Development Chain of Advanced CMC Materials for Dual-bell Rocket Nozzles

By L. Klopsch, C. Génin†, M. Frieß, F. Breede and D. Koch

German Aerospace Center (DLR)
Institute of Structure and Design
Pfaffenwaldring 38-40, D-70569 Stuttgart

† Institute of Space Propulsion
Langer Grund, D-74139 Lampoldshausen

A cold flow study was conducted as preparation for C/C-SiC material application as rocket nozzle structures. Three nozzles sharing a common inner contour were manufactured and tested in similar conditions. The flow separation behavior was compared between the reference configuration, the ceramic nozzle extension with metallic throat segment and the fully ceramic nozzle. The side load measurements show that a non-optimal interface to metallic and ceramic structures generates only a moderate increase. The higher wall roughness of the ceramic models leads to a nonlinear evolution of the separation position and an increase of the side load amplitude within an acceptable range.

1. Introduction

In the framework of the special research field SFB-TR40, cooperation between two DLR institutes in Lampoldshausen and Stuttgart has been started to investigate the application of C/C-SiC material for rocket nozzle structures. The present study concentrates on a cold flow campaign conducted at DLR’s P6.2 facility. A nozzle contour has been manufactured into three test specimens: a reference nozzle out of PMMA with a very low wall roughness, a first C/C-SiC nozzle extension with a metallic throat segment and ceramic extension, and a fully C/C-SiC nozzle. The study focused on the influence of contour roughness on flow separation in the nozzle. The inner surface of the final ceramic nozzle structure was not machined after the manufacturing process, hence a net-shape inner nozzle contour was achieved. A second point of interest for the present study was to investigate and optimize the interface between metallic and ceramic structures, as well as the implementation of measurement systems on the nozzle wall. The nozzles were tested under the exact same conditions.

The feeding pressure was progressively increased and decreased to simulate the complete start-up (and shut-down) process, until full flowing conditions. Aim of the study was to gain experience in the implementation and instrumentation of ceramic matrix material for nozzle structures, which will be used in future common hot flow test campaigns. [1]
2. Testing of nozzles

2.1. Test facility
The tests have been conducted at the blow down cold flow facility P6.2 at DLR in Lampoldshausen. The nozzle models were mounted on the horizontal rig and tested under ambient conditions. The feeding line provides dry nitrogen under pressure up to 55 bar. The resulting nozzle pressure ratio (NPR, feeding over ambient pressure ratio) was slowly ramped up and down from NPR 1 up to 55, corresponding to full flowing nozzle condition. Each test was conducted at least three times to ensure the repeatability of the measurements.

2.2. Instrumentation
The position and evolution of the flow separation from the wall was recorded through pressure ports placed along the nozzle contours. Small orifices in the wall (0.5 mm diameter) permitted to connect the flow with the pressure transducers through a system of small pipes fixed in the wall and Teflon tubes. Up to 25 positions were measured on each nozzle with a spatial resolution of 4 mm. The transducers have a measurement range of 0-1 bar. The total pressure and temperature were also recorded. A scanning rate of 1 kHz with a filtering at 160 Hz was used for the signals.

In addition to the pressure measurements, side load generation was monitored for the three models. A thin walled bending tube was placed upstream the nozzle convergent part [2]. The deformation of the tube was recorded using strain gauges placed on the tube in two directions (horizontal and vertical). The gauges were then wired into a full Wheatstone bridge in order to compensate the components of the signal which were not directly the bending. For each nozzle, a calibration procedure was realized with a series of mass attached to the nozzle end and suddenly released. The calibration permits an easier comparison of the side loads recorded for the different nozzles. The signals presented in this study are given in Newton and correspond to an equivalent force acting at the nozzle end. Fig. 1 shows the fully instrumented reference nozzle mounted at P6.2 facility.
Three nozzle models were used for this investigation. All three share a common inner contour designed as a TIC (truncated ideal contour) with a design Mach number of 5.3. Throat radius was $R_{th} = 10\text{mm}$ and total length was $L_{tot}/R_{th} = 11.75$. The reference nozzle was made out of PMMA with a wall thickness of 8mm and a very low wall roughness ($Ra = 0.8$). Two alternatives were chosen for the ceramic matrix nozzles: a first one featuring an interface at area ratio $\epsilon = 5$ between metallic and ceramic structures, CMC1, and a second full ceramic structure, CMC2. The nozzle model with an interface is representative to a real full scale application where the throat segment of the nozzle had to be metallic and conventionally cooled. The interface region may present contour irregularities, which can lead to increased side load when the separation position passes it.

### 3. C/C-SiC Nozzles

#### 3.1. Manufacture

Ceramic matrix composite materials have shown the potential to replace super-alloy materials in rocket nozzle application, e.g. Vinci or RL-10B [3–7]. The latter CMC nozzle structures are manufactured using rather time-consuming processing routes like chemical vapour infiltration (CVI) or polymer infiltration and pyrolysis (PIP).

The material in this study was manufactured using the liquid silicon infiltration (LSI) process, which exhibits a significantly shorter processing time and a lower final porosity than CVI- or PIP-materials. DLR's C/C-SiC nozzle structures are currently manufactured by filament winding technique and an adapted liquid silicon infiltration route. The manufacturing process can be divided into the following main fabrication steps: Design of the fiber architecture, wet filament winding of the nozzle green body, carbonization, re-infiltration and densification, siliconization and final machining.

In order to manufacture a nozzle structure with a complex divergent wall contour, con-
sisting of increasing diameters over the axial length direction, a sophisticated winding software is essential to simulate the desired fiber architecture. DLR is using the latest version of Cadwind V9 Expert filament winding software. The fiber preforms were generated using a three axis filament winding machine. According to the nozzle geometry the fiber architecture should be designed. This step includes the creation of single fiber layers with specific fiber orientations within the rules of filament winding. The nozzle preform was fabricated using wet filament winding technique. The continuous carbon fiber (Toray T800 12K) was impregnated with a high carbon yield phenolic precursor and was then wound onto the nozzle mandrel.

The CFRP green body was then removed from the mandrel and pyrolyzed for the first time to a C/C body. The carbonization process was formed at 1450 °C in inert gas conditions for 2 days. During carbonization the polymer matrix is converted to amorphous carbon. The carbonization leads to a volumetric contraction and mass reduction of the polymer creating a porous C/C composite. Generally, this process forms a pore and crack system, which represents the open porosity. To reduce the caused open porosity one re-infiltration process was performed after the first carbonization. This intermediate step is necessary to reduce the residual silicon after the siliconization process and improve the final material microstructure. The re-infiltration process was based on vacuum assisted resin infiltration method using foils to form a near net shape mould. [8]

During the last processing step the C/C nozzle structure was infiltrated with liquid silicon by means of capillary forces at 1450 °C. Thereby the molten silicon immediately reacts with the carbon surfaces to form silicon carbide. In general, the final microstructure can be characterized as encapsulated carbon fibers in carbon matrix segments surrounded by silicon carbide layer. For this reason the material is called C/C-SiC. Nevertheless, a certain amount of unreacted silicon remains inside depending on the morphology of the open porosity.
The processing time is in the range of 2-3 weeks. It should be noted that this manufacturing route provides CMC nozzle structures with a net-shape nozzle contour; hence no further machining of the inner nozzle contour was applied. Only the interface was machined. For this study a C/C-SiC nozzle extension with a metallic throat segment (CMC1) and a fully C/C-SiC nozzle (CMC2) were prepared. The transition from the metallic throat to the C/C-SiC nozzle (CMC1) was realized at an area ratio $\epsilon = 5$. The density of the resulting C/C-SiC nozzle was 1.8 g/cm$^3$ and exhibited a residual open porosity of less than 5%. The surface roughness of the inner nozzle wall was about 2-6 $\mu$m (Ra) and 10-30 $\mu$m (Ra), respectively. The gas permeability of this C/C-SiC material is about $1 \times 10^{-3}$ ml/bar/s. Fig. 3 shows a typical micrograph of the resulting C/C-SiC material. In this material carbon fibers are embedded in carbon matrix, forming dense C/C-bundles (dark grey). Those C/C-blocks are surrounded by a silicon carbide matrix and some residual silicon (light grey).

Fig. 4 shows the corresponding CAD design of the resulting C/C-SiC nozzle components. Holes were integrated into the nozzle wall to provide pressure measurements. Metallic tubes were glued into the holes of the CMC nozzle using a ceramic adhesive. The metal tubes were then connected to the pressure sensors.
3.2. Nozzle contour analysis

Nozzle contour measurements were performed after processing using computer tomography (CT) including CAD comparison. The inner nozzle wall contour showed a very good agreement with the required nozzle contour after processing. The deviation from the required nozzle contour was in the range of -0.25 to +0.25 mm.

After machining of the interface and mounting to the test bench flange the inner nozzle contour again was inspected using a 3D measuring arm (accuracy 0.0025 mm) to provide accurate contour data of the resulting nozzle contour.
It was found that within CMC1 the machining of the interface surfaces was not executed perfectly. In the mounted configuration a significant deviation was detected starting at the transition of the metallic throat section to the CMC1 nozzle, see Fig. 5. It was found that CMC1 was slightly tilted by 0.6° which resulted in a contour deviation in the range of -0.8 and +1.5 mm.

The full ceramic nozzle configuration (CMC2) on the other hand showed a very good contour agreement after mounting, see Fig. 6. The deviation was in the range of -0.2 and +0.3 mm. The contour data of the mounted configurations was then provided for flow calculations and pressure interpretations.

4. Results and Discussion

4.1. Wall pressure distribution

The pressure transducers placed along the nozzles wall yield information on the flow behaviour in the three test models. For a better understanding of the experimental measurement, the wall pressure distribution was determined using an in-house code based on the method of characteristics with the actual inner contour of both ceramic nozzles as input for the program. Fig. 7 shows the calculated (plain line) and experimentally measured (symbols) wall pressure distribution in the full flowing reference nozzle and CMC1. The experimental data presented here were recorded during a typical test and time averaged. The deviation from test to test is negligible (<1% of the pressure values).

In CMC1, a significant deviation is visible at X/Rth 3.2, which corresponds to the interface at area ratio 5 between the metallic and ceramic structures.
The wall pressure increases locally due to the recompression of the flow at the interface, presenting a backward facing step in the axial line, along which the pressure ports were placed. Behind the interface, hence behind the recompression shock, the wall pressure decreases slightly compared to the reference values. Further downstream, the difference between reference and CMC1 decreases, and the measurements are very similar.

The same procedure was applied to the second ceramic nozzle CMC2 which presents a much higher inner contour accuracy. The wall pressure distributions are presented in Fig. 8 for reference nozzle and CMC2, for both the experimental and the recalculated pressure values. Between the axial positions $X/Rth=4$ and 8, the pressure along the wall of CMC2 is slightly higher than along the reference nozzle wall.

The inner surface roughness of the ceramic structure CMC2 may increase the boundary layer thickness compared to the smooth reference nozzle and hence the local wall pressure. Toward the end of the nozzle, the pressure values are very similar for all three nozzle models.
The wall pressure evolution during the up-ramping process gives in addition information on the flow separation behaviour. Fig. 9 illustrates the position of the separation from the wall as a function of the NPR. As expected from earlier studies [9], the separation position evolution in the reference nozzle is almost linear in the most part of the nozzle and slows down toward the nozzle end. In both ceramic nozzles, for a given NPR value, the flow separation is shifted upstream compared to the reference nozzle. The difference decreases toward the nozzle end and all three nozzles are flowing full for comparable NPR values. The flow separation behaviour is in accordance with the similar wall pressure distribution in the last part of all three nozzle models.

The implementation of a ceramic matrix structure in a nozzle can lead to higher surface roughness, or small contour deviations at the interface. Both effects will slow down the start-up process as the separation point is shifted further up-stream for the same flow conditions. However the separation position moves faster toward the nozzle end, leading to full flowing conditions for similar conditions as in reference nozzles.
The side load measurements were post-processed using the calibration coefficients defined during the preparation phase of the test campaign.

As the evolution of the separation point along the nozzle contour is less smooth for the ceramic nozzles, it was expected to lead to an increase in the side load amplitude. Fig. 10 depicts the typical side load generation of all three nozzles over NPR during the nozzle start-ups. Both ceramic nozzles produce higher side loads compared to the smooth reference nozzle for most of the NPR conditions. When considering NPR values over 10, an increase of 22% to 25% on the averaged values appears for CMC1 and CMC2 respectively. The difference in peak values is more dramatic: an increase of 25% for CMC1 and up to 65% for CMC2 was measured. However, these values must be put in perspective with the side load generation at very low NPR. All three nozzles present a side load peak around NPR 5.

This peak is due to a partial flow re-attachment as the separation passes the throat region. This peak is known to be very high and is crucial for dimensioning of the system. CMC1 generates a peak of similar amplitude, which was expected, as the throat segment of CMC1 was made out of aluminium with low surface roughness. CMC2, however, presents a very different behaviour with half the amplitude of the reference peak and a start for lower NPR value (around NPR 2). Considering the overall side load generation, the maximal values are within the expected amplitudes of the reference nozzle.

Fig. 11 depicts side load generation and NPR variation over time during a test of the reference nozzle and of CMC1. It can be seen that the overall behaviour of side load generation is very similar over time with slightly higher amplitudes for CMC1. In addition, at the times t=10s and t=50s, corresponding to NPR value around 12, the side loads generated by CMC1 present an amplitude peak. This increase in side load corresponds to the passage of the separation front over the interface from metallic throat to ceramic extension. As the nozzle extension is slightly tilted from its own axis, the separation front becomes significantly asymmetrical while passing the interface. However, the side load increase remains lower than the maximal side loads at low NPR.
4.3. Shock system

The evolution of the shock system was recorded using imaging and video monitoring. In the present study, the focus is given on the video monitoring. The feeding gas expands through the nozzles and the flow temperature decreases. In some cases, the temperature of the nitrogen can undergo the condensation temperature in the core flow. The shock system out of the nozzle becomes then visible without the additional measurement technics needed for imaging. In addition, a significant condensation of the nitrogen in the core flow diminishes the quality of the images. The condensation superimposes then with the density variations in the exhaust flow.

Fig. 12 illustrates the shock system out of both ceramic matrix nozzles visible through nitrogen condensation in the core flow. The upper picture was taken out of CMC1 nozzle, the lower one out of CMC2, both of them for full flowing conditions. Visible is the condensate nitrogen, delimited by the oblique shocks and the Mach disk. In the flow out of CMC1, additional lines are visible in an almost axial direction and interacting with
the shock system. These weak internal shocks are probably due to the recompression of the flow at the interface between throat segment and ceramic extension. No deviation in the oblique shock or in the Mach disk can be noticed. In addition to the almost axial direction of these shocks, this indicates that no dangerous shock interaction like the one leading to RSS (Restricted Shock System) are to be expected from an interface, even a non-optimal one, at area ratio 5.

Conclusion
A cold flow test campaign has been conducted in cooperation between the institutes of Space Propulsion and of Structures and Design to investigate the flow behaviour in nozzles with ceramic matrix composite structures. Two configurations were of interest, a full ceramic nozzle and a structure with a metallic throat segment and a ceramic extension. The wall pressure measurements have shown an up-stream shift in the axial separation position in the ceramic nozzle structures compared to the smooth reference nozzle. The full ceramic nozzle generates a much lower peak at low NPR, but higher overall side loads. The maximal side load amplitudes remain in the range of the values generated by the reference nozzle. In conclusion, the ceramic structures have shown
a satisfying behaviour when implemented for nozzles. Future investigation will have to confirm the finding for hot flow conditions.

Acknowledgments

Financial support has been provided by the German Research Council (Deutsche Forschungsgemeinschaft - DFG) in the framework of the Sonderforschungsbereich Transregio 40. Computational resources have been provided by the Stuttgart High-Performance Computing Center (HLRS).

References