EDL HEATSHIELD EXPERIMENTS WITH DUAL-LAYER ABLATORS, ADVANCED MATERIALS AND VARIABLE HONEYCOMBS

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ABSTRACT

This paper discusses the Ablatives Laboratory's (ABL) on-going project to investigate ways to improve robustness and efficiency of charring ablators against planetary entry heating. Interim results for a three-year project sponsored by the NASA Hypersonics Program are given. As an example, we are investigating the application of silicon-carbide (SiC) fibers and microballoons in carbon-phenolic ablators as a means of reducing char recession from oxidation. And these same fillers are being applied to silica-silicone ablators to reduce recession from surface "melting", microballoon coalescing, and melt flow. (New technology SiC microballoons, specifically for ABL's use in ablators, are being developed by our industrial partner, Trelleborg Corp.) In addition, we are preparing and testing dual-layer ablators as a way of increasing surface layer density and durability but, by using low-density sublayers, minimizing net area weights. Also being studied are honeycomb reinforcement cell-size and bulk density as pertains to their effects on ablator performance and system weights. Final results for this project will be available upon completion in mid 2012.

1. INTRODUCTION

Today's challenge is to make ablative heatshield systems more robust yet lighter and more efficient for thermal protection of large Entry, Descent, and Landing (EDL) vehicles. Better ablators are especially important for manned exploration of Mars. One approach to try to meet this challenge is to improve upon heatshield systems already available. The three-year ABL technology project designed around this goal is focused on three elements: 1) investigate new resin systems as well as new ablator constituents to replace less-durable fillers currently in use; 2) fabricate and test dual-layer ablator systems with a higher-density, more robust top layer over a lower-density, more insulative sublayer of the same chemistry; and 3) produce, test and evaluate honeycombs with a range of cell-size and weight to better understand the dependence of ablator performance on reinforcement configurations. Primary ablator performance testing consists of: 1) arc-jet iso-q stagnation testing using the NASA/ARC's Interaction-Heating-Facility (IHF); 2) arc-jet aeroshear testing with a swept-cylinder design using the IHF; and 3) concentrated solar radiation testing using the Sandia Labs Solar Tower (ST) facility. The focus of this paper is summarizing project design and objectives, test methods and preliminary findings, as well as on-going work with an array of ablator experiments.

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2. PROJECT DESIGN AND OBJECTIVES

We are investigating newly available advanced ablator ingredients such as silicon-carbide (SiC) microballoons and fibers as well as polymide resin for enhancements to ablative system performance. Like phenolic, polyimide resin is a thermoplastic with high char yields. But polyimide is thermally stable to higher temperatures compared to phenolic. Its point of substantial degradation (pyrolysis) is roughly 100ºC higher than that of phenolic as shown in Fig. 1. Therefore, the bondline temperature allowable for a polyimide heatshield can be higher than phenolic. And a higher allowable bondline temperature results in a thinner TPS and reduced weight. Lower surface recession/erosion and lower in-depth temperatures are also expected for a given heating environment.

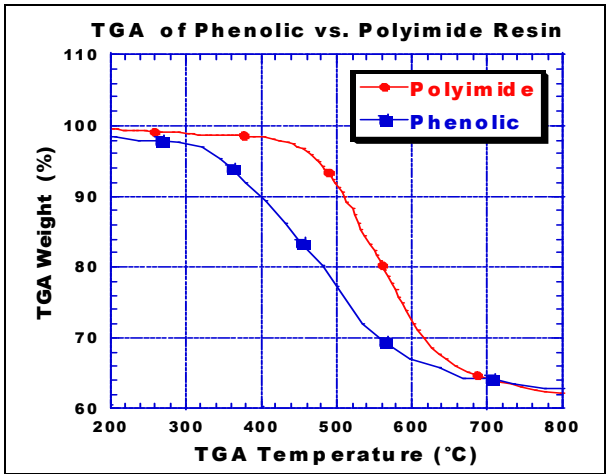

Fig. 1 – Data from Thermogravimetric Analysis (TGA) of Phenolic Resin and Polyimide Resin

Silica and carbon constituents in the form of fibers and microballoons have been used for decades. The viscosity of silica will drop and silica constituents will begin to "melt" and coalesce at heating rates as low as 130 W/cm^2. Also, carbon ingredients will combust in the presence of available oxygen. However, SiC ingredients are oxidation resistant and stable up to ~2700°C [1]. Therefore, SiC ingredients and poly-imide resin are expected to produce char layers that are more robust compared to those of existing phenolic and silicone ablators. A more robust char means less surface recession and less surface roughness that might otherwise augment atmospheric heating. Unitech Corp. manufactures the RP-46 high-temperature polyimide resin, Nippon Carbon Co. produces the Nicalon SiC fibers, and Trelleborg Corp. supplies developmental versions of SiC microballoons (specifically manufactured for this project).

Another project task, done in parallel, consists of producing and testing dual-layer thermal protective systems (TPS) based on established ablative materials. Shown in Fig. 2, these have a dense, robust surface layer with a low-density sublayer that is an efficient thermal insulator. Successful mating/adhesion of the layers is the biggest challenge in a dual-layer system. For the ABL dual-layer ablators (e.g., P28/P15), each layer has the same chemistry and uses the same resin system. This makes a simultaneous cure (same pressure and cure temperature) possible to "knit together" the layers with overlapping fibers secured within the resin. A dual-layer ablator may provide the lightest weight TPS solution for High-Mass Mars Entry Systems (HMMES) vehicles that will experience high levels of heating, aerodynamic shear and shock-layer radiation during aerocapture followed by a second phase of heating at lower levels during entry.

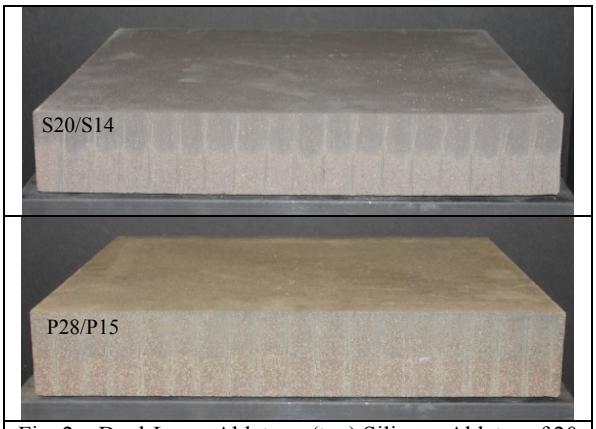

Fig. 2 – Dual-Layer Ablators: (top) Silicone Ablator of 20 lb/ft^3 over 14 lb/ft^3 (S20/S14) and (bottom) Phenolic Ablator of 28 lb/ft^3 over 15 lb/ft^3 (P28/P15).

In addition, we are producing and testing different cell-size honeycombs to understand optimal reinforcement designs for maximized ablator performance. The range of cell sizes and resulting bulk densities is shown in Fig. 3. (And a resin wet coat increases these bulk densities by another 25-35%.) Honeycomb composite (i.e., quartz phenolic) is a higher-density material with higher thermal conductivity than the typical ablators of interest that would fill the honeycomb for HMMES heatshields. Small-cells may prove to be beneficial for lowering surface recession from mechanical aeroshear erosion by containing char layers in smaller pockets. However, because of the honeycomb material's higher conductivity versus the ablator compound within it, small-cell honeycomb may have a bigger effect in degrading insulation performance compared to large-cell honeycomb. Therefore, honeycombs need to be optimized for ablator producibility and performance without adding unnecessary mass to the EDL vehicle's TPS. Typically larger-cell honeycomb is preferred over smaller-cell honeycomb due to ease in manufac-turing, provided adequate reinforcement is achieved.

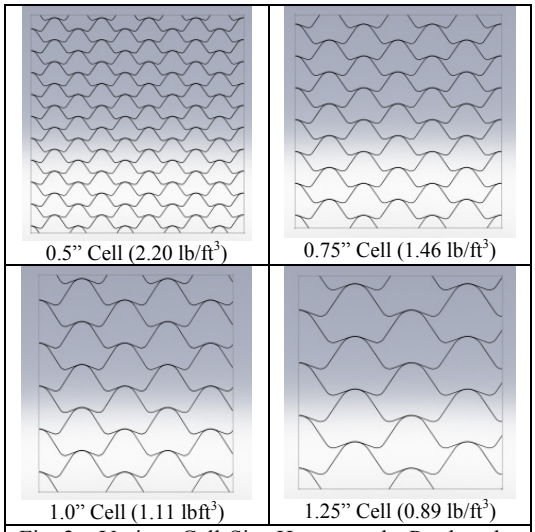

Fig. 3 – Various Cell-Size Honeycombs Produced by ABL for use in Experimental Samples

Our primary project plans are the following: 1) experiment with processing and manufacturing techniques to produce ablator panels for making experimental samples; 2) prepare instrumented test samples of these ablative systems for evaluation testing of their performance in high heating environments (arc-jet stagnation and aeroshear and solar thermal radiation); and 3) compare results for the experimental samples versus their matched controls. Our technical approach is to use established silicone and phenolic ablators as "test beds" for investigating new ingredients and new ablator designs (single parameter excursions on existing systems). For example, to evaluate a new fiber, we are supplanting the standard fiber in one or more of our ablator formulations, and then testing ablation performance of the resulting product versus the original (control) to look for and quantify potential improvements.

3. TEST METHODS

Three types of tests were conducted to evaluate the experimental test samples: stagnation and aeroshear testing at the NASA/ARC IHF arc-jet, and concentrated solar radiation testing at the Sandia Labs Solar Tower (ST) facility.

Stagnation testing (Fig. 4) focused on high heating rates in the range from 500 W/cm^2 to 1000 W/cm^2 and pressures between 0.25 atm and 0.75 atm. All samples were 4.0-in. diameter with a curved test surface of 4.0-in. radius (iso-q shape) and contained two type-K thermocouples in the bondline to an aluminum backplate. Samples were 1.125 - 1.250-in. thick. Our primary interest for this series pertained to ablator robustness (strong and stable char layer) under these severe, high heat-flux conditions. Experimental samples were compared to their controls for surface recession at the stagnation point, mass loss, and thermocouple temperature responses.

Fig. 4 – Experimental Phenolic Ablator Sample Containing SiC-Coated Carbon Microballoons – During Stagnation Testing in NASA/ARC IHF Arc-Jet

For the aeroshear test series we used the swept-cylinder test method (developed by NASA/ARC's Dave Driver) [2]. Ablator sample dimensions were approximately 6.0 x 3.0 x 0.8-in., with a curved test surface of 4.0-in. radius (simple curvature). Ablator thickness was 0.80-in. measured at the center ridgeline. Each sample was instrumented with two bondline thermocouples (ablators were bonded to an aluminum backplate). The primary interest in conducting aeroshear tests was relative surface erosion between the various samples and the reduction of surface erosion by different size honeycombs and different advanced ablator ingredients. Total mass loss was measured for each test condition. Mass loss numbers for advanced ingredient samples were compared to control samples, and mass loss numbers for various cell-size honeycombs were compared to each other. Sample backface temperature rise and ablator efficiency as a thermal insulator are also important. Thermocouple responses were measured and compared. Test conditions ranged from 100-350 W/cm^2 with pressures below 0.3 atm. Shear stresses were between 5.0 and 11.0 lb/ft^2 according to Computational Fluid Dynamic (CFD) simulations using the Data Parallel Line Relaxation (DPLR) code developed by NASA/ARC [3].

Solar radiation testing took place at the Sandia ST facility in New Mexico. The ST has a large field of 220 heliostats with reflective mirrors that track the sun and focus collected energy to targets on top of the tower. Total area of mirrors is 2.2 acres or 88,000 ft^2. Spot size on the target of less than 1.0-m (39.4 in.) in diameter can be achieved. TPS test articles within this concentrated spot can receive thermal radiation fluxes up to 300 W/cm^2 (for ideal conditions). For our tests, samples were 5.0-in. in diameter and were exposed to solar heat fluxes of 100-150 W/cm^2. Experimental sample thicknesses measured 1.2-in. for variable honeycomb and advanced ingredient samples and 1.95-in. for dual-layer samples. The purpose of these tests was to look at relative insulation performance and radiation-penetration depth between experimental samples and controls. Also, samples were evaluated for surface ablation responses and their resistance to spallation.

4. PRELIMINARY FINDINGS AND CONTINUING EFFORTS

We are only partially through the full scope of this project (in June 2011), thus about half of the sample sets have been tested to date. Presented below are some initial observations from the first round of completed testing. A second round of shear, stagnation, and solar radiation testing is scheduled for the third year of our effort.

Dual-layer ablators were only tested at the ST facility because of the need for one-dimensional heat transfer on a larger 5.0-in. diameter sample with a flat surface. (arc-jet stagnation samples were only 4.0-in. in diameter with an iso-q surface). Bondline thermocouples at 1.95-in. below the surface of phenolic and silicone dual-layer samples showed lower temperatures than bondline thermocouples of the control samples (Fig. 5). Bondlines for the controls were located at a depth with the same TPS area weight as the dual-layer samples (1.55-in. below the surface for P28 control and 1.64-in. below the surface for S20 control). Also, thermocouples between layers (0.95-in. below surface) of dual-layer phenolic and silicone samples showed higher temperatures versus their respective control sample's thermocouples (at same 0.95-in. depth). No separation of layers or spallation was observed. Samples still need to be sectioned to fully evaluate depth of radiation penetration.

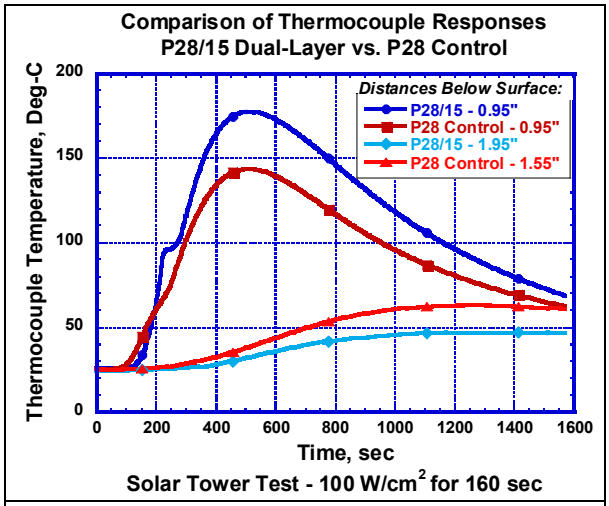

Fig. 5 – Comparison of Thermocouple Responses for Dual-Layer Phenolic Sample and its Phenolic Control – ST Radiation Test Conducted at 100 W/cm^2 for 160 sec.

ST tests showed a trend in bondline temperatures for various cell-size honeycombs. Samples with smaller-cell honeycombs showed somewhat higher thermocouple temperatures than those with larger-cell honeycombs. Most likely this is attributed to higher thermal conductivity of honeycomb material relative to the contained ablator. Such a trend was not as distinguishable in shear or stagnation testing. But shear tests showed less erosion in regions where honeycomb ribbons were more dense/close together. In places where ribbons were spread farther apart, deeper erosion channels developed (Fig. 6). These are preliminary results and completion of sample testing will provide more definite performance conclusions.

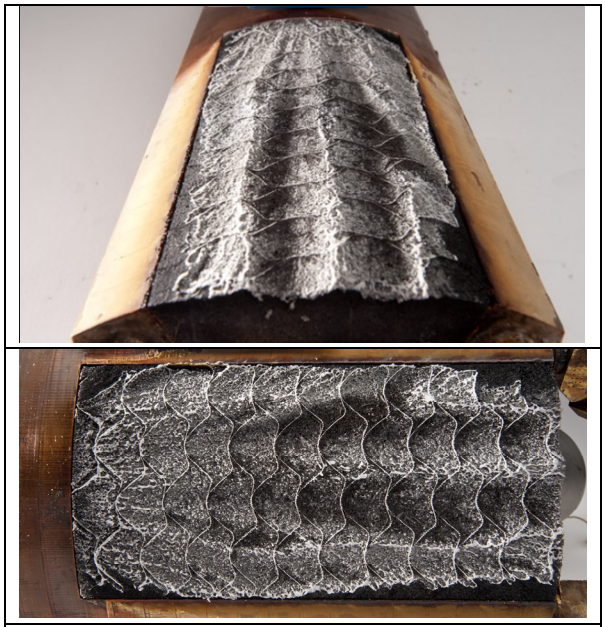

Fig. 6 – Post-Test Photos of Phenolic Aeroshear Sample with 0.75" Cell-Size Honeycomb – Tested at 110 W/cm^2 with Shear of Approximately 6 lb/ft^2 for 60 sec.

Trelleborg has made significant advancements in their production of SiC microballoons. Early microballoons received at the start of this project were two component: a SiC coating applied to a carbon micro-balloon substrate. Ablator samples made from the early microballoons did not show improved ablator performance. These samples had higher thermocouple temperatures, more recession and more mass loss compared to their control samples. During testing, we observed a greater level of particulates departing from the ablator surface of the early SiC samples (Fig. 4). We believe this was caused by the SiC coatings separating from their carbon microballoon substrates, thereby weakening the adhesion of this constituent within the resin matrix (Fig. 6). Aeroshear testing was not performed on samples containing the SiC-coated carbon microballoons for this reason. Trelleborg has since developed and delivered new, fully SiC micro-balloons (Fig. 7), which we expect will show improved ablator sample performance versus their respective control samples in the next round of ablation testing.

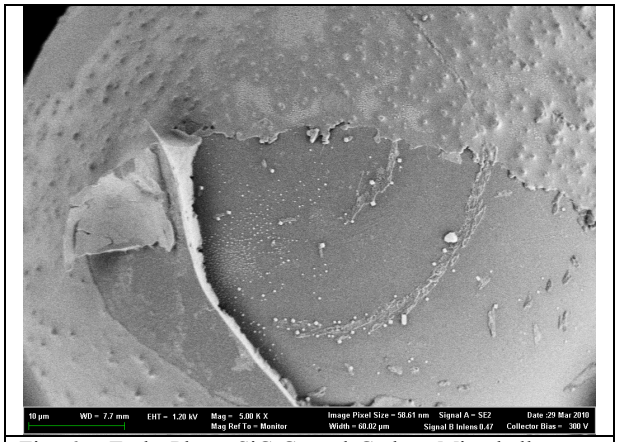

Fig. 6 – Early Phase SiC-Coated Carbon Microballoons – SiC Coating Separating from Substrate. (Scanning Electron Microscope Image Taken at Harvard University)

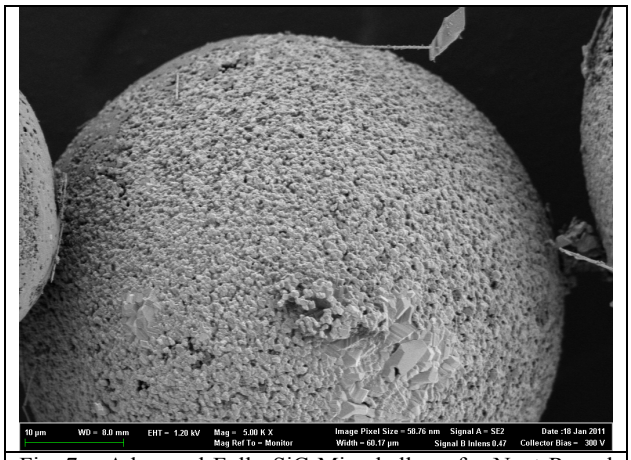

Fig. 7 – Advanced Fully SiC Microballoon for Next Round of Thermal Ablation Testing (Scanning Electron Microscope Image Taken at Harvard University)

Higher density phenolic samples (28 lb/ft^3) containing SiC fibers had lower thermocouple temperatures and showed improved insulation over their control. This was seen in stagnation, aeroshear, and radiation tests but lower thermocouple temperatures were more pronounced in aeroshear samples. (Aeroshear samples were thinner and thermocouples were located closer to the surface compared to stagnation and radiation samples – see Section 3.) Better insulation performance was also seen in 28 lb/ft^3 and 20 lb/ft^3 test samples where polyimide resin replaced phenolic resin. These responses were observed in all three types of tests but, again, were more noticeable in aeroshear samples. Fig. 8 below illustrates aeroshear test thermocouple responses for an experimental polyimide sample, an experimental phenolic sample containing SiC fibers and a control (all 28 lb/ft^3). More testing is planned at longer exposure times to thermally stress samples to a greater degree.

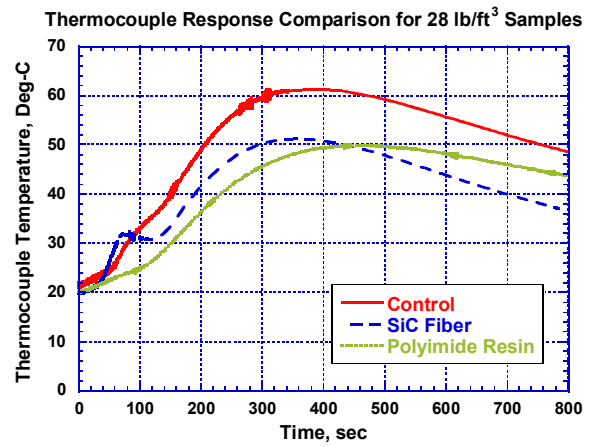

Fig. 8 – Comparison of Thermocouple Responses for 28 lb/ft^3 Samples: Phenolic Control Sample, Experimental Phenolic Sample with SiC Fiber, & Experimental Polyimide Resin Sample

5. SUMMARY

This paper discusses interim results and findings from our multi-year project to investigate ways to improve ablator performance through new materials, optimized honeycomb, and dual-layer systems. Final conclusions on ablator performance will be reached at project completion in mid 2012. Below is a summary of results and preliminary findings to date.

- As a result of ABL's recruitment of Trelleborg Corp. for this project, "fully" silicon-carbide microballoons (100% SiC) have been developed and are now available for use in ablators – new samples with these improved microballoons will soon go into the next phase of ablation testing.
- A SiC microballoon produced by the simpler method of applying a SiC coating to carbon microballoons was shown through testing to be insufficiently robust for use in ablative systems due to separation of the coating.
- Initial stagnation, aeroshear, and radiation tests on higher density phenolic samples containing SiC fibers showed improved insulation over their control.
- Initial stagnation, aeroshear, and radiation tests of experimental samples with polyimide resin (replacing phenolic resin) provided evidence of improved insulation over their control.
- Significant progress was made in the "learning curve" of how to produce ablators with new and different resins, fibers and microballoons.
- Advances were made in the relatively complicated processing required for dual-layer ablators (i.e., steps needed to achieve uniform layers)
- Honeycomb production methods for a range of different cell-sizes were improved.
- Initial thermal testing gave evidence that honeycomb cell-size influences ablator thermal insulation and aeroshear performance, in addition to affecting system producibility and weight.
- The first rounds of stagnation, aeroshear, and solar radiation test series were completed.
- Longer exposure times are planned for the next round of testing to thermally stress the samples to a greater degree. This is expected to bring greater visibility to differences in sample performance.

6. REFERENCES

1. Lewis R. J., *Hawley's Condensed Chemical Dictionary* (14th ed.), John Wiley & Sons, Inc., New York, 2001.

2. Driver D., et al, "Arc-Jet Testing in a Shear Environment for Mars Science Laboratory Thermal Protection System," 41st AIAA Thermophysics Conference, San Antonio, Texas, 24 June 2009.

3. Chun Y. T., "IHF Wedge Test – CFD Data," NASA/ARC E-mail sent to Jennifer Congdon, ARA Ablatives Laboratory, with Attached Excel Workbook Containing Centerline Solutions for the IHF Swept-Cylinder Simulations, 22 January 2008.