HIGH-TEMPERATURE STRUCTURES, ADHESIVES, AND ADVANCED THERMAL PROTECTION MATERIALS FOR NEXT-GENERATION AEROSHELL DESIGN

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ABSTRACT

The next generation of planetary exploration vehicles will rely heavily on robust aero-assist technologies, especially those that include aerocapture. This paper provides an overview of an ongoing development program, led by NASA Langley Research Center (LaRC) and aimed at introducing high-temperature structures, adhesives, and advanced thermal protection system (TPS) materials into the aeroshell design process. The purpose of this work is to demonstrate TPS materials that can withstand the higher heating rates of NASA's next generation planetary missions, and to validate high-temperature structures and adhesives that can reduce required TPS thickness and total aeroshell mass, thus allowing for larger science payloads. The effort described consists of parallel work in several advanced aeroshell technology areas. The areas of work include high-temperature adhesives, high-temperature composite materials, advanced ablator (TPS) materials, sub-scale demonstration test articles, and aeroshell modeling and analysis.

The status of screening test results for a broad selection of available higher-temperature adhesives is presented. It appears that at least one (and perhaps a few) adhesives have working temperatures ranging from 315-400^{o}C (600-750^{o}F), and are suitable for TPS-to-structure bondline temperatures that are significantly above the traditional allowable of 250^{o}C (482^{o}F). The status of mechanical testing of advanced high-temperature composite materials is also summarized. To date, these tests indicate the potential for good material performance at temperatures of at least 600^{o}F. Application of these materials and adhesives to aeroshell systems that incorporate advanced TPS materials may reduce aeroshell TPS mass by 15% - 30%. A brief outline is given of work scheduled for completion in 2006 that will include fabrication and testing of large panels and subscale aeroshell test articles at the Solar-Tower Test Facility located at Kirtland AFB and operated by Sandia National Laboratories. These tests are designed to validate aeroshell manufacturability using advanced material systems, and to demonstrate the maintenance of bondline integrity at realistically high temperatures and heating rates. Finally, a status is given of ongoing aeroshell modeling and analysis efforts which will be used to correlate with experimental testing, and to provide a reliable means of extrapolating to performance under actual flight conditions. The modeling and analysis effort includes a parallel series of experimental tests to determine TSP thermal expansion and other mechanical properties which are required for input to the analysis models.

INTRODUCTION

It is anticipated that the next generation of planetary exploration vehicles will rely heavily on robust aero-assist technologies such as aerocapture. The term "aero-assist" is used to describe the entire family of technologies that enable a spacecraft to use aerodynamic forces during atmospheric flight to manage and control spacecraft velocity. Aerocapture is a more specific term used to describe the procedure by which an entry vehicle uses atmospheric forces to decelerate into a desired orbit during a single atmospheric pass. Aerocapture is accomplished without the use of on-board propulsion, and is thus desirable because of the large reduction in fuel (mass) that must be carried to the target destination. The structural part of a spacecraft that is exposed to atmospheric loads and heating during entry is the aeroshell. Figure 1 depicts two example aeroshell geometries. Over the past several years, a variety of approaches

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have been considered to simultaneously achieve the goals of low aeroshell mass and reliable design. Conventional heatshield designs utilize an outer thermal protection system (TPS) material over a primary load bearing structure, generally of sandwich construction as shown in Figure 2.

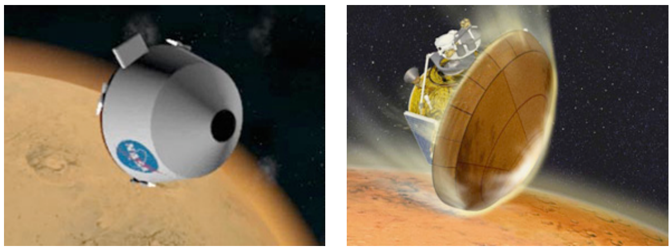

Figure 1. Blunt Cone and Sphere-Dome Aeroshells

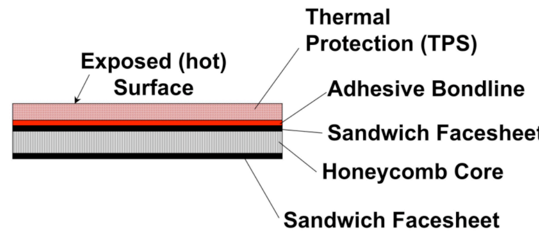

Figure 2. Aeroshell Structural Configurations

Sandwich aeroshells usually consist of either aluminum or graphite-epoxy facesheets with an aluminum honeycomb core. The thickness of the TPS material is directly related to the allowable temperature for the adhesive bondline. Generally speaking, aerocapture missions have assumed an allowable bondline temperature of 250^{o}C. The work summarized herein focuses on advanced structural systems, including adhesives and facesheet materials that may allow significantly increased bondline temperatures, and hence an important reductions in TPS mass. As this paper is intended to provide an overview the level of detail is necessarily cursory. In addition, although significant progress has been made, not all relevant test results are available at the time of this writing, and key elements related to large scale fabrication and testing are upcoming. Thus, this paper provides a status report that includes presently available results. Additional results and conclusions will be presented in the future.

HIGH-TEMPERATURE ADHESIVES

Conventional aeroshell design practice has generally assumed a maximum bondline allowable temperature of 250^{o}C. This assumption is based on both the maximum bondline allowable temperature, and on the degree of recession that is reasonable for the ablative TPS material. Higher bondline temperatures are only useful if more recession is allowed, which may not be practical for some TPS materials. The current program is examining the feasibility of using newer adhesive materials designed to withstand higher working temperatures. An advantage of aeroshell applications is that the adhesives must only withstand very high temperatures for relatively short time periods, and for only one, or at most a few, loading cycles. High-temperature adhesives, when combined with advanced ablative TPS materials that maintain their integrity at higher levels of recession, present the possibility of significantly reducing the thickness (and mass) of the TPS material. All mass savings translate directly into increased mass budgets for onboard science, and hence are desirable if they can be achieved without sacrificing reliability.

The current program began by considering the potential performance of approximately 20 adhesives at both room and elevated temperature. Through the consideration of vendor data, processing requirements, and expected working temperatures, this list was narrowed to 12 adhesives selected for actual testing. Screening tests of these adhesives are performed according to ASTM standard D3165 (Ref. 1) using tension loaded single-lap-shear joint assemblies. Tests using six specimen replicates are conducted at room and elevated temperatures of 315^{o}C (600^{o}F) and 400^{o}C (750^{o}F). A sketch of the ASTM test setup and a specimen prepared in a clamshell oven are shown in Figure 3.

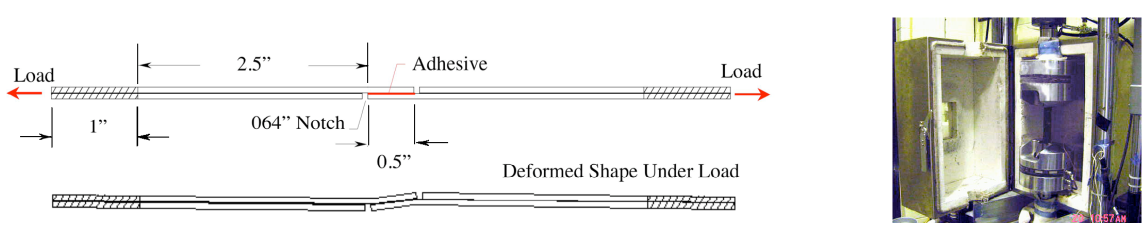

Figure 3. ASTM D3165 Lap Shear Specimen and Oven Test Configuration

Table 1 provides a list of the twelve adhesives that have either completed testing or will be tested in the near future. The table also gives manufacturer provided strength data (if available) and lap-shear results for those tests which have been completed.

Adhesive Designation	Manufacturer	Vendor Shear Strength	Test Results 75°F, 24°C (lbf)	600°F, 315°C (lbf)	750 °F, 400°C (lbf)
EA 9673	Loctite Corporation	2 ksi (75 °F) 600 psi (600 °F)	**1047.38**	**99.42**	**N/A**
EA 9369	Loctite Corporation	2.2 ksi (RT) 1.5 Ksi (550 °F)	**In Progress**	**In Progress**	**In Progress**
FM 680-1	Cytec Engineered Materials	3 ksi (75°F) 1.2 ksi (700 °F)	**1044.07**	**811.77**	**224.58**
HT 424	Cytec Engineered Materials	3.55 Ksi (75°F) 2.0 ksi (500 °F)	**In Progress**	**In Progress**	**In Progress**
PETI-8	NASA LaRC	2.5-3 ksi (75°F)	**2104.64**	**97.69**	**74.40**
RP-50	NASA LaRC	Not Available	**In Progress**	**In Progress**	**In Progress**
FM 57	Cytec Engineered Materials	3.6 ksi (75°F) 1.65 ksi (550 °F)	**1137.30**	**513.56**	**In Progress**
Q-Sil 1000	Quantum Silicones, Inc.	Not Available	**97.56**	**23.18**	**N/A**
PETI-5	NASA Langley	Not Available	**In Progress**	**In Progress**	**In Progress**
CV10-2568	NuSil Technology	Not Available	**In Progress**	**In Progress**	**In Progress**
EP45HTT	Master Bond Inc.	2.5 ksi (75°F)	**In Progress**	**In Progress**	**In Progress**
RTV 560	General Electric	Not Available	**In Progress**	**In Progress**	**In Progress**

Table 1. Adhesives, Available Vendor Strength Data, and Test Results to Date

Due to a variety of issues related to specimen surface preparation and testing, obtaining good test data has been a challenge. During the early part of the test program, many specimens exhibited adhesive failures as shown in Figure 4a, indicating failure to obtain a good bond at the adhesive to titanium-surface interface (Ref. 2). In the case of an adhesive failure, the adhesive material essentially pulls away from one or both sides of the test specimen. The desired "cohesive" failure mode is one that demonstrates failure of the adhesive itself. This mode is shown in Figure 4b. After several refinements to the specimen processing procedures, all recent tests have exhibited cohesive failures and the complete sequence of tests outlined in Table 1 is expected to be completed in early 2006.

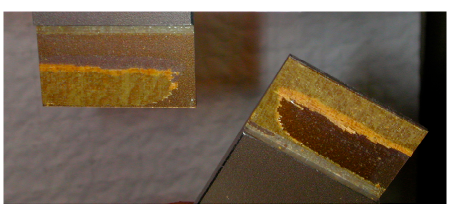

Figure 4a. Adhesive Specimen Failure

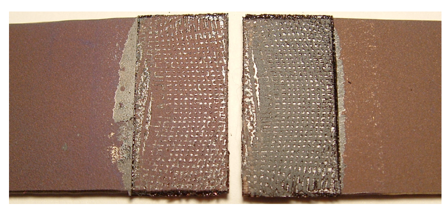

Figure 4b. Cohesive Specimen Failure

At the onset of this part of the program, it was known that few if any adhesives have demonstrated performance at temperatures higher than approximately 275^{o}C. This is born out by the results shown for EA 9673 and PETI-8 which indicate that these adhesives retain less than 10% of their strength at 315^{o}C and are unlikely to perform well at temperatures approaching 400^{o}C. The notable standout in Table 1 is the Cytec Engineering adhesive FM 680-1 which retains 77% of its room-temperature strength at 315^{o}F and 22% at 400^{o}C. Based upon cursory testing unrelated to this program, it is believed that the NASA Langley adhesive RP50 also has strong potential to perform well at the highest test temperature, however lap-shear results for this adhesive using the ASTM standard test are not complete at the present time. The actual adhesive strength required is highly dependent upon the structural loads associated with specific missions. However, for at least some applications, even somewhat large decreases in strength may be acceptable because the aeroshell may be lightly loaded, and the highest temperatures may occur for only a short time. The primary requirement will be that the adhesive material retain a portion (but not all) of its strength at elevated temperature.

HIGH TEMPERATURE COMPOSITE MATERIALS

The performance of several candidate high-temperature polymer-matrix composite (PMC) material systems is also being investigated. The systems discussed here consist of T650-35 composite fibers (selected because of their good high-temperature performance) combined in prepreg form with a high-temperature resin system. The resin system (or matrix) holds the fibers together after curing, and typically is the limiting factor for high-temperature applications. For the highest temperature applications, the most promising resin systems are advanced polyimides with working temperatures of at least 315^{o}C (600^{o}F). One such resin system is RP46, developed at NASA Langley Research Center (LaRC) in 1991. The resin has a glass transition temperature of 400^{o}C and is characterized by low toxicity, low moisture absorption, and good micro-cracking resistance. For its class, RP46 is relatively low cost and easy to process. A similar high-temperature polyimide is AFRPE-4 (Ref. 3). RP46, AFRPE-4, and several other candidate materials are being studied under the current program.

An extensive test program has been developed to evaluate candidate PMC material systems. The properties determined include tensile stiffness and strength, compressive stiffness and strength, in-plane shear modulus, and inter-laminar shear strength. Unidirectional laminates, cross-ply laminates, and eight-harness satin (8HS) woven fabric laminates have been tested using ASTM standard methods at room and elevated temperatures. Test coupons are cut from 12" x 12" composite panels fabricated on site at LaRC. Test temperatures, laminate types, and measured material properties are listed in Table 2.

Laminate	Test Type (ASTM Standard)	Property
$[0]_8$, $[90]_{16}$, or 4-ply weave	Tension (D3039) RT, 600°F, 650°F	Modulus E1 or E2, Strength
$[0]_{20}$, $[90]_{20}$, or 10-ply weave	Compression (D695) RT, 600°F, 650°F	Modulus E1 or E2, Strength
$[0]_{20}$, $[90]_{20}$, or 10-ply weave	Double-Notch Shear (D3846) RT, 600°F, 750°F	Inter-laminar Shear Strength
$[\pm 45]_{5s}$ cross ply	Tension (D3518) RT, 600°F, 650°F	In-Plane Shear Modulus

Table 2. High-Temperature Composite Laminates, Test Types, and Measured Properties

Tensile modulus of elasticity and ultimate tensile strength are determined from tension tests using ASTM standard D3039 (Ref. 4). Bonded electrical resistance strain gages are used to measure strain in the transverse direction and extensometers are used to measure strain in the axial direction. Detailed results for specific materials are not presented here. However, some results for a typical example polyimide material are shown in Table 3 where each result shown represents the average of six replicate tests. The results demonstrate that (for the example material) modulus does not degrade significantly for temperatures approaching 315^{o}C, whereas the tensile strength decreases by approximately 25%.

Temp (°C, °F)	Tensile Modulus (MSI)	Strength (KSI)
24, 75	17.7	198
315, 600	17.0	149

Table 3. Tensile Modulus (E1) of Typical High-Temperature Polyimide ($[0]_8$ laminate)

Compressive modulus of elasticity and strength are determined by performing compression tests using the method described by ASTM standard D695 (Ref. 5). End-loaded "dog-bone" specimens are mounted in a support fixture that prevents buckling and induces failure in the specimen mid-section. Typical results for one material are shown in Table 4. The results show a moderate decrease in modulus for a test temperature of 315^{o}C, where as the compressive strength is reduced by approximately 55%.

Temp (°C, °F)	Compressive Modulus (MSI)	Strength (KSI)
24, 75	16.2	125.5
315, 600	15.6	56.9

Table 4. Compressive Modulus (E1) of Typical High-Temperature Polyimide ($[0]_{20}$ laminate)

Inter-laminar shear strengths are determined in accordance with ASTM standard test D3846 (Ref. 6) by applying a compressive load to a double-notched specimen. Typical results are shown in plot form in Figure 4 for three different laminates. In addition to showing a decrease in strength at 315^{o}C (600^{o}F), this plot indicates significant sensitivity to increases in temperature above 315^{o}C.

Testing of high-temperature polyimide materials is nearly complete at the time of this writing. However, additional test data (particularly for temperatures above 315^{o}C) is still being taken. In addition, the current large-article fabrication program (described later) is considering alternate resin systems (modified polycyanates) that may perform well at intermediate temperatures (250-500^{o}C) but are easier to process than polyimides. As is the case for the adhesives, the actual material stiffness and strength required is mission dependent. Again, the primary requirement will be that the composite material retain a portion (but not all) of its strength at elevated temperature. The ability to determine structural requirements without "over designing" is one of the goals of the analysis efforts described later in this paper.

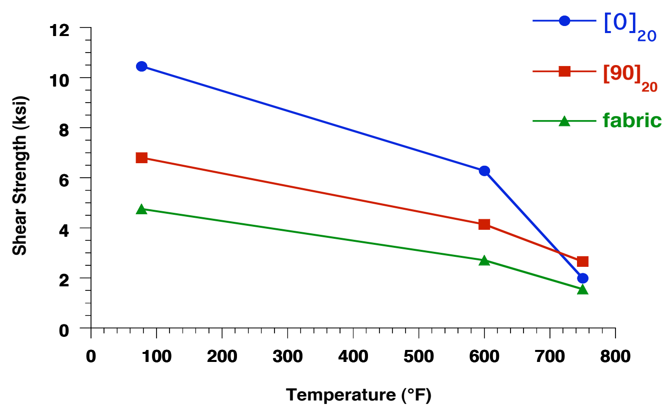

Figure 4. Effect of Temperature on Inter-laminar Shear Strength

POTENTIAL MASS SAVINGS

The mass savings that can be realized through application of the composite materials and adhesives described in the previous sections is dependent on specific mission requirements the ability of the TPS material to perform well and maintain its integrity at reduced thicknesses. Several advanced TPS ablative materials are being investigated by the Applied Research Associates (ARA) Ablatives Laboratory for application to aeroshell missions in general, and also for integration with the high-temperature adhesives and materials discussed in the preceding sections. Details related to the performance and testing of these ablators can be found in References 7 and 8. The ARA ablators are both silicone based and phenolic based with high levels of fibers for increased robustness and efficiency. Four materials from ARA's lightweight family of ablators have been selected as candidates for aerocapture of an orbiter at Saturn's moon Titan (one of the reference missions of NASA's in-space propulsion program), and have been part of an extensive test program conducted independently of the work presented here. Three of these ablative materials are silicone based, namely; SRAM-14, SRAM-17, and SRAM 20. The fourth ablator is phenolic based, namely; PhenCarb-20 (PC-20). Based upon the thermal resistance and density of these ablators, and the heating rates expected for aerocapture at Titan, analyses were conducted to determine the TPS thickness required for each material assuming allowable adhesive bondline temperatures of 250^{o}C (482^{o}F), 325^{o}C (617^{o}F), and 400^{o}C (750^{o}F). The results of these analyses are shown in Figure 5. The percentages shown in the figure indicate the percentage reduction in TPS thickness as compared to the thickness required at 250^{o}C (shown only for the SRAM-20 and PC-20 materials). The analyses indicate that for an allowable bondline temperature of 325^{o}C the required TPS thickness (and hence mass) is reduced on the order of 18%, and for an allowable bondline temperature of 400^{o}C, the thickness reduction approaches 30%. It is noted that the analysis presented here does assume some mass penalty to account for internal insulation that may be required to protect the spacecraft payload as the temperature of the aeroshell forebody is allowed to increase.

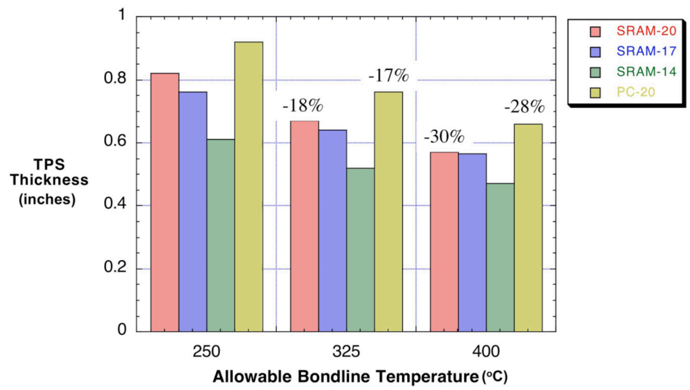

Figure 5. Required TPS Thickness as a Function of Allowable Bondline Temperature (Titan Orbiter)

EVALUATION CRITERIA

The precise selection of composite material, adhesive, and TSP material is again mission dependent. Table 5 summarizes some of the evaluation criteria that are being considered. The items in red are considered pass/fail criteria. These are specific criteria that must be met for any particular mission application. For example, bondline integrity must be maintained at or below a specific temperature. Similarly, for specific missions, it is possible to specify numerical requirements for shear strength and stiffness (modulus). The items in black in the table are either less quantitative, or aren't primary mission drivers. These however are useful for ranking competing design concepts when other factors do not discriminate between two approaches. For example, ease of processing can discriminate between two adhesive materials which are both capable of meeting mission requirements.

Adhesives	Composites Materials/ Resin Systems	Ablative TPS Materials
Lap-shear strength (Titanium)	Coupon-Level:	Characteristics:
Lap-shear strength (composite)	Tensile Modulus, Strength	Strength and CTE
Lap-shear strength (comp/TPS)	Compressive Modulus, Strength	Surface Integrity
Bondline Integrity	Shear Strength	Recession/Shape Change
Above, at target temperature	Wet Properties	Insulation Efficiency
Above, at very high temperature		Char and Pyrolysis Depth
Predictability/Repeatability	Panel/Subscale-Level:	Mass Loss
Ease of Processing/Cost	System Performance	Other properties:
Toxicity/Compatibility	Test/Analysis Correlation	Test/Analysis Correlation
Shelf Life	Manufacturability/Cost	Manufacturability/Cost
Vendor stability/reliability		Repairability

Table 5. Evaluation Criteria for Aeroshell Design Options

The adhesives and materials testing described earlier are intended to provide some of the data required to evaluate the criteria in the above table. ARA has evaluated many of the criteria related to specific TPS material characteristics. Other criteria in Table 5 will be evaluated through large panel and subscale aeroshell testing as described in the next section

LARGE PANEL AND SUB-SCALE AEROSHELL FABRICATION AND TESTING

A final and critical requirement is to demonstrate system level integration and performance via large panel and sub-scale aeroshell test articles. This work is intended to address issues associated with the integration of high temperature composite materials, high-temperature adhesives, and advanced TPS materials into representative aeroshell structural systems. Note that the incorporation of any one of these advanced technologies into the aeroshell design process represents a significant challenge. Over the next 18-24 months, fabrication and testing is planned for a series large-panel (24"x24") test articles and sub-scale aeroshell (1.0 meter diameter) prototypes. These test articles will be manufactured by ATK Space Systems in San Diego, CA. The test articles will consist of various primary structural configurations, adhesives, and TPS materials. Approximately twenty flat-panel 24"x24" test articles and four aeroshell prototypes will be built. The aeroshell prototypes will have curvatures representative of what is anticipated for full-scale flight vehicles. Figure 5 shows cross section and cut-away views depicting the approximate geometry of the 1-meter aeroshells that will be fabricated and tested.

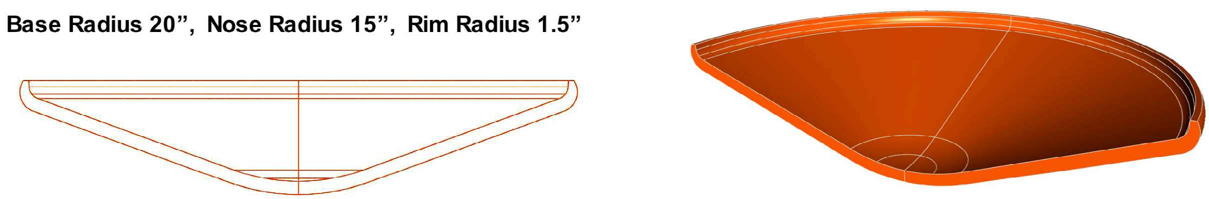

Figure 5. Geometry of 1.0-meter Aeroshell test Articles

The large panels and 1.0-meter aeroshell test articles will be subjected to thermal radiation testing at the Sandia Solar Tower test facility at Kirtland AFB, New Mexico. This facility uses a large array of solar collectors (heliostats) to generate radiation fluxes of up to 300 W/cm^2 uniformly over surfaces as large as 1 meter. Testing of such large specimens in not practical or possible using arc-jet facilities. The Solar Tower facility consists of 220 heliostats and a 200-ft tall tower that receives the collected energy at one of several test areas. The total collection area of all 220 heliostats is 88,000 ft^2 or roughly 2.2 acres. For the test articles described here, the specimens will be mounted to specially designed fixtures placed on the roof of the solar tower. The fixtures can be raised and lowered to allow for test setup and placement of the test articles into the concentrated "spot" of energy produced by the collection of heliostats. Because the heliostats are computer controlled, the test radiation can be shaped in terms of both intensity and time. This allows for rapid heating of the test article bondlines at rates that are typical of realistic entry conditions and would be difficult to duplicate using other test methods.

AEROSHELL MODELING AND ANALYSIS

In addition to the fabrication and experimental work already described, a significant effort is also underway to develop details analytical models that can used to both verify ground test results, and to extrapolate performance conditions to actual flight vehicle conditions. These models should be capable of predicting (for ground testing and flight) critical parameters that include, aeroshell deformation under thermal and dynamic loading conditions, bondline stresses/strains, and stresses in the facesheets and core of the aeroshell primary structure. Of particular interest is the prediction of bondline shear stresses that may be induced because of aeroshell deformation and/or mismatches between the coefficients of thermal expansion of the aeroshell primary structure and the bonded TPS material. The analysis portion of the program focuses on the large-panel and subscale test articles presented in the previous section. This portion of the program is a work in progress. Figure 6 provides a preliminary look at one of the finite element models that is currently under development for the sub-scale aeroshell prototypes. The boundary conditions of the model are capable of simulating either flight conditions or the mounting configuration that will be used for Solar Tower testing (most likely a three point statically determinate mounting fixture attached to the back face of the aeroshell)

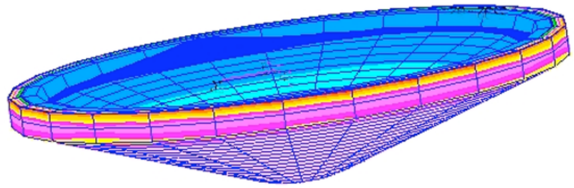

Figure 6. Aeroshell Finite Element Model

An important aspect of the modeling and analysis effort involves obtaining realistic input values for the mechanical and thermal properties of the materials, and for the time histories of the temperatures seen at various locations through the thickness of the aeroshell structure. Many of the mechanical properties for the composite facesheet and honeycomb core materials are available either in the open literature or can be obtained from the manufacturer. Some of the properties of the ablative TPS materials are not as readily available. Notable among these are the TPS material coefficients of thermal expansion (CTE). To obtain this information, a small experimental and analysis effort has been undertaken to both predict and measure the CTE of several TPS material specimens. A finite element model of the TPS material has been generated and is shown in Figure 7.

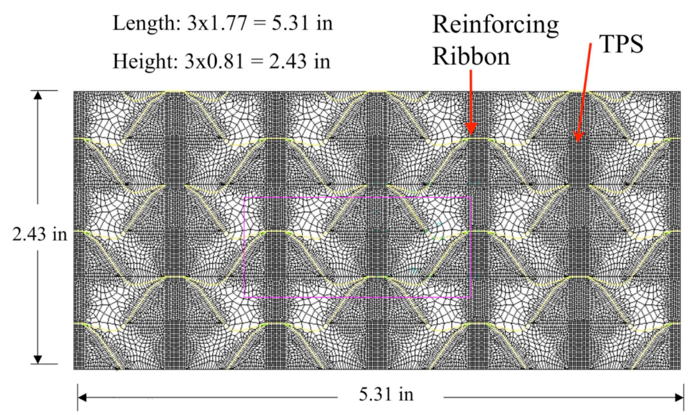

Figure 7. TPS Finite-Element Model for Predicting TPS/Ribbon CTE

The model includes both virgin TPS material and the reinforcing ribbon that is used to stabilize the material. The two-dimensional model is capable of predicting the "equivalent" CTE of the combined TPS and ribbon material. Predictions from this model have been verified using the experimental setup shown in Figure 8.

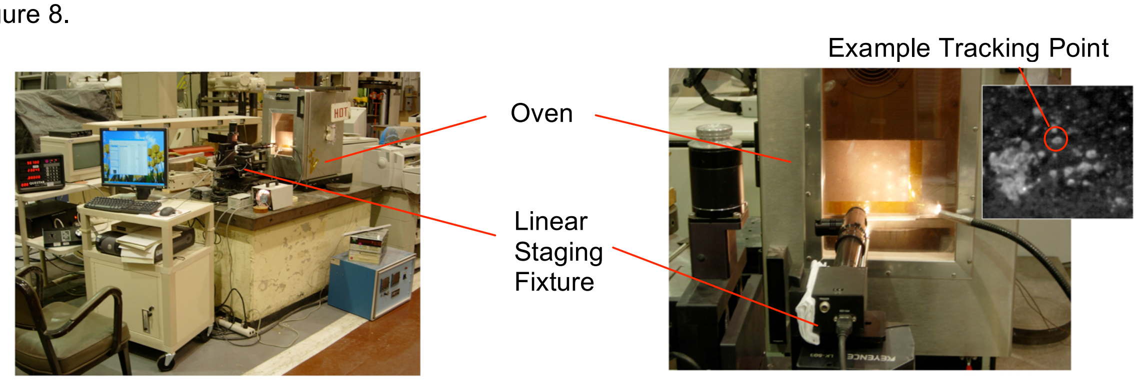

Figure 8. TPS Finite-Element Model for Predicting TPS/Ribbon CTE

In this test, TPS coupon samples are placed in an oven and heated slowly to various temperature intervals. A laser pointing device mounted to a precision 2-dimensional linear staging fixture is used to track the movement of several predetermined points on the surface of the TPS specimen. By inspecting the movement of the points over various temperature regimes, it is possible to determine the CTE of the TPS material. The goal is to calibrate and verify the finite-element model shown in Figure 6, with enough confidence such that the model can be used to predict the CTE of different TPS materials without experimental testing in every case. Thus far the analytical model and experimental measurement scheme have been used to successfully predict and measure the CTE of SRAM-20 TPS material, with an agreement between analysis and test of approximately 15%.

SUMMARY AND CONCLUSIONS

This paper has provided a summary of an ongoing development program consisting of several components intended to advance the state-of-the-art of aeroshell design. Test results to date indicate that high-temperature composite resin systems can yield acceptable performance at temperatures approaching 315^{o}C (600^{o}F) or higher. These material systems should be suitable for the primary sandwich facesheets of future aeroshell structural systems. Test results to date for a range of high-temperature adhesives indicate that working temperatures approaching 400^{o}C (700^{o}F) are feasible. It is very likely that adhesive working temperatures significantly in excess of the commonly assumed 250^{o}C allowable are feasible. This is especially true when the short time durations associated with aerocapture heating are considered. Analysis indicates that by combining high-temperature composite materials and adhesives with new families of ablator materials, it may be possible to reduce aeroshell TPS mass by as much as 30%. However a wide range of evaluation criteria were briefly presented, all of which must be considered in order validate the feasibility of new aeroshell design approaches. A critical step in this process will the thermal radiation testing of several large-panels (24"x24") and four 1.0-meter aeroshell prototypes at the Sandia Solar Tower Test Facility. The facility is capable of subjecting the test articles to large heat fluxes that produce heating rates representative of what is expected under flight conditions. Tests of the large articles will be used to validate analytical models, to verify the manufacturability of large components using new materials, and to demonstrate bondline integrity under severe heating conditions.

ACKNOWLEDGMENTS

The work presented in this paper was originally awarded under the aerocapture element of the ROSS (Research Opportunities in Space Science) 2002 NASA Research Announcement. It is part of NASA's In-Space Propulsion (ISP) Program, with overall management carried out by the Marshall Space Flight Center (MSFC). The management team at Marshall consists of Michelle M. Munk (Lead Systems Engineer for Aerocapture) and Bonnie F. James (Program Manager). Adhesives testing under this program is being carried out by Wichita State University under the direction of Dr. Charles Yang. The lead engineer at ATK Space Systems for test article fabrication is Mr. Mark Pryor. The entire effort has significant scope and all progress has been the result of significant collaboration by everyone involved.

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