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# Carbon-Carbon Nozzle Extension Development in Support of In-Space and Upper-Stage Liquid Rocket Engines

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**Upper stage and in-space liquid rocket engines are optimized for performance through the use of high area ratio nozzles to fully expand combustion gases to low exit pressures, increasing exhaust velocities. Due to the large size of such nozzles, and the related engine performance requirements, carbon-carbon (C-C) composite nozzle extensions are being considered to reduce weight impacts. Currently, the state-of-the-art is represented by the metallic and foreign composite nozzle extensions limited to approximately 2000°F used on the Atlas V, Delta IV, Falcon 9, and Ariane 5 launch vehicles. NASA and industry partners are working towards advancing the domestic supply chain for C-C composite nozzle extensions. These development efforts are primarily being conducted through the NASA Small Business Innovation Research (SBIR) program in addition to other low level internal research efforts. This has allowed for the initial material development and characterization, subscale hardware fabrication, and completion of hot-fire testing in relevant environments. NASA and industry partners have designed, fabricated and hot-fire tested several subscale domestically produced C-C extensions to advance the material and coatings fabrication technology for use with a variety of liquid rocket and scramjet engines. Testing at NASA's Marshall Space Flight Center (MSFC) evaluated heritage and state-of-the-art C-C materials and coatings, demonstrating the initial capabilities of the high temperature materials and their fabrication methods. This paper discusses the initial material development, design and fabrication of the subscale carbon-carbon nozzle extensions, provides an overview of the test campaign, presents results of the hot fire testing, and discusses potential follow-on development work. The follow on work includes the fabrication of ultra-high temperature materials, larger C-C nozzle extensions, material characterization, sub-element testing and hot-fire testing at larger scale.**

## Acronyms

| | | |
|---|---|---|
| ACC | = | Advanced Carbon-Carbon |
| AM | = | Additive Manufacturing |
| APS | = | Air Plasma Spray |
| C | = | Carbon |
| C-C | = | Carbon-Carbon |
| CCNE | = | Carbon-Carbon Nozzle Extension |
| CTE | = | Coefficient of Thermal Expansion |
| CVI | = | Chemical Vapor Infiltration |
| DIC | = | Digital Image Correlation |
| EMCC | = | Enhanced Matrix Carbon-Carbon |
| F | = | Fahrenheit |
| Hf | = | Hafnium |
| IML | = | Inner Mold Line |
| IR | = | Infrared Thermography |

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| | | |
|---|---|---|
| IRAD | = | Independent Research and Development |
| LH2 | = | Liquid Hydrogen |
| LOX | = | Liquid Oxygen |
| $MoSi_2$ | = | Molybdenum Disilicide |
| MSFC | = | George C. Marshall Space Flight Center |
| NDE | = | Nondestructive Examination |
| OML | = | Outer Mold Line |
| PAN | = | Polyacrylonitrile |
| Pc | = | Chamber Pressure |
| PIP | = | Polymer Infiltration and Pyrolysis |
| SiC | = | Silicon Carbide |
| $SiO_2$ | = | Silicon Dioxide |
| SBIR | = | Small Business Innovation Research |
| STTR | = | Small Business Technology Transfer |
| SiC | = | Silicon Carbide |
| TS115 | = | MSFC Test Stand 115 |
| TW | = | Tape Wrap |
| UHTC | = | Ultra-high Temperature Ceramic |
| $ZrB_2$ | = | Zirconium Diboride |
| ZrC | = | Zirconium Carbide |

## I. Introduction

Carbon-carbon (C-C) composite nozzle extensions are of great interest for use on (a) launch vehicle upper stage liquid rocket engines, (b) in-space liquid and nuclear thermal propulsion systems, and (c) lunar/Mars descent/ascent liquid propulsion systems. The development projects presented here are part of a larger NASA and industry effort aimed at advancing the readiness level of United States (U.S.) C-C technology to the point that large-scale domestically-manufactured C-C nozzle extensions (CCNE's) can be considered as viable candidates for use on U.S. cryogenic liquid propulsion rocket engines. The CCNE technology being developed is intended to support the needs of the commercial space transportation industry, as well as those of NASA and the Department of Defense (DoD). For NASA, CCNE technology development is aimed primarily at satisfying requirements of the Commercial Crew and Cargo Programs, as well as those of the Science and Human Exploration and Operations Mission Directorates.

Upper stage and in-space liquid rocket engines are optimized for performance through the use of high expansion area ratio nozzles to fully expand combustion gases to low exit pressures while increasing the gas exhaust velocities. Currently, the state-of-the-art is represented by the metallic and foreign composite nozzle extensions used on the Atlas V, Delta IV, Falcon 9, and Ariane 5 launch vehicles' upper stage engines. The initial two flights of the NASA Space Launch System (SLS) Exploration Upper Stage (EUS) will make use of the Boeing Interim Cryogenic Propulsion Stage (ICPS) and its Safran Ceramics (France) polyacrylonitrile- (PAN-) based CCNE [1,2,3].While a few U.S. domestic development programs have investigated the use of carbon-carbon extensions for liquid engines, there have been very limited domestic flight programs that make use of C-C nozzle extensions. The RL10B-2 is the only U.S. liquid engine that has flown with a C-C composite nozzle extension – it uses a French material made by Safran Ceramics (formerly Snecma Propulsion Solide or Herakles).

While the requirements and operating conditions for cryogenic liquid upper stage engines are considerably different from solid rocket motors, current efforts to develop large C-C composite nozzle extensions are based upon the technology developed under prior solid propulsion programs of the 1970's and 1980's[3]. Such programs led to the development of uncoated C-C exit cones for intercontinental ballistic missiles (Peacekeeper and Midgetman) and for solid motor upper stages (Inertial Upper Stage and Star 48 Payload Assist Module). The only flight-proven coating for C-C components in the 1980's was the silicon carbide coating system used on the Space Shuttle Orbiter's wing leading edge structural subsystem (LESS) panels, and that technology was not applicable to solid propulsion systems because of their extremely high operating temperatures (1950-3000ºC or 3542-5432ºF). The breakup of the Soviet Union in December 1991 led to the cancellation of many DoD programs, which in turn led to

the collapse of most of the U.S. C-C industry[4,5]. Thus, when the Centaur upper stage program became interested in CCNE's in the 1990's, there were few U.S. options, and what is now Safran Ceramics was selected to develop the RL10B-2's CCNE [6,7].

As a consequence of both the large size required for nozzle extensions and the related liquid engine performance requirements, CCNE's are being considered for a variety of reasons, including:

- The use of CCNE's enables approximately a 50% reduction in mass (weight) versus that of comparable metallic or ablative nozzle extensions.
- Using C-C composite nozzle extensions significantly improves thermal margins versus that of comparable metallic nozzle extensions. As uncooled metallic nozzle extensions are limited to temperatures of around 2000ºF (1093ºC) [Ref: 8,9], CCNE's offer improved performance capabilities and efficiencies through greater thermal capabilities – increases of 500 to 1000ºF are achievable, enabling upper use temperatures of 3000ºF (1649ºC). New and emerging C-C materials may enable CCNE designs that offer increases of up to 2000ºF, with upper use temperatures of 4000ºF (2204ºC) being possible.
- Substantial reductions in overall costs are possible with CCNE's when compared to metallic nozzle extensions and foreign composite nozzle extensions. Even greater cost and mass reductions may be possible if the regeneratively-cooled portion of the metallic nozzles can be shortened and longer CCNE's used.
- Finally, the possible use of state-of-the-art coatings and mixed matrices (carbon plus refractory carbides/borides) may further increase the potential capabilities of advanced C-C nozzle extensions and may lead to higher thermal performance.

Primarily as a result of the lack of new liquid upper stage engine development programs in the 1970's and 1980's, as well as the difficulties experienced by the solid upper stage motor programs of that time period, little consideration was given to CCNE's for liquid engines until the Delta III Program decided to use C-C composite extensions on the Centaur Upper Stage's RL10B-2 engine. While ultimately solved, the problems experienced by the solid upper stage motor community with processing variability for multiple motor programs and the in-flight loss of two Star 48 motors in 1984 also surely led to a reluctance to consider CCNE's for liquid engines[10]. As noted above, the collapse of the Soviet Union led to a greatly reduced U.S. C-C industry. Thus the RL10B-2 had a choice of a niobium alloy (C-103), a HITCO Carbon Composites 2D C-C, and a Safran Ceramics 3D C-C – the Safran material was chosen primarily due to weight considerations (the C-103 option) and, although solved, delamination concerns (the HITCO option). To date, the Safran Ceramics C-C nozzle extensions for the Delta IV Centaur Upper Stage have performed flawlessly[11]. The Safran nozzle extension makes use of a pseudo-3D needled Novoltex preform that is densified through chemical vapor infiltration (CVI). The NASA Constellation and Space Launch System Programs' J-2X engine development effort initially baselined Safran Ceramics' 3D Novoltex preform C-C material, but ultimately switched to a metallic approach because of a variety of cost, technical, and programmatic reasons[12].

With, until recently, a lack of liquid upper stage engine application opportunities and the high costs associated with developing, qualifying, and certifying new nozzle extensions, there has been insufficient impetus for a C-C nozzle extension to be developed for an upper stage engine. To be fully flight qualified, more effort is required in the areas of material processing (including stable, reliable, sources for precursor materials), material characterization/databases, modeling capabilities, engine hot-fire testing, and viable paths to flight certification. Finally, there must be a clear industry need and pull for development of domestic C-C nozzle extension technology. The recent onset of new commercial space company launch vehicle programs and increases in the number of liquid rocket engines being developed has provided a significant push to develop liquid engine CCNE's both to reduce costs and to provide higher performance through weight savings.

NASA, along with various industry partners, has been working to advance the domestic supply chain for high temperature C-C nozzle extensions. Such composite nozzle extension development work has been funded primarily through (a) investments under the NASA Small Business Innovation Research (SBIR) Program, to advance C-C material readiness levels, develop advanced concepts, and to acquire test data for extensions; (b) internal NASA program funding; and (c) independent research and development (IRAD) investments by engine manufacturers and the domestic C-C industry. Although significant program funding has remained elusive, these small investments have allowed the industry to slowly progress forward, providing more viable candidate materials for future flight programs. Significant program investments would allow more fully maturing candidate C-C materials and the associated processing technology, thus enabling a path to flight certification.

The manufacturing process development work, sub-element testing, material characterization, and hot-fire test campaigns described in this paper, as noted previously, are part of a much larger development program being pursued jointly by NASA and industry. Under the larger program, the technology readiness level (TRL) of CCNE technology will be advanced through (a) design, analysis, and modeling; (b) manufacturing process and database development; and (c) evaluation activities including coupon, sub-element, component, and hot-fire testing. Recent efforts in this technology area have addressed specific individual issues, but have not performed the integrated detailed work needed to incorporate the technology into flight programs. Goals of this larger overall program include significantly reducing the cost of fabricating and testing C-C extensions and building industry confidence that C-C-based materials are viable options for upper-stage and in-space engines.

## II. Overview of Recent NASA-Funded C-C Nozzle Extension Development Efforts

Mass (weight) reduction, improved engine performance, and reduced cost are the primary reasons for the recent surge in interest in using carbon-carbon (C-C) composite nozzle extensions on a variety of NASA, DOD, and Commercial Space propulsion systems. As was discussed in the introduction, most of the relevant C-C fabrication technology for rocket propulsion applications was originally developed for solid rocket motors, most notably for ballistic missiles and payload assist modules. As many of these applications for C-C composites were abandoned in the 1985-1995 timeframe and little consideration was given to the use of C-C for liquid rocket engines (other than for the RL10), little, if any, progress or development occurred until roughly 10 years ago. Around the 2005-2010 timeframe, NASA again became interested in C-C materials for rocket propulsion applications. Most of the NASA-funded work initiated in that timeframe was accomplished through the Constellation Program's J-2X engine program and through a variety of NASA SBIR/STTR (Small Business Innovation Research / Small Business Technology Transfer) projects. Since then a variety of small NASA, DOD, and industry efforts have investigated specific technology issues, but an overall, coordinated, integrated effort has not been pursued – as noted above, that is something NASA is working towards doing now. Additionally, the various non-propulsion efforts being conducted for hypersonics, heatshields, and brakes continues to contribute to the overall state-of-the-art for domestic high-performance carbon-carbon composite materials.

Primarily as a result of the demonstrated success of the C-C nozzle extensions used on the Delta IV's RL10B-2 upper stage engines, Safran Ceramics was selected as the supplier of the CCNE for the J-2X engine in 2007. While the J-2X Program eventually switched to a metallic nozzle extension, the engine development program did stimulate significant interest in C-C nozzle extensions, including possible domestically-sourced options. Since then, both through SBIR/STTR projects and MSFC in-house technology development tasks, NASA has continued to advance the state of CCNE technology.

Development work under SBIR and STTR contracts has concentrated primarily on two broad technology areas: attachment concepts and material fabrication methods. Attachment concept projects have been led by Materials Research and Design (MR&D), Orbital Technologies Corporation (ORBITEC), Plasma Processes, and Ultramet; while materials development projects have been led by Allcomp, Carbon-Carbon Advanced Technologies (C-CAT), MR&D, Plasma Processes[13], Southern Research, and Ultramet.

The attachment concept approaches have followed two main tracks, those of (a) intermediary composite transition materials and (b) integrally-bonded metallic attachment components. As a means of dealing with the generally substantial differences in coefficients of thermal expansion (CTE) between metallic nozzles and C-C nozzle extensions, intermediary hybrid composite nozzle extension transition materials have been developed. The use of silicon carbide (SiC) fiber / carbon (C) matrix composites and carbon fiber / zirconium carbide (ZrC) matrix composites have been examined as means of transitioning from metal components to C-C components. While still of relatively low technology readiness level (TRL), such concepts show promise. An example of an Ultramet/C-CAT demonstration component incorporating a C-ZrC transition region is shown in Figure 1a. Methods for integrally bonding metallic attachment components to C-C and carbon/silicon-carbide (C-SiC) composites have included the use of plasma spraying, high-pressure cold spray, and electrochemical deposition techniques.

A range of material fabrication approaches have been investigated through both SBIR and STTR efforts across the agency. This group of 12 projects has examined a significant range of technologies and resulted in the fabrication of demonstration test articles, some of which have undergone some limited hot-fire engine testing. Technologies explored include the following: (a) mixed matrices and filler materials; (b) fibers derived from a variety of organic precursors – rayon, polyacrylonitrile (PAN), and lyocell; (c) two-directional (2D) fabric layup

architectures – gore and involute; and (d) coatings – electrochemical, pack-cementation, functionally-graded chemical vapor deposition, and plasma spray. These technologies, and in some cases combinations of technologies, have been pursued to enable higher temperature capabilities, improved efficiencies, and longer duration nozzle extension firing times, as well as to better accommodate the CTE mismatch between the nozzles and their extensions. While current nozzle extension operating temperature limits are approximately 2000ºF, the development goal of some of the new materials is to be able to push use temperatures up to roughly 4000ºF without significant loss of material (through erosion, oxidation, spallation, ablation, etc.). Examples of some of the demonstration test articles fabricated under these SBIR/STTR projects are shown in Figure 1a-e.

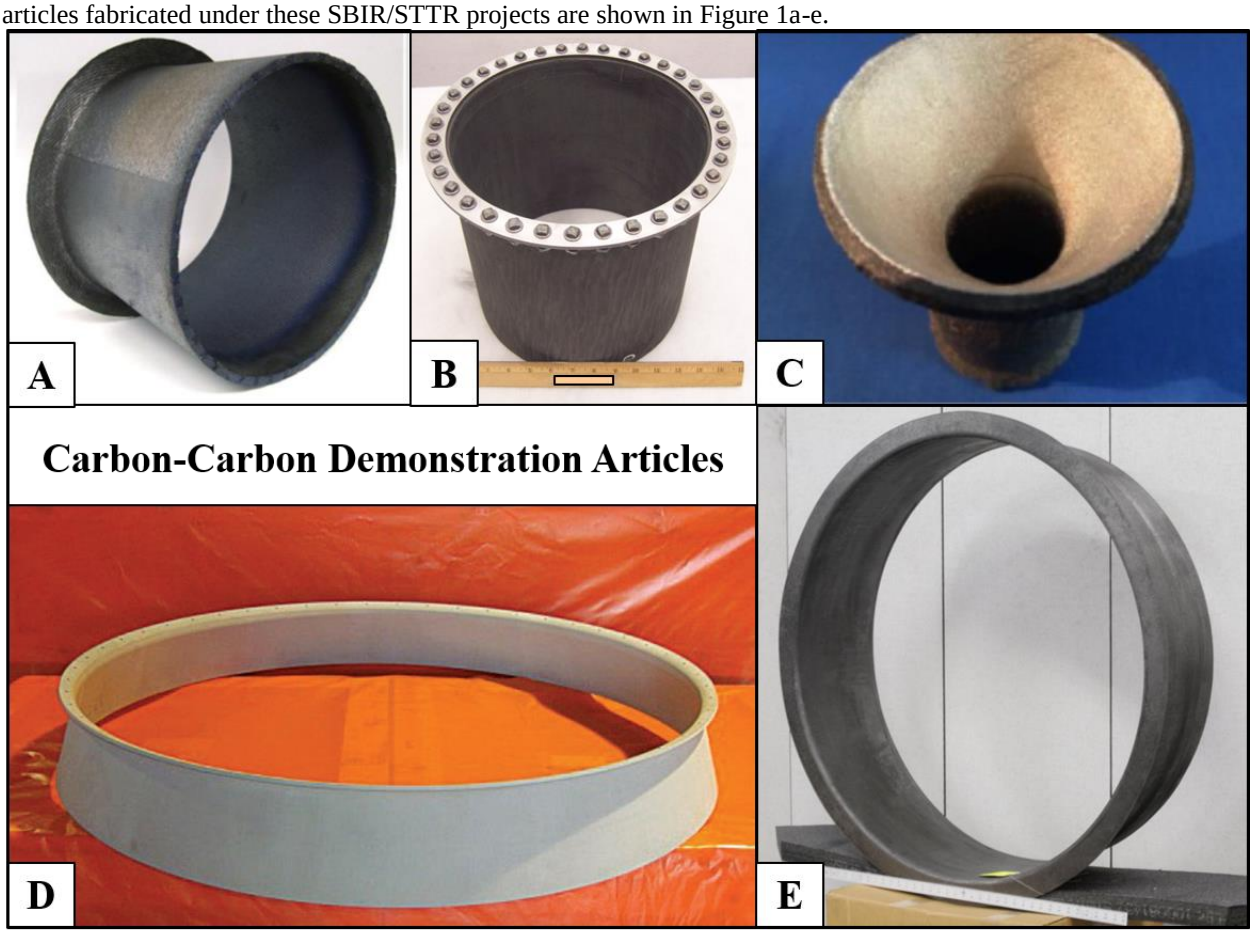


**Figure 1. (a) PAN-based 2D hybrid C-ZrC/C-C fabricated by C-CAT and Ultramet. Exit plane diameter= 11 in. (b) Rayon-based 2D involute C-C fabricated by Orbital ATK. Exit plane diameter = 11 in. (c) PAN-based iridium-lined 2D involute C-C integral combustion-chamber/nozzle assembly fabricated by Orbital ATK and Plasma Processes. Exit plane diameter = 3 in. (d) PAN-based 2D C-C fabricated by C-CAT, using their "high-melt" and pack-cementation coating systems. Attachment flange diameter = 44 in. (e) Lyocell-based 2D C-C fabricated by C-CAT. Cylinder diameter = 42 in.**

A variety of domestic C-C composite materials are available that could potentially meet the needs of NASA, DoD and industry for cryogenic liquid rocket engines. In addition to the SBIR/STTR projects discussed above, MSFC has been conducting a number of projects over the past several years in cooperation with industry partners in order to explore some of these domestic material options for nozzle extension and combustion chamber applications. A series of test campaigns (discussed later in this paper), have made use of a small 1.2K-$lb_f$ liquid engine test facility at MSFC in order to inexpensively and rapidly screen composite nozzle extension materials through hot-fire engine tests. This facility enables, through the use of a liquid-oxygen/gaseous-hydrogen (LOX/GH2) combustion environment, testing of nozzle extension materials at temperatures ranging from 2200º to 4000ºF for durations up to 180 sec. To date, tests have been performed with Orbital ATK (OATK) and C-CAT nozzle extensions. Tests are

planned in the future with Allcomp under a SBIR program. In addition to testing with LOX/GH2, testing with LOX/methane and LOX/kerosene can also be performed. As a means of assessing scale-up issues and making direct material comparisons, a pair of moderate-size C-CAT nozzle extensions fabricated with PAN- and lyocell-based fiber will be tested using a 35K-$lb_f$ liquid engine at MSFC in the near future. Tag-end rings excised from these two 25-in. exit-plane-diameter extensions have also been used to assess nozzle extension material properties and how they compare to flat-plate data (discussed in a later section, below).

The rocket propulsion industry has also been investigating the potential use of large domestically-fabricated composite propulsion system components. Northrup Grumman has an U.S. Air Force program exploring C-C and C-SiC composites for manufacturing the thrust chamber for a first-stage boost engine concept that will include fabrication and hot-fire testing[14]. Aerojet Rocketdyne is currently evaluating a domestic source (C-CAT) for C-C nozzle extensions (see demonstration article shown in Figure 1d). A 33" axial length, 62" diameter C-C nozzle extension was fabricated under a NASA Phase III SBIR program and currently being evaluated in shaker testing and hot-fire conditions[15]. Other industry partners have shown interest in the advancement of domestic C-C materials in support of their missions.

Potential domestic fabricators of high temperature composite nozzle extensions include:

- Allcomp, Inc. – City of Industry, CA
- Carbon-Carbon Advanced Technologies, Inc. (C-CAT) – Kennedale, TX
- GE Aviation – Newark, DE
- HITCO Carbon Composites, Inc. – Gardena, CA
- Orbital ATK (OATK) – Magna and Promontory, UT
- Physical Sciences Inc. (PSI) – Andover, MA

Aside from the companies listed above that have worked development of the C-C material (in conjunction with the coating), other companies have invested resources into development of anti-oxidation coatings relative to C-C materials. These include:

- Exothermics – Toledo, OH
- OATK COI Ceramics (COIC) – San Diego, CA
- Plasma Processes, LLC – Huntsville, AL
- Ultramet – Pacoima, CA

## II. Subscale Hot-Fire Testing

### A. Overview

In 2014, MSFC created a subscale nozzle test rig to offer affordable, long duration hot-fire testing for screening nozzle material systems. It was successfully used testing Orbital ATK 2D C-C materials in 2014 and later tested with follow-on OATK materials and C-CAT materials in 2016[16,17]. The thruster used on this test rig uses a simple, coaxial injector supplied with liquid oxygen/gaseous hydrogen (LOX/GH2) to create high temperature environments. The chamber used in the OATK test series was a heritage (1960's) design with a slotted copper alloy liner slipped into a stainless steel housing. Using deionized water to cool the chamber allowed the thruster to be operated for long durations. The initial test series with OATK successfully screened a variety of materials, but it also revealed that flow separation was significant due to the aggressive nozzle half-angle and the attachment area ratio used with the existing chamber, as observed in Figure 2.

To provide better flow expansion, prior to testing the nozzle extensions from C-CAT, a new main combustion chamber (MCC) was designed for this thruster. The chamber design was updated to use a GRCop-84 (Cu-8Cr-4Nb) liner fabricated with powder-bed additive manufacturing. The hotwall profile of the new liner provided an updated expansion angle to achieve full flow on attached nozzle extensions. A new stainless steel housing was fabricated to accommodate the updated liner design and allow for interchangeable liners. The GRCop-84 alloy and the redesigned coolant channel sizes allowed the new chamber to be operated at higher pressures and temperatures, if desired.

Table 1 offers a comparison of the vintage hardware and the updated assembly with the new chamber design. In addition to changing the liner material and its contour, the barrel section of the chamber was shortened to fit in the available AM machine. The liner was fabricated at MSFC with Selective Laser Melting (SLM) in the Concept

Laser machine, which offered a build box of 250mm x 250mm x250mm. The new chamber length was still adequate to allow for proper propellant mixing of the tested hardware. The convergent radii were maintained per the heritage hardware since heat flux data was known and the throat diameter was maintained. The divergent radius and nozzle contour was completely redesigned aft of the throat.

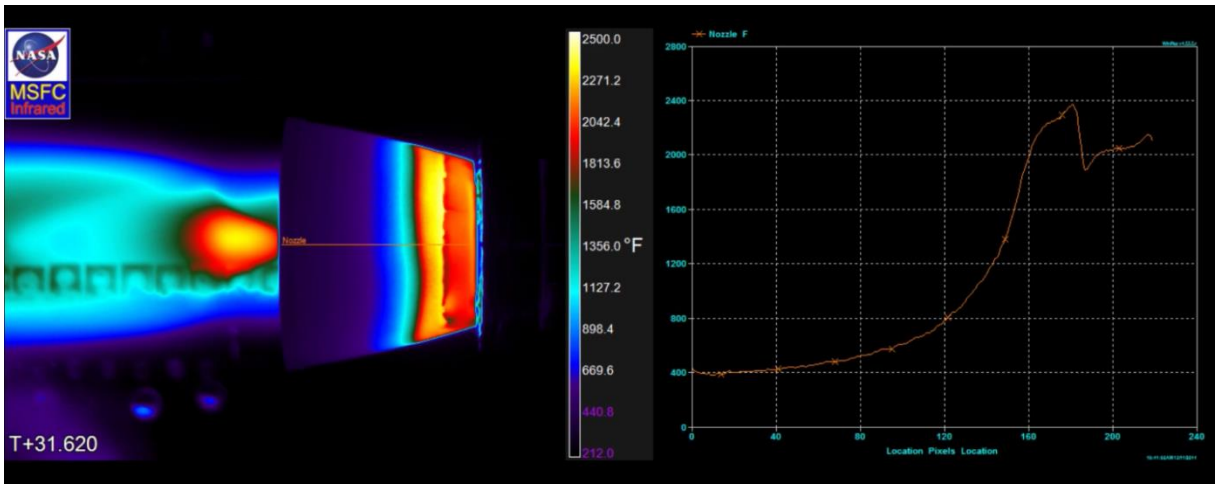


**Figure 2. Flow Separation Observed with Infrared (IR) Thermography on NASA heritage chamber during OATK testing. [IR Imaging: Derek Moody and Darrell Gaddy /MSFC]**

**Table 1. Comparison of Heritage and New Jacket MCC Designs for Test Facility.**

| | Heritage Design | New Design |
|---|---|---|
| Thrust Chamber Assembly, Drawing Reference | MER00060-101 | MER01446-001 |
| Main Combustion Chamber, Liner | MED04227-1 | MER00664-001 |
| Maximum Chamber Pressure, Pc (psia) | 850 | 1350 |
| Water Coolant Inlet Pressure, (psia) | 1000 | 2000 |
| Chamber Barrel Diameter (in) | 2.25 | 2.25 |
| Chamber Barrel Length (in) | 6.77 | 5.26 |
| Divergent Radius, Rd/Rt | 2 | 0.5 |
| Throat Diameter (in) | 1.2 | 1.2 |
| Nozzle Attach area ratio (AR) | 8.1:1 | 4.4:1 |

The new workhorse chambers were originally designed to enable testing of a series of regeneratively cooled nozzles and radiatively-cooled nozzle extensions (at reduced mixture ratios). The target nozzle size was approximately 6" for length and 5.5" for aft diameter. A divergent radius, $R_d/R_t$, of 0.5 was assumed, which provided the appropriate nozzle length, and the full length contour was optimized to an area ratio of 27:1. An attachment area ratio (AR) of approximately 4:1 was selected, which balanced the heat flux on the nozzle and extensions and also maximized the required length of the nozzle. The contour was designed to test at sea level conditions. The full AR of the nozzle was expanded to 27:1 with a wall pressure of 5.5 psia assuming a chamber pressure, Pc, of 750 psia. However, the chambers were designed and analyzed to a Pc of 1350 psia, which would allow for a longer nozzle in future testing. Ideally, the new design maintained continuity to the vintage hardware, so that the test rig could be operated in a similar manner but with higher Pc capability, if desired. Figure 3 offers an image of the redesigned thruster with the nozzle extension attached. Flange attachment configurations have varied depending on the material being tested. The test rig allows mainstage test durations up to 180 seconds at MSFC TS115.

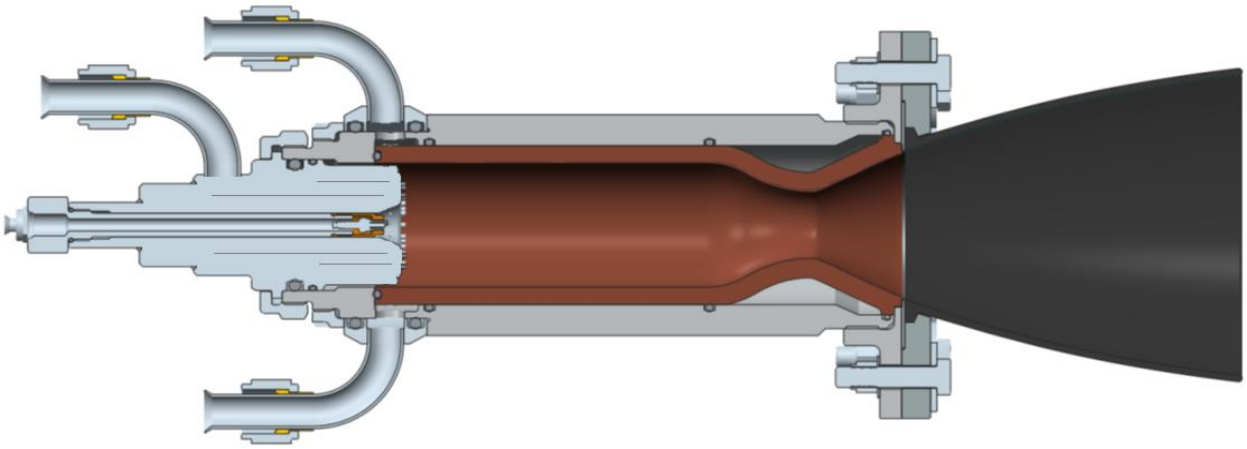

**Figure 3. 1.2K $lb_f$ Thruster with Additively-Manufactured GRCop-84 Liner and C-C Nozzle Extension.**

### B. Orbital ATK (OATK) Configuration and Testing

The OATK C-C nozzle extensions are based on tape wrapped (TW) carbon phenolic preforms that are then converted into C-C via heat treat and pitch densification. This process has demonstrated reduction in the manufacturing time of 2D C-C from 12–14 weeks to about six weeks using combined carbonization/graphitization and impregnation/carbonization cycles. This process has enabled bulk densities to be obtained that are equivalent to prior much-longer-duration processing methods after only three impregnation/carbonization cycles with uniform densities and pore structures throughout the composites. A C-C tape wrapped nozzle can be seen in Figure 4. The billets are tape wrapped slightly over-sized, rough machined to a blank and then converted to C-C using the rapid process to a density appropriate for the oxidation protection to be applied. If methods are used such as silicon-carbide (SiC) via polymer infiltration and pyrolysis (PIP) then a more open porosity is developed in the C-C than if using a plasma-sprayed coating. The cone blanks are machined to final configuration prior to application of the oxidation protection, since none of the oxidation protection methods used significantly change the dimensions of the cones.

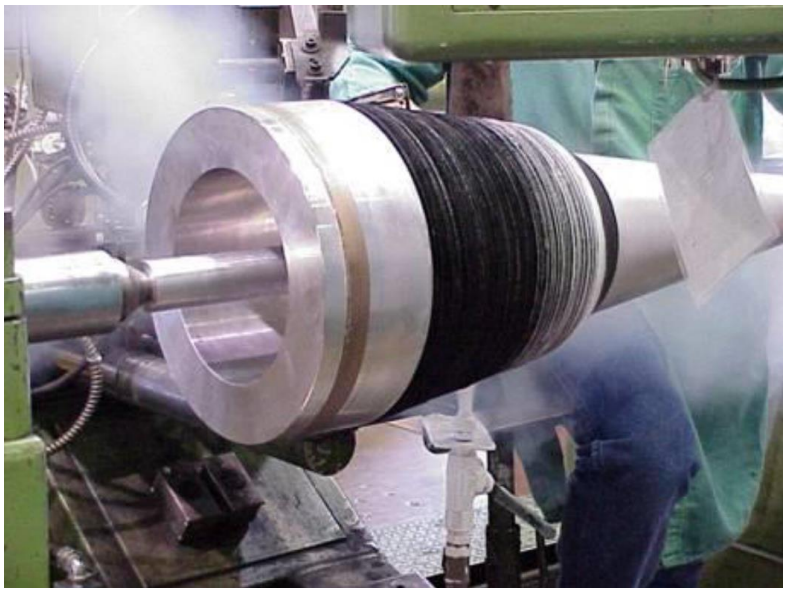

**Figure 4. OATK C-C Extension using tape wrap fabrication.**

The OATK C-C nozzles were machined to final thicknesses which included varying thickness near the flange to allow for the split ring and an aft stiffener ring. The tape wrap process does allow for thicker C-C wall thicknesses up to approximately 0.8" using the rapid densification process. A Grafoil seal was used to interface with the chamber and a carbon phenolic split ring for attachment. An air plasma sprayed (APS) zirconia-based insulator was applied on the outer surface of the C-C cones between it and the carbon phenolic split ring attachment. The configuration and installation in the test stand can be seen in Figure 5.

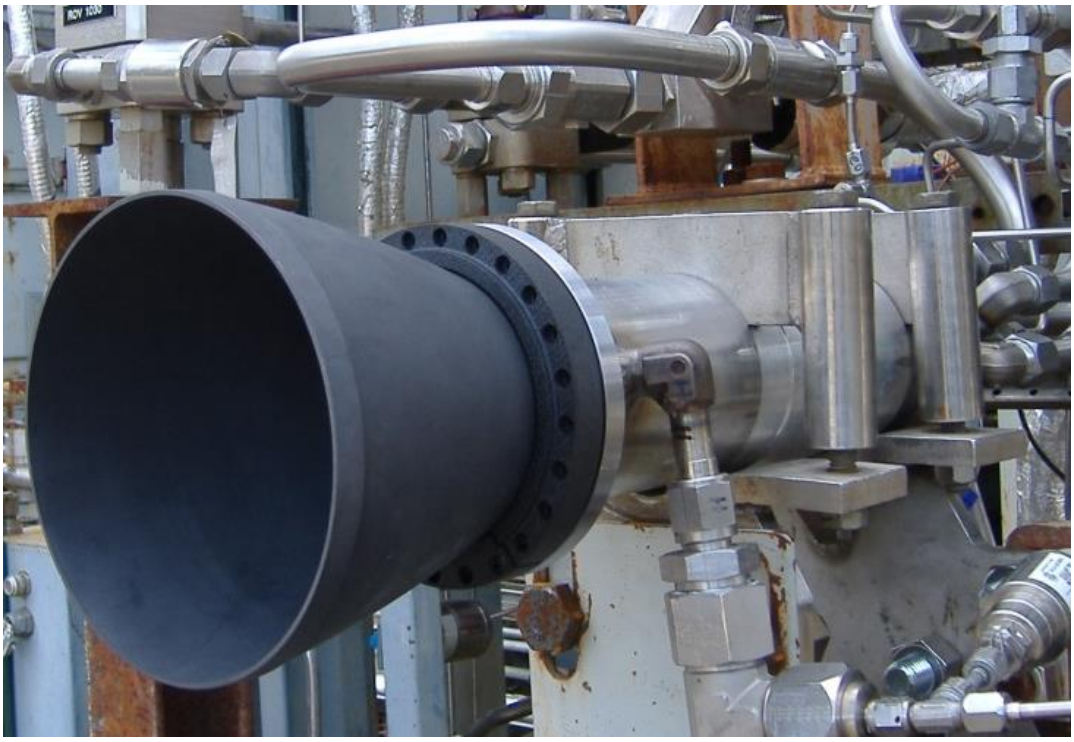

**Figure 5. OATK C-C Extension Installed in Test Stand 115 at MSFC.**

The OATK C-C extension hot-fire configurations focused on anti-oxidation coating development. The coating evaluations traded fabrication costs, based on coating application time, and performance of the coatings verses the baseline of an uncoated C-C composite. The coatings used a variety of processes for anti-oxidation coating application. The shortest lead-time coating development was conducted using vacuum plasma spray (VPS) and air plasma spray processes by Plasma Processes, LLC. The first formulation constituted an agglomerated blend based on zirconium-diboride and SiC. The second coating consisted of an agglomerated blend of a molybdenum disilicide-($MoSi_2$-) based Plasma Processes proprietary coating. Both coating formulations were developed on prior efforts. They were down-selected for use in this application based on their good performance both in exposures to oxidizing environments in static air furnaces and during plasma torch testing. Coatings on C-C in high temperature applications have limitations due both to thermal expansion differences and interactions with the C-C substrate, and to reactions with the turbulent oxidizing gas flow.

A second series of nozzle extensions used coating systems developed by OATK COIC in San Diego, CA. These extensions used a SiC infusion via PIP using preceramic polymers and ultra-high temperature ceramic (UHTC) fillers. A baseline extension configuration had no UHTC fillers, while the two additional cones used zirconium-based fillers and hafnium-based fillers. This PIP method has a significant advantage in that the SiC infiltrates throughout the entire C-C article, significantly improves mechanical properties (tensile and shear capability), and reduces thermal conductivity relative to the bare C-C. It does require more processing time than the VPS and APS methods, but with improved performance.

Another lower-cost coating method investigated during development was metal melt infiltration, which was completed by Exothermics. This coating method makes use of a silicon-based material and results in partial conversion to SiC.

### C. C-CAT Extension Design and Fabrication

The contour for the C-CAT extensions was based on the new truncated ideal nozzle contour and was offset radially at the MCC/extension interface. The baseline C-C extension was designed with an acreage wall thickness of approximately 0.15", which allowed for a 2-stage layup and had acceptable structural margins at the forward end. Although variants of C-C material constituents and processing techniques were used, the design remained the same for all nozzle extensions, thus enabling commonality in tooling. The forward end of the nozzle extensions included an outboard step to allow for a 2-piece split ring to engage and interface with the MCC. A Grafoil seal was used at the joint of the extension-split ring and the MCC.

A graphite split ring was designed to interface the extensions to the AM chamber. These split rings were originally machined from GES PFI-25 graphite from Graphite Products Corp, but later changed to their PFI-45 graphite. The graphite split ring interfaced and engaged with the extension along two surfaces and was selected for temperature and thermal expansion compatibility with the nozzle extension. The structural analysis of the assembly showed a tensile and compressive bending moment at the interface in the split ring due to the startup loads, which required the additional thickness of the extension at the forward end. The skewed-shock plan method was used for the side load analysis and to determine design loads during start up[18]. A Grafoil GTB (inhibited 98% graphite) seal was captured between the split ring and the MCC flange. The Grafoil seals were all custom cut to the required diameters from 0.060" thick sheet. A tantalum split ring backer was used on the nozzle extension-side of the graphite split ring to prevent any fractured graphite from shedding. Larger washers were used with the attachment assembly to distribute the load. The entire assembly can be seen in Figure 6.

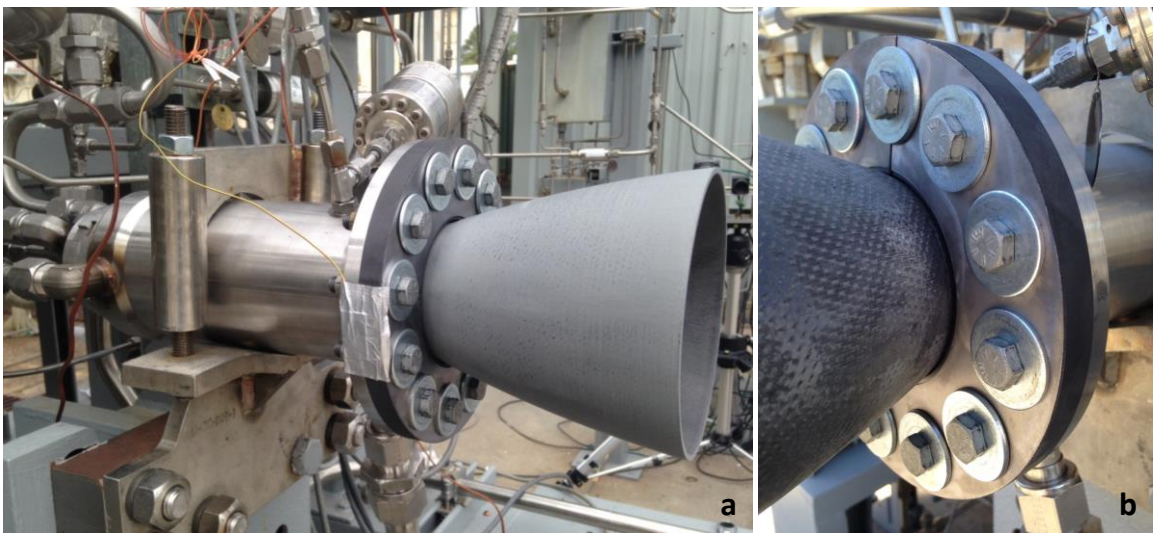


**Figure 6. C-CAT extension installed on the thrust chamber assembly; a) Full Assembly at MSFC TS115 with SiC conversion coated extension, b) View of tantalum backer split ring, graphite split ring and interface with ACC-4 uncoated extension.**

The extension was centered during assembly operations using a 3D plastic printed centering tool and the Grafoil seal was easily aligned with the groove in the split ring. Following the initial fitup, the split ring, extension and Grafoil were removed and inspected. The Grafoil seal showed a clear indication of compression at the MCC liner interface and the split ring did not show any signs of damage.

A series of C-C nozzle extensions were designed and fabricated with variants in the material and oxidation-protection coatings as seen in Figure 7. Common tooling was used for fabrication of all the extensions. The configurations of the nozzles were as follows:

- CCAT 40 6C-FF, T-300 3K heat treat, fiber-filled resin, ACC-6, no coating
- CCAT 40 6C-C-FF, T-300 3K heat treat, fiber-filled resin, ACC-6, silicon carbide (SiC) conversion coating
- T-300 3K 4000 ˚F heat treat, fiber-filled SiC-enhanced resin, ACC-6 *(Experimental Material)*
- T-300 3K 4000 ˚F heat treat, ACC-6, zirconium diboride ($ZrB_2$)/hafnium-carbide (HfC)-enhanced matrix *(Experimental Material)*[19]

**Figure 7. C-CAT 2D C-C extensions prior to hot-fire testing. From left to right: ACC-6/SiC-coated, EMCC ACC-6 SiC-enhanced matrix, ACC-4 Uncoated, EMCC *ACC-6 $ZrB_2$/HfC -enhanced matrix***

The material systems chosen for this effort were selected based on the desire to satisfy two main objectives. The first objective, addressed with the manufacture and testing of the SiC conversion coating, was in the validation of the use of the SiC-coated C-C systems which are currently being considered for production nozzle extension designs in LOX/LH2 rocket applications. The second goal was in testing a selected set of experimental enhanced matrix C-C (EMCC) material systems, with the goal of identifying a viable uncoated solution which eliminates the cost and schedule risk associated with a standard SiC-conversion coating process. For baseline comparison, an extension was manufactured to provide performance data for standard uncoated C-CAT 40 ACC-6 material. The nozzle did experience a manufacturing issue, which resulted in a crack during machining, but was still tested to obtain baseline data, as later discussed.

The current state-of-the-art oxidation protection available for the C-CAT C-C material system is based on a SiC pack cementation process which was first developed for the reinforced carbon-carbon nose cap and wing leading edges of the Space Shuttle orbiter[20]. It has since been used in many applications, though this effort represents the first time that C-CAT has fabricated a SiC-coated nozzle extension designed to be tested at temperatures and in environments similar to those of current production nozzle extension designs. A series of processing operations create a robust and reliable barrier to oxidation in air at temperatures of up to 3300 °F for short durations, and 2750 °F for indefinite operation; however, the coating process can account for a relatively large percentage of the overall cost of a C-C part, and can significantly affect schedule during manufacture. The conversion coating step also requires the availability of a high temperature furnace, which can be a limiting factor when considering the fabrication of large-scale oxidation resistant C-C structures.

For these reasons, C-CAT has in recent years developed several EMCC material systems, which have shown promise in providing oxidation resistance without the need for the SiC conversion coating process. The two EMCC material systems selected for testing were originally developed by C-CAT for use in the harsh conditions inherent in high Mach number hypersonic flight, at temperatures exceeding the maximum effective range in air for the standard SiC conversion coating[21].

All four nozzle extensions were manufactured using satin weave fabric with T-300 fibers, 3000 filaments per tow, heat-treated prior to layup. The heat-treated carbon fabric was then pre-pregged using varying resin systems, each one based on a phenolic base with varying fillers and refractory particles added as enhancements. Identical male layup tools were fabricated for each nozzle extension using a high-density graphite material.

To address the inherent challenges in manufacturing a closed shape in C-C with a relatively large thickness and small diameter, layup for each nozzle extension was split into two stages, with intermediate pyrolysis and processing steps performed between each stage. A gore layup was used, with each ply being uniformly divided into axially-oriented strips butt spliced together to form a full ply. To minimize the impact that the presence of the butt splices might have on in-plane strengths, a pattern for offsetting the clocking of the splice pattern of each ply was devised, which was tailored to maximize the distance between butt splices through the thickness of each article.

The acreage region of all four C-CAT nozzle extensions was laid up with each gore segment spanning the entire length of the extension, beginning at the flange outer circumference and terminating at the aft edge. The thickness of

the flange was built up using additional plies interleaved with the gore plies, which provided the stock material required to machine the final geometry of the flange. The ply schedule used was tailored to target a quasi-isotropic laminate in the acreage region after final machining, with two additional plies being laid up on the outer surface to ensure sufficient stock material to hit the desired thickness. However, the decision was made to machine only the flange and neck regions which would interface with the split ring retainer on the test stand, and simply skim the remaining OML surface to remove the bag-side surface roughness. For layup of the flange, the primary target was to maintain a balanced layup, with equal numbers of positive and negative 45° buildup plies. Layup was performed in the same way for all four nozzle extensions.

After layup was completed, each nozzle extension was then pyrolyzed and densified six times using C-CAT's standard process, which consists of repeated cycles of phenolic resin impregnation, cure, and pyrolysis processing steps. Final machining of all articles occurred following the fourth densification and an intermediate heat treatment step. The machining process was performed by first mounting each nozzle extension on a mandrel, which had been machined using the 2D template and tracer lathe used for the fabrication of the male graphite layup tools. A tracer lathe template, with the OML contour of the nozzle extension design, was then used to machine the neck and flange regions. It was at this point in the fabrication process that the uncoated nozzle extension was damaged.

### D. Subscale Hot-Fire Testing of OATK and C-CAT Nozzle Extensions

Testing on C-C extensions was completed from 2014 to 2016 at MSFC TS115. A summary of the testing is shown in Table 2. A total of 43 tests were completed over this time period to characterize the C-C material and coating systems in a LOX/GH2 environment. MSFC completed mainstage testing on the various OATK and C-CAT nozzle extension and coating configurations. The primary goal of testing was to complete hot gas exposure of the nozzle extensions at a temperature approximately 2400-3500+˚F for extended durations. Chamber pressures (Pc) of 550-750 psig and mixture ratios from 3.5 to 5.9 were utilized.

Two infrared cameras, mid-wavelength and high-wavelength, were setup to one side of the nozzle extension. A series of high speed, low speed and still cameras were also position around the test article to capture visual data during the testing. Cameras were positioned to allow for external and internal views of the extensions.

**Table 2. Summary of hot-fire testing completed on C-C extensions.**

| Base Material | Anti-Oxidation Protection *coating* | Accumulated Duration *sec* |
|---|---|---|
| OATK TW Rapid Densification 3 Cycles | Bare | 10 |
| OATK TW Rapid Densification 3 Cycles | COIC-SiC, No Filler | 90 |
| OATK TW Rapid Densification 3 Cycles | PPI ZrB2+SiC, APS | 30 |
| OATK TW Rapid Densification 3 Cycles | Exothermics Si-Partial SiC | 155 |
| OATK TW Rapid Densification 3 Cycles | PPI MoSi2-based, VPS | 30 |
| OATK TW Rapid Densification 3 Cycles | COIC-SiC + Hf-based Filler | 720 |
| OATK TW Rapid Densification 3 Cycles | COIC-SiC + Zr-based Filler | 480 |
| C-CAT 40 ACC-4 | Bare | 240 |
| C-CAT 40 ACC-6 | SiC Conversion | 2050 |
| C-CAT EMCC ACC-6 | None, SiC-enhanced resin EMCC | 10 |
| C-CAT EMCC ACC-6 | ZrB2/HfC enhanced matrix EMCC | 64 |

The Orbital ATK extensions and coatings completed two rounds of testing in 2014 and again in 2016. Test time was accumulated during both test series for those nozzle extensions where anti-oxidation coating and flange attachment methods were being evaluated. Hot-fire testing of the OATK nozzles can be a seen in Figure 8.

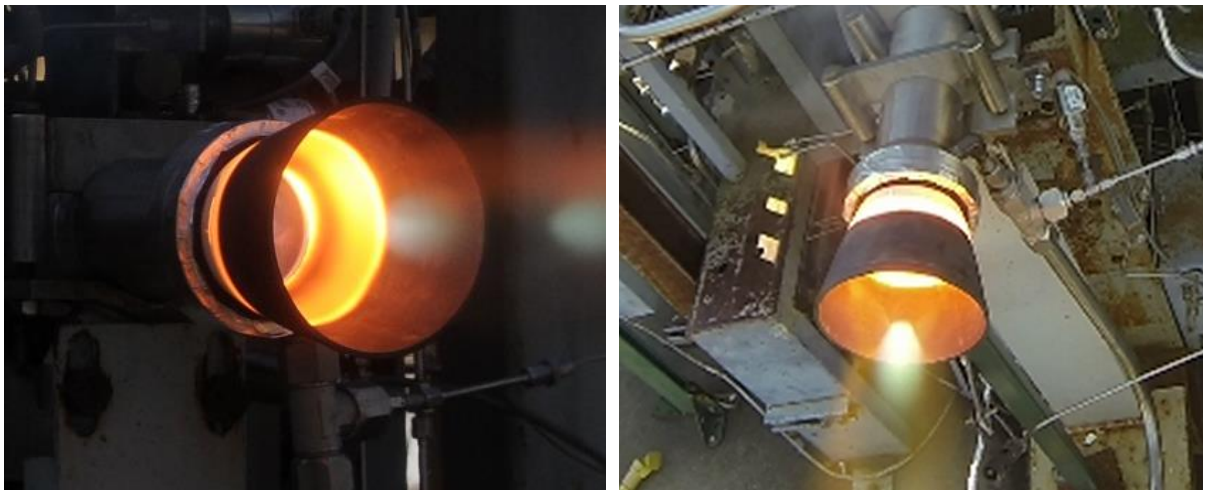

**Figure 8. Hot-fire testing of OATK C-C nozzle extensions at MSFC Test Stand 115.**
[Photo credit: David Olive / MSFC]

For purposes of further discussion, three of the OATK nozzles will be highlighted in this section. These nozzles include the COIC SiC-based coating with the Hf-based and Zr-based filler and also the Exothermics Si-partial SiC-based coating. The COIC SiC-based coating with Zr-filler can be seen in Figure 9. Some of the white residue on the inner surface can be attributed to the triethylaluminum and triethylborane (TEA-TEB) ignition; there is also some minor erosion present. The COIC Zr-based extension had minimal weight loss (<6%) on the IML and outer mold line (OML) surfaces. The region of flow separation can be observed visually through discoloration on all the OATK test nozzle extensions.

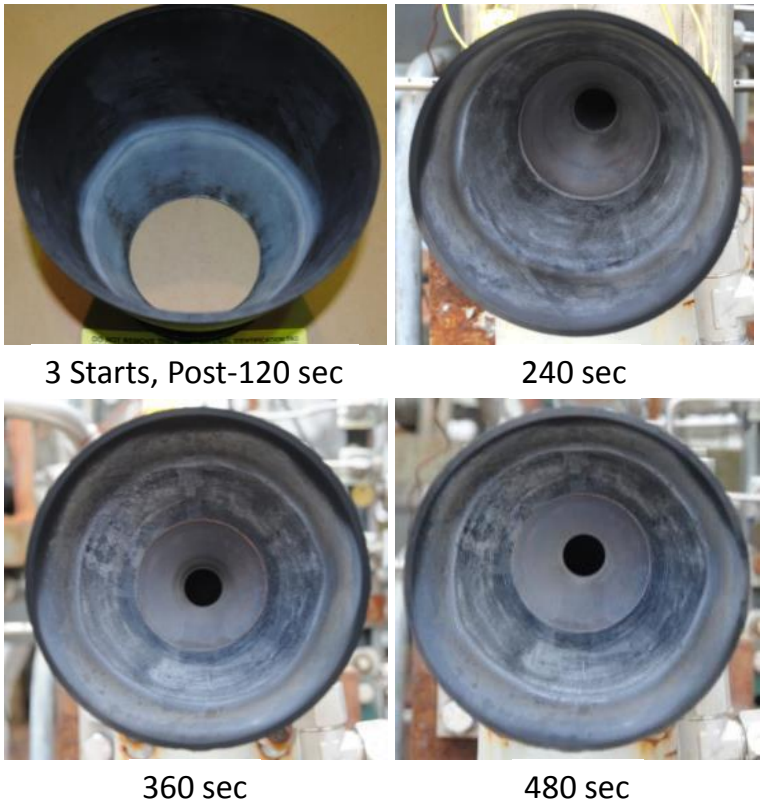


**Figure 9. OATK COIC Zr-based filler nozzle extension progression of accumulated hot-fire testing time.**

The OATK COIC Hf-based filler extension accumulated a total of 720 seconds of hot-fire testing with <23% weight loss at mixture ratios >5.5. The progression of accumulated time on this nozzle can be seen in Figure 10. A significant amount of erosion can be seen in the region of flow separation, which was predicted entering the test series. Accelerated erosion of the local IML surface in the area of flow separation was observed following an accumulated time of 360 seconds. The SiC-based Exothermics extension can be observed in Figure 11 and experienced <4% weight loss following 155 seconds of accumulated hot-fire time.

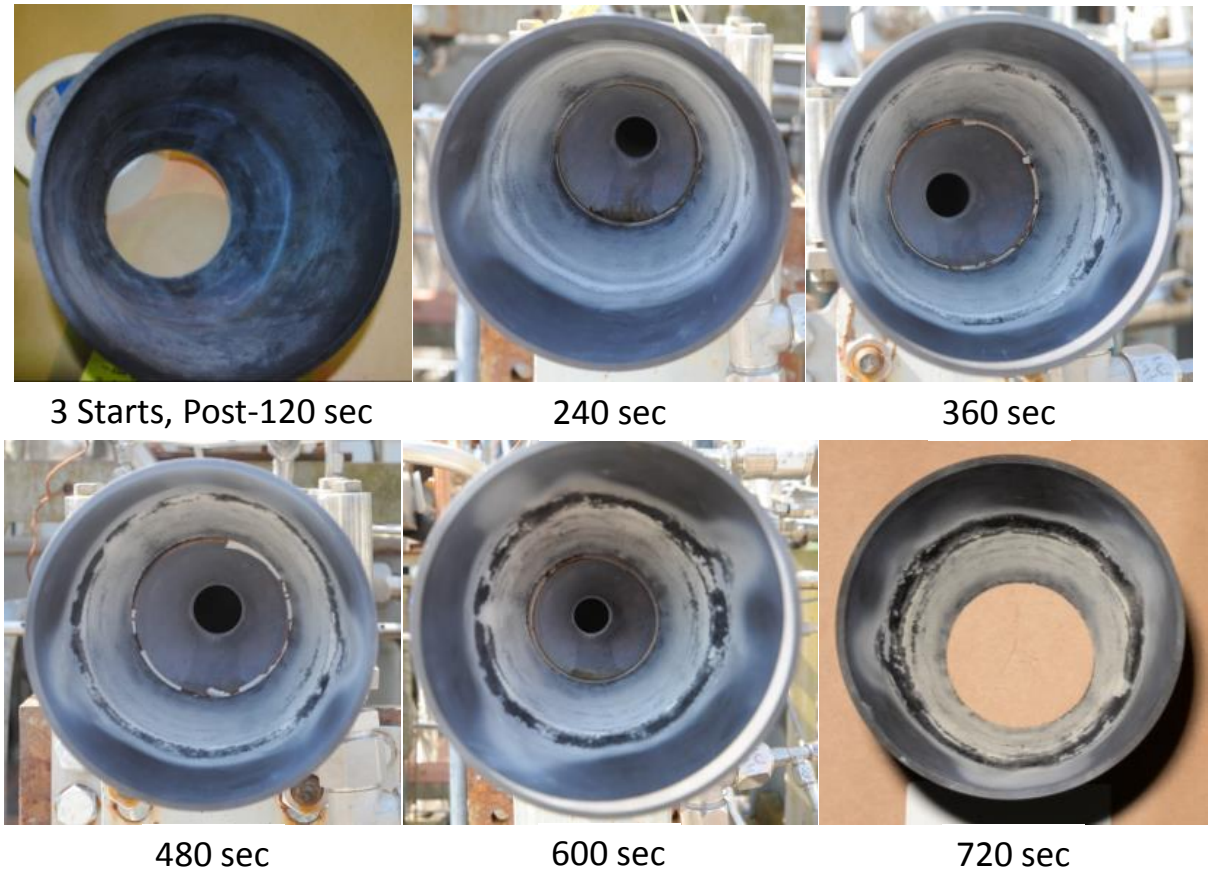


**Figure 10. OATK COIC Hf-based filler nozzle extension progression of accumulated hot-fire testing time.**

**Figure 11. OATK extension with Exothermics partial SiC/SiC-based coating system after hot-fire testing.**

The C-CAT nozzle extensions completed testing in 2016. Prior to each test, the nozzle extension was installed with a new split ring, a tantalum backer ring, and a new Grafoil seal (as discussed in the design section). A total of 25 mainstage tests were completed on the C-CAT nozzle extensions, although some tests were shut down early due to facility redlines. There were no premature shut downs due to the nozzle extensions, chamber, or interface hardware.

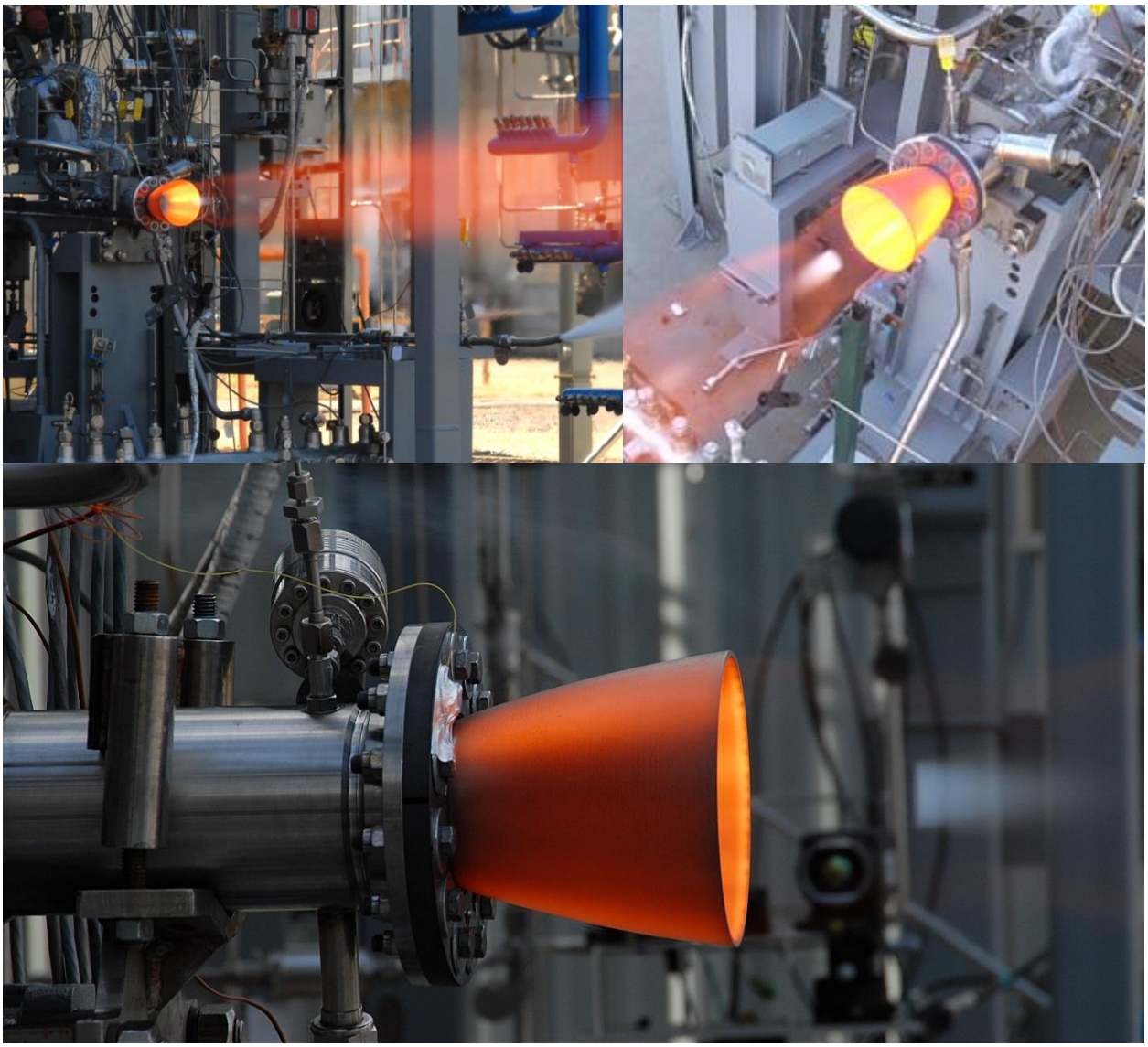

**Figure 12. Hot-fire testing of C-CAT C-C extensions at MSFC TS115. Streaks can be observed in some of the camera images. [Photo credit: David Olive/MSFC]**

### 1. C-CAT ACC-4, No Coating

In the first test of the uncoated C-CAT extension, the interior ply at the location of the machining-induced crack noted previously exhibited some erosion, additionally some minor erosion of the IML and OML surfaces was observed. The unit remained attached to the chamber assembly for the full 120 seconds of mainstage firing. The crack was located at the 3 o'clock position looking forward. In the second test of the extension, the aft end began to erode further, but it still remained attached to the assembly. Overall it performed much better than expected, considering it was of a lower density than the other units and had no coating applied to its structure.

Significant vapor and air entrainment flows were observed in the high speed video along the OD surface and at the aft end of the nozzle. This additional oxygen from the air entrainment caused the carbon matrix to break down on the OML surface of the nozzle extension and accelerated oxidation at the aft end, leading to significant aft-end erosion. There was little erosion on the internal surface of the nozzle extension toward the forward end following 240 seconds. This demonstrates that the combustion environment was not as oxidizing for carbon materials as theorized at these conditions and that the nozzle extension could survive if a coating experienced some spallation. Only minor erosion was observed on the IML of the nozzle extension following 240 seconds of accumulated test time, including at the forward end just aft of the interface with the chamber, which was the hottest region.

**2. C-CAT ACC-6 with SiC Conversion Coating**

C-CAT extension with the SiC conversion coating completed a series of 18 test firings and accumulated a total of 2,050 seconds. There were no indications of any issues with the material or no evidence of erosion during the test series on either the internal or external surfaces, although some discoloration was observed. The early indications of material changes were likely due to glassifying of the coating, or transitioning from SiC to $SiO_2$, which provides further oxidation protection. This condition did not affect the performance of the nozzle extension and demonstrated robustness in the ACC-6 material and coating. A progression of the ACC-6 extension hot-fire time accumulation with SiC conversion coating can be seen in Figure 13.

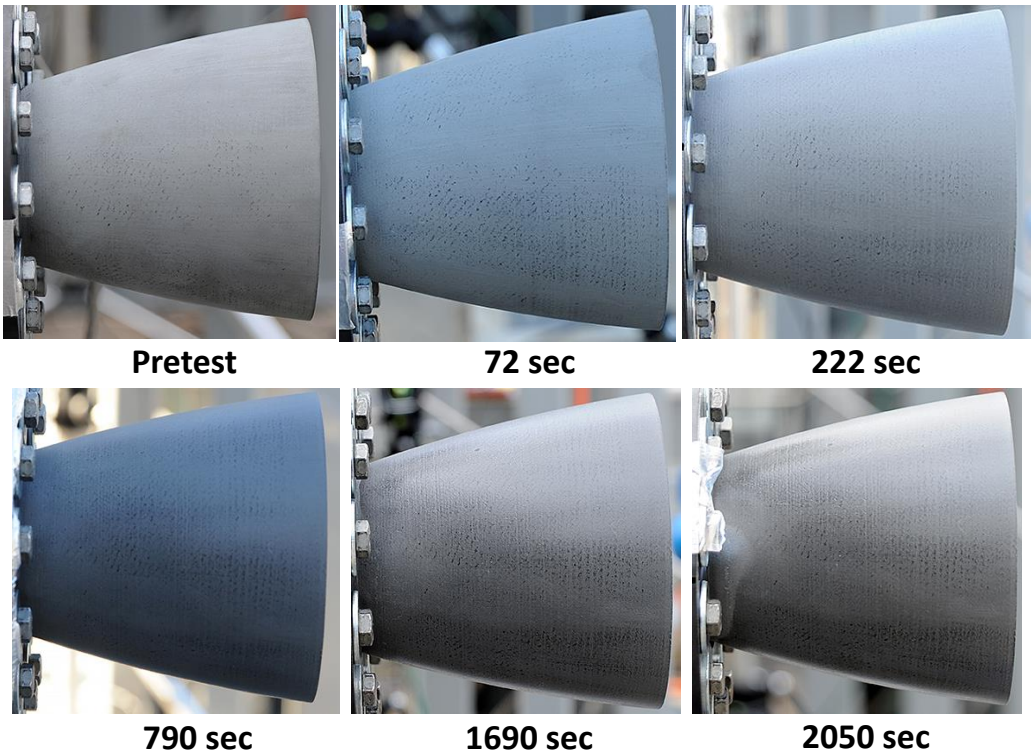


**Figure 13. Progression of C-CAT SiC conversion coated extension testing.** ***Note: Lighting conditions were changing throughout the testing, there was no color change. Also, the clocking was slightly different for the first 72 seconds (3 tests) but remained consistent thereafter.***

Following several tests on the C-CAT SiC conversion-coated nozzle extension, an early shutdown and initiation of the water deluge system caused significant water impingement on the hot extension. This occurred at 7.8 seconds into mainstage operation with the nozzle extension at approximately 1200°F. During post-test inspections, the nozzle extension appeared mostly dry following this off-nominal operation. There were minor water spots observed on the extension, but no visible evidence of moisture. To mitigate any additional moisture concerns with the material, it was dried at 225-250°F to help evaporate any trapped moisture.

The extension completed a structured-light dimensional scan after an accumulated time of 790 seconds[22]. The scan data was compared to the pre hot-fire condition dimensional scan data. The minor variations observed in the data indicated a slight increase in material thickness (~0.001”), but it was determined this was likely due to camera alignment issues and limits with the structured-light scanning technology for the scanning resolution employed. Therefore, the extension was determined to be unchanged.

A later test was also cut off early at approximately 0.5 seconds into mainstage operation due to a facility valve anomaly; the shutdown sequence was similar to a nominal test though. The nozzle extension was inspected, but the split ring and Grafoil seal were not replaced following this test. A minor leak was observed during the following mainstage test. Inspection of the nozzle extension following this test indicated some surface roughening in both the flange area and at about 0.75” aft on the surface acreage due to external burning and subsequent oxidation from the joint leak. This did not affect performance of the extension.

### 3. C-CAT Extension ACC-6, Fiber Filled SiC Enhanced Resin

The C-CAT SiC enhanced-resin EMCC extension completed a 10-second mainstage test. In the final seconds of this test, material was seen being expelled from within the nozzle extension, and post-test inspection confirmed that 2-3 plies of the IML surface had been stripped away in two large areas visible in Figure 14. The damage seemed to have initiated near the aft-end edge and along the butt splice interfaces of adjacent gore segments, where it was evident that the splices had failed via in-plane buckling. The areas where no material was lost, but where significant blistering was present, can be seen in the zones of discoloration in Figure 14. This process of buckling and subsequent blistering appears to have been the primary mode of failure which eventually resulted in the localized shedding of the affected plies during the final seconds of the test.

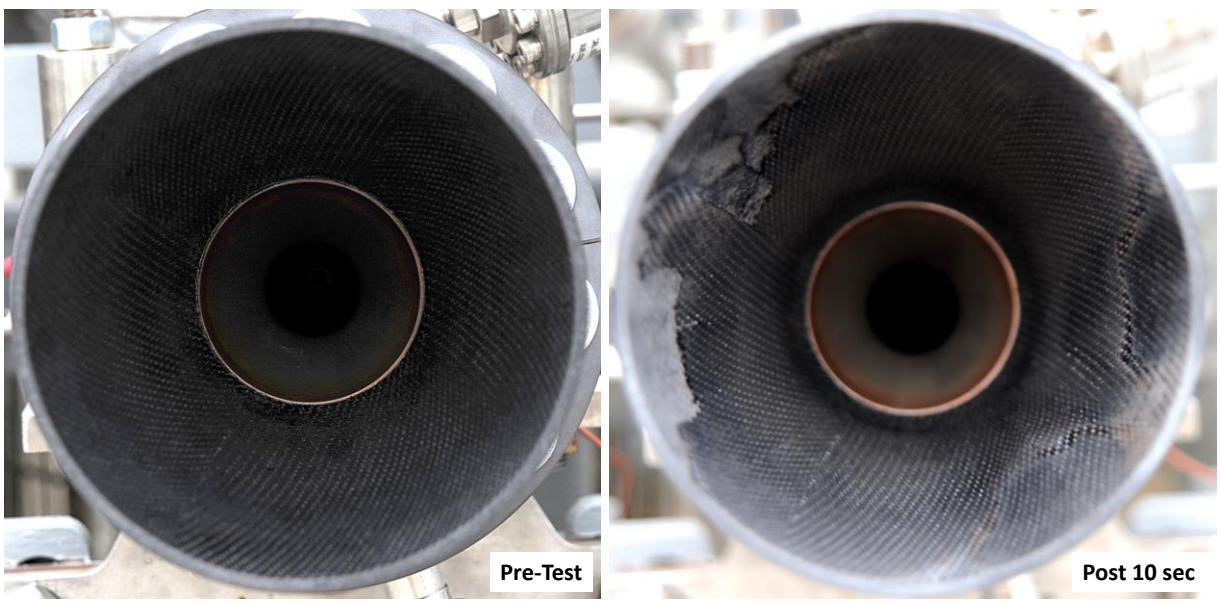


**Figure 14. C-CAT EMCC extension with SiC-enhanced resin; Before (left) and after (right) initial 10-second test**

The failure of the IML surface ply in buckling suggests that the issues were related to thermal shock, resulting from the relatively large through-thickness temperature gradients present during the initial moments of the test. The camera footage from testing and the post-test inspection suggest that the thermal expansion of the IML ply and a through-thickness thermal conductivity which proved insufficient for the relatively high heat fluxes at the wall during startup led to the buildup of in-plane stresses, producing the buckling failures observed. After the formation of the blisters along the surface, the affected areas of the exposed ply were no longer in solid thermal contact with the substrate, leading to further increases in temperature and ultimately a localized total failure of the ply.

### 4. C-CAT Extension, ACC-6 zirconium diboride ($ZrB_2$)/hafnium-carbide enhanced matrix

A 10 second test was performed on C-CAT EMCC zirconium diboride/hafnium-carbide extension, which produced results similar to those observed for the EMCC SiC-enhanced resin, with the same pattern of ply buckling and subsequent shedding of material in areas along the inner surface shown in Figure 15. However, lower levels of overall damage were noted, and the buckling was observed to have occurred along the center line of the gore segments, rather than along the butt splices.

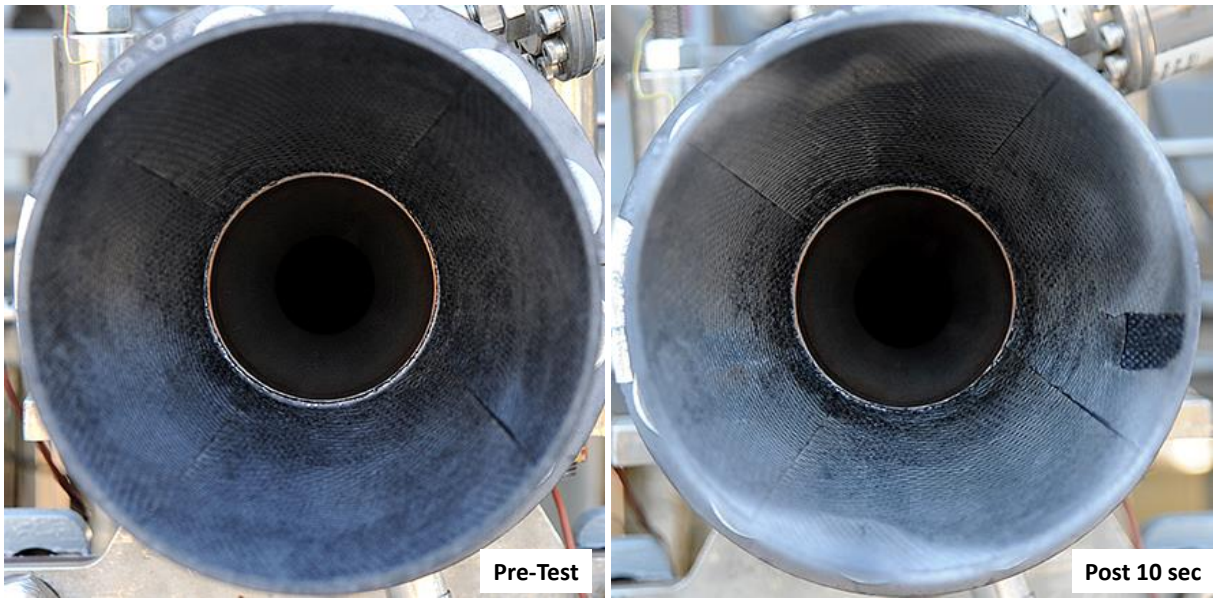


**Figure 15. C-CAT EMCC Extension with Zirconium Diboride/Hafnium-Carbide matrix; Before and after the initial 10-second test.**

In spite of the damage already present, a second test, 53.9 seconds in duration, was performed on this same EMCC extension. Predictably, post-test inspection revealed that additional sections had been stripped away, with failures initiating in the areas where blisters had already formed during the initial 10 second test. However, significant material loss was also observed along the entire circumference of the aft edge, which appeared to be due to gradual recession, rather than complete and sudden disbonding of the affected plies. While quantitative thermal imaging data of the IML surface is not available, it does appear that this area corresponds to a visibly higher temperature zone which can be seen along the aft-end edge of the C-CAT SiC conversion-coated extension, which may account for the increased recession observed. Composite infrared (IR) thermography images can be seen in Figure 16, which shows the first test of each C-CAT extension at start + 10 seconds plus two additional tests.

Two primary plausible explanations have been identified which address the significant differences observed between the test results for the C-CAT coated and uncoated nozzle extensions. One likely important way in which the fabrication process for the uncoated EMCC nozzle extensions differed from that of the SiC conversion-coated extension, and likely affected their performance in testing, was the fact that the uncoated extensions did not receive a final high-temperature heat treatment cycle after completing densification, while the SiC conversion-coatied extension did undergo a high-temperature heat treatment as part of the final coating process. Small and relatively thick closed-shape 2D C-C structures such as these nozzle extensions are inherently very stiff, and have significant built-in residual stresses due to through-thickness shrinkage experienced during processing and the high temperatures used for pyrolysis. Given the geometry of the nozzle extensions and the harsh test conditions, a final heat treatment might have been necessary and sufficient to ensure successful survival through the initial thermal shock in testing. In the past, issues of spallation of coating due to thermal shock in arc jet testing have been successfully addressed through the application of a heat treatment step prior to testing, providing further reason to believe that doing the same for these uncoated extensions might have proved beneficial.

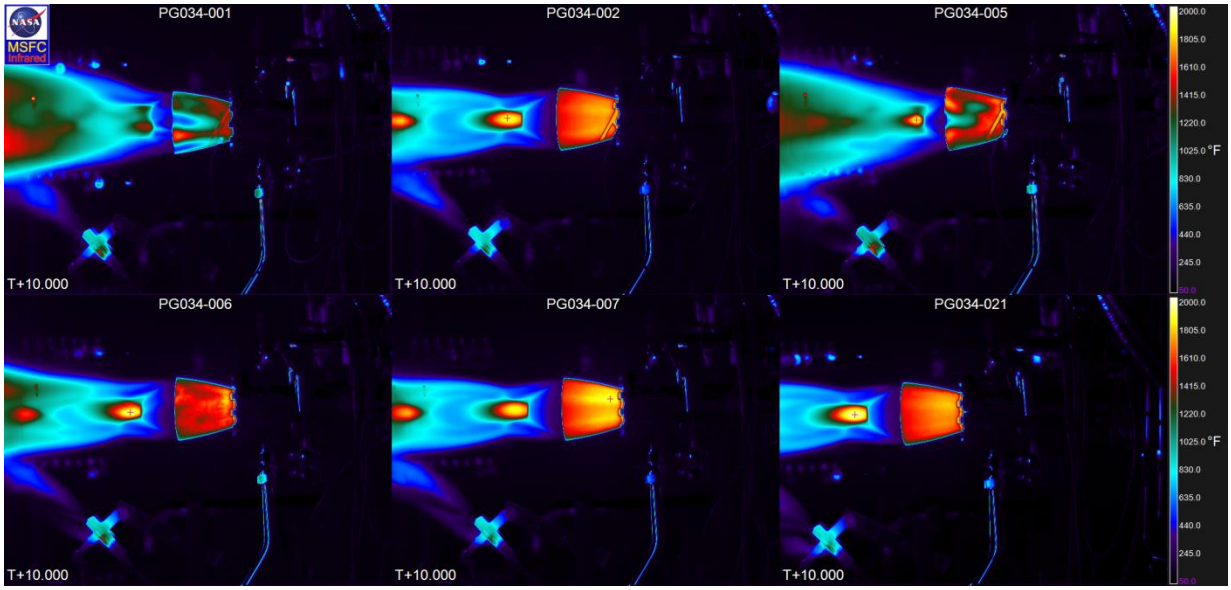


**Figure 16. Comparison of infrared (IR) thermography imaging for C-CAT extensions at start +10 seconds with various amounts of streaking observed.** ***Note: Tests -002, -007, -021 are with the SiC conversion coating.***

Another possibility is that the cracks that are inherent in the SiC coating layer played an important role in ensuring the survival and success of the SiC conversion-coated nozzle extension. The conversion coating process introduces cracks within the coating layer, which form due to the mismatch of coefficients of thermal expansion between the C-C substrate and the newly-converted layer of SiC. During cooldown from the conversion-coating process temperature, this layer of SiC tends to contract more than the C-C beneath, leading to the formation of craze cracks along the surface. It is possible that these cracks in the coated surfaces provided sufficient room for the IML surface to grow independently of the C-C substrate without generating the same in-plane stresses which led to the buckling observed in the C-CAT EMCC uncoated extensions, despite the presumed presence of similar through-thickness thermal gradients during the initial moments of hot-fire testing.

## III. C-C Coupon, Subelement, and Measurement Support Testing

### A. 35K Nozzle Extension Design and Proposed Hot-fire Testing

C-CAT and NASA jointly completed design of 35K-$lb_f$ sized nozzle extension hardware as part of the chamber testing planned under the Low Cost Upper Stage Propulsion (LCUSP) program[23]. The LCUSP program is advancing additive manufacturing of the GRCop-84 copper alloy for liquid engine hardware using selective laser melting and application of a bimetallic deposition jacket. The LCUSP program is developing a liquid oxygen/liquid hydrogen (LOX/LH2) chamber providing high heat fluxes with a Pc of 1400 psig. In order to allow for a C-C extension to be tested in this environment, it would have to replace the regen nozzle in a thrust chamber assembly (TCA) only test series. This is due to the high area ratio of the regen nozzle, which would result in the composite nozzle extension not flowing full if it were integrated at the aft end of the regen nozzle. The regen nozzle area ratio was maximized to allow for sea-level testing without flow separation. The test setup for this C-C subscale nozzle extension would include the LCUSP chamber and injector, a film coolant ring at the aft end of the MCC, and the C-C nozzle extension attached to the film coolant ring (see Figure 17a, below). Film cooling is necessary due to the low area ratio attachment of the composite extension onto the LCUSP chamber.

Under a SBIR Phase III program, C-CAT fabricated two composite nozzle extensions for testing with the LCUSP hardware: (1) a PAN-based ACC-6 SiC conversion-coated C-C nozzle extension, (2) a lyocell-based C-C nozzle extension. These extensions are shown below in Figure 17b and Figure 17c. As a means of understanding the properties and capabilities of these composite nozzle extensions, the tag-end rings removed from the aft ends of the extensions were used for a series of tests performed at Southern Research, which are described in the next section.

**Figure 17. (a) LCUSP engine with a C-C composite nozzle extension. (b) PAN-based ACC-6 C-C nozzle extension with a SiC conversion-coating – fabricated by C-CAT. (c) Lyocell-based C-C nozzle extension, uncoated – fabricated by C-CAT.** ***Note: The two nozzle extensions have the same dimensions, as they were fabricated with the same tooling at Carbon-Carbon Advanced Technologies (C-CAT).***

### B. Coupon and Subelement Testing of LCUSP Nozzle Extension Tag-End Ring Materials

Initial investigations into the feasibility of using lyocell-based carbon-carbon composite materials for upper-stage liquid rocket engines began in 2012 with a Small Business Technology Transfer (STTR) project. Carbon-Carbon Advanced Technologies, Inc. and Southern Research jointly conducted this study, which investigated a variety of processing parameters aimed at developing a lyocell-based C-C with mechanical and thermal properties appropriate for a composite nozzle extension. As the STTR results were promising, when the opportunity arose to fabricate a pair of composite nozzle extensions for testing with the Marshall Space Flight Center LCUSP hardware (presented in the previous section), polyacrylonitrile- (PAN) and lyocell-based composites were chosen. The C-C nozzle extensions were fabricated through a SBIR Phase III effort. As a means of assessing both the quality and potential performance capabilities of the pair of C-C extensions, a mechanical and thermal properties test effort was conducted using tag-end ring material. This mechanical/thermal properties assessment was performed under the NASA Space Launch System (SLS) Program.

After tag-end rings (approximate dimensions: diameter = 27 in.; height = 4 in.) were removed from the aft ends of the pair of composite nozzle extensions, the materials were examined by a variety of nondestructive evaluation (NDE) techniques. These NDE methods included: (a) three-dimensional structured-light scanning, (b) computed tomography (CT), and (c) infrared thermography (IRT). All of the NDE methods indicated that the two tag-end

rings were of high quality from a dimensional uniformity standpoint and that they were free of significant defects or anomalies. Additionally, x-ray radiography inspections were performed on the individual test specimens excised from the two tag-end rings. The individual test specimens were also found to be free of significant defects.

Southern Research performed a series of 16 tests with each of the two tag-end rings, for a total of 32 tests. For both the lyocell-based C-C tag-end ring and the PAN-based C-C tag end ring, the following tests were performed: (a) two conical ring hoop tension tests, (b) six axial (longitudinal) compression tests, (c) six interlaminar tension tests, and (d) two hoop thermal expansion tests. Figure 18 shows the PAN-based C-C tag-end ring prior to the sectioning and machining of test specimens, as well as typical post-test images of a conical ring hoop tension specimen, an axial compression specimen, and an interlaminar tension specimen. *Note: all test specimens (except for the hoop tension specimens) were machined flat prior to testing by removing just enough material to enable material property tests to be conducted without having to deal with the complications caused by curved surfaces.* The axial (longitudinal) compression specimens were 2.25-in. long dog-bone specimens, while the interlaminar tension specimens were approximately 1-in. diameter cylindrical button specimens.

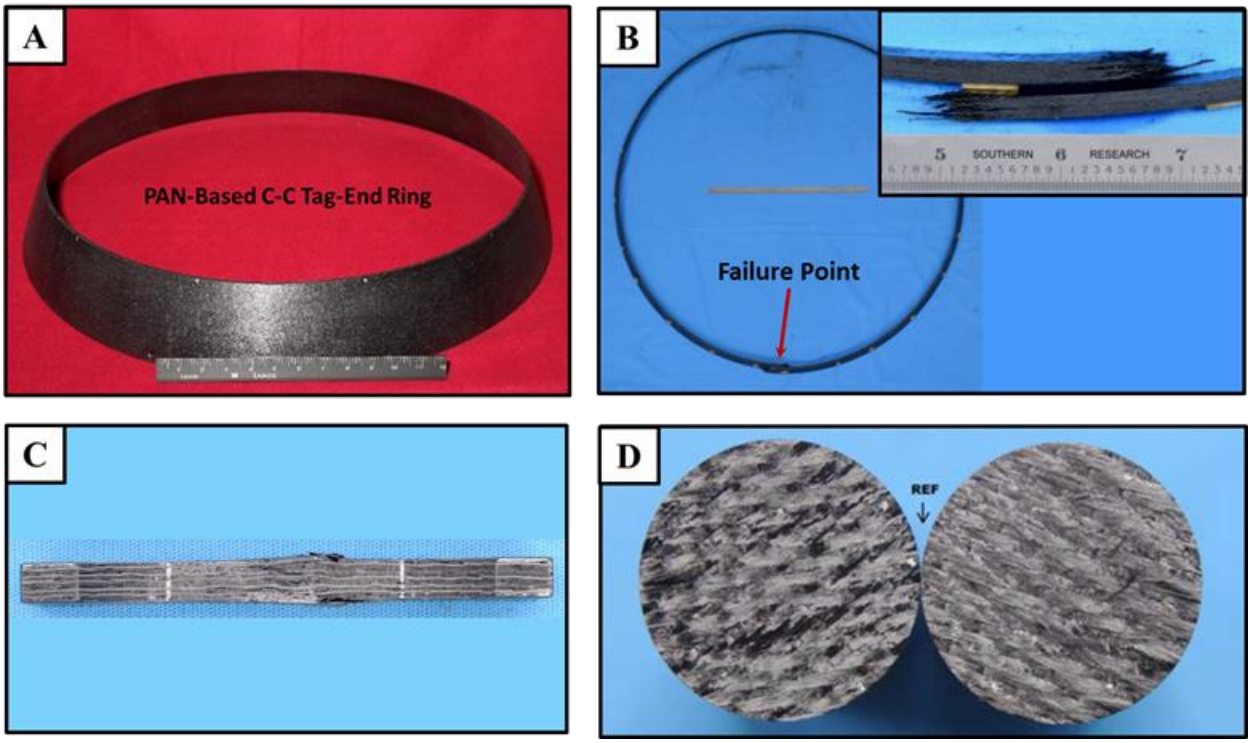


**Figure 18. (a) The nominally 27.5-in. diameter, 4-in. high, tag-end ring sectioned from the aft end of the PAN-based ACC-6 SiC conversion-coated C-C nozzle extension; (b) a post-test conical-ring hoop tension specimen viewed in the axial direction – note fibrous nature of failure region and the overall contraction of the post-test specimen; (c) a post-test axial (longitudinal) compression specimen viewed from the side – note failure region near center of test specimen gauge region; and (d) both pieces of a post-test interlaminar tension specimen viewed in the through-the-thickness direction – failure occurred in within the C-C composite material and not at/near the interfaces with the test fixtures.** ***Note: All of the test specimens shown were excised from the tag-end ring shown in (a).***

The conical ring hoop tension tests were performed through hydrostatic loading of the inner surfaces of the test specimens machined from the tag-end rings. Each hoop tension specimen had a height (axial direction) of 0.5 in. A pair of rings was tested for both of the C-C material types – the approximate average diameters of the two rings for each material were 26.0 and 26.5 in. Prior to hoop tension testing, an analytical assessment was performed by Materials Research and Design (MR&D) to ascertain the best means of fixturing and loading the test specimens, which presented some challenges as the specimens were conical sections of nozzle extensions and not simple right

circular cylinders. Analysis indicated that testing could be performed with the primary loading being in the hoop direction and only minimal generation of stresses in other directions. The conical ring hoop tension tests results are summarized in Figure 19a. From the figure, it can be seen that both materials (lyocell- and PAN-based) yielded similar strain-to-failure results, with the PAN-based C-C being considerably stronger, but also much stiffer. The axial compression results (shown in Figure 19b) indicate that the two C-C materials have similar compressive strengths, but that the lyocell-based C-C offers considerably greater strain-to-failure capability due in part to its lower modulus. Although not being presented at this time, the interlaminar tension and circumferential thermal expansion test results also indicated significant differences between the two types of C-C composite materials.

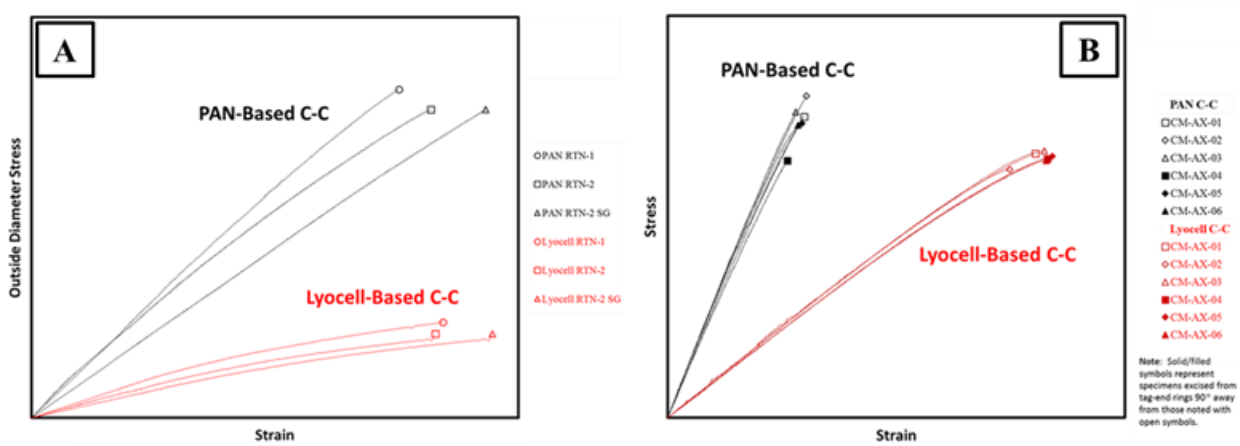


**Figure 19. (a) Conical ring hoop tension tests results for both the PAN- and lyocell-based C-C materials. Two ring specimens were tested at room temperature for each material. Two sets of results are shown for the second test of each material because strain was measured by two different techniques for those specimens – with longitudinal and hoop strain gauges, and with circumferential wires used to measure total hoop strain. Both materials yielded similar strain-to-failure results, with the PAN-based C-C being considerably stronger, but also much stiffer. (b) Axial compression test results for both the PAN- and lyocell-based C-C materials. Two groups of three specimens each were tested at room temperature for both C-C materials – the groups were excised from the tag-end rings at locations approximately 90º apart (solid vs. open symbols on graph). The two C-C materials have similar compressive strengths, but that the lyocell-based C-C offers considerably greater strain-to-failure capability due in part to its lower modulus.**

### C. Digital Image Correlation Supporting C-C Extension Development

Digital image correlation (DIC), specifically the GOM ARAMIS system, is an integral technology being used as part of C-C nozzle extension development. This optical non-contact deformation measurement technique can obtain full surface time-domain displacement, acceleration, and strain data at room and elevated temperatures to evaluate local and global deformations and stresses. The system uses two high-speed cameras that are calibrated in 3D-space using a reference carbon-fiber calibration artifact. After establishing the camera positions and accounting for any lens distortions, the target specimens (nozzle extensions) are speckled with a random black and white stochastic pattern. A variety of paints are used for room and high temperature applications and speckling is often aided with a vinyl template[24]. The speckle pattern allows the software to calculate unique tracking points and surface locations across the component with respect to time, and subsequently the surface strains and displacements, by building a mesh.

Data was collected during subscale hot-fire testing for the first firing of the uncoated C-CAT extension (prior DIC data collection was conducted on metallic nozzles as discussed in another publication[21]. The data collection on this nozzle extension was limited to an initial 120 second test due to spalling of the paint from overheating. The paint was not reapplied. VHT FlameProof™ white paint (SP101) was used for this testing, thus allowing for the elevated temperature testing. The C-C material was used as the contrasting black background. Visibly, at room temperature, the white paint had good contrast with the black C-C material. During heating of the nozzle extension the contrast inversed where the C-C material was high intensity and the paint was low intensity. This data was

collected in the visible spectrum with no filtering. The visible imaging from the high speed cameras can be seen in Figure 20.

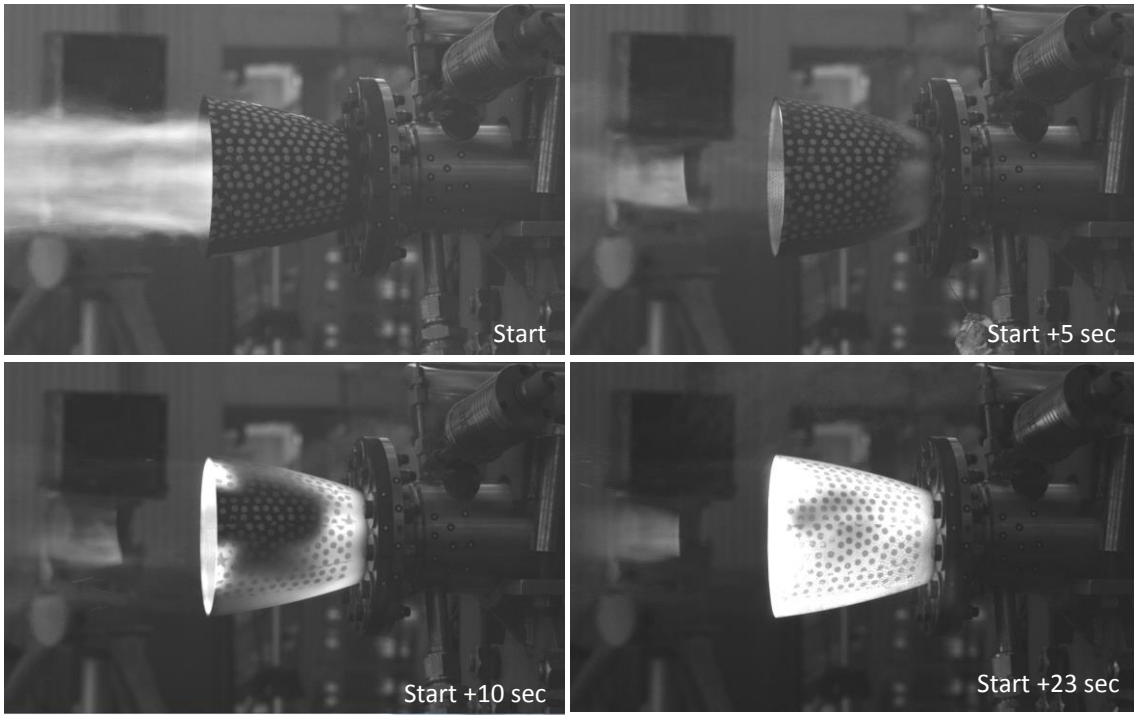


**Figure 20. High speed images collected during digital image correlation assessments. It was observed that the contrast speckle pattern inversed during surface temperature increases causing issues with resolving the DIC data for the duration of the test.**

The DIC software had issues with resolving and applying the mesh to the extension since the contrast pattern inversed during the test as the nozzle increased in temperature. It was shown that data could be resolved at high temperature after the inverse of colors was accounted for and the extension was at thermal equilibrium, as seen in Figure 21. There were not any significant strain or displacement events during this time period, so the absolute values were unknown. A baseline pre-test image to compare against for displacements and strains was also not possible since the stochastic pattern had inversed. This did demonstrate the feasibility of using the DIC system at elevated temperatures; the system is being further evaluated in other research and development applications of C-C nozzle extensions. Additional ultraviolet (UV) wavelength and non-visible techniques are being considered for future C-C hot-fire testing applications[25,26]. Alternative paints or patterning techniques are also being considered to allow for a consistent contrast pattern across all temperatures during testing.

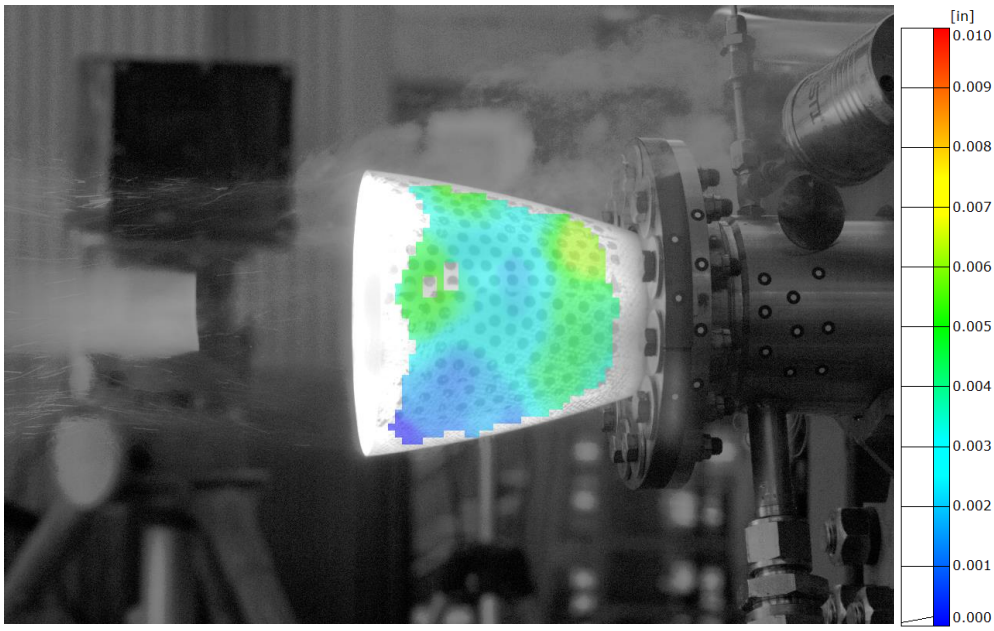


**Figure 21. DIC imaging of the nozzle extension at elevated temperatures. Data was limited though as the paint spalled. Note: A baseline strain could not be obtained since the paint inversed color during heating.**

Digital image correlation has also been used as part of C-C extension evaluations during large-scale lab testing. A DIC system identical to that used during the hot-fire testing was setup to support boost-phase shaker testing. A full-scale 33" axial length C-CAT ACC-6 SiC conversion-coated nozzle extension fabricated under a NASA SBIR, was speckled using the room temperature paints and the high speed system was employed to gather data using the DIC technique and its use with the C-C extension. Response data was collected during various shock and boost simulation testing to determine overall deformation, mode shapes, accelerations and frequency response of the nozzle extension. An image from this testing can be seen in Figure 22.

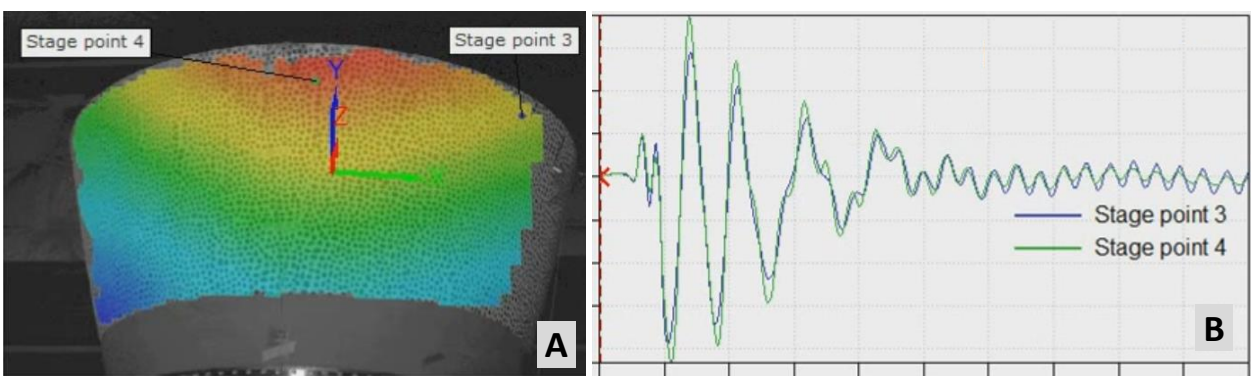


**Figure 22. DIC applied to full-scale nozzle extension during simulated boost load shaker testing. a) Full surface displacements using DIC during testing, b) Discrete point data shown graphically from testing.**

## IV. Conclusions

NASA has been investing in and evaluating C-C materials for use in upper-stage and in-space propulsion applications. There are significant opportunities to make use of these materials for weight savings on future missions. A variety of industry vendors and partners are advancing the materials, coatings, and analysis techniques required for the application of composite nozzle extensions, although additional development is still required to further advance the materials into flight applications.

Hot-fire testing at MSFC TS115 has enabled the advancement of C-C materials and the development of data to facilitate processing changes to further optimize the nozzle materials. The testing was low cost and allowed for significant data and visual information to be collected quickly in a relevant environment. MSFC maintains this test capability for applications like this, which allows for a variety of rapid hardware change-outs and the ability to change test conditions in order to meet customer requirements.

The OATK extensions performed well in the hot-fire environment and showed minimal signs of erosion. Times of 480 and 720 seconds were accumulated on the COIC Zr- and Hf-based fillers, respectively. These extensions will be non-destructively and destructively evaluated to better understand the minor erosion observed. The Exothermics SiC-based coating also performed well and may be further evaluated in the future.

The C-CAT ACC-6 extension with the SiC conversion coating performed well in hot fire testing. This extension accumulated 2,050 seconds of hot-fire time. The nozzle extension will be further evaluated through non-destructive inspections and potentially destructive testing to further evaluate and fully understand the material's capabilities in this oxygen/hydrogen engine environment. The C-CAT experimental-material extensions experienced ply lifts during hot-fire testing, likely due to the high thermal gradients across the extensions' walls. Testing provided performance data on these materials, enabling potential changes to processing conditions to address the observations from test. Despite the ply lifts, the nozzle extensions maintained their structural integrity and continued testing demonstrated that the materials have potential for future applications.

MSFC has completed fabrication of additional moderate-scale nozzle extensions sized for a 35K-$lb_f$ thruster, in addition to test specimens for subcomponent testing[27]. These nozzle extensions include both the C-CAT ACC-6/SiC conversion-coated material and also the C-CAT lyocell-based material. These nozzle extensions will complete hot-fire testing at MSFC in mid-2017.

High temperature composite C-C nozzle extension design, analysis, processing, inspection and testing techniques are being advanced to make these materials viable candidates for use on upper stage and in-space liquid rocket engines. Application of these composite materials provides the opportunity to significantly reduce weight to provide additional engine performance, as well as to reduce costs when compared to foreign suppliers. Infrared thermography was an extremely valuable technique to collect full-surface temperature data. It is recommended that thermography continue to be used for C-C extensions during test to help characterize performance and any potential failures.

The feasibility of using the digital image correlation ARAMIS measurement system for collecting data at elevated temperatures on C-C materials was demonstrated. This data was collected in the visible spectrum with no filtering. Additional ultraviolet (UV) wavelength and non-visible techniques may be considered for future composite applications. Alternative paints or patterning techniques may also be considered to enable a consistent contrast across all temperature regimes during testing.

C-C nozzle extensions have the potential to enable significant cost and weight savings for NASA and commercial space partner missions, but require additional development. These development areas include further material testing and characterization, material processing development and scale-up, coatings for extended duration missions, development of ultra-high temperature materials, non-destructive evaluation techniques and support measurement systems for evaluations both during and after hot-fire testing.

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