Testing of DLR C/C-SiC for HIFiRE 8 Scramjet Combustor

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Ceramic Matrix Composites (CMCs) have been proposed for use as light-weight hot structures in scramjet combustors. Previous studies have calculated significant weight savings by utilizing CMCs (active and passive) versus actively cooled metallic scramjet structures. Both a C/C and a C/C-SiC material fabricated by DLR (Stuttgart, Germany) are being considered for use in a passively cooled combustor design for HIFiRE 8, a joint Australia / AFRL hypersonic flight program, expected to fly at Mach 7 for ~ 30 sec, at a dynamic pressure of 55 kPa. Flat panels of the DLR C/C and the C/C-SiC materials were installed downstream of a hydrogen-fueled dual-mode ramjet combustor and tested for several minutes at conditions simulating flight at Mach 5 and Mach 6. Gaseous hydrogen fuel was used to fuel the ramjet combustor. The test panels were instrumented with embedded Type K and Type S thermocouples. Zirconia felt insulation was used during some of the tests to reduce heat loss from the back surface and thus increase the heated surface temperature of the C/C-SiC panel ~ 177°C (350°F). The final C/C-SiC panel was tested for 3 cycles totaling over 135 sec at Mach 6 enthalpy. Slightly more erosion was observed on the C/C panel than the C/C-SiC panels, but both material systems demonstrated acceptable recession performance for the HIFiRE 8 flight.

INTRODUCTION

The HIFiRE Program is a collaboration between the Defence Science & Technology Organisation (DSTO) of Australia and the United States Air Force through its Air Force Research Laboratory (AFRL). The primary objectives of the HIFiRE program are to investigate fundamental hypersonic phenomena and to develop and demonstrate component technologies which enable the sustained operation of aerospace systems within the atmosphere at speeds greater than Mach 5. The current manifest of the HIFiRE program includes nine flights yielding basic scientific data with analyses relevant to the design of future aerospace systems.

Completed flights in the HIFiRE program, such as HIFiRE 1, have produced significant data on high-speed boundary layer transition**.** The launch technology used in HIFiRE is based around the sounding rocket approach developed during the HyShot Program at The University of Queensland [1]. Thus far, HIFiRE test technology has been used to test partially complete scramjet flowpaths that remain attached to the second stage booster. Furthermore, the trajectory for the tests have been ballistic, with the scramjet experiment conducted upon re-entry to the atmosphere at very high flight path angles. In contrast, the HIFiRE 8 vehicle, shown in Figure 1, is intended to cruise at Mach 7 under scramjet power for 30 seconds at approximately zero flight path angle. A significant upgrade in the use of high-temperature materials is required for key components of HIFiRE 8 (relative to earlier HIFiRE flights), including the scramjet combustor.

Figure 1: Schematic of the HIFiRE 8 flight vehicle

State-of-the-art scramjet combustors utilize actively cooled metallic structures. However, ceramic matrix composites (CMC), due to their high-temperature capabilities, have the potential to provide a passive alternative for at least a portion of the flowpath. Due to the relatively short flight time (~30 sec) and single use nature of the HIFiRE 8 flight, a scramjet combustor constructed using a passive CMC material is being considered. Toward this end, flat panels of the DLR C/C-SiC were tested in the NASA Langley Direct Connect Supersonic Combustion Test Facility (DCSCTF) [2] using the Durable Combustor Rig (DCR) test article. In addition to the C/C-SiC, the DLR C/C material was also tested.

TEST FACILITY & TEST ARTICLE

Tests of the DLR test articles were conducted in the Direct Connect Supersonic Combustion Test Facility (DCSCTF). The facility is located in a 16- by 16- by 52-ft test cell within Building 1221D at the NASA Langley Research Center in Hampton, Virginia. The facility has historically been used to test ramjet and scramjet flow paths at stagnation enthalpies duplicating that of flight at Mach numbers between 3.5 and 7.5. The facility is of a direct-connect, or connected-pipe, configuration such that the entire facility test gas mass flow passes through the flow path model; the flow at the exit of the facility nozzle simulates the flow entering the isolator of a ramjet or scramjet in flight. The stagnation enthalpy necessary to simulate the flight Mach number for the test is achieved through hydrogen-air combustion with oxygen replenishment to obtain a test gas with the same oxygen mole or mass fraction as atmospheric air (0.2095 or 0.2314, respectively).

CHARACTERIZATION OF THE TEST FLOW CONDITIONS

Using the inflow conditions, the amount of fuel added (ϕ_{H2} = 0.58), and estimates of the viscous drag and heat loss, the 1-D flow properties in the duct can be calculated. In the region where the CMC panel was installed (x = 43 in.), the total temperature of the flow was T_t = 3900°R (2167K), the static temperature was T = 3200°R (1778K), the static pressure was P = 15 psia (103 kPa) and the Mach number was M = 1.35. Based on a calculation of the facility boundary layer performed using the Van Driest II method, the heat load applied to a wall at T_w = 540°R (300K) in the region of the CMC panel was q_{dot} (T_w=300K) ~ 1.4 MW/m^2. Due to the higher total enthalpy, more fuel could be added in the combustor without disrupting the test, and typical fueling was at ϕ_{H2} = 1.01. At x = 43 in., the 1-D flow properties for these tests were T_t = 4500°R (2500K), T = 3800°R (2111K), P = 14 psia (96 kPa) and M = 1.35. The estimated heat load seen by the CMC panel in this case was estimated to be q_{dot} (T_w=300K) ~ 1.9 MW/m^2.

FABRICATION OF DLR C/C-SIC COMPOSITE PANELS

Ceramic matrix composites have been proposed for use as thermal protection materials and hot structures. At the Institute of Structures and Design of DLR in Stuttgart, a specific CMC variant, C/C-SiC has been developed consisting mainly of carbon fibers embedded in a silicon carbide matrix [3]. The fabrication of C/C-SiC CMC composites at DLR is divided into three steps, as indicated in Figure 2. In the first step, a carbon fiber reinforced plastic (CFRP) component is produced which can be performed in different ways. The preferred approach is resin transfer molding (RTM) or using autoclave technology, but warm pressing or filament winding are also acceptable processes. After the curing, the composites are tempered for 4 hr at 240°C to complete the polymerization of the matrix. It is essential to use a resin (e.g. phenolic) with high carbon yield in this step to create a matrix with sufficient carbon content in the subsequent step.

In the second step, the CFRP composite is carbonized under inert atmosphere (nitrogen) at a temperature of 1650°C to convert the polymer matrix to amorphous carbon. The result is a C/C component.. The pyrolysis results in a macroscopic shrinkage of about 10% mainly in thickness and a microscopic network of cracks within the C/C composite is formed. The fiber bundles remain practically intact.

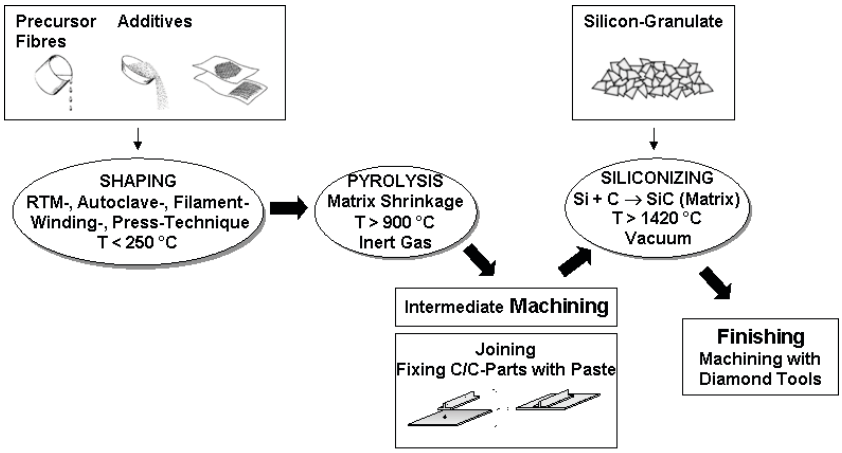


Figure 2: Schematic diagram of fabrication process

In the third step, the C/C component is siliconized via melt infiltration. The component is placed into a coated graphite crucible and solid silicon is added as granulated pure metal. After heating up to over 1420°C (melting of silicon) the porous C/C component is filled with liquid silicon due to the capillary effect of the micro-cracks and the low viscosity of the molten silicon. In an exothermic reaction between the molten silicon and the carbon matrix, silicon carbide is formed along the micro cracks encapsulating the carbon fiber bundles. The siliconizing is carried out under vacuum at a temperature of 1650°C. The resulting C/C-SiC composites contain three material phases. These are the carbon phase consisting of carbon fibers and residual carbon matrix, silicon carbide as the main matrix constituent and a small share of unreacted free silicon.

TEST RESULTS

The test matrix is shown in Table 1. Three C/C-SiC and one C/C panels were tested, with multiple tests per panel. The test hardware was allowed to cool for approximately 20 minutes between tests. The facility run number is shown, followed by the simulated flight Mach number, either Mach 5 or 6. The actual (aerodynamic) Mach number of the flowfield within the DCR was ~ Mach 2. As mentioned previously, two TC's were embedded in the panel from the back surface. In each of the panels tested, there was one Type K and one Type S thermocouple installed. Shown in the table are the temperatures of the TC at the end of each test. The facility total temperature and pressure are also shown in the table, along with the equivalence ration (ER) of the injected hydrogen fuel. Finally, the fuel-on time and the total test duration are shown.

Table 1: Test Matrix

Panel	Run No.	Simulated Flight Mach No.	Temperature at end of Test, °R		P_0	T_0	ER	Fuel-on Time [sec]	Total Test Duration [sec]
			Type K	Type S	[psia]	[°R]			
C/C HP635-7	68	5	859	858	96.1	2131		n/a	20
	69	5	1507	1450	96.1	2117	0.556	35	40
	70	5	1611	1548	95.9	2130	0.741	35	40
	71	6	1002	1031	89.4	2546		n/a	20
	72	6	1878	1796	91.8	2611	0.986	40	45
	73	6	1026	1015	88.8	2558		n/a	20
	74	6	1987	1800	91.6	2594	1.003	39	44
	75	6	2051	1835	90.8	2626	1.023	39	44
C/C-SiC #4	52	5	1005	972	92	1939		n/a	20
	53	5	1248	1193	91.6	1957		n/a	40
	54	5	997	971	91.8	1989		n/a	20
	55	5	1214	1329	92.6	2020	0.53	14	20
	56	5	1044	1062	94.2	2035		n/a	20
	57	5	1737	1834	94.5	2070	0.58	32.5	38.5
	58	5	1076	1087	94.2	2070		n/a	20
	60	5	1010	999	94.6	2059		n/a	20
C/C-SiC #3	62	6	1281	1291	90.6	2624		n/a	20
	63	6	1295	1319	90.7	2647		n/a	20
	64	6	2025	2206	90.6	2648	1.01	30	40
C/C-SiC #1	76	6	1382	1317	89.6	2591		n/a	20
	77	6	2352	2515	91.9	2599	1.009	39	44
	78	6	2342	2504	91.2	2639	1.039	39.5	44.5
	79	6	2336	2462	91.9	2654	1.047	39.5	44.5

In an effort to increase the hot surface temperature on the final test panel (Runs 76-79), zirconia insulation was placed on the cool surface of the panel (backside). The insulation increased the hot surface temperature by ~177°C (350°F).

A plot of the data from the two embedded TC's is show in Figure 3 (for Run 79). The Type S TC reads a higher temperature, and was located upstream of the Type K TC. The facility total pressure (PTOTAL1) and fuel supply pressure (FUEL1P) are also shown on the figure. A photograph of the test panel post-test is also shown on the figure.

After the final run, the test panel was removed from the carbon steel fixture. Despite the high heating that the panel was subjected to during the last test and the melting of the steel sidewalls, the panel showed practically no damage, just a slight stain where the melted steel came into contact with the panel.

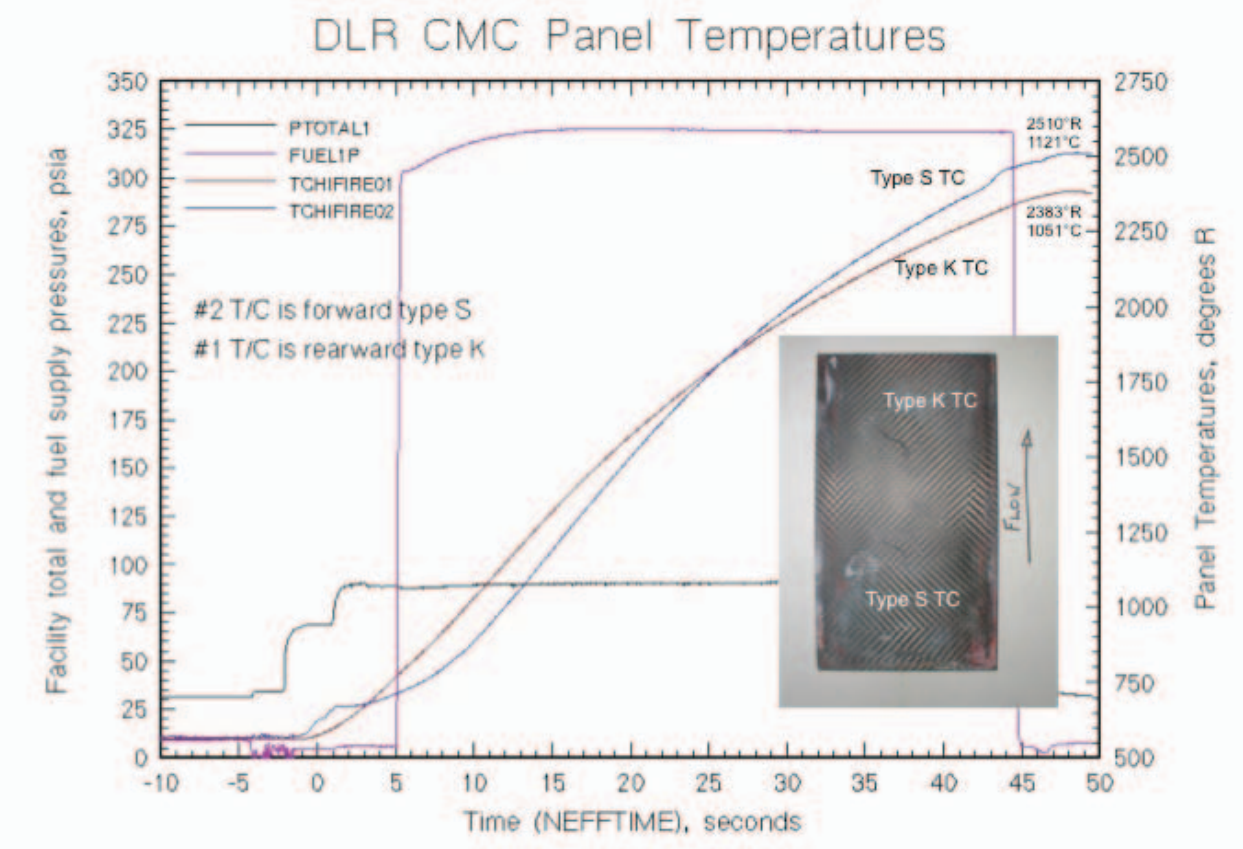


Figure 3: C/C-SiC panel test temperatures during Run 79

Very little recession was measured on the test panels. The largest recession was 0.051 mm. Several locations have zero recession. Table 2 shows the pre- and post-test thickness measurements, taken prior to the first run with panel C/C-SiC #1 and taken again after the last run with C/C-SiC #1. As indicated by the recession measurements and the post-test photographs of the panel, the panel survived the series of tests with negligible deterioration.

Table 2: Pre-and post-test thickness measurements for C/C-SiC panel #1

	Thickness (mm) / Measurement location							
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Pre-test	8.026	8.052	8.052	8.103	8.306	8.280	8.052	8.052
Post-test	8.026	8.001	8.026	8.077	8.306	8.255	8.026	8.052

After the C/C-SiC panels were tested, a single C/C panel from DLR was also tested. A plot of the two embedded TC's is show in Figure 4. The Type S TC reads a higher temperature, and was located upstream of the Type K TC. The facility total and fuel supply pressures are also shown on the figure. A photograph of the test panel post-test is also shown on the figure.

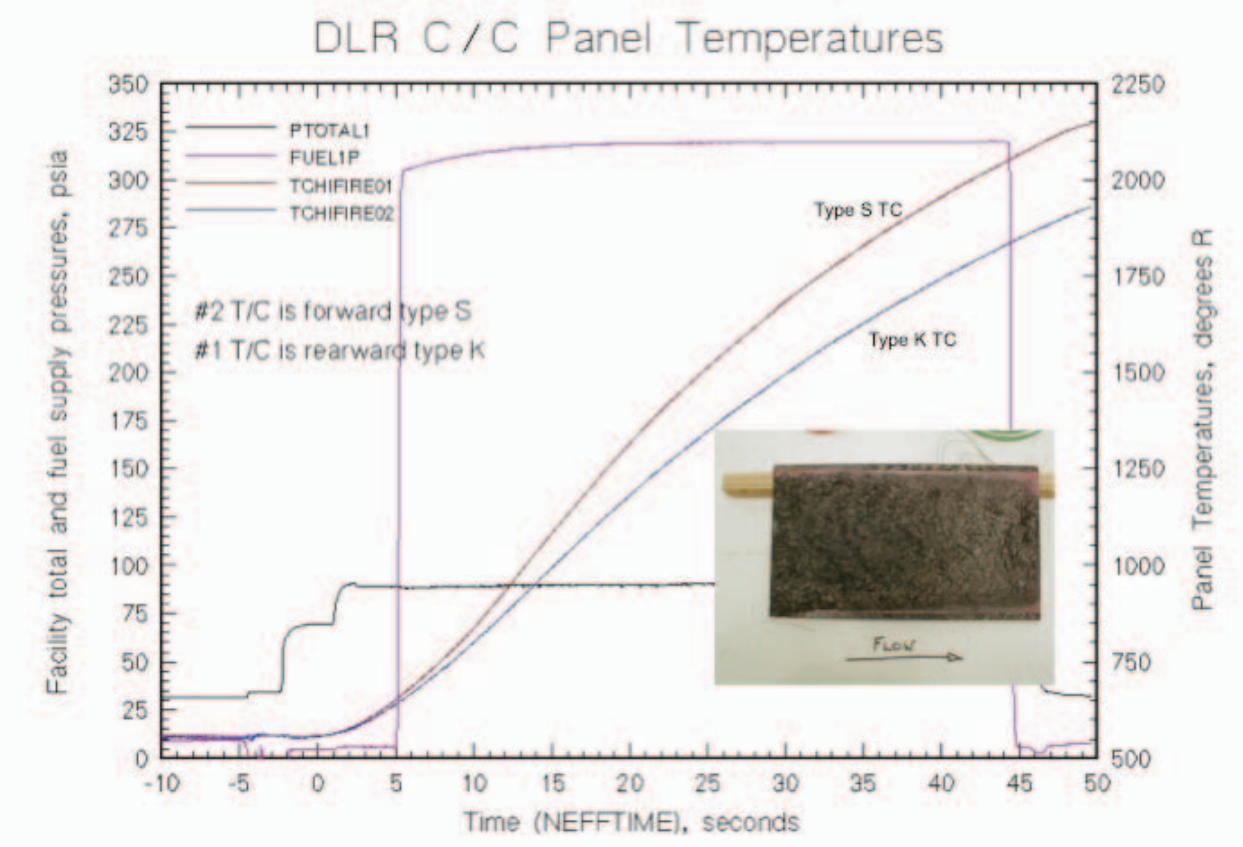


Figure 4: C/C panel test temperatures during Run 75

As shown in Table 1, the panel was tested for 100 seconds at Mach 5 conditions, and 193 seconds at Mach 6 conditions. The measured recession data is shown in Table 3.

Table 3: Pre-and post-test thckness measurements for C/C panel HP635-7

	Thickness (mm) / Measurement location							
	1	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Pre-test	10.008	10.008	10.008	10.008	10.084	10.109	9.957	9.931
Post-test	9.881	9.881	9.957	9.881	9.830	9.627	9.855	9.881

POST-TEST INVESTIGATION OF C/C-SIC PANELS

Several of the test panels were fabricated from C/C-SiC material as described previously. The surface that was to be exposed to the exhaust flow of the combustor was intentionally not machined in order not to remove the as-fabricated SiC layer that forms as the result of the material processing. The backside of the panels was machined and grooves for thermocouple installation were created. Post-test investigations were performed with samples from all three tested C/C-SiC panels. Since the findings were consistent between the three panels, the process is described for panel #1 only.

A number of samples were prepared on the centerline of the panel and on a line that was 20 mm from the side edge of the panel. The panel was cut into 19 pieces, and each piece was numbered accordingly. The samples were prepared for investigations in the SEM, i.e. they were embedded in a packing material and the surface to be investigated was ground and polished. For the investigations presented here, two samples from each panel were prepared. These were the cut-outs #5 and #17 from the centerline, as shown in Figure 5. Cut-out #5 was upstream on the panel closest to the combustor. Cut-out #17 was on the downstream end of the panel close to the nozzle exit.

Figure 6 shows a scanning electron microscope (SEM) image with the typical C/C-SiC microstructure. Carbon fiber bundles are separated by crack volumes that are filled with SiC at the boundaries, and Si in the case when the width of the pore or crack is comparatively large. The surface of the sample is not flat due to the fact that it was not machined, so the topology of the fiber textile is seen. The area on the top part of the image where numerous bright spots appear is the packing material used for embedding the sample.

There is no evidence of significant oxidation which might have resulted in degraded fibers or matrix near the surface. What can be seen is a thin bright layer on the surface that was identified as silicon dioxide (or silica) using electron diffraction spectroscopy (EDX) analysis.

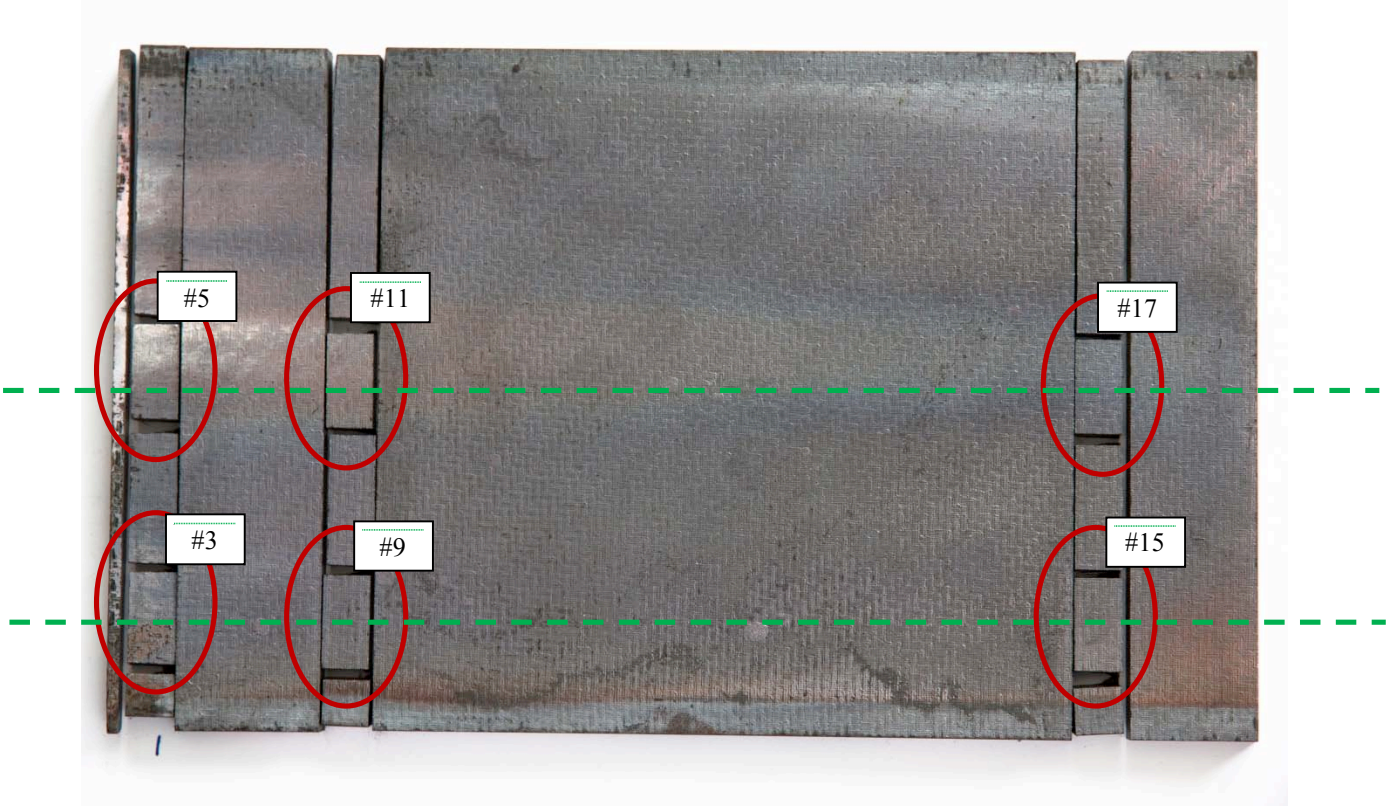


Figure 5: Panel 1 with cut pattern for the sample preparation

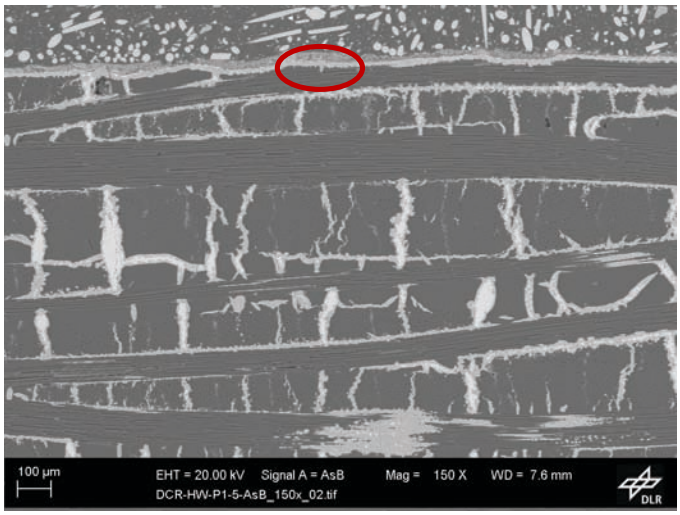
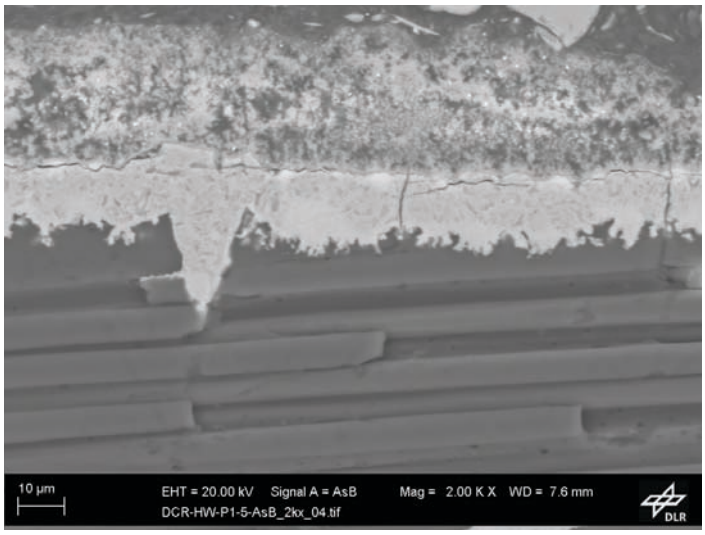


Figure 6: SEM image of sample #5 from panel 1. There is a thin layer on the surface that is identified as silica. The circle indicates the area that is shown in higher magnification in Figure 7.

The close-up of the detail highlighted in Figure 6 is shown in Figure 7. The different material phases can be distinguished very well. There is a fiber bundle with silicon carbide on the surface. On the surface, above the silicon carbide is a thin layer of silica of around 20 µm thickness. The SiO_2 layer on the surface does not have a constant thickness in every location over the sample. There are some spots where no SiO_2 layer can be found and there are locations with a thinner layer compared to Figure 7, but in general, there is a SiO_2 layer over most of the sample.



Silicon dioxide, SiO_2

Silicon carbide

Carbon fibers and carbon matrix

Figure 7: SEM image of the SiO_2 scale on top surface of the sample

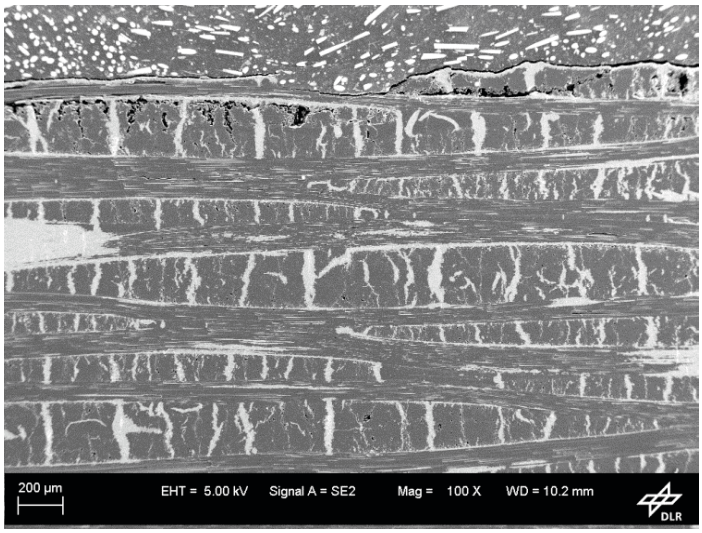


Figure 8: SEM overview image of sample #17 from panel 1, exposed surface is on the top

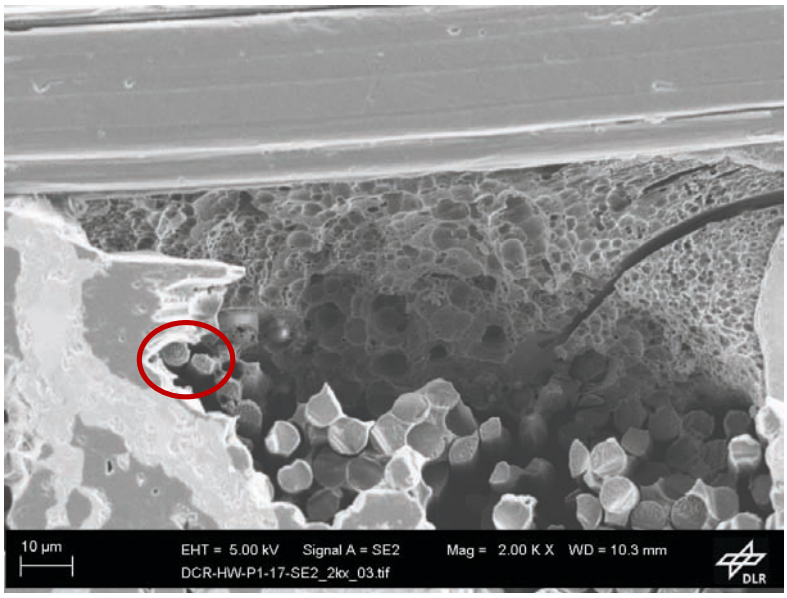


Figure 9: Close-up SEM image showing some indication of matrix oxidation in the pore.

Figure 8 shows the surface of sample #17 from the end of the panel. There are a few cracks and pores in the two topmost layers. These could be the result of oxidation of the carbon since close-up images show signs of oxidation

in the pores at fiber ends and on the matrix; however, the amount of pores was significantly smaller in other samples, so in part they can also be the result of sample preparation as there is a tendency of sample material to break out at the edges due to the relative softness of the surrounding packaging. Figure 9 shows a close-up SEM image showing some indication of matrix oxidation in the pore. Note the clean-cut ends of most of the fibers as the result of the preparation. The circle indicates the region detailed in Figure 10.

Figure 10: SEM image of fiber ends that have a rough cross-section which is interpreted as the effect of oxidation.

The conclusion is that there was some oxidation on the surface of the panel, but limited to only the topmost one or two layers, with no significant degradation of the panel.

CONCLUDING REMARKS

The DLR C/C and C/C-SiC materials were tested at NASA Langley Research Center in a high-enthalpy direct-connect test facility at conditions simulating flight at Mach 5 and Mach 6 for several minutes. The C/C-SiC survived the high-temperature scramjet combustor environment with very little erosion. The C/C material experienced slightly more erosion, but still only a small amount. SEM analysis of the tested panels indicated very little oxidation of the exposed surface. The HIFiRE 8 flight, for which the materials were tested, is planned for Mach 7 for ~ 30 sec. Due to the successful performance of the test panels, the DLR C/C-SiC material is being considered for use as a passive combustor on the HIFiRE 8 flight vehicle.

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REFERENCES

1. Smart, M.K., Hass, N.E. and Paull, A., “Flight Data Analysis of the HyShot 2 Flight Experiment”, AIAA Journal, Vol. 44, No. 10, pp 2366-2375, 2006.
2. Guy, R.W.; Rogers, R.C.; Puster, R.L.; Rock, K.E.; and Diskin, G.S.: *The NASA Langley Scramjet Test Complex*, (AIAA-96-3243), AIAA, ASME, SAE, and ASEE, Joint Propulsion Conference and Exhibit, 32nd, Lake Buena Vista, FL, July 1-3, 1996.
3. Krenkel, W.: Ceramic Matrix Composites, ISBN 978-3-527-31361-7 – Wiley-VCH, Weinheim, 2008