASA.Gov*

<sup>(3)</sup>NASA Glenn Research Center, MS 49-3, Cleveland OH 44135, USA, *MaryAnn.Meador@NASA.Gov*

<sup>(4)</sup>Ohio Aerospace Institute, 22800 Cedar Point Road, Cleveland OH 44142, USA, *Haiquan.N.Guo@NASA.Gov*

<sup>(5)</sup>University of Vermont, 33 Colchester Ave., Burlington VT 05404, USA, *Douglas.Fletcher@UVM.Edu*

## ABSTRACT

The Hypersonic Inflatable Aerodynamic Decelerators (HIAD) project has invested in development of multiple thermal protection system (TPS) candidates to be used in inflatable, high down-mass, technology flight projects. Flexible TPS is one element of the HIAD project which is tasked with the research and development of the technology ranging from direct ground tests, modelling and simulation, characterization of TPS systems, manufacturing and handling, and standards and policy definition. The intent of flexible TPS is to enable large deployable aeroshell technologies, which increase the drag performance while significantly reducing the ballistic coefficient of high-mass entry vehicles. A HIAD requires a flexible TPS capable of surviving aerothermal loads, and durable enough to survive the rigors of construction, handling, high density packing, long duration exposure to extrinsic, in-situ environments, and deployment.

This paper provides a comprehensive overview of key work being performed within the Flexible TPS element of the HIAD project. Included in this paper is an overview of, and results from, each Flexible TPS research and development activity, which includes ground testing, physics-based thermal modelling, age testing, margins policy, catalysis, materials characterization, and recent developments with new TPS materials.

## 1. INTRODUCTION

Development of flexible TPS for HIAD began in 2007 with exploratory funds for a demonstration flight project, moderate fundamental analysis, and ground testing of commercial-off-the-shelf (COTS) materials. Since 2007, HIAD with Flexible TPS has developed into a strong technology development effort and flight integration investment for NASA. HIAD as a program encompasses the Inflatable Reentry Vehicle Experiment (IRVE-3), Flexible Systems Development (FSD), and Advanced Technologies, and is enabling

other flight projects including the High-Energy Atmospheric Reentry Test (HEART). FSD is responsible for advancing inflatable structures (IS) and flexible TPS (FTPS). This paper provides an overview of FTPS activities and insight into the objectives and challenges of each element.

## 2. FLEXIBLE TPS OVERVIEW

FTPS is responsible for advancing forebody aeroshell TPS for inflatable aerodynamic decelerators. The Flexible TPS project is responsible for the development of FTPS materials, manufacturing components, stitching and seaming technologies, ground test techniques, modelling FTPS, materials characterization, lifecycle testing and thermal margins for design and tests.

FTPS activities are broadly characterized by three principle activities: ground testing, material development and characterization, and FTPS modelling. Each effort supports multiple technology products including a comprehensive FTPS margins policy, validated FTPS thermal model, manufacturing capabilities, and advancing FTPS materials. The ground test effort includes all facility testing of FTPS systems in shear and stagnation conditions. The objective of the ground test effort is to develop test techniques for evaluating FTPS in flight-relevant conditions of heat flux, surface pressure, and aerodynamic shear force. The objective of material characterization activities is to measure thermophysical properties of FTPS components, namely the outer fabrics, insulators, and gas barrier layers. [1] The material characterization effort is also working to quantify catalytic efficiency of the outer fabrics in order to realize a thermal margin resulting from super-catalytic assumptions currently used throughout FTPS development activities at NASA. Finally, the material characterization effort has focused on developing lifecycle test capabilities in order to quantify the effects of expected in-situ conditions, including long duration stowage at high density resulting in degraded TPS performance, exposure to vacuum environments, and

launch vibrations. The objective of FTPS modelling is to develop a robust physics based thermal model, validated against ground-test data, and able to predict thermal performance to within  $\pm 60^{\circ}\text{C}$  of test.

Flexible thermal protection materials are essential to the development of deployable aeroshell systems. FTPS's generally multilayer systems consisting of one or more types of materials. In general, there are one or more layers of a porous outer reinforcing fabric capable of withstanding high temperatures followed by one or more layers of a porous insulating material. The stack of outer fabric and insulation is backed up by a thin, impermeable material called a gas barrier which is analogous to the bondline for a rigid ablative TPS.

Several materials are being evaluated by the FTPS element of the HIAD program for use as the outer fabric and insulation layers. The outer fabric materials being evaluated are Nextel BF-20 and a silicon carbide (SiC) trade named Nicalon. The Nextel BF-20 can withstand heat fluxes up to  $30\text{ W/cm}^2$  and is considered to be a first generation FTPS. The Nicalon SiC material can withstand heat fluxes up to  $50\text{ W/cm}^2$  and is considered to be a second generation FTPS. The insulation materials being evaluated are Pyrogel 3350, Pyrogel 2250, and Saffil. The material used for the gas barrier is an aluminized Kapton layer laminated to a layer of Kevlar and is designated AKK.

A significant amount of progress has been made in the FTPS program in the past year. The thermal modelling activity has migrated from utilizing an artificial thermal contact resistance to force the analysis to match the test data to a physics-based model that correctly accounts for conduction, advection, pyrolysis, radiation, and permeability effects. [2] Ground testing performed early [2] in the TPS development timeline for HIAD, focused on shear tests in the 8-foot high temperature tunnel (8'HTT), which is a valid environment for low heating sounding rocket programs, but is not for higher heat flux Earth or Mars entry environments. After an extensive facilities survey, FTPS has determined that Boeing's Large-Core Arc Tunnel (LCAT) facility is able to achieve relevant conditions and affords the largest trade space of environments. [1] While material characterization previously focused only on thermophysical properties, FTPS is now taking active states into account and is attempting to characterize the effects of handling and pack and deploy scenarios.

### 3. GROUND TESTING

It is important to evaluate and characterize a TPS to determine if the performance is satisfactory for a specified vehicle and flight trajectory. A combination of ground testing and analytical analysis is required to fully evaluate the TPS. Ground testing can typically only match a few of the many variables important for a proper simulation of the flight regime during any

individual test. Analysis can typically characterize a full flight trajectory, but requires ground testing results for verification and validation.

This section will provide a brief overview of current aerothermal ground testing efforts to evaluate the TPS performance. Previous TPS testing efforts are described in. [1]

#### 3.1 Aerothermal Ground Testing

A development effort has been underway to develop aerothermal stagnation and shear test techniques appropriate for evaluating FTPS. Testing flexible systems is very different than testing rigid TPS and requires unique model holder hardware to accommodate the flexible materials and instrumentation. Recent aerothermal development and testing efforts have been performed in the Boeing LCAT facility located in, St. Louis, Missouri. Detailed descriptions of the model holder hardware development effort and test techniques have previously been described. [3]

During stagnation testing, it is important to match the flight surface pressure, heat flux, and heat load. It is also desirable to match the correct flight enthalpy; however, it is usually difficult to match more than two or three of the flight variables during a single test. Shear testing adds additional complexity because additional parameters become important, such as shear force, boundary layer thickness, and flow state (laminar or turbulent).

It is also desirable to test the TPS under the exact flight profile with increasing and decreasing conditions as experienced in flight. All of the arc jet facilities in the U.S. capable of testing FTPS samples of appropriate sizes can only simulate a small portion of the flight profile during any single test and the LCAT facility is no exception. Performing a profile test adds complexity to the testing and since only a very limited portion of the flight profile can be simulated, testing is usually performed at constant heat flux, pressure, and shear conditions. Therefore, specific points from the flight trajectory must be selected for testing. To fully evaluate the TPS, numerous test are performed at various conditions to cover the full flight envelope.

Flight trajectories for the HEART and IRVE-3 vehicles are presented in Fig. 1 with the max heating, max pressure and max-max test points identified. For screening of flexible TPS materials in the early stages of development, a max-max test point concept was used where the arc jet test condition simultaneously simulated the maximum heating and maximum pressure the FTPS experiences during flight, but would result in shear forces larger than flight. This test methodology is an over-test of the FTPS and had the potential to result in false negatives, but provided a convenient method to rapidly screen multiple material systems while limiting the number of tests. The

concept is that if a material can successfully withstand the max-max test condition for the full heat load then it should be able to survive the max heat flux with reduced pressure, or the max pressure point with reduced heat flux conditions. As FTPS materials are down selected to fewer systems, the testing becomes more refined, focusing on simulating the maximum heating point and the maximum shear points individually at the full heat load. In addition, lower heating conditions are simulated for the maximum heat load which results in longer run times and more heat soak to the FTPS-structural support interface.

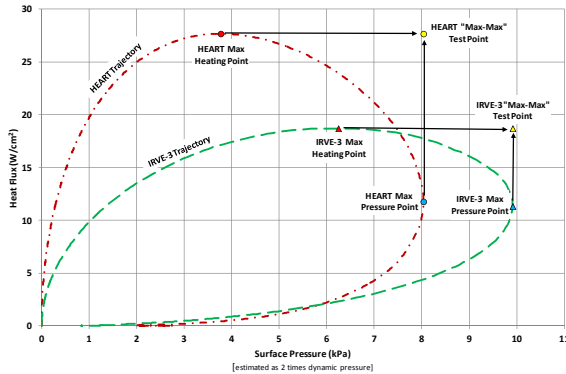


Fig. 1 HEART and IRVE-3 Flight Trajectories Showing Pressure and Cold Wall Heat Flux Relationship and Various Test Points.

Stagnation testing results in a pure thermal evaluation of the FTPS while shear testing evaluates the structural performance of the FTPS via aerodynamic shear forces while subjected to the proper thermal loads. Matching the correct pressure is important for FTPS. Unlike rigid, non-porous TPS, the test gas infiltrates the layers of the FTPS materials because they are porous. Testing of the individual material properties has shown that the interstitial pressure has a significant effect on the thermal conductivity of the material. Therefore, if the surface pressure is not matched during testing, the FTPS will not exhibit the correct thermal transport properties. In addition, if an oxidizing material is used as the insulator, the pressure is also important so the correct partial pressure of oxygen is present in the material. Otherwise, the oxidation characteristics of the material will not be properly simulated.

The test techniques that have been developed and demonstrated in the LCAT facility have successfully captured the thermal performance of the material at the interface of each material layer using thermocouples. In addition, pyrometers and infrared cameras are used to measure the surface temperature and temperature distribution. A temperature-time history of a FTPS is shown in Fig. 2.

### 3.2 Aeroelastic Ground Testing

Another area of ground testing and analysis is being initiated to evaluate the aeroelastic performance of the flexible material that can result from the aerodynamic forces during entry. The 8'-HTT at NASA Langley has been identified to perform initial evaluations of the FTPS under simulated aerothermal loads at elevated temperatures. A flat plate wedge test article has been developed with a 2-foot by 2-foot cavity in the centre of the flat plate where a TPS test article can be suspended and supported for evaluation. These initial tests will evaluate the test concept and provide initial data for analytical modelling efforts.

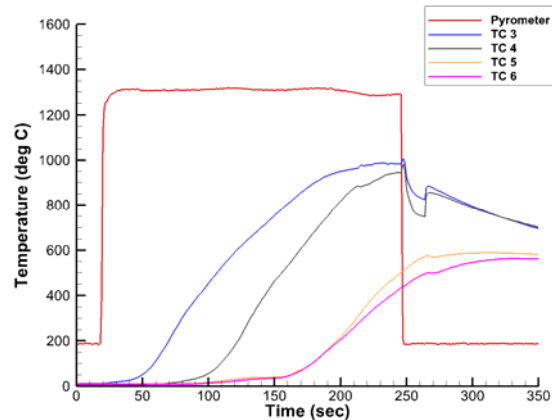


Fig. 2 Temperature-Time Results of 50W/cm<sup>2</sup> TPS, tested for 200 seconds, in the LCAT Facility.

Aerothermal ground testing development efforts are continuing to improve testing efficiency so more samples can be tested in a given period and development of new instrumentation techniques to improve thermal measurements and to measure the pressure at various locations in the FTPS.

### 4. THERMAL MODELING

Developing a thermal model to accurately predict the thermal response of a FTPS presents numerous challenges since multiple physical phenomena must be accounted for in the model. Insulation materials may decompose and char at high temperatures thus producing pyrolysis gas. Since all but the gas barrier are porous, flow through porous media and advection heat transfer must be included in the model. Solid conduction within the insulation materials is not straight forward since gas conduction and radiative transport within the material may be significant.

A thermal model to predict the temperatures through the thickness of a FTPS is being developed using the commercially available finite element software COMSOL®. COMSOL has the ability to include multiple, physical phenomenon and can be run on a multiprocessor Windows® platform or Linux computational cluster. COMSOL can also be

customized to include user defined differential equations or relations such as the Arrhenius equation for material decomposition.

The current version of the thermal model is a 1-D transient solution which incorporates measured thermophysical properties of all constituent materials as a function of temperature and pressure. COMSOL has the ability to solve transient heat transfer problems which include flow through porous media as well as radiation in participating media. The current model includes decomposition and pyrolysis gas flow but remains inactive until permeability data can be collected. Through analysis of thermal conductivity test data for the pyrogel 3350 and 2250, it was determined that a diffusion model for the thermal conductivity was adequate so radiation in participating media did not have to be included in the COMSOL model.

Several arc jet tests have been performed in Boeing's LCAT facility with various heat flux boundary conditions, surface pressures, and material layups. The first LCAT test series used panel test specimens at a 5° angle of attack with respect to the arc jet exit nozzle. The IRVE-3 vehicle heat shield is made up of two layers of Nextel BF-20, two layers of Pyrogel 3350, and one layer of AKK. A schematic of the IRVE-3 layup with thermocouple locations is provided in Fig. 3. A calculation with the COMSOL model is compared to LCAT test data for this IRVE-3 layup and is shown in Fig. 4. The heat flux for this particular run was 18.4 W/cm<sup>2</sup> at a pressure of 6.37 kPa and is consistent with the anticipated environment for the IRVE-3 vehicle. The arc jet flow was cut off when the back face thermocouple reached 450°C. Fig. 4 verifies that the current thermal model is predicting the response of the LCAT test data well. The largest difference occurs between the AKK and second layer of pyrogel 3350 at the TC4 location. The model over predicts this test condition by 54°C at the TC4 location and in general, looking across the multiple test conditions and layups, the model consistently over predicts the temperature at the AKK-pyrogel interface between 40-60°C. Clearly there is room for improvement and the near term focus will be to close the gap between the predicted and measured temperatures. While the current model does not predict the temperatures exactly, it is a conservative prediction and is an improvement compared against the previous version of the model.

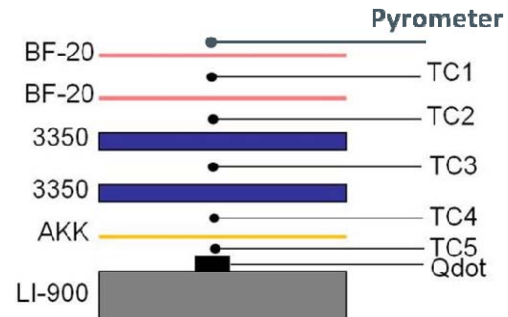


Fig. 3 IRVE-3 LCAT Layup

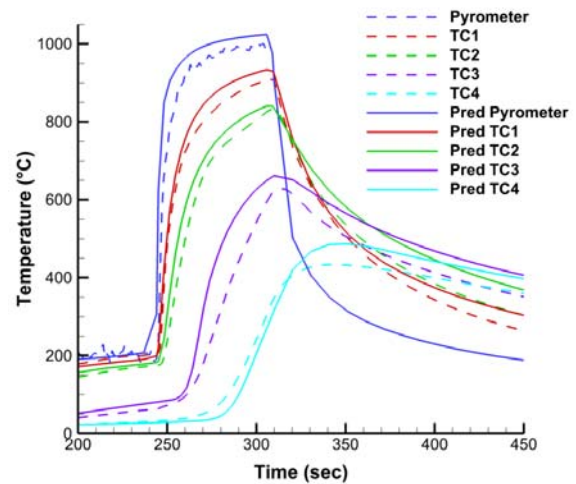


Fig. 4 COMSOL analytical model comparison to LCAT test data [4]

#### 4.1 Thermal Margin Management

In the design of a FTSP there are several sources of uncertainty. Uncertainty is present in the environment the FTSP is exposed, specifically, the trajectory the vehicle flies and the aerodynamic heating encountered. There is also uncertainty in the thermal response of the FTSP to that environment, in the measured material properties, and even the ground test data. The ground test data represents one uncertainty that feeds into the thermal model since the heat flux applied to the test samples can vary by up to 20% of measured values. There is also uncertainty in the test instrumentation, material being tested, and test conditions, all of which feed into the test methodology. The uncertainties associated with predicting the thermal response of a FTSP can be rather large because of the complex physical processes involved in pyrolysis gas generation and flow, char layer formation, and subsurface radiation. The requirement for the AKK not to exceed the bondline, which is the interface of the TPS to the support structure, temperature be maintained with 3σ probability necessitates that the TPS design must include some margin to account for the temperature prediction being uncertain. The typical way to apply

margin to account for these types of uncertainties is to increase the thickness of the nominally sized TPS. For FTPS materials, it is the insulator thickness or the number of discrete layers of insulator material that is increased. In previous heat shield designs, margin was calculated and applied using a root-sum-squared (RSS) approach. In the RSS approach, the trajectory, aerothermal, and thermal response uncertainties are compounded together to determine the margined TPS thickness. For example, the RSS method currently being used on NASA's Orion program is to run three different TPS sizing cases. The first case uses a  $3\sigma$  dispersed trajectory, the next case adds a  $3\sigma$  factor on heating to the dispersed trajectory from the first case, then the final case uses the dispersed trajectory from the first case but sizes the TPS to a  $3\sigma$  low bondline temperature. The resulting thicknesses from these three cases are root-sum-squared together and an additional thickness to account for recession and manufacturing tolerance uncertainty is added at the end to arrive at the final thickness. Although conservative, this method is flawed in two respects. First, this method cannot quantitatively determine the probability the margined TPS will exceed its bondline temperature limit. Second, it compounds  $3\sigma$  uncertainties from three different disciplines resulting in a TPS thickness that is well beyond a  $3\sigma$  thickness and hence adds unnecessary mass.

The approach being implemented by the HIAD program represents a paradigm shift in how thermal margin is calculated. [5] Instead of using an RSS approach, an end-to-end Monte Carlo simulation approach will be utilized. Since Monte Carlo simulations are routinely performed to calculate the  $3\sigma$  dispersed trajectory, in the HIAD approach, an aerothermal and thermal response Monte Carlo simulation will be indirectly linked to the trajectory Monte Carlo simulation. Each resulting trajectory from the flight mechanics code Monte Carlo simulation will be used by the COMSOL model for bondline temperature evaluation. In the COMSOL model, for each of these trajectories, the pertinent thermal model input parameters and the aerothermal loads will be statistically varied according to their own distributions and the bondline temperature will be determined. The COMSOL model will be re-run using the same trajectories and same statistical variation for the aerothermal and thermal parameter inputs using a different layup consisting of an additional layer of insulation material and will be repeated for several different layups. The result of these calculations will be the probability that each of the layups of increasing number of pyrogel layers will exceed its bondline temperature. The margined thickness is then the layup that gives the desired  $3\sigma$  or greater probability of exceeding the bondline temperature. The margin is then the difference between the nominally sized TPS

thickness and the calculated  $3\sigma$  thickness. The Monte Carlo process is illustrated in Fig. 5.

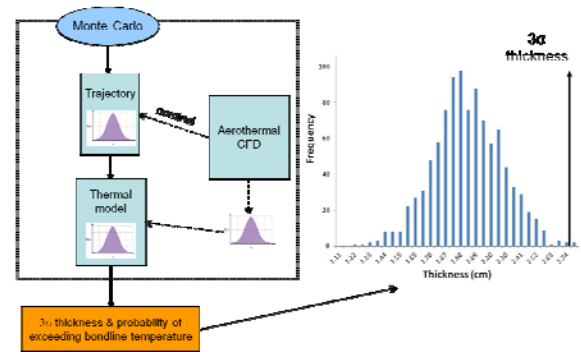
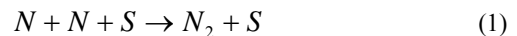


Fig. 5 Monte Carlo sizing process

## 5. MATERIAL CHARACTERIZATION

### 5.1 CATALYSIS

Surface catalyzed recombination reactions can increase local heating rates by up to a factor of two, and so knowledge of the catalytic efficiency of materials is critical for the design of thermal protection systems. In fact, current design practice invokes the fully catalytic assumption for conservative margin on the design. This practice will not change until greater progress is made in the reliable quantification of surface-catalyzed reaction efficiencies. With this perspective in mind, an investigation of the catalytic efficiency of thermal blanket materials that may be used to protect inflatable structures is underway. A noteworthy aspect of this investigation is the use of spatially resolved laser-spectroscopic techniques to measure species and temperature gradients in the reacting boundary layer above the material to quantify the catalytic efficiency. The investigation addresses both homogeneous and heterogeneous recombination in high temperature air plasma by measuring the atomic species (N and O) fluxes toward the surface, and differentiates NO production from  $N_2$  and  $O_2$  by direct detection of NO near the surface. All of the measurements are carried out in an Inductively Coupled Plasma (ICP) test facility at flight relevant boundary layer edge and material surface temperatures. Following a brief description of the facility and a description of the measurement approach, preliminary results are presented for surface-catalyzed reaction efficiency for the reaction:



Investigations of the surface-catalyzed reaction efficiencies of the candidate materials are carried out in the recently developed 30 kW ICP Torch Facility (Fig. 6 and Fig. 7) at the University of Vermont. [6]

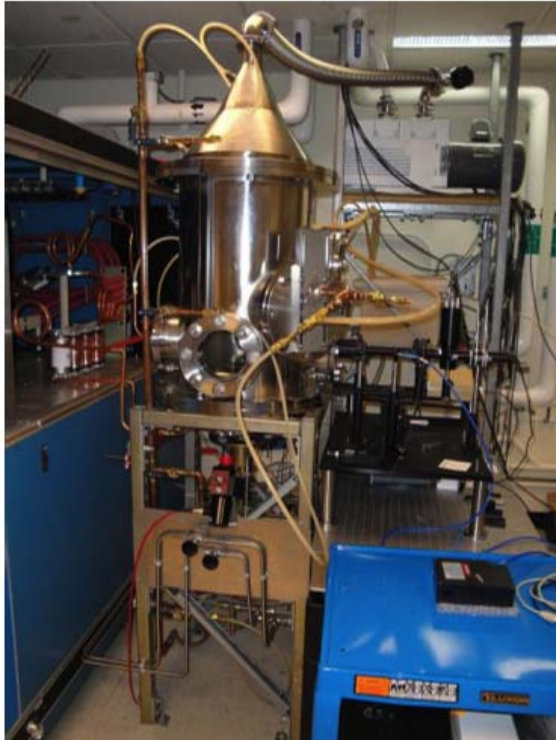


Fig. 6 ICP Torch Facility

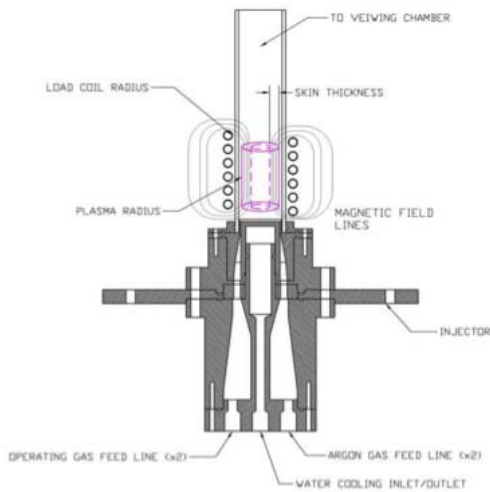


Fig. 7 Schematic of injector block and load coil

The 30 kW ICP Torch Facility at the University of Vermont was designed specifically to accommodate spectroscopic measurement techniques. Two large windows on either side of the facility are installed for simultaneous laser-beam and emission spectrometer access, as needed, with a third window installed perpendicular to the other two for laser-induced fluorescence (LIF) detection access. The laser system is not shown in Fig. 6, but a schematic representation of the optical configuration is shown below in Fig. 8.

A frequency-doubled Nd:YAG laser pumps a dye laser containing red dyes such as DCM. The output of the dye laser is then frequency tripled through a series of nonlinear optical crystals to generate ultraviolet light at wavelengths from 207 to 226 nm. These wavelengths access two-photon absorption transitions of atomic nitrogen and oxygen as single photon absorption transitions of nitric oxide. The UV output of the system is split in two paths; one going to a microwave discharge flow reactor and the second to the ICP Torch Facility. The flow reactor is operated at about 0.5 torr pressure and room temperature, and is used to provide an independently measured population of oxygen or nitrogen atoms, depending on the target species in the plasma flow. For each LIF measurement in the ICP flow, a simultaneous measurement is recorded in the flow reactor and the flow reactor measurements are then used to calibrate the ICP measurements. By moving the laser beam and detection optics in the ICP flow relative to the test material surface, the relative species concentration and translation temperature gradients are measured, as illustrated in Fig. 9. If additional measurements are made to quantify the fluorescence lifetime, beam diameter, and detection efficiency in both the test chamber and the flow reactor locations, then the relative species concentration measurements can be converted to absolute values. [7]

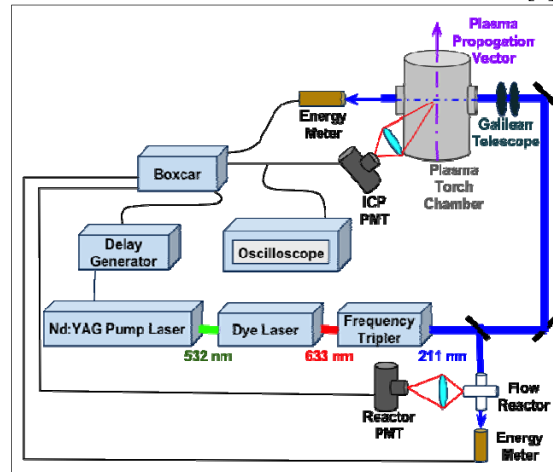


Fig. 8 Optical configuration for LIF measurements

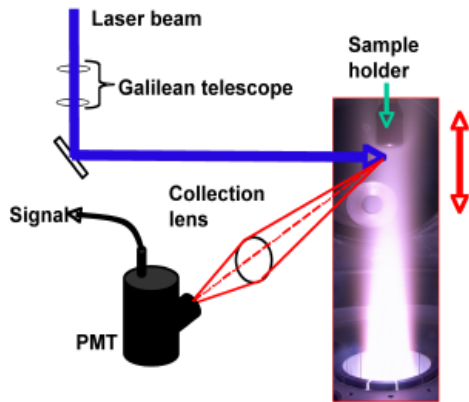


Fig. 9 Boundary layer survey illustration

At each measurement location, the laser is tuned over a narrow wavelength range around the absorption transition of interest. An example laser scan is shown in Fig. 10, where the relative two-photon LIF signal is plotted as a function of dye fundamental wavelength. This example is for the 211 nm transition for N. Two spectra are shown, and both are normalized by the square of the laser pulse energy, that is measured on both beam paths. In the figure, the narrower spectrum comes from the lower temperature flow reactor measurement, while the broader spectrum is obtained from the ICP plasma stream. The difference in line width for the two spectra is due to the Doppler broadening that scales with the square root of translation temperature. However, the line widths for both spectra also contain significant contribution from the laser line width. The laser contribution is determined from fitting a spectral model of the transition width at the known temperature conditions of the flow reactor (which is shown by the smooth curve), and this laser line width is then used in a similar model of the ICP spectrum to extract the temperature from the resulting Doppler width. In each spectrum, the integral of the LIF signal over wavelength is proportional to the nitrogen atom concentration. Thus, relative nitrogen atom concentrations are obtained directly from the spectral integrals of the LIF signals in the boundary layer over the material sample. Again, these relative measurements can ultimately be converted to absolute values if additional measurements are performed.

Determining the catalytic efficiency of flexible thermal protection materials is challenging. Unlike typical materials samples, flexible materials are porous, and so potentially offer a significantly increased surface area over solid materials. The overall approach for the test strategy is based on comparing the atomic species gradients for the flexible material with those of a solid surface with the same elemental composition. Thus, initial tests are focused on determining baseline atomic species density distributions in the boundary layer over the solid material that will comprise the substrate (SiC) for the flexible TPS sample tests.

Nitrogen recombination was addressed first owing to its chemical simplicity. All tests are conducted with the same nominal power supply conditions, a chamber static pressure of 160 torr, and a heat flux of about 60 W/cm<sup>2</sup>. Currently, the surface temperature for the SiC sample is on the order of 1300 K. Additional tests are performed using a quartz surface, which has a low catalytic efficiency [8], to have an indication of the relative catalytic efficiency of SiC.

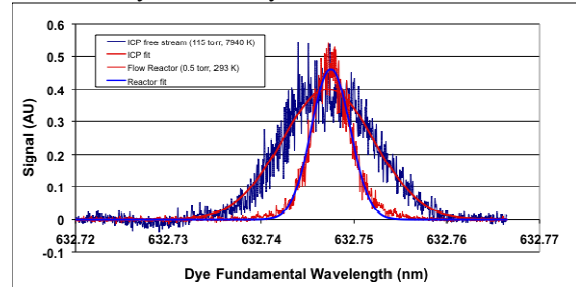


Fig. 10 Example N atom LIF signals from the ICP Torch Facility and flow reactor.

Temperature gradients for the material tests are compared below in Fig. 11 and are shown as a function of distance from the surface along the central stagnation streamline. Temperature values are determined from the spectral fits of the two LIF spectra as described above. We estimate the uncertainty in these measurements to be on the order of 500 K at present. The surface temperature of the water-cooled quartz is less than 500 K. The two temperature gradients agree reasonably well, indicating that the test conditions were quite similar for the two surveys. Also shown on the figure are temperature distributions that were calculated using the VKI Boundary Layer Code [9] for a non-catalytic surface.

A comparison of the relative nitrogen atom concentration gradients measured for the tests with the two materials is provided in Fig 12. As described above, the measured values are obtained from the spectral integral of the LIF signal, assuming that the detection configuration does not change with measurement location. The material is located at the left, indicated by zero on the horizontal axis. The measurements start from 2 to 5 mm away from the surface and the relative concentrations increase noticeably near the surface for the two materials. This is a strong indication that surface catalyzed reactions happen infrequently, if at all. If the materials were highly catalytic, then the consumption of atoms at the surface by recombination would enhance diffusion and the gradient would follow a downward trend toward the surface. As an indication of relative surface catalytic efficiency relative N concentration gradients calculated using the VKI Boundary Layer Code are shown by the group of red curves for different values of the surface-catalyzed reaction efficiency. There is little difference in the measured relative nitrogen

concentration gradient for SiC and quartz, indicating that the SiC also has low surface-catalyzed recombination efficiency.

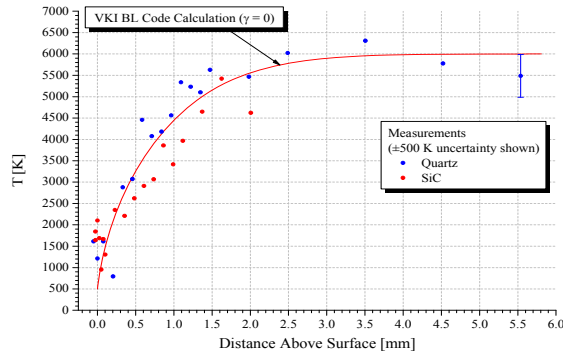


Fig. 11 Temperature gradients measured by two-photon LIF for the tests in nitrogen plasma.

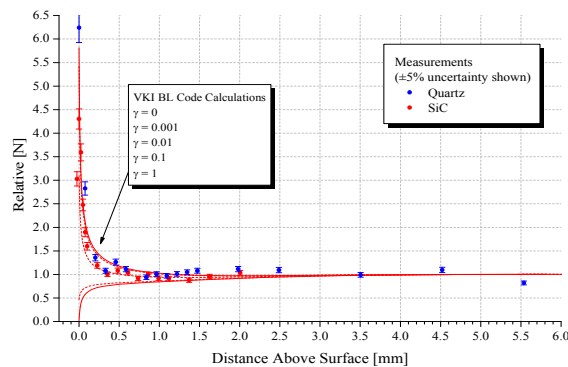


Fig. 12 Relative nitrogen atom concentrations in boundary layer above quartz and SiC.

Further tests to obtain the boundary layer species and temperature gradients for a catalytic material (nickel-coated copper surface) and for the solid piece that will be backing the flexible material sample are ongoing. These tests will complete the study of homogeneous nitrogen atom recombination. At that point, the laser dye will be changed to enable studies of oxygen recombination and NO production from recombination in air plasmas. Efforts are also underway to develop a water-cooled load coil to enable sustained operation at higher heating levels.

## 5.2 LIFECYCLE TESTING

The principal goal of this testing is to develop test techniques and procedures to characterize aging effects of HIAD FTPS subjected to flight conditions and configurations. These procedures must be flexible to allow for variation in evaluation parameters, but accurate so that the end-result is consistent for a particular set of evaluation parameters. In order to determine the effects of aging on key attributes, FTPS is developing characterization test methods to support flight configuration analysis and determination of acceptable material and system performance. Within

this secondary goal is the generation of materials' property data utilizing both virgin and aged materials. This effort is still in development, therefore what is presented here is what FTPS is notionally pursuing as a coarse TPS evaluation path being implemented through commercial contracts.

The FTPS will be used to demonstrate that the article has sufficient robustness to allow the inflatable aeroshell to be hard-packed for launch, remain stowed for several months, and then deployed without sacrificing performance. The final FTPS article will be used in a series of tests to assess the effects of manufacturing features, anomalies, and flight integration on thermal performance.

Products of lifecycle testing will include FTPS performance curves as a function of conditioning cycles and parameters (to be developed with NASA technical personnel) and a definition of a standardized aging regimen for future testing. The FTPS materials to be tested will be comprised of several layers held together with stitching threads. These material systems shall be conditioned (aged) as a complete system. Individual layers or elements may be separated out for characterization, depending on the method of characterization.

By studying the aging behaviour of several candidate FTPS stack-ups (material combinations), we add another figure of merit (aging tolerance) to the factors that guide the selection of the FTPS system for HIAD missions. This selection may not always be straightforward, since a particular stack-up may be less tolerant of compaction, but offer superior thermal performance, enabling lower packing density, thereby remaining "in the box" for system selection.

There are several steps that will occur in the development of a standard aging procedure. Each step will be documented at the conclusion of the development stage of this project such that a standardized aging regimen will be available for the community in further testing efforts.

The initial condition of the system will be characterized as accurately as possible, as each manufacturer of articles will have different construction methods.

In order to evaluate the tolerance of a particular material system to compaction, each stack-up will be folded according to NASA provided detail, then placed in a vacuum bag and compressed to the desired density for an extended period of time. Test articles will be subjected to 15, 20, and 25 psf compaction force for this element of testing, and then later evaluated by tensile testing to generate quantitative data and to define the "knee" at which point the property retention degrades significantly.

Subsequent to packing, each specimen will be transferred to the aging facility in order to simulate long term exposure to vacuum environment and



temperatures that would be experienced by an aeroshell prior to deployment. This facility, which requires development during this program, will be designed to handle multiple specimens simultaneously. The facility will operate at a vacuum of 1 Torr or higher and be capable of exposing the specimens to temperatures ranging from near liquid nitrogen (-192°C) up to 200°C. This condition and variants of this condition will be able to be held and monitored for a customer defined time period or a known thermal profile. The condition of the aged specimens will be recorded visually and as a function of mass loss subsequent to an aging regimen.

It is expected that variations in this procedure will include the length of total vacuum exposure, thermal soaks, both cold and hot, and packing/unpacking cycling (which would be done at the vacuum bagging stage).

Subsequent to aging, specimens will be tested as whole structures or as individual layers in a number of modalities, described briefly below.

### 5.2.1 FTPS Material Characterization

System and layer level coupons shall be evaluated for these performance parameters at varying levels of conditioning:

1. *Thermal conductivity* – Specimens, depending on the temperature range needed for testing, can be tested using either the guarded hot plate technique or the radial inflow technique. Both techniques are capable of controlling the amount of compression on the specimens to properly simulate the system as it will be in use.
2. *Tensile strength/elasticity* – Uniaxial tension tests will be completed on individual layers. The uniaxial test is suitable for a fabric and can be used for the outer layers and the inner AKK layer. In addition, a uniaxial test may become useful when assessing the effect of seams in the non-woven insulators (particularly when NDE post-test inspection is performed). These tests will be performed at ambient conditions to assess degradation due to aging. Data capture and post test NDE will be performed to help assess the mechanisms and effects of damage.
3. *Gas Permeability* – Permeability measurements will be made for structures and individual layers at relevant temperatures. The permeability of the materials in this program range from very low (AKK) to reasonably high (outer fabric layer and non-woven insulators). In addition, the temperature allowables for each of these materials is different. A recommended set of techniques will be developed to test these materials in relevant isothermal conditions. Particular attention will be paid to specimen loading techniques, assessment of damaged areas of aged specimens, and definition of appropriate flow rates.

4. *Areal Mass Loss and other NDE techniques*– As mentioned above NDE inspection, including mass loss, optical inspection, and scanning electron microscopy, if needed, will be performed on virgin and aged materials during the steps that will be developed for the aging regimen.

5. *Specific Heat* – Both adiabatic and ice calorimetric methods will be used to determine material specific heats. The combination of these two test techniques are capable of heat capacity determination through the usable temperature range of all of the materials used in HIAD systems. Specific heat should not be sensitive to damage or condition, and will be performed on virgin materials only for analysis inputs.

Both virgin and damaged materials will be tested to add to the database, in order to find where degradation no longer occurs, and help with the definition of an aging procedure. Specimen generation techniques will be developed for each test and recorded such that a consistent, known procedure is carried forward for future testing. Due to the handling sensitivity of some of these materials, this is a necessary component of the testing that must be understood for a successful aging regimen design.

## 6. ADVANCED TPS DEVELOPMENT

Aerogels are a class of porous solids, typified by having very small pore sizes and very high porosity, which makes them superior thermal insulators among other things. [10] Because silica aerogel monoliths are fragile and brittle, they are limited in application. [11] FTPS for inflatable aerodynamic decelerators for entry, descent and landing operations [12,13,14] require superior insulation which is also flexible and foldable as well as high temperature stable. Pyrogel composite aerogel insulation manufactured by Aspen Aerogels, which is the baseline insulation for HIADS, overcomes some of the issues with the brittle nature of silica aerogels by trapping the aerogel in a flexible, thermally stable batting. Nevertheless, aerogel particles continuously shed from the batting, leading to dusty environments and reduction of properties with handling over time. In response to the need for more flexible, non-shedding insulation, we have developed a family of three-dimensional cross-linked polyimide aerogels. The polyimide aerogels are potential candidates for these applications because they are stable up to 400 °C and thin films of the aerogels are flexible with good tensile properties. [15,1] The polyimide aerogels are fabricated from polyamic acid oligomers capped with anhydride, cross-linked with either 1,3,5-triaminophenoxybenzene (TAB) [18] or octa(aminophenyl)silsesquioxane (OAPS). [16] Different backbone structures in the oligomers lend different properties to the polyimide aerogels,

including mechanical strength, density, thermal stability and moisture resistance.

Both types of cross-linked polyimide aerogels (TAB or OAPS cross-linked), unlike other recently reported linear [17, 18] polyimide aerogels are produced with very little shrinkage during the processing. The cross-linked polyimide aerogels have thermal conductivity comparable to silica aerogels of similar density (as low as 14 mW/m-K) but are much stronger with a compressive modulus ranging from 1 to 100 MPa, and tensile strength as high as 8-9 MPa. One formulation of the flexible thin film aerogels was recently tested in a layered insulation system meant to simulate heat loads for planetary re-entry and was able to survive a laser heat flux load of 20 W/cm<sup>2</sup> for 90 seconds, while maintaining a 500 °C difference in temperature measured by thermocouples on the top and under the bottom-most insulation layers. [1]

In general, using ODA as diamine gives aerogels with the most flexibility but they are very moisture sensitive. Using PPDA as diamine gives the most thermally stable aerogels but they shrink the most during processing resulting in the highest density. Using DMBZ as diamine gives the lowest density aerogels because they shrink the least during processing and they are also the strongest, but they make brittle films. To design polyimide aerogels with the best combination of properties, we have examined properties of the OAPS aerogel using ODA in combination with either rigid diamine, PPDA or DMBZ as shown in **Error! Reference source not found.** In this regard, DMBZ or PPDA are used to replace up to 100 mol % ODA to optimize the mechanical properties, thermal stability and resistance to moisture of the polyimide aerogels. From this optimization study, it was determined that the best combination of properties is achieved with formulations using 50 % ODA and 50 % DMBZ as diamine. Fig. 13 shows pictures of a (a) 100% ODA aerogel and (b) a 50% ODA / 50 % DMBZ aerogel after exposure to moisture. As shown, the ODA aerogel shrivels in contact with water, while that made with 50% ODA and 50% DMBZ is unchanged. This combination is also nearly as flexible as those made from 100% ODA as seen in Fig. 13c.

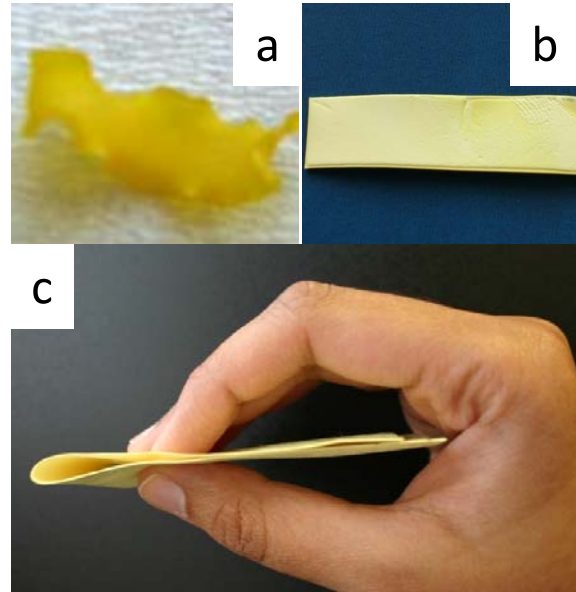


Fig. 13 a) Polyimide aerogel thin film made using 100 % ODA after soaking in water overnight and drying in air; b) thin film made using 50% DMBZ and 50% ODA after soaking in water and drying in air; c) demonstration of flexibility of a film made using 50% DMB

Thermally, the polyimide aerogels compare favourably with Pyrogel 2250 or Pyrogel 3350 silica aerogel blankets. Using thermal gravimetric analysis, Pyrogel 3350 begins to outgas at 350°C, while the polyimides have onsets of decomposition in excess of 500 to 600°C. In addition, the polyimide aerogels do not break down or shed dust particles when handled. Currently, additional enhancements to the properties of the polyimide aerogels by the addition of nanoclay or carbon nanotubes are under investigation.

## 7. CONCLUSIONS

A comprehensive overview of key work being performed within the Flexible TPS element of the HIAD project was presented. An overview of each Flexible TPS research and development activity, including ground testing, physics-based thermal modelling, age testing, margins policy, catalysis and materials characterization, and recent developments with new TPS materials. Ground testing utilizing the Boeing LCAT facility has yielded positive results with a large test envelope and methodology in both shear and stagnating conditions. Thermal modelling of TPS systems has advanced far beyond previous thermal resistance modelling methods, and is enabling advances which take into account permeability and pyrolysis effects. Each new modelling enhancement improves not only the state of the art for predictive capability, but also enhances TPS margins allowing for lower mass FTPS improvements over previous RSS methodologies for flight projects. Material characterization efforts are beginning to answer key

questions which allow for reducing incident heating requirements by virtue of quantification of outer fabric catalytic efficiencies, and a methodology for answering questions about handle-ability, and degradation of TPS performance for flight articles. Significant achievements towards improving polyimide based insulators have been made which are enabling higher bond line temperatures, and offer the promise of improvements to heat load capability in the future.

The original goal of FTPS, to investigate the viability and survivability of COTS materials in FTPS layups, has expanded over the last four years to include development, characterization, modelling, and manufacturing components. Over the next two years, Flexible TPS will be advancing manufacturing components by building a 6-meter TPS aeroshell, and will be advancing FTPS materials for high heat flux (>30 W/cm<sup>2</sup>) with a 3-meter advanced FTPS in 2013. Flexible TPS will explore large scale manufacturing, investigating alternate weaving technologies allowing for seamless aeroshells. Flexible TPS will continue to characterize materials, develop manufacturing standards, and margins requirements, which will enable future flight projects. Flexible TPS will continue to leverage advances in materials and explore potential on-ramp opportunities which allow for 75-100 W/cm<sup>2</sup> FTPS. Flexible TPS expects to begin testing of a 75 W/cm<sup>2</sup> TPS solution in 2013.

## REFERENCES

1. Del Corso, J. A.; Cheatwood, F. M.; Bruce, W. E.; Hughes, S. J.; Calomino, A. M. *Advanced High-Temperature Flexible TPS for Inflatable Aerodynamic Decelerators*, 21st AIAA Aerodynamic Decelerator Systems Technology Conference and Seminar, Dublin Ireland, May 23-26, 2011.
2. Del Corso, J. A., Bruce III, W. E., Liles, K. A., Hughes, S. J., *Thermal Analysis and Testing of Candidate Materials for PAIDAE Inflatable Aeroshell*, 20<sup>th</sup> AIAA Aerodynamic Decelerator Systems Technology Conference and Seminar, Seattle, May 4-7, 2009.
3. Bruce III, W. E., Mesick, N. J., Ferlemann, P. G., Siemers III, P. M., Del Corso, J. A., Hughes, S. J., Tobin, S. A., Kardell, M. P., *Aerothermal Ground Testing of Flexible Thermal Protection Systems for Hypersonic Inflatable Aerodynamic Decelerators*, 9<sup>th</sup> International Planetary Probe Workshop, Toulouse France, June 18-22, 2012.
4. Internal work performed by Roy Sullivan, and Eric Baker, NASA Glenn Research Center.
5. Wright, M. J., Bose, D., Chen, Y. K., *Probabilistic Modeling of Aerothermal and Thermal Protection Material Response Uncertainty*, Stanford Workshop on Uncertainty Analysis in Complex Multi-Physics Applications, July 25-26, 2008.
6. W. P. Owens, J. Uhl, A. J. Lutz, M. Dougherty, and D. G. Fletcher, *Development of a 30 kW Inductively Coupled Plasma Torch Facility for Aerospace Material Testing*, AIAA-2010-4322, 10th AIAA/ASME Joint Thermophysics and Heat Transfer Conference, Chicago, June, 2010.
7. D. G. Fletcher, A. J. Lutz, J. M. Meyers, W. P. Owens, and M. Dougherty, *Species and Temperature Gradient Measurements in the Boundary Layer Over ICP Test Samples*, Paper No. 2139316, 7th ESA Symposium on the Aerothermodynamics of Space Vehicles, Brugges, May 2011.
8. J. Marschall, *Experimental Determination of Oxygen and Nitrogen Recombination Coefficients at Elevated Temperatures Using Laser-Induced Fluorescence*, AIAA 97-3879, August, 1997.
9. P. F. Barbante, *Accurate and Efficient Modeling of High Temperature Non-equilibrium Air Flows*, PhD Thesis, Univ. Libre de Bruxelles, Bruxelles, (2001).
10. Hüsing, N.; Schubert, U. *Angew. Chem., Int. Ed.*, 1998, 37, 22-45.
11. Moner-Girona, M.; Roig, A.; Molins, E.; Martí'nez, E.; Esteve, J. *J. Appl. Phys. Lett.* 1999, 75, 653-655.
12. Braun, R. D.; Manning, R. M. *J. Spacecraft and Rockets* 2007, 44, 310-323.
13. Smith, B. P.; Tanner, C. L.; Mahzari, M.; Clark, I. G., Braun, R. D.; Cheatwood, F. M. Dillman, R. A.; *IEEEAC*, 2010, 1276.
14. Smith, B.P.; Clark, I.G.; Braun, R.D *Aerospace Conference, IEEE*, 2011, 1-16.5.
15. Meador, M.A.B.; Malow, E. J.; Silva, R.; Wright, S.; Vivod, S. L.; Guo, H. *ACS Appl. Mater. Interfaces*, 2012, 4, 536-544.
16. Guo, H.; Meador, M. A. B.; McCorkle, L.; Quade, D. J.; Guo, J.; Hamilton, B.; Cakmak, M.; Sprowl, G. *ACS Appl. Mater. Interfaces* 2011, 3, 546-552.
17. Rhine, W.; Wang, J.; Begag, R.; *Polyimide aerogels, carbon aerogels, and metal carbide aerogels and methods of making same*. US patent 7074880 B2, 2006.
18. Chidambareswarapatter, C.; Larimore, Z.; Sotiriou-Leventis, C.; Mang, J. T.; Leventis, N. J. *Mater. Chem.* 2010, 20, 9666-9678.