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Particle size in SRM plume: assessment of collection method

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Abstract

The present work aims at giving an overview of the current experimental activity status of an intrusive technique for particles capturing in supersonic-high temperature flows for the use in solid rocket motors plume. The innovative technique was conceived in the EMAP (Experimental Modelling of Alumina Particulate in Solid Booster) framework, an initiative financed by the European Space Agency aiming at the characterization of the alumina exiting from rocket nozzles in terms of size, temperature, and spatial distribution. This kind of information are of paramount importance for the environmental impact assessment of space launch activity. Experimental tests are still ongoing and the present paper discusses some of the critical aspects, solutions, and open questions arising during the hot fire testing of this innovative intrusive technique.

1. Introduction

The EMAP project (ESA contract No. 4000114698/15/Nl/SFe) is an international joint effort financed by ESA involving research groups from DLR (Germany), FOI (Sweden), and POLIMI (Italy). The aim of the project consists in gaining a better understanding of aluminum metal particles when they are exhausted from the nozzle of a rocket motor after combustion. It is known that particles in the upper atmosphere can reside for long time, depending on their size. Particulate at stratospheric level can interact with the solar irradiation and can alter the so-called radiative forcing (already assessed for soot residues generated at high altitude).[1] These particles can even play a role in the depletion mechanism of ozone by acting as substrate for chlorine.[2]

As for all manned activities, space launch operations are not exempt from environmental impact. An AGARD meeting in 1995 tried to answer to questions about the pollution introduced by rocket use, globally. The conclusions suggested that the impact was minimal, compared to other manned activities.[3] Nowadays, the sensibility over environmental issues is much different. The perspectives of a strengthening space economy is rising the concerns about real environmental effects of repeated launches with increasing rates from same locations (space ports).

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Plume detailed characterization represents the starting point for the elaboration of a reliable model for atmosphere interaction analysis. For aluminized rocket propellants, the characterization should provide the knowledge on the particle size distribution and the composition (namely, the residual aluminum). In this respect, EMAP project has been conceived to implement a set of characterization methods for gathering of these information from small-scale rocket. Among the others, the Space Propulsion Laboratory of Politecnico di Milano (SPLab-POLIMI) has developed a probe for the collection of condensed combustion products directly at the nozzle exit. Description of probe features and development process was addressed in past works.[4] As the fire tests are ongoing and analysis data are not yet available, the present paper discusses an overview of the project some initial lessons learned from both cold and hot-flow operations of the probe.

2. Overview of applied measurement techniques

In the following, a short overview is given on the measurement equipment in the *vertical test section Cologne (VMK)*. VMK is a blow-down type wind tunnel facility with an open test section for tests in the subsonic and supersonic flow regime.[5] The combination of having the wind tunnel nozzle vertically aligned and featuring an open test section is advantageous for the current tests since it offers the space which is required for the highly instrumented experiments in the frame of ESA-EMAP.

The image in Fig. 1 provides proof that the term 'highly instrumented' is justified. It shows the inside of VMK with the wind tunnel model integrated in the wind tunnel nozzle. The wind tunnel model mimics the base region of a space launcher. To simulate a flight-realistic exhaust plume, a solid rocket motor is integrated in the base model. The rocket motor expels the hot jet in the upward direction through a nozzle. Simultaneously, the wind tunnel provides an ambient flow at Mach 0.6. In other words, a co-flow between a cold ambient flow and a hot solid propellant exhaust jet is investigated.

For this reason, the wind tunnel model is surrounded with measurement equipment. Just downstream from the nozzle exit of the rocket motor, one can find the rocket plume collector (RPC). The RPC is used to collect particles for the determination of the particle size distribution. It is protected from the hot exhaust gas by a protective shield, which only opens for 0.5 s during each run. The aerodynamic particle sizer (APS) is even farther downstream and serves the same purpose by means of the scattering intensity of light on particles and by aerodynamic measurements. The heat flux is captured with two different methods: First, an infrared camera (IR) acquires the temperature evolution of the base, and second, a Gardon gauge points at the rocket exhaust plume to collect data of its heat flux radiation. The temperature of the rocket exhaust plume itself is assessed with spectroscopic measurements. Measurements by means of Fourier-transform infrared spectroscopy (FTIR) and by spectroscopy in the ultraviolet-visible range (UV-Vis) are conducted. Moreover, in the frame of this project, AEM was developed, which stands for *alumina emission measurement*. The idea is to determine the phase state of the alumina particles inside the plume by using their thermal emission. These spectroscopic methods also provide information of the various species in the plume. For *particle image velocimetry* (PIV), a pulsed laser system illuminates the particles in the rocket exhaust plume. Cameras on either side perpendicular to the laser sheet capture the scattered light. The PIV camera targets as region of interest about one to two shock cells of the jet shock train. The camera for DIPSD is focused on a very small range (order of 5 mm) of the exhaust plume. On the one hand, the intention of DIPSD is to directly gather data of the particle size by imaging glare points (*direct imaging particle size determination*). On the other hand, it might also be used to deduce information about the velocity by cross-correlating particle images. The results of the velocimetry methods can then be cross-checked with highly accurate Laser-2-focus (L2F) measurements. High-speed schlieren (HSS) imaging concludes the list of measurement techniques for the exhaust plume. It is used to determine the density gradient field, and in particular, to visualize expansion and shock patterns in the flow field. The conditions of the rocket motor are monitored with two pressure sensors, and the ambient Mach number with a pitot-static/Prandtl tube. Note that all measurement equipment is put in boxes or wrapped to protect it from the corrosive exhaust plumes. A complete overview to the project and the more details to the measurement systems are provided in.[6]

3. Supersonic Probe

The operational version of the probe merges the concept of a supersonic collection methodology[7] and a scrubber segregating the particles from the gas with a quenching liquid sprayed by sprinklers.[8] The logical scheme of the probe is shown in Fig. 3. A straight inlet duct captures the flow. After that, a secondary inert gas, nitrogen, is radially injected enabling a progressive deceleration and cooling of the primary flow in such a way that any strong shock is avoided or delayed until the particles reach a temperature lower than the alumina melting one. After a straight channel, which ensures the time and space for complete mixing, the flow enters into a conical divergent channel where the

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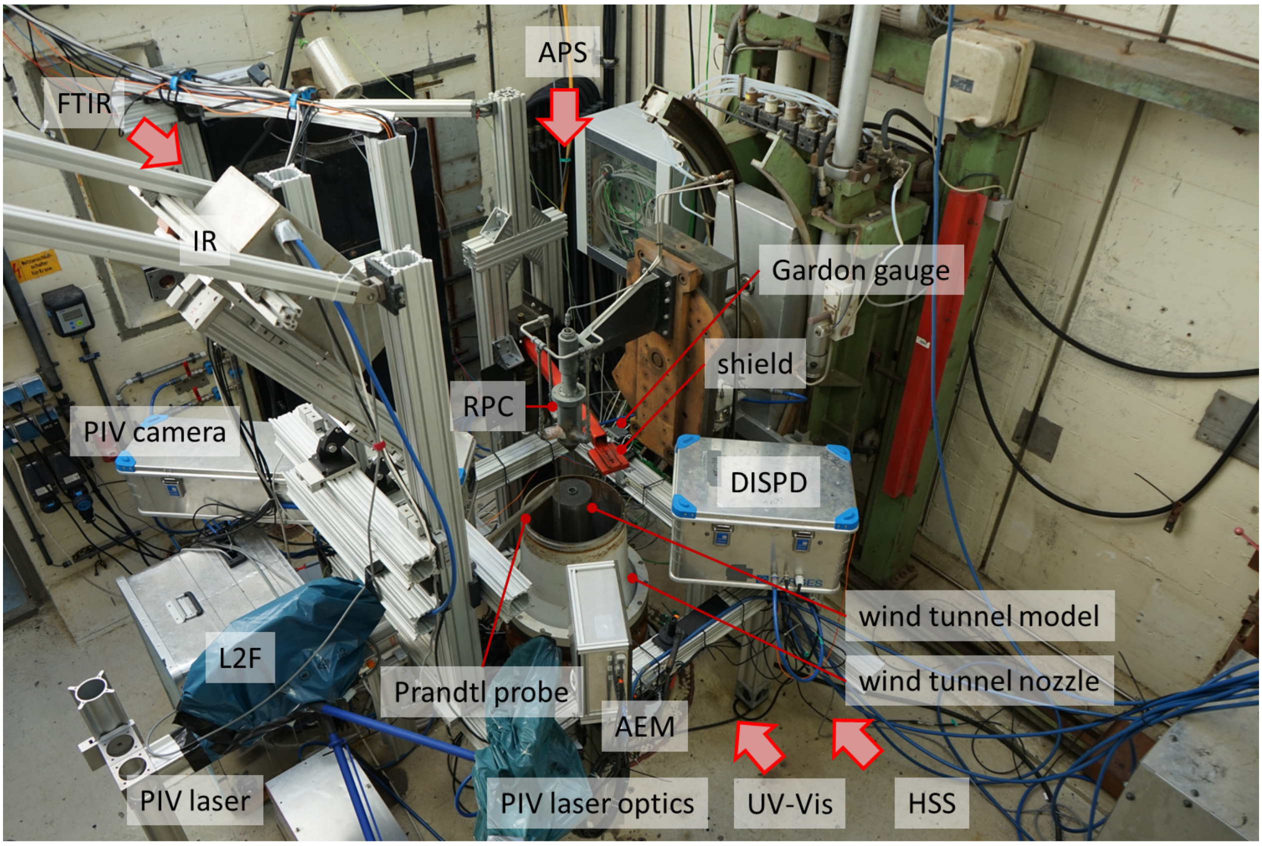

Figure 1: Top view on the wind tunnel and measurement setup (courtesy of DLR AS-HYP,[6]).

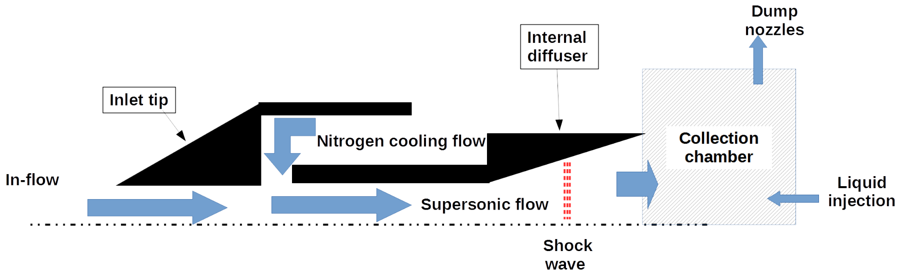

Figure 2: Logical scheme of the supersonic rocket particle collector (RPC).[4]

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supersonic-to-subsonic transition, if not yet occurred, is caused by a shock wave. The presence and position of the shock is defined by the global design which ensures a passive control of the downstream pressure. A more in-depth discussion concerning the design methodology can be found in Carlotti et al.[4]

The architecture of the probe tries to answer to the need of collecting particles which are representative of the population exiting from the nozzle. Other authors in the past collected residues from the nozzle using several options (e.g. quenching plates, motors placed inside closed volumes, cryogenic vessels, ...). Data sets are non homogeneous, though. When collecting particles post-combustion effects and mixing with the external atmosphere should be minimized to avoid potential chemical alteration. Moreover the collection should freeze the shape of the particles with reduced mechanical stresses. Finally, the population should be large enough to grant a statistically reliable data set. In this respect, the probe was conceived for the collection of the samples just downstream the nozzle. In addition, it provides cooling of the incoming flow with radial injection of nitrogen secondary gas as well as quenching and capturing of particles through liquid spray. Nitrogen cooling gas was selected on the basis of its inert nature, as already adopted by Gallier and co-authors[9] whereas the quenching liquid is a chlorine-based hydrocarbon derivative, which proved its efficacy in experimental quench-bomb tests.

The severe environment where the RPC is placed requires *ad-hoc* measures for thermal protection. The probe is made of steel and is directly exposed to the flow. Only the tip is produced with compact graphite, capable of withstanding high thermal stresses. The thermal design was conceived to sustain the flow temperature for exposure time in the range 0.5 s to 1 s. Passive thermal protections are applied to the body. A layer of aramidic fiber is applied to the metal directly facing the hot flow. High temperature refractory paste is applied to nitrogen pipes and connections. High temperature silicon sealing is used to fill the gaps between the components. In addition, a movable shutter is operated in front of the probe as a protective thermal shield to protect from longer exposures and removing transients.

Many uncertainties are involved in the probe design. The correct functioning of the probe is granted by defined pressure levels of the cooling nitrogen and inside the collection chamber, in turn influenced and defined by the mass flow rates and cross sections at stake. Nitrogen injection has been modeled to occur in sonic conditions. Hence, changing the area of injection will modify the pressure level and the mass flow of the nitrogen. Moreover, the nitrogen mass flow has to be sufficient to define a proper deceleration and cooling of the main flow ingested by the flow. The final properties of the flow are in turned influenced by the position of the shock wave, set by the pressure level inside the collection chamber, imposed by knowing the amount of mass flow incoming, the amount of quenching liquid sprayed, and the dump nozzle area. It is clear that the temperature level might influence the amount of evaporating tetrachlorethylene, in turn modifying the gaseous mass flow rate dumped by the nozzle and hence the pressure level in the combustion chamber. Moreover, the severe environment where the probe is inserted may erode the leading edge of the inlet duct, hence increasing the amount of the ingested flow and modifying all the conditions downstream. The influence of these parameters on the probe behavior has been discussed elsewhere: the readers are encouraged to refer to Carlotti et al.[10] A sensitivity and uncertainty analysis has been conducted considering eventual variations with respect to the nominal conditions of the ingested supersonic flow (i.e, Mach number, temperature, pressure and specific heat ratio), inlet cross section, nitrogen injection cross section, dump nozzle area and amount of evaporated tetrachlorethylene highlighting the robustness of the probe and counteracting effects of the input variable canceling each other and resulting in a global high working efficiency.

4. Assessment of critical components

4.1 Collection method

The collection method was based on a conical liquid spray acting in counterflow with respect to the ingested gas. The spray should be capable of capturing the particles and suspend the in the quenching medium in an annular region of the probe. The liquid is then removed after each test. A scheme of the capturing device is reported in Fig. 3.

The proof of concept of the collection methodology was achieved throughout a cold flow experimental campaign at a representative Mach number (i.e., M = 3). The tests were performed at the vertical wind tunnel facility (VMK) of DLR (Cologne). The VMK operates with a contour nozzle granting a Mach number equal to 3 at the exit section and expanding a flow at ambient temperature and at different total pressures. An in-house seeding generator for dispersing solid particles was implemented for the testing of the particles-collection methodology. Water was used in place of tetrachloroethylene to simulate the quenching liquid. As shown in Fig. 4(a), non symmetrical injection of nitrogen was implemented. This solution caused a spinning flow inside the probe capable of enhancing the mixing with the ingested mass flow rate. The probe ready for the test is shown in Fig. 4(b). In the picture the tip is metallic (instead of graphite) because during cold flow tests high temperatures were not a concern.

The required nitrogen mass flow rate (set for these test to 0.042 kg/s, c.a. 10 times the ingested supersonic mass flow) was controlled by a Bronkhorst flow meter (Bronkhorst IN-Flow F-116BI-IIU-90-V). A relay switch circuit

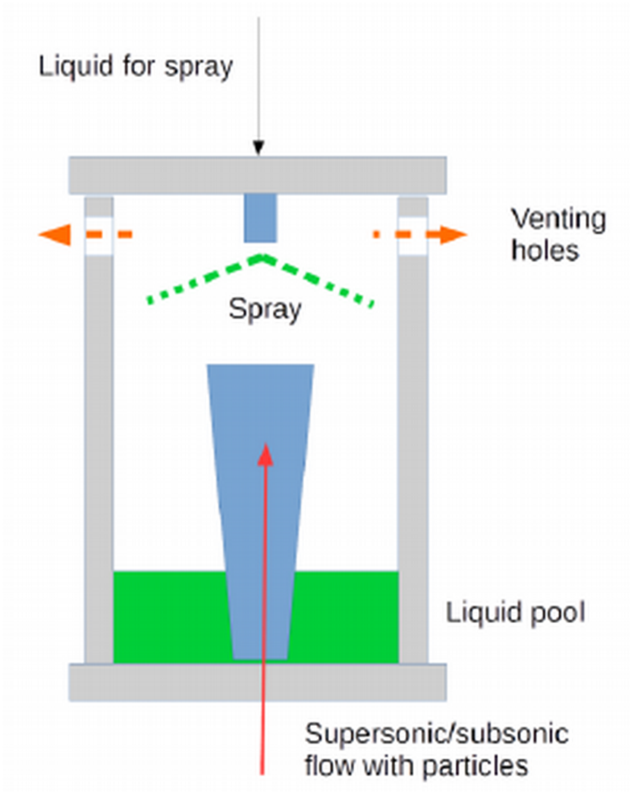
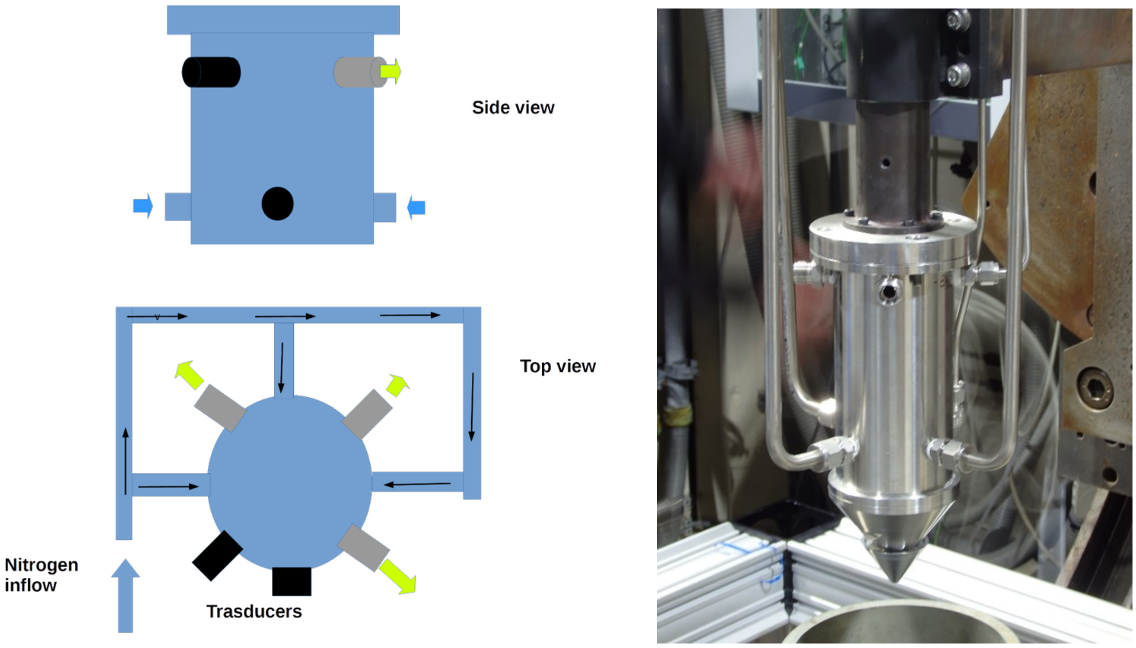


Figure 3: Scheme of the particle capturing method



(a) Scheme of nitrogen supply

(b) Probe in place

Figure 4: Experimental setup at the VMK wind tunnel

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Table 1: Validation of collection: comparison original and collected particle diameters

	D(3,2), [μm]	D(4,3), [μm]
original	5.693	17.032
collected	5.572	16.993

controlled by Arduino dictated the opening and closure of an electrovalve which assured the correct functioning of the injector for 1 second. Eventual pressure transducer could be inserted both in the nitrogen stagnation chamber and in the collection chamber for pressure monitoring. Inert micrometric particles have been used as reference to seed the flow. Sieved magnesium oxide powder was used. The particle size distribution was obtained using laser diffraction methodology with Malvern Mastersizer 2000 instrument with water dispersion. Table 1 summarize the tests comparing both the volume-mean and the surface-mean diameter of particles seeding the channel and after collection and treatment procedure. Numbers demonstrate that the collection method is capable of capturing a representative population of the particles seeding the flow. Data were reported for a total wind tunnel total pressure of 25 bar.

For the hot test the main concern was related to the evaporation of the quenching liquid. The temperature of the gaseous mixture entering the collection chamber is expected to be significantly higher than the liquid boiling temperature (i.e., 394 K). The mass evaporation interacts with the pressure in the quenching chamber, as the vapor will vent through the exhaust holes in addition to the expected gas mass. In this respect, an uncertainty analysis described in Sec. 3, highlighted no significant risks connected to the evaporation mechanism. In addition, if complete evaporation of the liquid spray occurs, the particles in the main stream may not be quenched and collected, exiting through the dump nozzles with the main flow. Initial outcomes of hot fire tests assessed that neither the quenching mechanism nor excessive evaporation were affected. As a qualitative indication, the image of the collected liquid is reported in Fig. 5. The liquid is gray instead of transparent, meaning that suspension of particles is present in there. The liquid was treated and the result of the process is not yet available.

Figure 5: Collected liquid from a hot fire test

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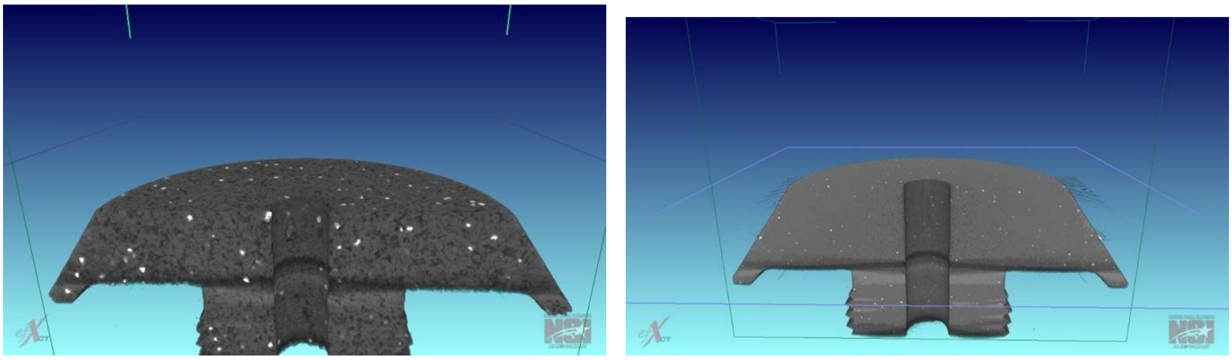
4.2 Graphite tip

One of the most critical components of the probe is represented by the graphite tip. The item is the one that suffers from the highest thermal load, the strongest gradient, and the toughest thermal shock. The design of the component was a compromise between general strength and ease of operations. At the end a screw fixing was considered the best option for fast replacement during campaign.

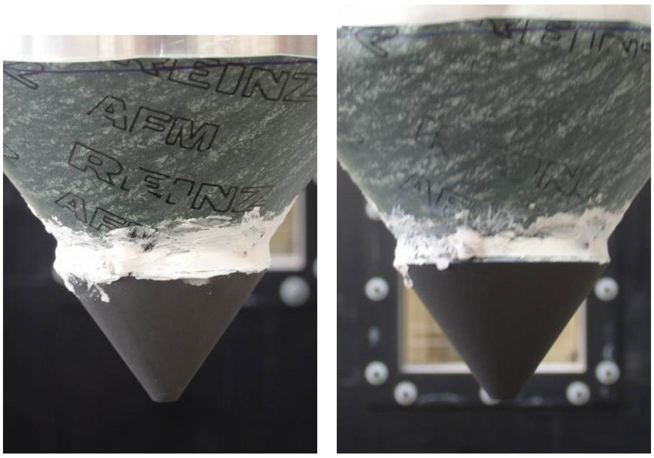
The choice of the material spanned between two types of graphite, one extruded and a grade of electrographite. Both were used for prototype manufacturing. The main difference for the production consisted in the size of the crystal grains. Whereas the extruded graphite had a grain size of 800 µm, the electrographite featured 7 µm. The difference can be easily appreciated in two tomographic analyses performed on the two materials, reported in Fig. 6. Porosity and impurity (bright spots) can be observed for the extruded graphite while the other image shows more homogeneous material. The porosity of the extruded graphite created manufacturing problems on the sharp leading edge of the tip and was discarded. Electrographite demonstrated very good machining properties, enabling also the construction of the thread to fasten the tip to the holder.



(a) Extruded (b) Electrographite

Figure 6: Tomography of tips made by different graphite grades

The cold flow tests showed that the choice of the material was substantially valid to withstand high Mach number incoming flow and particle wearing. Tests were performed at the VMK with the complete thermal protection assembly. The integrity of the tip after the flow tests was complete. Images reported in Fig. 7 show the tip assembly before and after a test. Particle flow duration was in the order of few seconds. The reader can see that the printing on the thermal protection was partially removed while the tip does not show apparent damages.



(a) Before (b) After

Figure 7: Tip and frontal thermal protection during cold tests

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Finally, the hot flow tests revealed a pitfall in the design concept of the tip. The graphite tip broke into pieces after the first two tests. From the images it is possible to appreciate that the threaded fixture of the tip was subjected to fracture and detached from the supporting steel element. The issue was attributed to the different thermal expansion of graphite and metals, and the contemporary fragile nature of graphite itself. The thread caused a fixed interface and did not allow different expansions, triggering the crack and the failure. The detail of the problem is shown in Fig. 8. The issue was solved by changing the method to apply the tip from a threaded to a glued connection. A comparison between tip exposed to the nozzle flow of a propellant containing 5 % of aluminum and new one is visible in Fig. 9. A crust of aluminum oxide appears at the surface. The wearing is limited and non-symmetrical because of the protective shutter movement. Visible cracks are not present. The internal channel is completely smooth.

Figure 8: Tip suffering from thread failure

Figure 9: Comparison between tip status: left post-firing (left) and pre-firing (right)

4.3 Passive thermal protections

For a matter of precaution, steel components exposed to the hot flow are protected by means of passive thermal protections. A flat foil for high temperature of aramidic material is attached to the tip holder. The material is named AFM 37/8 and it is a highly cost-effective yet high-performance material for low to moderate sealing requirements. It consists of aramide fibers and other asbestos substitutes that are resistant to high temperatures and are processed with high-grade elastomers under elevated pressure and temperature. It is commonly used for sealed joints that are subjected to low or medium mechanical stress. Technical details are shown in Table 2. The attachment of the foil to the metal

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Table 2: Technical details for the AFM 37/8 thermal protection.

Density, [g/cm^3]	1.7-1.9
Tensile Strength (1.5 mm), [N/mm2][a]	> 7
Compressibility, [%][b]	8-15
Recovery, [%][b]	< 50
Max. Operating Nominal Pressure, [bar]	60
Ignition Loss, [%][c] < 38	

[a]: acc. to ASTM F 152, across grain.

[b]: acc. to ASSTM F 36, Procedure G.

[c]: acc. to DIN 52 911.

surface was the issue faced during the tests. Currently, the most effective solution seems to be a bi-component epoxy resin, after testing thermosetting tapes and acrylic glues.

Part of the nitrogen pipes are exposed to the hot flow of the test and are not protected by the protective shield because they are wider than the shutter. One-component sealant based on silicates, specifically designed for free flames at temperatures (up to 1200 °C of continuous exposure) was painted over joints and pipes. The surfaces must be clean, dry, and degreased, eliminating any trace of rust that may be present on metallic surfaces. Sand paper should make the surface minimally rough to grant better adhesion. This solution demonstrated long endurance to flow and minimal wear. Multiple tests do not produce alteration of the protective role.

Finally, corners and gaps have been filled with high temperature silicone (up to 300 °C of continuous exposure). These are parts that are not directly exposed to the flame and require both elastic property of the sealing medium and good adhesion property to polymers, metal, and graphite. Short term exposure during shield opening did not alter the property of this silicone.

The complete assembly of thermal-critical components is visible in Fig. 10. In the picture the black graphite tip is attached to the supporting tip holder with the high-temperature silicone glue (in red color). The insulating foil (green color) wraps the metal. On the pipes, whitish high-temperature silicate paste was hardened some hours before testing.

Figure 10: Probe ready for test

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5. Final remarks

An innovative intrusive technique for supersonic hot-flow particle collection from rocket nozzle has been developed in the frame of the EMAP project. Whereas the test campaign is still ongoing at the VMK supersonic wind tunnel of DLR-Cologne, the concept and most of the critical design components have been refined and validated under relevant operating conditions. Only the characterization of the collected liquid is missing. This will occur in the incoming months. The outcomes will integrate in the global dataset that DLR is producing with a number of other diagnostics, as described in the introduction of this paper, obtaining an unprecedented description capability of the plume exiting form a rocket nozzle.

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