QUALIFICATION OF DOMESTIC LYOCELL BASED PHENOLIC IMPREGNATED CARBON ABLATOR (PICA-D) FOR NASA MISSIONS

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ABSTRACT

Phenolic Impregnated Carbon Ablator (PICA) is a low-density ablator that has been used as the planetary entry heatshield for several NASA missions since the late 90's. Its low density and efficient performance characteristics have proven effective for use from Discovery to Flag-ship class missions, from Sample Return missions such as Stardust, OSIRIS-REx to large Mars Lander missions such as Mars Science Lab (MSL) and Mars 2020. The rayon-based carbon precursor raw material used in PICA preform manufacturing has experienced multiple supply chain issues. The challenge involved in finding a replacement fiber source is in processing as well as in the final performance of the ablator. Each replacement necessitates the requalification of the PICA. This has happened at least twice in the past 25 years, and a third substitution is now needed.

Due to the obsolescence of the input foreign rayon fiber source, a new variant of PICA has been developed using a domestic rayon-like fiber source, Lyocell. Due to its flight heritage and proven capability, PICA is baselined as the Thermal Protection System (TPS) for Dragonfly and Mars Sample Return Sample Retrieval Lander mission and is being considered for the backshell of the Mars Sample Return Earth Entry System. All three missions are due to be launched between 2026 and 2028. The challenge this time is to ensure the PICA made with domestic material is a suitable replacement to the heritage PICA used in MSL and Mars 2020 so that the design of the heatshield can be matured without much risk. Results are presented from the recent efforts of 22 PICA-D billets that were Lot Acceptance Tested. Thermal, mechanical, and representative environment arc-jet tests have been conducted. Testing of PICA-Domestic (PICA-D) indicates very comparable performance with respect to "heritage" PICA materials and thus PICA-D is expected to be a sustainable and nearly a "drop-in" replacement solution for future NASA missions.

Index Terms— Phenolic Impregnated Carbon Ablator (PICA), PICA-D, Low Density Carbon Phenolic

1. INTRODUCTION

PICA, invented in the mid 1990's at NASA Ames Research Center, is the lowest density carbon-phenolic based ablative thermal protection system (TPS) with flight heritage [1]. PICA is composed of a rigid carbon fiber based preform (derived from rayon) that is infused with a porous phenolic matrix. PICA is a relatively straight forward ablative TPS to manufacture and to model its response in aerothermal entry environments, given that it has a limited number of constituents. Fiber Materials Inc. produces carbon FiberForm™, a rigid, low density porous carbon foam-like material, using a proprietary process from chopped carbon fibers. The carbon fibers are derived from commercially available rayon by a high temperature conversion process allowing controlled removal of volatiles (carbonization) and graphitization. The PICA process involves infusing the carbon FiberForm™ with a lightweight phenolic resin matrix. The phenolic resin infusion and curing yields a high surface area phenolic phase filling the void space between filaments resulting in a low conductivity and low density ablative TPS that has proven performance at heat-fluxes approaching 1500 W/cm^2 and stagnation pressures approaching 1atm.

The timely invention of PICA enabled the Stardust mission [2], the very first mission to bring back samples from outside the Earth-Moon system. During entry, the sample return capsule was protected from the entry environment by a single-piece near-net-shape cast PICA heatshield. OSIRIS-REx, the first US mission to bring back samples from an asteroid (Bennu) to Earth, also uses a single piece PICA heatshield very similar to the Stardust design. In addition to Stardust and OSIRIS-REx, PICA was used as the heatshield

Table 1 – History of rayon replacement and

Timeline	Mission/ Project	Precursor Type	Rayon Sustainability	Changes /Updates to PICA
Early 2000's	**Stardust** Near Net Shape 0.8m	Liberty rayon	US source – production ceased in the 90s	Developing process to fabricate NNS within the project density specification required
2010	**Orion CEV ADP** Billets	Multiple sources – settled on SINACE	Multiple international sources evaluated	Optimized densification process for billets, tested the bounds of the density specification and the influence on performance / properties
2012	**MSL** Billets	SINACE rayon	International source – production ceased in ~ 2017	Leveraged ADP data to allow use on MSL
2016	**OSIRIS Rex** Near Net Shape 0.8m	SINACE rayon	International source – production ceased in ~ 2017	Spec tightened over Stardust for NNS casting range . Phenolic adjustments based on lessons learned from ADP/MSL
2018-2019	**M2020** Billets	SINACE rayon – source depleted	International source – production ceased in ~ 2017	Leveraged MSL
2016	**PICA-D Development** Billets	Lyocell w/ Ti Dulling Agent	Domestic/international sister plants. Greener processing	MSL specification range – Eliminated use of "re-grind"
2017	**PICA-D Development** Near Net Shape 0.8m	Lyocell w/ Ti Dulling Agent	Domestic/international sister plants. Greener processing	Leveraged OSIRIS REx/MSL – No re-grind
2018	**PICA-D Development** Billets	Lyocell	Domestic/international sister plants. Greener processing	MSL/M2020 specification range – No re-grind
2018	**PICA-D Development** Near Net Shape 1.5m	Lyocell	Domestic/international sister plants. Greener processing	Leveraged OSIRIS REx/MSL – No re-grind
2019-2021	**PICA Capability Sustainment**	Lyocell	**Facility Upgrades**	**Commercial FiberForm Line Ended. NASA only product now. Upgrades to electrical, FiberForm and PICA Infusion equipment for future missions.**

material in a tiled configuration for both the Mars Science Lab (MSL) and the Mars 2020 missions. Based on successful mission use across destinations ranging from Earth return to Mars entry, PICA has been selected as the heatshield TPS option for the Dragonfly New Frontiers mission to Saturn's moon Titan. PICA has also been proposed for a Lunar Sample Return mission and as the backshell TPS for missions to multiple destinations such as Venus and Saturn, where the environments require a carbon-based ablator.

Although PICA only has two constituents and a relatively straightforward process, it has faced multiple supply chain issues as shown in Table 1. The original PICA was manufactured using FiberForm™ derived from Liberty rayon (a US based source). Liberty rayon manufacturing was discontinued in the mid 2000s. Since the FiberForm™ commercial product line depended on a rayon precursor, FMI evaluated an alternate rayon, Sniace, (a Spanish source). In 2016, FMI learned that the Sniace rayon used for MSL PICA was discontinued, they acquired enough in anticipation of PICA needs for the Mars 2020 mission. FMI informed NASA of the imminent supply chain issue and NASA secured the remaining limited quantity of Sniace rayon for future SMD use. In addition, NASA started to evaluate whether domestically produced Lyocell-based PICA would be comparable to heritage PICA [3-4]. In 2016, the Planetary Science Division (PSD) of NASA's Science Mission Directorate (SMD) funded an effort proposed by NASA Ames to contract with FMI and conduct an exploratory study to procure and convert a small batch of Lyocell rayon into PICA (PICA-D, where D stands for domestic) and perform limited arc jet and material property testing. The results from these tests were reported in an IEEE paper and provided high confidence that PICA-D could be a replacement for "heritage" PICA [5]. This initial work on Lyocell-derived PICA was key in informing the viability of Lyocell as a PICA precursor. The next phase of the PICA-D development effort, conducted in 2018-2019, had two objectives: 1) to further characterize the Lyocell derived PICA-D, maturing it for future "drop-in" mission use and 2) explore expansion of PICAs capabilities. That work is summarized in the 2019 MS&T Proceedings [6].

In 2019 FMI notified NASA that the company would cease production of commercial FiberForm™. At that time, NASA Ames halted all remaining PICA-D testing and again worked in partnership with the Planetary Science Division to fund an effort maintain PICA fabrication capabilities at FMI. This effort, termed PICA Capability Sustainment (PCS) sought to refurbish aging commercial FiberForm™ equipment, create a NASA / Aerospace line for TPS FiberForm™ (using Lyocell) and automate the PICA-D fabrication processes at FMI to ensure PICA-D TPS availably for future NASA missions. Facility modifications were completed in 2020 then began an extensive certification / qualification effort by FMI and NASA. Results are presented in this paper from the recent 2020-2021 efforts whereby 22 PICA-D billets were fabricated, Lot Acceptance Tested (LAT) per the PICA fabrication specification and compared to previous eras of PICA. Thermal, mechanical, and representative environment arc-jet tests have been also conducted at NASA Ames. Testing of PICA-D alongside Mars 2020 PICA indicates very comparable performance with respect to "heritage" PICA.

2. PICA CAPABILTY SUSTAINMENT

Recently, FMI made a strategic decision to cease manufacturing FiberForm™ for the commercial market and is reallocating their space for other purposes. FMI had identified continued manufacturing of PICA for NASA as a clear corporate goal, but it did require movement of some equipment to reduce the FiberForm™/PICA footprint and required the upgrading of equipment to bring the process up to modern standards (including safety standards). FMI had identified several tasks for the PICA-D TPS. These tasks were aimed at creating a state-of-the-art, dedicated FiberForm™ fabrication facility to meet NASA's needs, improved safety and environment of FMI employees, improved reliability, and improved process control of the PICA-D processes.

In support of TPS manufacturing sustainability and process improvements of Lyocell-derived PICA-D, NASA planned a 3-year project with FMI to improve and upgrade the FiberForm™ manufacturing facilities and PICA infusion vessels at FMI in Biddeford, Maine. The result of this work was a state-of-the-art, dedicated TPS fabrication capability for PICA-D (and Heatshield for Extreme Entry Environment or HEEET which uses similar infusion process) Thermal Protection Systems. The anticipated work was completed in 4 phases, some of which were in parallel.

2.1. Phase 1: Planning, Shutdown and Storage

The scope of tasks in the first phase were related to existing equipment that needed to be moved to new, dedicated fabrication areas. FMI had several prior FiberForm™ production areas that were vacated. Movable equipment was cleaned and put into storage and the current state of manufacturing equipment and procedures was documented. In preparation for contractor activities remaining equipment underwent extensive PM (preventive maintenance) while vacated areas were cleaned and idle equipment/structures were removed. In parallel, FMI conducted engineering design requirements for replacement equipment design and building safety improvements.

2.2. Phase 2: Building, Electrical and Safety Upgrades

The scope of tasks in the second phase were related to building electrical and safety upgrades as well as process improvements to existing PICA infusion vessels. NASA and FMI cost shared to install electrical system upgrades to meet regulatory compliance in carbon fiber processing and FiberForm™ casting, and billet carbonization areas.

2.2.1 Fiber and FiberForm™ Production Areas

In the carbon fiber and FiberForm™ production areas specifically, FMI installed an OSHA compliant dust collection system in FiberForm™ mixing and machining areas. FMI cleaned and performed safety upgrades to FiberForm™ casting system and material staging area. The commercial casting system was completely rebuilt and FMI installed a new casting system material transport system to meet OSHA specifications. Casting area controls, system interfaces, automation, programming, and data collection were designed and built from the ground up to meet new Aerospace Grade FiberForm™ requirements for TPS use.

2.2.2 PICA Vessel and Infusion Process Areas

In the phenolic infusion process areas, the two existing vessels #1 and #2 remained in place. Around the vessels, however, FMI removed all non-compliant electrical services and equipment. NASA and FMI shared the cost of several PICA vessel upgrades for safety, ergonomics, and electrical and environmental compliance. In addition to hardware/facility upgrades, FMI installed a process control and archiving system for the PICA vessels to meet PICA (and Heatshield for Extreme Entry Environment -HEEET) material specification requirements. FMI overhauled the automated resin feed systems for Vessel #1 and Vessel #2, to meet PICA (and HEEET) material specification requirements and FMI installed pressure, vacuum, and temperature control functionality to the Programmable Logic Controller (PLC) on both Vessels #1 and #2.

2.3. Phase 3: Production Start-Up

Building, electrical and safety upgrades to the FiberForm™ and infusion production areas were completed in December of 2020 which allowed the third phase to begin. In January 2021 FMI began the startup of the new FiberForm™ production equipment for the fabrication of aerospace grade carbon substrates in support of NASA's future missions. Once the casting area was operational, FMI fabricated Qty (10) 8-inch and Qty (12) 6-inch thick FiberForm™ billets that subsequently went through the PICA infusion process in 4 separate vessel runs, 2 runs in each infusion vessel.

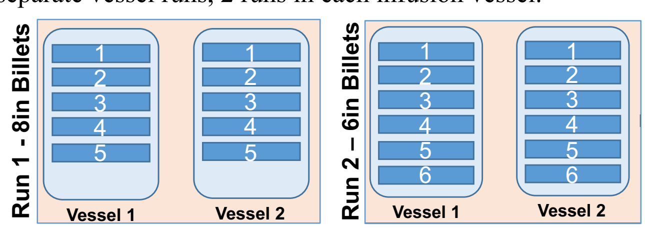

Figure 1 – PICA-D qualification vessel loading

2.4. Phase 4: Qualification of PICA-D

In the fourth and final phase of PICA-D qualification, FMI evaluated the structural and thermal material properties of the 22 PICA-D billets per the heritage PICA Lot Acceptance Test (LAT) matrix shown in Table 2. The purpose of testing all 22 PICA-D billets was to ensure that all the PICA-D material being processed through the dedicated/upgraded equipment at FMI had demonstrated material properties similar to heritage materials.

Table 2 – Standard Lot Acceptance Tests for PICA/PICA-D billets

General	Test Type	Tests per billet
	Density @ RT, TTT	5
	TGA – RT to 900C in N2	3

Structural	Test Type	Tests per billet
	IP tension	3
	TTT Tension	3

Thermal	Test Type	Virgin Property Testing @ 0.2atm Ar	
		38°C	177°C
	TTT Conductivity	2	2
	IP Conductivity	2	2

3. PICA-D LAT PROPERTY TESTING

3.1. IP and TTT Tension Testing

The Tension testing of PICA material complied with ASTM E-4, "Verification of Testing Machines"; ASTM E-83, "Verification and Classification of Extensometers"; ASTM D 1623, Type B, "Standard Test Method for Tensile and Tensile Adhesion Properties of Rigid Cellular Plastics"; and FMI IPOS 08-02-04 Part M Revision A, "In-Plane and Thru-Thickness Orientation Tensile Testing Procedure for FMI PICA Materials". Test specimens were bonded to T6-6061 aluminum loading blocks using epoxy resin in an alignment fixture, and following cure were subsequently placed into the test equipment. Test equipment included an Instron Model 1115 Electromechanical 10,000 lb capacity test machine; Instron model 2511-301 1,000 lb. load cell; MTS model # 632-59C-01 Elevated Temperature Contacting Extensometry System; and data acquisition computer system with National Instruments LabVIEW 2011 software.

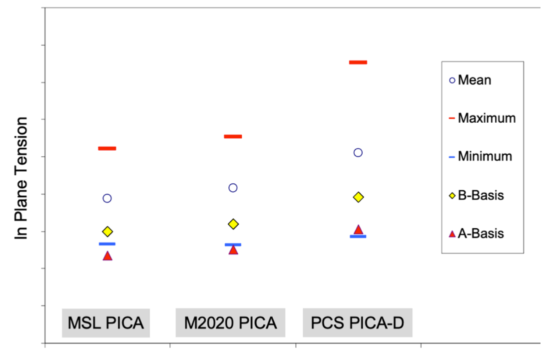

Figure 2 – In Plane Tension of PICA-D compared to MSL and Mars 2020 PICA

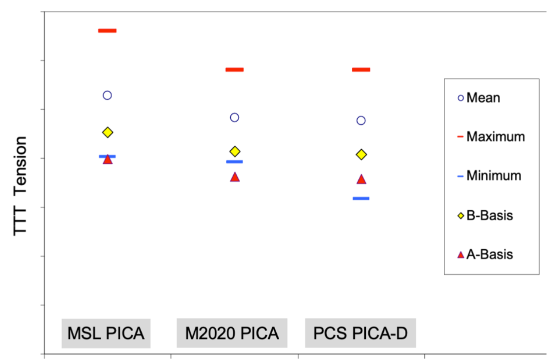

Figure 3 – Through the Thickness (TTT) Tension of PICA-D compared to MSL and Mars 2020 PICA

As shown in Figures 2 and 3, the PICA-D tension results are very comparable LAT results to production heritage MSL and Mars 2020 PICA. In-plane tension (Figure 2) is higher than heritage PICA but meets the spec minimum requirements and results are acceptable. No other test anomalies were identified in the minimum values of tension strength, variance of tension strength, or the other measurements with respect to heritage PICA test results.

3.2. Thermal Conductivity Testing

Thermal conductivity measurements were performed by the comparative rod analysis method (ASTM E1225, "Standard Test Method for Thermal Conductivity of Solids by Means of the Guarded-Comparative-Longitudinal Heat Flow Technique") and FMI IPOS 08-08-20, "Thermal Conductivity Test Procedure: Comparative Flat Slab Method". The test specimens were instrumented with thermocouples in grooves on the top and bottom faces and were mounted between two metering samples of the reference material (Pyrex - NIST standard). This stack was longitudinally positioned between a set of top and bottom heaters and associated heat sinks.

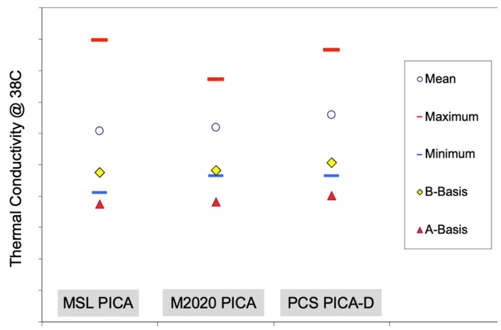

Figure 4 – Through the Thickness (TTT) Thermal Conductivity at 38C of PICA-D compared to MSL and Mars 2020 PICA

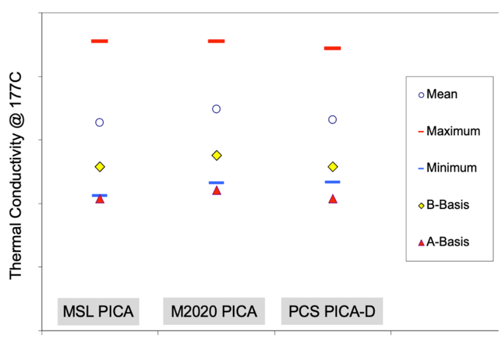

Figure 5 – Through the Thickness (TTT) Thermal Conductivity at 177C of PICA-D compared to MSL and Mars 2020 PICA

A thermal guard heater was placed around the test stack to minimize radial heat flow, and insulation was installed to surround the setup. The entire stack was enclosed by a glass bell jar, and then evacuated and backfilled with the test gas, 0.2 atm argon. Measurements and calculations were conducted in accordance with ASTM E1225. As shown in Figures 4 and 5, the PICA-D conductivity data at 38C (100F) and 177C (350F) exhibits very comparable LAT results to production heritage MSL and Mars 2020 PICA.

4. PICA-D THERMAL ARCJET TESTING

In parallel to the LAT testing at FMI, thermal arcjet testing was conducted at NASA Ames in the 13-inch nozzle of the Interaction Heating Facility (IHF). Figure 6 shows a cross-section of the test articles which were 6-inch diameter IsoQ stagnation models with center plugs containing 5 in-depth thermocouples. Thermocouple plugs were manufactured from the same TPS billet used in the stagnation acreage. TC 1 and 2 were R-type and TC 3-5 were K-type. The TPS on each model was bonded with RTV-560 to an LI-2200 insulator that interfaced with the facility sting arms. A total of 8 models were fabricated: 6 PICA-D models and 2 Mars 2020 PICA models, where half were tested at 2 different conditions termed "High" and "Low".

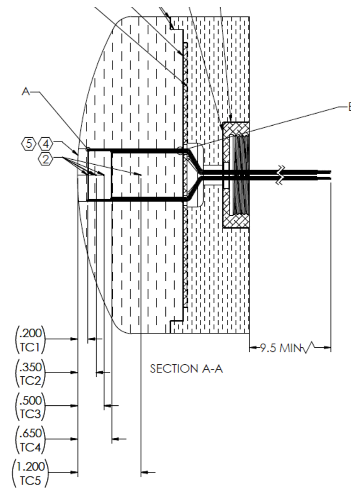

Figure 6 – Arcjet condition 2 calibration and CFD comparison

The two test conditions were calibrated using both a cold wall 4-inch hemi and a 4-inch IsoQ calorimeter.

- Condition 1 "High" (4-inch Hemi calibration): Heat Flux = 370 W/cm^2 , Pressure= 12 kPa
- Condition 2 "Low" (4-inch Hemi calibration): Heat Flux = 180 W/cm^2 , Pressure= 7 kPa.

The test gas was an air/argon mixture containing approximately 8% argon by mass. For each condition, based on the calorimeter measurements and arcjet run parameters, the flow field through the nozzle and around the calibration model was calculated using the Data Parallel Line Relaxed (DPLR) code [7].Calibration comparison to Computational Fluid Dynamics (CFD) predictions is shown in Figures 7 and 8.

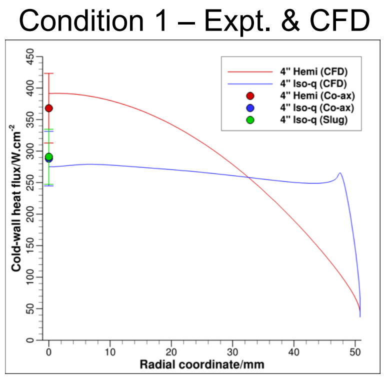

Figure 7 – Arcjet condition 1 calibration and CFD comparison

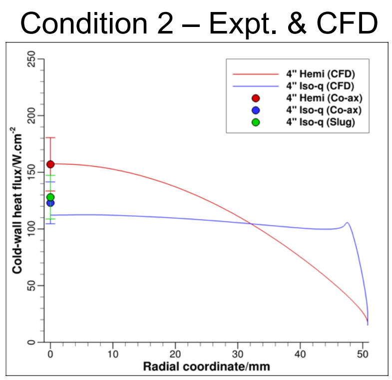

Figure 8 – Arcjet condition 2 calibration and CFD comparison

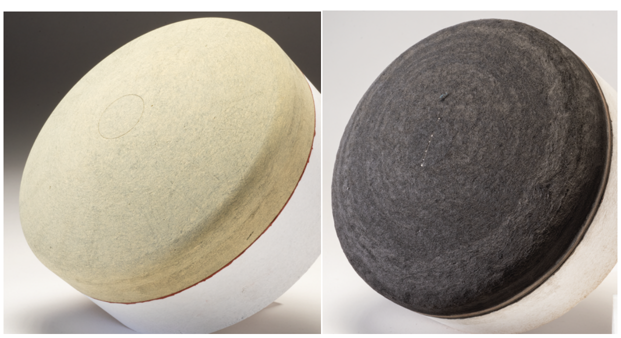

Figure 9 – Pre and post-test photos of the PICA-D thermal response arcjet model.

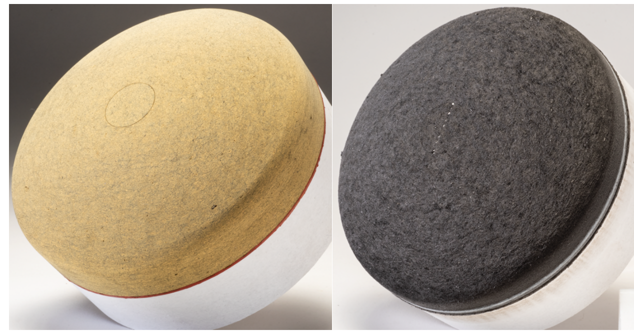

Figure 10 – Pre and post-test photos of the Mars 2020 PICA thermal response arcjet model.

Across all arcjet runs at both conditions of testing, all 40 in-depth TCs provided data. Representative pre- and post-test photographs of the instrumented models are shown in Figures 9 and 10. All models showed smooth recession without cracks, spallation or other surface defects.

The one-dimensional thermal response of PICA and C-PICA was calculated using FIAT, Version 4c, with the dynamic chemistry option. Based on past experience with environment and data uncertainties in the arcjet, calculations were performed using the nominal environments with +/-10% uncertainty on the heat transfer coefficient. Figures 11 and 12 provide temperature data from the stagnation samples as symbols and FIAT predictions for the nominal environment as solid black curves. For the "high" condition, the temperature predictions agree with the data for surface temperature as well as with in-depth temperature. For the "low" condition the surface temperature prediction is slightly below the data, but FIAT matches the in-depth temperatures well.

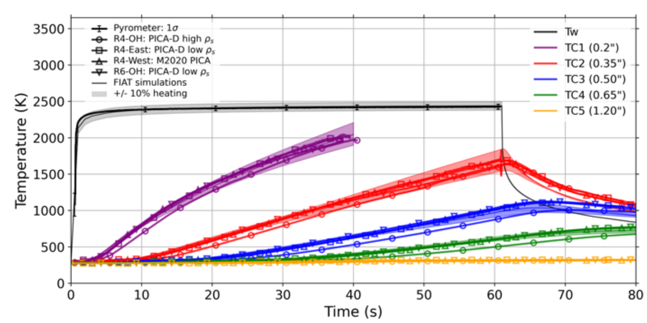

Figure 11 – Comparison of experimental in-depth temperature data and FIAT predictions for High Condition 1.

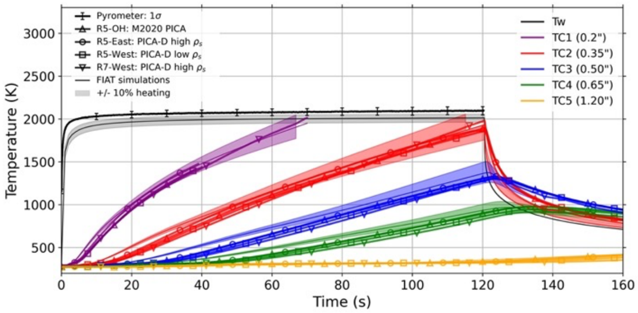

Figure 12 – Comparison of experimental in-depth temperature data and FIAT predictions for Low Condition 2.

The average recession values for PICA-D and MARS 2020 PICA are listed in Table 3 with an error of +/- 0.25 mm. In general, the average recession of PICA-D is slightly less than Mars 2020 PICA at both test conditions. FIAT prediction of recession at 100% heating overestimates measured recession which is conservative when designing for flight.

Table 3 – Comparison of measured and predicted recession for Conditions 1 and 2.

Condition	Measured Recession [mm]		FIAT Simulated Recession [mm]		
	PICA-D	MARS 2020 PICA	90% Heating	100% Heating	110% Heating
Cond 1 High	4.7	5.2	5.0	5.6	6.2
Cond 2 Low	6.7	6.8	7.5	8.4	9.3

6. SUMMARY

Phenolic Impregnated Carbon Ablator (PICA) is a low-density ablator that has been used as the planetary entry heatshield for several NASA missions since the late 90's. Due to the obsolescence of the input foreign rayon fiber source, a new variant of PICA has been developed using a domestic rayon-like fiber source, Lyocell. Testing of PICA-Domestic (PICA-D) was underway when in 2019 FMI notified NASA that the company would cease production of commercial FiberForm™. At that time, NASA Ames halted all remaining PICA-D testing and again worked in partnership with the Planetary Science Division (SMD-PSD) to fund an effort maintain PICA fabrication capabilities at FMI. This effort, termed PICA Capability Sustainment (PCS) sought to refurbish aging commercial FiberForm™ equipment, create a NASA / Aerospace line for TPS FiberForm™ and automate the PICA fabrication processes at FMI to ensure PICA TPS availably for future NASA missions.

Thermal, mechanical, and representative environment arc-jet tests have been conducted. Testing of PICA-D indicates very comparable performance with respect to "heritage" PICA materials and thus PICA-D is expected to be a sustainable and nearly a "drop-in" replacement solution for future NASA missions. Since Lyocell is manufactured in the US in very large quantities and the need is in the commercial sector, Lyocell based PICA is anticipated to be a sustainable source for the foreseeable future.

7. ACKNOWLEDGEMENTS

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