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Research and Test Activities on Advanced Rocket Propellants at DLR's Institute of Space Propulsion in Lampoldshausen

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Abstract

The present paper gives a comprehensive overview and summary of research and test activities conducted at the test facility M11 and the physical-chemical lab at DLR in Lampoldshausen. The focus of the research is on advanced rocket propellants and new materials for space technologies. In addition, the activities regarding supersonic flows and cooling of SCRamjets will be shown and discussed. Also the use of machine learning methods for rocket engine control are presented. The activities on advanced rocket propellants include research on ADN (Ammonium dinitramid)-based propellants, hydrogen peroxide, mono- and bipropellants based on nitrous oxide (HyNOx), green hypergolic bipropellants as well as gelled and nitromethane based propellants. For each propellant or propellant combination, the main research and test results of DLR internal projects are summarized. Furthermore, selected results of EU and ESA projects regarding advanced propellants and research conducted at DLR Lampoldshausen are presented.

1. Introduction

Research on advanced rocket propellants is mandatory to find high performance, easy to use, non-toxic and low cost propellants for future space applications. Conventional propellants as hydrazines or mixed oxides of nitrogen are highly toxic, corrosive and/or carcinogenic. Thus, the use of these conventional propellants require high safety measures, what results in high handling, storage, transportation and testing costs. To lower the cost of space flight, reduce the testing, production and qualification efforts as well as the turnaround times, green propellants are obligatory.
To find suitable green propellants for different kinds of application, DLR's Institute of Space Propulsion conducts research on ADN (Ammonium dinitramid)-based propellants [1–3], hydrogen peroxide [4], mono- and bipropellants based on nitrous oxide (HyNOx) [5–9], green hypergolic and non-hypergolic bipropellants based on hydrogen peroxide [10–12], gelled [13–17] as well as nitromethane-based propellants [18]. In addition to the research on green propellants, SCRamjet research regarding cooling methods and flow field investigations are conducted at a dedicated test position. The activities are and were embedded in DLR- internal, ESA- and EU-projects. During the last decade, the staff of DLR gained hands-on experience on all of the above mentioned propellants and propellant combinations. The activities included lab-scale analysis in the physical-chemical lab as well as combustion and thruster testing at the test facility M11 [19, 20].

2. Overview of the Physical-Chemical Laboratory

This section presents the activities of the physical-chemical laboratory at DLR Lampoldshausen. The laboratory activities are mainly carried out by the Chemical Propellant Technology (CTT) department. Section 2.1 introduces the facility as well as the analytical and spectroscopic equipment. In the later sections, specific research activities of the laboratory are further explained. Moreover, the infrastructure is also used to e.g. perform material or propellant compatibility tests of new green propellants or oxidizers.

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2.1 Physical-chemical Lab: Equipment and Facility

Figure 1 shows an insight into the physical-chemical laboratory. In total, the laboratory in building M3 is distributed over five different rooms. Here, wet chemical synthesis, material investigations as well as analytical and spectroscopic measurements take place. In the following, some selected methods are described in detail.

Figure 1: View into one of the laboratories in building M3 at DLR Lampoldshausen

One of the most important tasks of the physical-chemical laboratory is the precise identification of the composition of gaseous and liquid samples. On the left in Figure 2, one of four gas chromatographs with which the laboratory is equipped, is shown. Here various gas samples from large-scale test facilities as well as research test stands can be analyzed. The right side of Figure 2 shows an ion chromatograph which can be used to analyze aqueous liquid samples very precisely for their ionic compounds.

Figure 2: left: Gas chromatograph (Perkin Elmer); right: ion chromatograph (Metrohm)

Two further important methods for identification of unknown samples are FTIR spectroscopy (Fig. 3 left) and Raman spectroscopy. These two complementary methods can identify functional groups of sample constituents by exciting molecular vibrations.

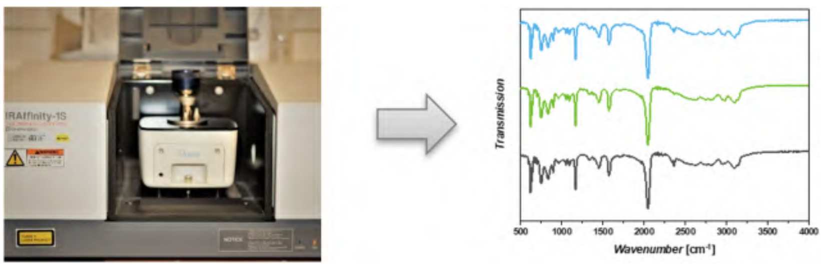

Figure 3: left: FTIR-spectrometer (Shimadzu); right: FTIR-spectra of three similar thiocyanate ionic liquids.

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However, due to the different selection rules, both methods provide different information which often complement each other to form a complete picture together. In sum, both methods are used for the identification of solids, liquids and surfaces. On the right side of Fig. 3, FTIR-spectra of thiocyanate ionic liquids are shown.

For thermal analyses, a simultaneous analyzer (Fig. 4 left) is available with which both thermogravimetry and dynamic scanning calorimetry measurements can be performed. A corresponding spectrum is shown on the right side in Fig. 4. In this case, thermal decomposition of a fuel under inert condition is studied. Due to two different furnaces, wide temperature ranges (−120 °C to 1500 °C) of the measurements are possible. In addition, the measurements can be performed under selectable gas atmospheres (N_2, O_2, He, etc.) as well as in suitable crucible materials (e.g. Al, Al_2O_3 or steel). Within this approach, the oxidation behavior of e.g. metals or organic compounds can be studied under inert or oxidative conditions.

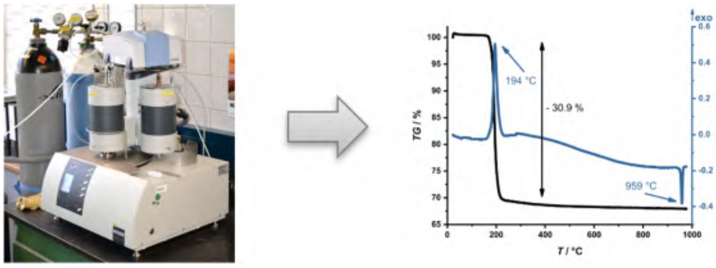

Figure 4: left: simultaneous thermal analyzer (NETZSCH); right: TG-(black) an DSC-curve (blue)

For the analysis of very small samples (e.g. particles or fragments) and structures, a scanning electron microscope is available, which is shown in Figure 5. Here, a resolution of up to 3 nm can be achieved. In addition, the integrated EDX (energy dispersive X-ray spectroscopy) measurements are of great interest for many applications, through which the atomic composition of unknown samples can be determined.

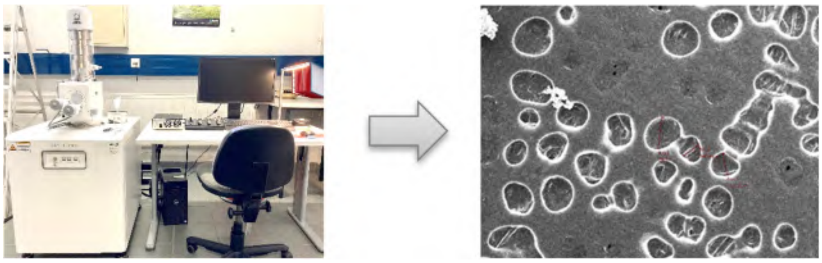

Figure 5: left: SEM (scanning electron microscope; Jeol); right: SEM picture.

Further, the physical chemical lab is equipped with the following devices: bomb calorimeter, glovebox, rheometer, tensiometer, density meter, microviscosimeter, atom absorption spectrometer and a nanosizer. This bunch of analytical methods and infrastructure allows us to determine e.g. thermodynamic quantities of materials and enable us to work with air and water sensitive compounds. This wide selection of analytical and spectroscopic equipment allows us not only support other departments of DLR as well as external customers with all kinds of specific requests; but also, to develop own approaches for future propellants for space applications. The topics of the latter activities are explained in more detail in the next chapters and sections.

3. M11 Test Facility Overview

The test facility M11 allows combustion tests with all kinds of advanced propellants at five test positions [19,20]. Each of these five test positions is dedicated to a specific research area: At the M11.1 a H_2/O_2 air vitiator can produce supersonic flow conditions for SCRamjet research [21,22]. The M11.2 is equipped with a vacuum chamber, vacuum pumps and a two staged ejection system to allow high-altitude simulation and testing of green propellant thrusters for orbital propulsion [19]. The M11.3 is a facility for basic research on the combustion of hybrid propellant combinations. The M11.4 is designed for testing of gelled and nitromethane-based propellants, but also serves as flexible test platform for different customers. The M11.5 is separated into two test positions which allow testing of hybrid rocket engines up to 10 kN as well as testing of green propellants.
The test positions M11.1, M11.3 and M11.4 are controlled via an Siemens SPS control system, while the data acquisition is carried out by a NI and LabView based externally developed software system. The test positions M11.2 and

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M11.5 offer flexible and adaptive Adwin measurement and control systems which were programmed and developed in-house.
The test bench infrastructure allows a supply with gaseous hydrogen, nitrogen, oxygen, methane and pressurized air with up to 200 bar. Furthermore a nitrous oxide infrastructure as well as gaseous hydrocarbon supply lines are available at the M11.5. Due to safety reasons, the specific propellants as hydrogen peroxide, the ionic liquids, gels or nitromethane-based propellants are fulled into dedicated run tanks prior to the testing activities and de-fuelled at the end of each test day. Depending on the hazard analysis in advance to each test campaign, the testing activities are conducted under surveillance of the fire brigade, safety department or in restricted areas at the test bench. All test activities are remote controlled via the specific measurement and control system. In most cases the test sequences are programmed in advance to the test day or campaign, but they can also be modified during the specific test activity.
To handle the various propellants, the test bench personnel is trained according to the German explosives law. Furthermore the test bench is designed for the worst-case scenario and an explosive decomposition of a specific amount of propellant is allowed without endangering the staff. Nevertheless, several propellant dependent safety procedures are in place to avoid an unwanted decomposition or ignition.

Figure 6 shows an aerial view of the M11 test facility with the different test positions, the machine shops and the office buildings. A propellant production facility allows on-site propellant manufacturing and subsequent hot firing without transportation across the Lampoldshausen site.

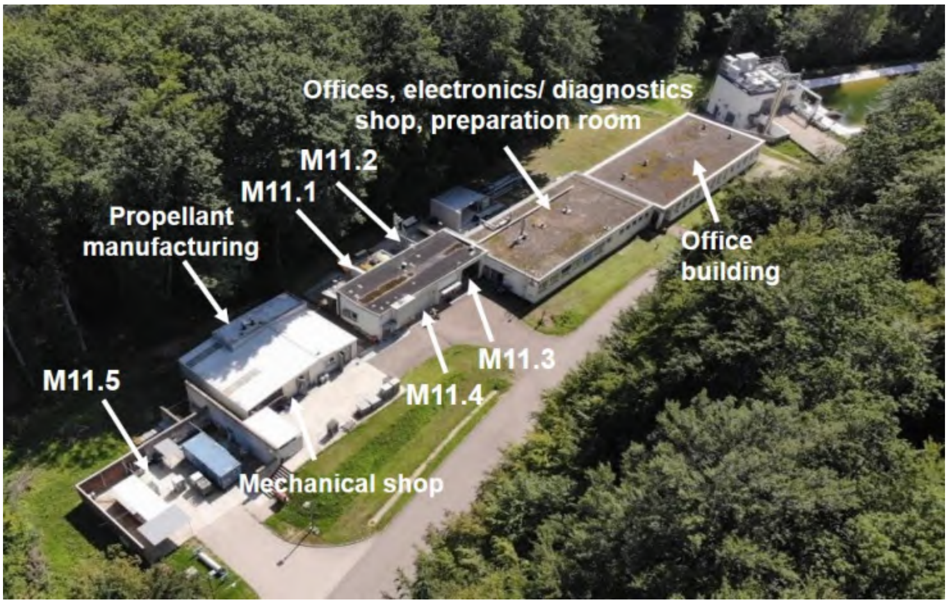

Figure 6: Aerial view of DLR's M11 test facility in Lampoldshausen

Each of the following sections gives a short overview of the test activities, projects and the safety measures which help conducting the experiments.

4. Scramjet Research

The test bench M11.1 is equipped with a chemical air vitiator for ramjet and scramjet research. The test cell was built as part of the M11 complex with laboratories and offices in the 1960s. In the 1970s research activities focused on solid fuel ramjet (SFRJ). In the 1980s, investigations in hydrogen as fuel for ramjet and scramjet lead to the first version of a powerful chemical air vitiator. This air vitiator was able to establish combustor inlet flow velocities up to Mach 2 with a Laval nozzle which simulates flight conditions of Mach 6 at a flight level of 30 km. In the 90s the M11.1 was involved in projects like LAPCAT, JAPHAR and the "Saenger II" two-stage-to-orbit vehicle (TSTO) [23] with research in supersonic combustion. For more details see [20, 24].

Between 2012 and 2015 the air vitiator was extensively refurbished and modified to the setup used today. A mass flow of 5 kg/s at supersonic flow conditions can be provided at boundary conditions of 1500 K stagnation temperature and 25 bar stagnation pressure. 11 hydrogen/oxygen burners, which can be combined into different burner patterns heat up pressurized air. This mixture of vitiation gases and pressurized air is then enriched with makeup oxygen to adjust the chemical gas composition to ambient air. The hot gas mass flow rate can be adjusted by activating different burner

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patterns and the supply pressures according to the boundary conditions. This approach enables hot gas mass flow rates between 0.5 and 5 kg/s at stagnation pressures between 1.5 and 25 bar.

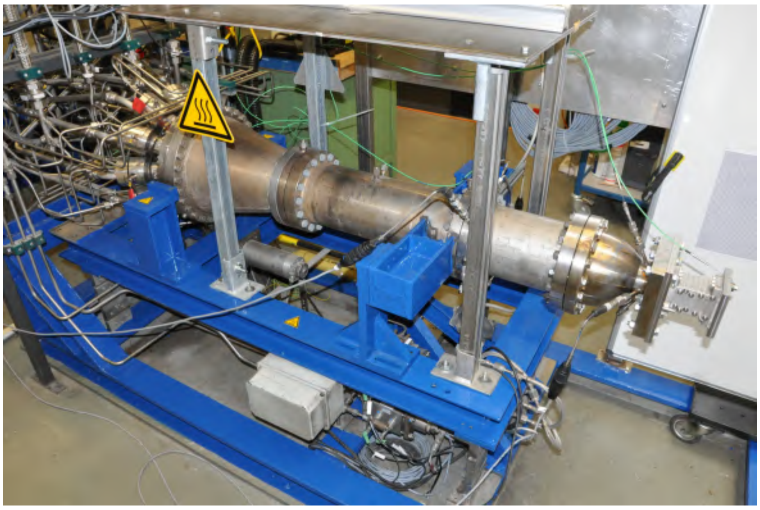

Figure 7: Air Vitiator at M11.1

The M11.1 can simulate ramjet and scramjet conditions within a wide range of boundary conditions with run times up to at least a minute. From 2016 to 2018, an extensive test campaign was performed including more than 1,500 hot runs at the air vitiator facility. The research topic of this campaign was the applicability of transpiration cooling systems to a model scramjet combustion chamber including different types of porous wall materials (sintered steel and CMC ceramics) and coolants (nitrogen and hydrogen) at various test boundary conditions (see [25–28]). In a transpiration cooling system coolant enters the hot gas main flow through a porous wall section, which provides direct cooling to the wall. Additionally, this system provides a coolant film / coolant boundary layer, which protects the downstream wall surface for a limited run length. This cooling approach is a promising method to control the high heat loads on internal engine surfaces, which is one of the main challenges connected to high speed flight propulsion. Some unique gas dynamic behavior is caused by the introduction of a coolant secondary flow into the hot gas main flow.

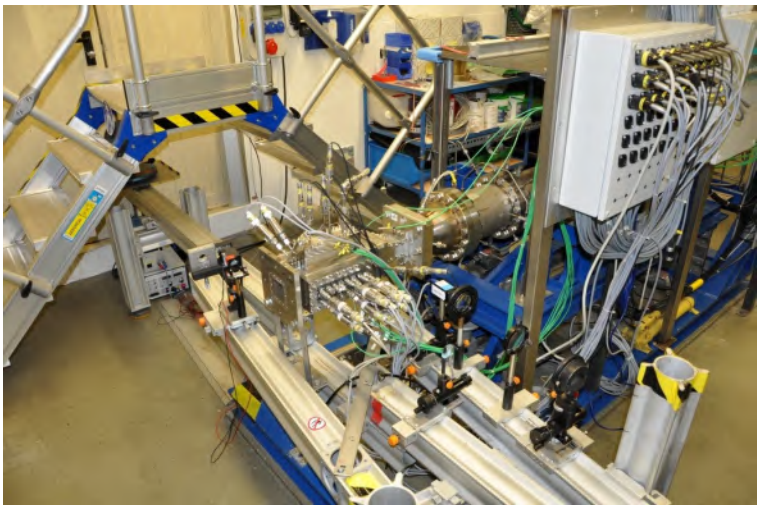

Figure 8: Scramjet Combustion Chamber Setup

This includes extensive shock-shock and shock-boundary layer interaction (SBLI) phenomena if a wedge-shaped shock generator/flame holder is introduced, as well as thermal choking, unstart and, in case of non-inert coolants, SBLI induced self-ignition of the coolant. For more details see [25–28]. For the 2016 to 2018 measurement campaign a plenum on top of the combustion chamber supplies the main flow through a porous section with gaseous coolant (hydrogen or nitrogen). Different test configurations featuring different optical accesses, measurement inserts and a horizontally positionable wedge shaped shock generator were used at the model combustion chamber.

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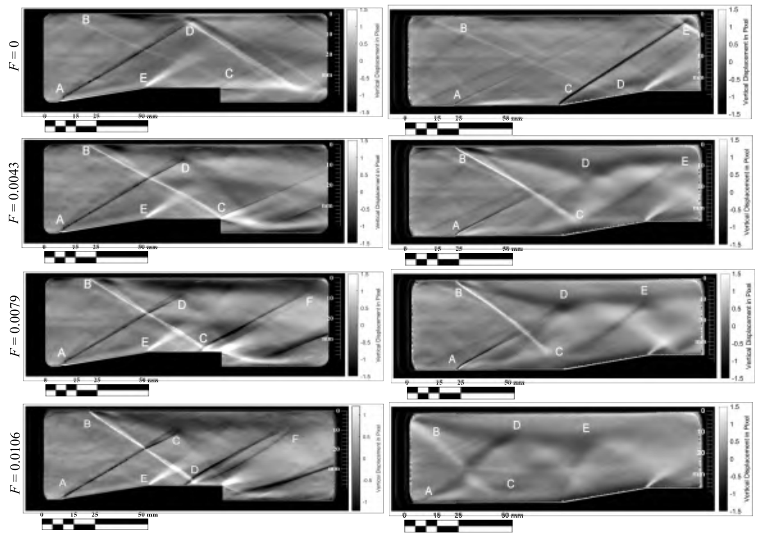

Figure 9: Typical SBLI Pattern (1200 K, 10 bar Stagnation Values), Wall Material: Sika-R 150, BOS Images

Advanced optical measurement techniques such as background oriented schlieren (BOS) using an in-house developed code and highspeed BOS were introduced to the test bench during the transpiration cooling campaign to visualize the flow field (see Fig.9). The test campaign proved a strong influence of the shock generator and its position on the flow field, which reduced the cooling efficiency up to 20-25 %. Additionally, hydrogen was identified as a superior coolant that provides sufficient cooling efficiency even in strong interactions in the flow field. However, it is prone to self-ignition during certain boundary conditions.
During 2018 and 2019 the test runs for an ESA GSTP program addressing liquid film cooling in small rocket engines were conducted. This program, called “ExLiFiCo” [29], included several international partners: ESA ESTEC Noordwijk (NL), ArianeGroup Ottobrunn (GER), Numeca Brussels (BE), VKI Von Kármán Institute of Fluid Dynamics, Brussels (BE) and DLR Institute of Space Propulsion, Lampoldshausen (GER). The experiments were conducted in the lower operational range of the air vitiator facility (500-600 K, 0.5-1.4 kg/s, 2 bar stagnation values) and at subsonic flow conditions. The air vitiator provided the boundary conditions for an experimental film cooling chamber with various optical accesses (see Fig.10) and a film injector. Ethanol was used as a substitute for hazardous hydrazine fuels to simulate the coolant.

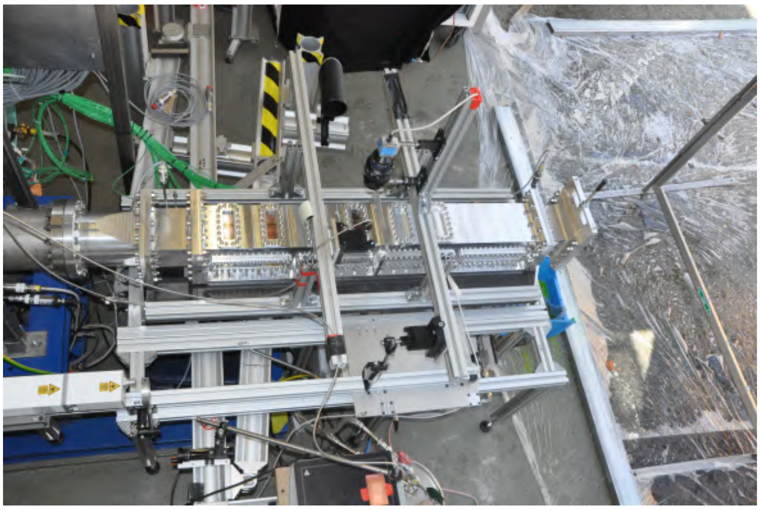

Figure 10: ExLiFiCo Film Cooling Experiment with LIF Setup Attached to M11.1 Air Vitiator

In summary more than 250 test runs were performed during this campaign with sophisticated measurement techniques including background oriented schlieren (BOS) and laser induced fluorescence (LIF) specially tailored to the requirements of the experiment (see [29] for details). More & Less (MDO and Regulations for Low-boom and Environmentally Sustainable Supersonic Aviation) is a European funded project to establish global environmental

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regulations of supersonic aviation. The main goals are to optimize trajectories and operations of supersonic aircrafts by high-fidelity environmental modelling, optimize aeropropulsion technologies to reduce sonic boom and emissions, to investigate boundary conditions on sonic boom variability and to develop validated modelling tools for generating and propagation of sonic booms. The project started at the beginning of 2021 with a duration of 4 years. The contribution of the M11.1 are ramjet combustion tests with hydrogen and bio-fuels, so called SAF (Sustainable Aviation Fuels), which consist of hydrocarbons. The aim is to validate numerical models by experimental data. For prediction of the arising emissions by combustion a system to measure the emissions is under development. In the frame of the project an injector was designed to inject the fuel into the combustion chamber. The injector is positioned at the bottom of the chamber and provides a cavity causing a recirculation zone for the vitiated air to enhance the residence time. The fuel is injected directly into the cavity for mixing with the main gas flow. A glow plug at the bottom of the cavity ignites the fuel-air mix. Downstream of the cavity a ramp guides the gases back into the main flow. Pretests with Nitrogen and Hydrogen without ignition were accomplished. These tests serve to investigate the impact of injected fuel to the main flow. The injector is equipped with temperature and pressure sensors in the cavity and on the ramp, respectively. The ramp and the nozzle for fuel injection is exchangeable to vary the mass flow in a wide range at supersonic conditions and to vary the residence time for mixing the main flow with the fuel. Since tests with the injector are done without ignition, the next step will be to establish a steady combustion under varying boundary conditions. Combustion in stable state is essential for emission measurements.
Future project and activities will be testing of high-temperature materials, load tests for components of supersonic vehicles and the continuation of the film cooling experiments to gain more data.

5. Hybrid Propellants Research

Hybrid rocket engines embody many of the key benefits of the other chemical propulsion systems, while eliminating some of the main drawbacks. Their main advantages derive from the architecture of this kind of engines. The physical

Figure 11: Combustion flame of a paraffin-based slab

separation of fuel and oxidizer makes them inherently safe, with zero TNT-equivalent for storage and handling with respect to solid motors. This also contributes to drastically reduce the costs of the engine. Moreover, they can be easily throttled, shut-off and restarted. The presence of only one liquid propellant reduces the complexity of the whole system (less piping and valves) compared to liquid engines, which furthermore contributes to lower the total costs. The

(a) Atmospheric combustion chamber set-up with optical diagnostic at M11.3

(b) Pressurized combustion chamber with optical access at M11.3

Figure 12: Hybrid combustor testing at M11.3

intrinsic safety and simplicity of hybrid systems make them suitable for different applications, from space tourism to kick-stages. In the last years, they found their way to the market in the small and micro launchers area thanks to the NewSpace sector.

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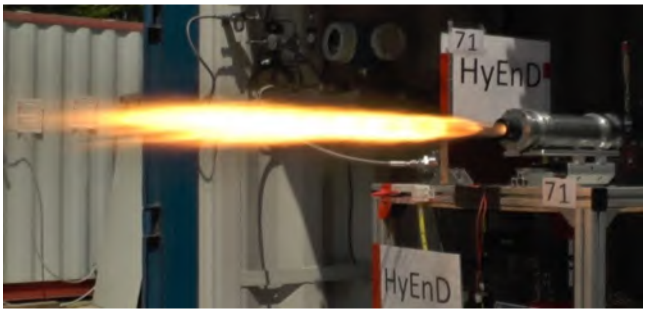
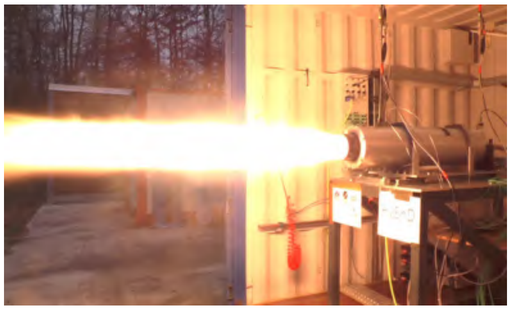
Activities in the hybrid propulsion field have been performed at the test complex M11 in Lampoldshausen since its erection in the 60s [20]. Both burning tests, as well as theoretical investigations with various propellant combinations, were conducted. Most of the used propellant combinations and components were developed, produced or modified in the chemical laboratory of the M11.
Detailed investigations in the model combustor were conducted by using different solid fuel with and without metal additives in combination with different oxidizers, such as liquid oxygen (LOX), liquid fluorine (LF2), and FLOX (liquid oxygen mixed with liquid fluorine). Since the 70s, research activities on solid fuel ramjet (SFRJ) propulsion also started. Since 2010, a renewed interest in hybrid rocket propulsion, due to the discovery of liquefying propellants, gave

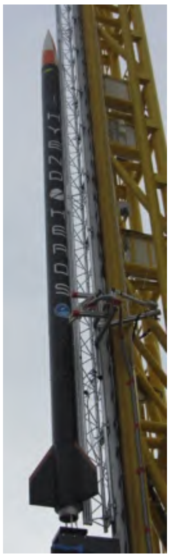


(a) Firing test of 500N (top)



(b) Firing test of 10 kN N_2O/Paraffin-based hybrid engine at M11.5

rise to new research activities at the test complex M11, which focused on the understanding of the entrainment combustion process of paraffin-based fuels [30, 31] and the optimization of hybrid engine propulsion performances [32]. Optical combustion tests are performed at the test bench M11.3 with the aim of characterizing the combustion flame behavior of paraffin-based fuel slabs in combination with gaseous oxygen. An image of the typical boundary layer flame with Kelvin-Helmholtz waves over a paraffin-based fuel slab is shown in Fig. 11.
The optical access from both sides of the combustor enables the use of different visualization techniques for the observation of the combustion and ignition processes at atmospheric and super-critical pressures. A photo of the high-speed video imaging set-up mounted with the atmospheric combustor is shown in Fig. 12a, while a firing test with the pressurized combustion chamber is shown in Fig. 12b. A further small model combustor for cylindrical fuel grains allows investigations of the regression rate and performance of the engine under real operating conditions. Since 2013, bigger



(a) HEROS 3 Rocket on launch pad

(b) Firing test of the 10 kN LOX/Paraffin-based hybrid engine at M11.5 [33]

Figure 14: 10 kN LOX/Paraffin-based hybrid engine

scale hybrid engines (up to 10 kN of thrust) based on paraffin-based fuel and nitrous oxide are tested at the test bench

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M11.5. Here a test container is used which was built in 2013 in the framework of the DLR STERN program, where student groups can test and develop their engines. The HyEnD group from the University of Stuttgart started to test their first 500 N hybrid engine here in 2013 (see Fig. 13a), which was scaled-up to 10 kN in the following 3 years (see Fig. 13b). The developed and flight-optimized engine was the propulsion system of the sounding rocket that broke the world altitude record (>32 km) for hybrid student rockets in 2016 from ESRANGE in Kiruna (see Fig. 14b) [34]. Firing tests of hybrid engines from the student group continues till the present days with a 10 kN engine based on polymeric fuels and nitrous oxide.
The test bench M11.5 is also used from the NewSpace start-up HyImpulse since 2018 for testing its small-scale 10 kN hybrid engine burning paraffin-based fuel and LOX (see Fig. 14b). The scale-up version of this engine will propel the orbital launcher that will bring payloads up to 500 kg into LEO [33].

6. Green Propellants Research

The research on green propellants at DLR in Lampoldshausen was initiated in 2009 with the start of the EU FP7 project GRASP [35,36]. In the frame of the project, different green propellants were investigated and tested. At DLR's Institute of Space Propulsion and the M11 test facility, combustion and component tests with FLP-106 [37] were conducted. In 2014 the research on green propellants consisting of nitrous oxide and hydrocarbons (called "HyNOx") was initiated [5–7, 38–40]. The activities were included in DLR's "Future Fuels" project [9, 13] and are ongoing in the recently started "NeoFuels" project. From 2015 to 2017 the EU Horizon 2020 project "Rheform" was coordinated by DLR's Institute of Space Propulsion [2]. Here the ADN-based monopropellants FLP-106 and LMP-103S have been extensively studied [41–44]. To kick-off research activities regarding hydrogen peroxide, the DLR-internal project "H2O2@TRS" was founded in 2017. As part of the project, a hydrogen peroxide monopropellant thruster was successful fired together with ArianeGroup [4]. Furthermore, in cooperation with Hochschule Aschaffenburg a sputtered iridium catalyst was developed and tested at the M11 test facility [45]. In 2020 the ESA project "High Performance Propellant Development" was acquired, where a green propellant mixture consisting of nitrous oxide and ethane was studied in detail [46]. In addition, work on nitromethane-based, liquid propellants began in 2020. Here the focus lays on additives and inhibitors which reduce the impact sensitivity of nitromethane, respectively the sensitivity to adiabatic compression [18]. Moreover, the DLR-internal project "AGILE" started in 2021 and focuses on hypergolic bipropellants based on hydrogen peroxide [10,12,47,48], ionic liquids [49] and AI-based control of rocket engines [50].
Recently, in 2022, the ESA-project "GreenRAIM" was kicked off. Here green mono- and bipropellants based on H_2O_2, N_2O and ADN shall be studied. The propellants will first be tested and hot fired in experimental propulsion systems, these propulsion systems will subsequently be modelled in Ecosim Pro ESPSS. If the propellant behavior is described sufficiently in these reference systems, the gained Ecosim models can be used to describe and simulate future propulsion system.

In parallel to the mentioned projects, test activities as contract work are conducted at the M11 test facility. Here e.g. water propulsion systems are investigated [51], Hydrogen Peroxide and fuels are studied as bipropellant combination [52], torch ignition systems are tested [53] or hybrid rocket engines are developed [54,55]. Furthermore, regarding the field of green propellants, cooperation with ArianeGroup, Bradford ECAPS, Dawn Aerospace, HyImpulse, OHB and Bayern-Chemie (MBDA) help to foster the exchange in between industry and academia.
The overarching aims of the mentioned research and test activities are:

- Gain hands-on experience in different kinds of green propellants
- Find solutions for all kinds of technical problems and increase the technology readiness level (TRL) of the propellants and corresponding propulsion system hardware
- Gain experience in the development of thruster hardware
- Assess and evaluate the different chemical and physical propellant properties
- Support the European space industry and increase the competitiveness of the European space sector
- Distribute the gained knowledge to a wider audience
- Support and foster the development of green propellants and the connected technologies

Following this aims, our vision is that future spacecrafts will be powered by a propellant developed at DLR in Lampoldshausen.

6.1 Nitromethane based propellants

Research in nitromethane as a rocket propellant is already carried out since the 1930s [57]. After the Second World War, it was explored as a propellant by the U.S. Army and Navy. Workers characterized and optimized ignition and

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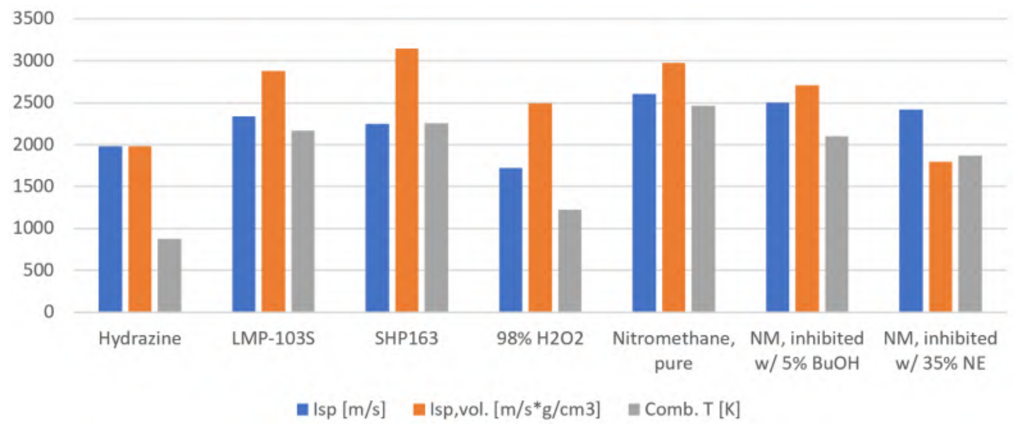

Figure 15: Overview of the theoretical rocket performance of green propellants and nitromethane. Calculated with NASA-CEA [56], p_{CC} = 10 bar, ϵ = 100 : 1, flow frozen at throat

combustion behavior, and sought to find additives that help to decompose the propellant during combustion in order to lower L^* requirements on combustion chambers [58, 59]. Later the Ministry of Defense of Israel also became interested in the use of nitromethane. Makovky et al. [60, 61] quantified the effects of additives such as nitroethane and nitropropane on combustion. Additionally, they made the first steps in the identification of the decomposition mechanism. With the advent of hydrazine the interest in nitromethane however almost ceased [57]. The research focused on the use of nitromethane for small thrusters with catalyst-based ignition returned as soon as hydrazine's carcinogenic properties were discovered [62, 63]. Since then research interest has continuously increased. Its combustion kinetics were analyzed in detail by Kelzenberg et al. [64] and especially with respect to high-pressure deflagration by Boyer et al. [65, 66].

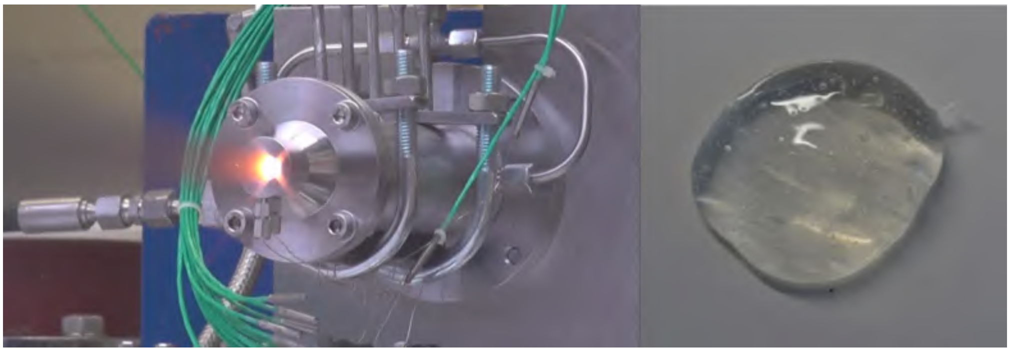

Figure 16: Left: Test firing of a nitromethane-based gelled monopropellant at the M11.4 test bench, Right: gelled monopropellant sample produced in the M3 laboratory

At DLR work with nitromethane began in the early 2000s. Since then rheological properties and combustion behavior of gelled nitromethane-based propellants are analyzed in cooperation with the Fraunhofer ICT and industry partners. Further details on the overall program can be found in the publications by Kirchberger et al. [67, 68] and Ciezki et al. [69]. Rheological properties and their influence on the flow and spray behavior of gelled propellants were characterized and modeled by Madlener et al. [14], Negri et al. [70] and Teipel et al. [71] in great detail. In [72] the combustion of nitromethane gels (see Figure 16) in small-scale combustion chambers is analyzed. Later a special focus was placed on the optical characterization of propellant injection and ignition in a windowed combustion chamber depicted in Figure 17. For further details on the optical combustion chamber setup see [73].

As mentioned before, With the advent of the "NewSpace"-age, the demand for alternatives to liquid hydrazine-based orbital propulsion is ever increasing. From Figure 15 it becomes evident that nitromethane is a very promising green propellant candidate. This is because of it's good rocket performance: compared to state-of-the-art green monopropellants nitromethane has superior mass-specific impulse and comparable volumetric impulse and combustion temperature. However, nitromethane is also a liquid explosive [74]. Especially if trapped under heavy confinement for example within tubes and heated [58, 60, 75]. Unfortunately, these are exactly the conditions common in injector passages or other elements of rocket thrusters. Detonations in a propulsion system are not only unacceptable in space

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missions but also may ruin the test bench used as shown in Figure 18. An additional issue in using pure nitromethane for propulsion is its low impact sensitivity under heavy confinement. Wharton and Harding [76] quantified the impact sensitivity to be below 1 J for pure nitromethane. According to [77] this is below the threshold where a substance is deemed too dangerous for road transportation.

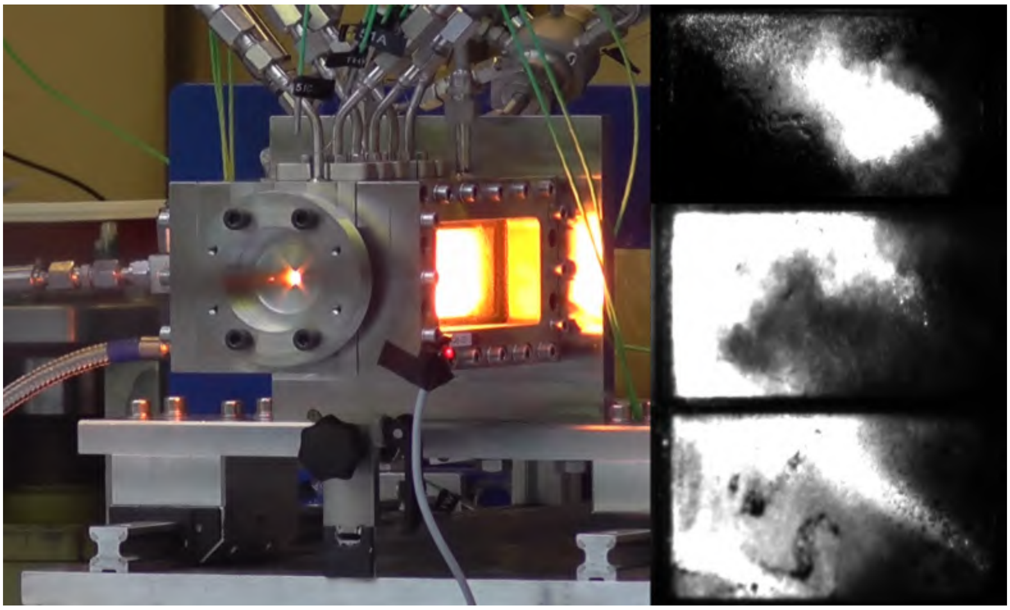

Figure 17: Left: Hot test with a gelled nitromethane propellant in the combustion chamber with optical access at the M11.4 test bench. Right: video stills of the ignition of a nitromethane-based gelled propellant

Our approach to mitigate the detonation risk and to decrease impact sensitiveness was to add phlegmatizing diluents to pure nitromethane. This technique is already known to work for increasing the critical diameter for detonation wave propagation in nitromethane. Workers quantified this effect by adding benzene, xylene, nitroethane, chloroform, carbon tetrachloride, and other substances [78, 79]. The use of n-butanol as a phlegmatizing agent was mentioned by [57, 80]. As a quick and easy way to quantify the effect of diluents, we used a BAM-Fallhammer impact device. This device is used to measure the impact sensitiveness of explosive substances. We tested the liquid specimen exactly according to Section 13.4.2 Test 3 (a) (ii) BAM Fallhammer of [77]. Additionally, in order to ensure good repeatability and consistent results we used a very narrow test matrix, i.e. we gradually increased the inhibitor content in the tested mixtures by small wt.% increments (1% in n-butanol, 5% in nitroethane). Our goal was to increase the impact sensitiveness to at least 10 J in order to comply with internal rules and industrial requirements for large-scale test-bench use.

Figure 18: Deflagration event and its aftermath in a 2009 hot test with an early nitromethane-based gel at the M11.4 test bench

In table 1 the impact energies and inhibitor content of some of the identified nitromethane-based green monopropellant candidates are depicted. In Figure 15 the respective mass-specific and density impulse, and combustion temperature values are depicted. From the diagram it is clear that even though some performance compared to pure nitromethane is lost, the inhibited mixtures still offer higher mass-specific impulse than other state-of-the-art green propellant mixtures such as LMP-103S and SHP-163. The volumetric impulse and adiabatic combustion temperature values are within a similar range in these propellants. The rocket performance values were calculated by means of the NASA-CEA tool [56]. The propellant compositions for LMP-103S and SHP-163 were picked from [81] and [82] respectively.

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Table 1: Effect of the addition of n-butanol and nitroethane on the impact sensitiveness of nitromethane

Inhibitor admixture to nitromethane	Impact sensitiveness
5 wt.% n-butanol	$10 < S_i \leqq 15$ J
35 wt.% nitroethane	$10 < S_i \leqq 15$ J

A side effect of adding phlegmatizing substances to nitromethane is that the adiabatic combustion temperature is lowered. This may help ease the requirements for construction materials. On the other hand, a lower combustion temperature may hinder important decomposition steps as pointed out by Kelzenberg et al. [64].

To sum up, we successfully used inhibitors to significantly decrease the risk potential of nitromethane-based propellants without sacrificing their performance characteristics. As a next step, a test-bench campaign is planned at the M11.4 test position in order to characterize the combustion behavior of these mixtures.

6.2 Hydrogen Peroxide Monopropellants

Propellants based on hydrogen peroxide are a promising alternative to commonly used space propellants based on hydrazine as well as for the corresponding hypergolic combinations. Highly concentrated hydrogen peroxide can deliver propulsive performance as monopropellant due to its exothermic decomposition. Further H_2O_2 can be applied as liquid green oxidizer in bipropellant applications. A hydrogen peroxide compatible feed system is available at the M11 test facility which can be utilized at different test positions. At the M11.2 a 1 N ArianeGroup thruster was tested with hydrogen peroxide under high altitude conditions. The thruster is designed for hydrazine and had a flight-like configuration. For the test a platinum-based catalyst was applied. In the test campaign, successful firing with the thruster were demonstrates and the performance of the thruster under different operation condition was determined [4, 83].

Further, at DLR a monopropellant catalyst demonstrator (MoCa) was designed and commissioned. MoCa is a 5 N monopropellant thruster for testing novel catalysts which is shown in Figure 19. It is equipped with an injector, the catalyst, a heater, three thermocouples at different positions of the catalyst bed, a thermocouple behind the catalyst and two pressure sensors, one at the begin of the catalyst bed and one behind the catalyst. With the demonstrator tests are planned for the evaluation of different catalysts for hydrogen peroxide and nitrous oxide in the ESA GreenRAIM program.

Figure 19: MoCa installed at M11.2 inside the vacuum chamber

The search for suitable catalysts for the effective decomposition of hydrogen peroxide poses still a challenge when it is used as a monopropellant. In cooperation with the Aschaffenburg University of Applied Sciences, Al_2O_3 catalyst pellets sputtered with iridium were investigated. The laboratory setup in Fig. 20 allowed the influence of the pellet coating on the decomposition behavior of H_2O_2 to be analyzed quantitatively [45].

6.3 HIP_11

The development process of green hypergolic propellant combinations is conducted in several steps. As starting point of the development of novel hypergolic combinations, hydrogen peroxide was determined as the oxidizer of choice. For the fuels, the class of room temperature ionic liquids was selected. The advantage of room temperature ionic liquids

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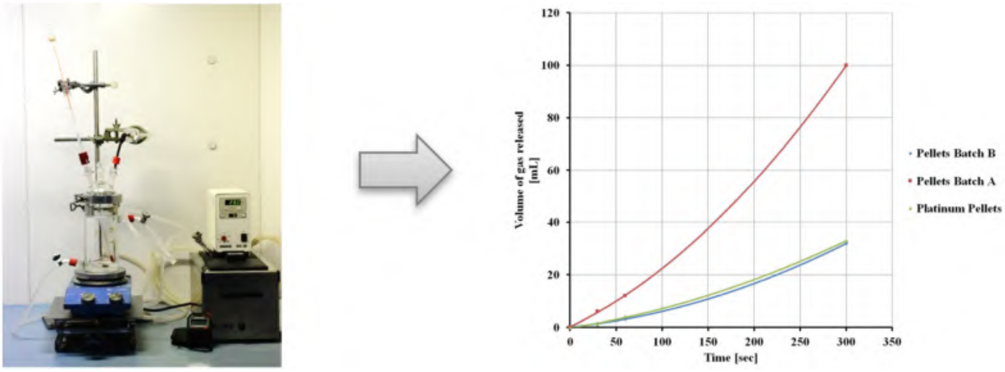

Figure 20: left: Apparatus for collecting gas of decomposed H_2O_2; right: Experimentally determined amount of released gas as a function of time [45].

is the high versatility: the properties can be directly tuned by changing of the molecular structure. Furthermore, ionic liquids have a neglectable vapor phase which vastly facilitates handling procedures.
The development and evaluation of fuel candidates is conducted in the following procedure. In a first step a screening of suitable substances is conducted. Promising candidates then are purchased or synthesized. The hypergolic behavior with hydrogen peroxide is determined in a lab scale drop test. At a drop test, one component of the propellant falls into a pool of the other component. If the two components are hypergolic with each other, an ignition can be observed shortly after initial contact of the two components. This process is recorded with a high-speed camera and the ignition delay time (IDT) can be determined. In this step, the effect of additives on the IDT can also be studied. A short IDT is essential for hypergolic propellants to provide reliable and fast ignitions. The drop tests are very different from a later application of a propellant in a thruster, therefore, the hypergolic ignition should be verified under flowing conditions. For this, the Hypergolic Ignition Test Setup (HIT) was developed [84, 85]. The hypergolic ignition of different injectors such as impinging or swirl injectors can be investigated with the propellant candidate. If a propellant provides fast ignition under flowing conditions, the performance of the propellant can be evaluated using a 40N battleship thruster.
In 2018 development activities regarding green hypergolic propellants with hydrogen peroxide started. In a first screening, commercially available ionic liquids were considered. Suitable ionic liquids were purchased and tested for hypergolicity with hydrogen peroxide in drop tests [10, 11, 47, 86]. Thiocyanate ionic liquids were found to be hypergolic with hydrogen peroxide. The ionic liquid EMIM SCN has an IDT of around 30 ms with 96 % hydrogen peroxide. With a copper additive, the IDT can be reduced to 13.9 ms. This propellant is called HIP_11 for Hypergolic ionic propellant developed at test facility M11. It also demonstrated reliable hypergolic ignition with an 2 on 1 impinging injector [85]. HIP_11 was also successfully tested using a battleship thruster [12]. Figure 21 shows a successful ignition with the 2 on 1 impinging injector and the battleship thruster. Further performance evaluation is ongoing at the moment. Research is also being conducted into the further development of fuels for this kind of hypergolic propellant combinations. The focus remains on thiocyanate-based ionic liquids, but newer generations of fuels are expected to work without metal-containing additives. For this reason, work has been carried out to investigate the influence of the cation structure on the hypergolic ignition behavior of the fuels [48]. Recent work on protic ionic liquids has created a promising combination of ionic liquids, HIM_35, which will soon be subjected to further investigation on the M11 test bench [49, 87].

6.4 HyNOx Mono- and Bipropellants

At DLR hydrocarbon/nitrous oxide propellants (called HyNOx propellants) are studied as premixed monopropellants and in conventional bipropellant configurations [38]. The work is conducted in the frame of DLR's internal "Future Fuels" and "NeoFuels" projects [9, 13] and in the ESA activity "High Performance Propellant Development" [46]. During the last years several milestones were achieved, e.g. flame arresters were successful developed and tested [7,8], porous injectors evaluated, different ignition systems studied, heat loads analysed [6,88] and the propellant performance was evaluated [5,6]. Furthermore safety tests regarding material compatibility, thermal stability, solubility and adiabatic compression were performed [46]. In addition, it was shown that a regenerative cooling system supports steady state operation of the thruster and that additive manufactured thrusters can be operated successfully . Finally, hot firings of a premixed propellant in an experimental propulsion system were performed [46].

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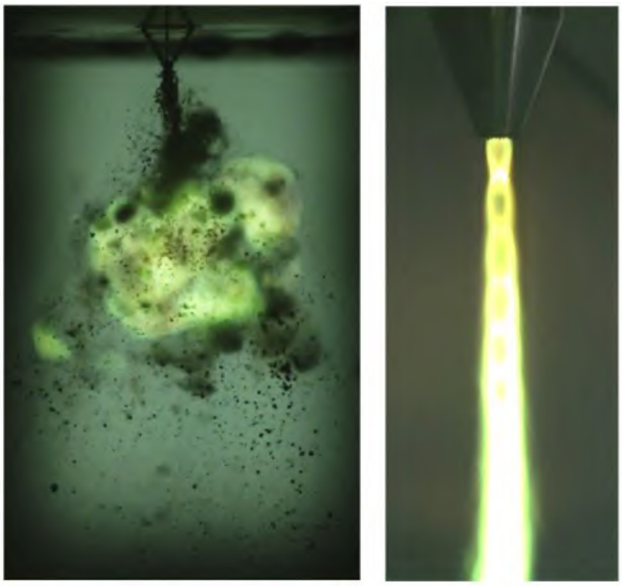
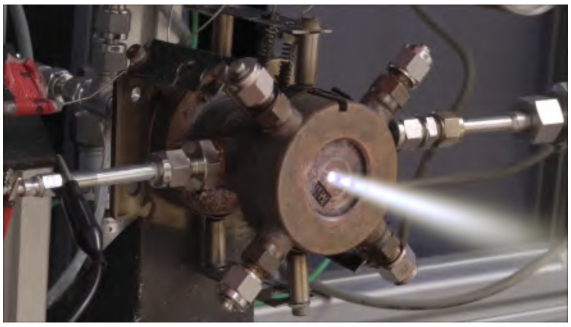
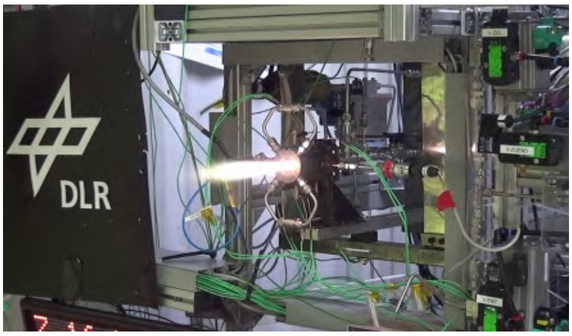


Figure 21: Hypergolic ignition of HIP_11, left: injector test, right: thruster firing



(a) Thruster operating with premixed HyNOx in a monopropellant-like system (see [46])



(b) Thruster using HyNOx propellant in conventional bipropellant system

Figure 22: Hot runs of thrusters in mono- and bipropellant configuration

6.4.1 HyNOx Monopropellants

Premixed nitrous oxide fuel blends promise several advantages: self-pressurized propulsion systems, non-toxic, cheap and easily available components as well as a high performance ($I_{sp} \approx 300$ s). However, these propellants also bring some major challenges, which have to be solved before they can safely be used in a propulsion system. One main challenge is the safe handling, fuelling and firing of a premixed propellant without the danger of explosion. A second challenge are the high combustion temperatures (≈ 3000 K) of these propellant combinations. Since 2014 DLR's Institute of Space Propulsion is working on HyNOx propellants together with DLR's the Institute of Combustion Technology [38]. The combination of basic combustion research, numerical simulations and experimental hot firings proved to be very successful. So the basic combustion research led to the development of appropriate reaction mechanisms [89–93] which were then used to design suitable flame arresters [7, 8, 94]. By using these flame arresters, numerous successful test campaigns were conducted where ignition methods, heat loads, chamber geometries and combustion efficiencies were studied [5, 6, 46]. In a second step, the reaction mechanisms were then used to simulated the combustion inside the chamber and compare the results to the experimental data. By using this approach, the experimentally obtained pressure, heat flux and thrust data could be reproduced and e.g. the flame position and precise heat flux distribution could be derived from the simulations.
In the frame of the ESA activity "High Performance Propellant Development" the system and safety aspects of a premixed HyNOx propellant and the corresponding propulsion system was evaluated in greater detail. So, the thermal stability of the premixed propellant was asses, by heating the propellant up to 75 °C reaching a pressure of 135 bar. Furthermore, the material compatibility of HyNOx with polymers and metals was studied, showing a good compati-

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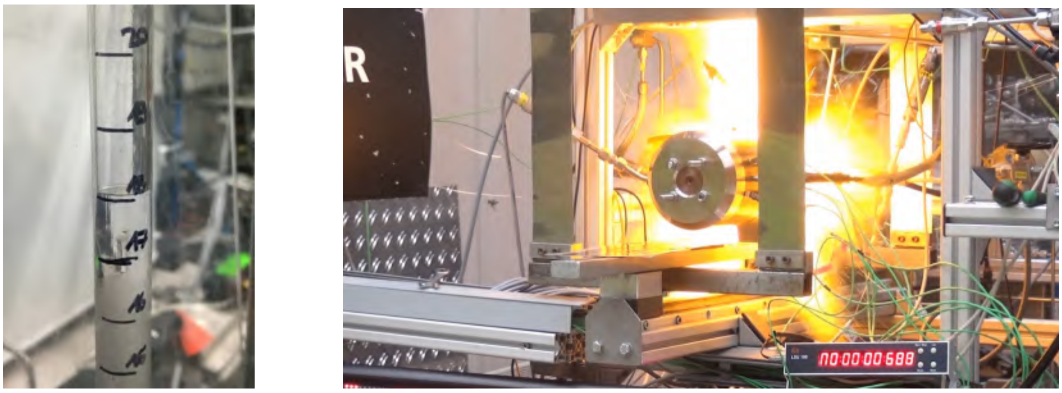
bility with the metals and some limitations regarding FEP and Kalrez. In addition, the propellant did not show any sensitivity to adiabatic compression, most likely due to the high vapor pressure of the propellant. In a next step, the miscibility and stability of the propellant was successful demonstrated. Figure 24a shows the premixed, liquefied HyNOx propellant in an in-house developed mixing setup. After the initial propellant investigations, premixed HyNOx was used to demonstrate vacuum ignition of a thruster for a variation of the mass flow and mixture ratio. In a last step, a monopropellant-like propulsion system was built, here the premixed propellant was fed from a single run tank. The system was successfully operated in pulse and steady state firings [46].

(a) Research thruster with regenerative cooling during 10 minute steady state firing

(b) Operation of thruster under vacuum conditions, burning a premixed N_2O/C_2H_6 propellant [46]

Figure 23: Hot runs of experimental monopropellant thrusters

Despite all these positive results it should never be forgotten that the premixed propellant is an highly energetic material. If an unwanted ignition occurs, the propellant is able to explode or detonate. So all operations with the premixed propellant should be conducted remote controlled and with enough safety margins. During the many years of testing activities with the premixed propellant, nobody was ever harmed as restrictive safety procedures were followed. But even if nothing critical ever happened, the propellant showed its energetic characteristics on several flame flashback events during thruster operation. In this events, connected feeding lines, pressure sensors and fittings were destroyed or damaged. Figure 24b shows such a flame flashback event where the thruster feeding line did burst.



(a) Liquid, premixed HyNOx propellant

(b) Flame flashback and destruction of feeding line during initial testing with premixed, liquid HyNOx

Figure 24: Liquid propellant production and flashback issues

6.4.2 HyNOx Bipropellants

In parallel to the evaluation and assessment of the premixed HyNOx propellants, N_2O and hydrocarbons are also used in conventional bipropellant systems. Regarding the bipropellant systems, different ignition and cooling methods, the heat transfer to the chamber walls, the combustion efficiency and different (porous) injection systems are studied. Up to now the thrusters were successfully tested under atmospheric and vacuum conditions, here steady state firings of to 10 minutes were conducted. For the developed 22 N thruster a typical throughput of 20 kg propellant and reliable re-ignition was also demonstrated. In addition, the thruster showed a reliable operation for a wide range of mixture

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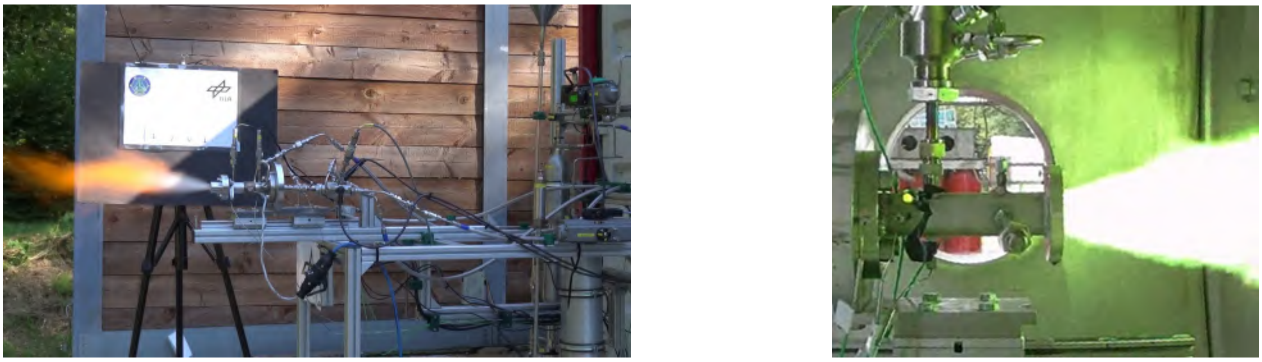
ratios and was throttleable in between 50 % and 140 % of thrust. Figure 22b shows a picture of a bipropellant thruster operating under atmospheric conditions.

6.5 ADN-based Propellants

Another replacement for hydrazine are liquid, aqueous propellants based on ammonium dinitramide (ADN). Compared to hydrazine, ADN-propellants offer simplified handling, especially when loading at the launch site. During the PRISMA mission the man-hours for loading hydrazine were three times more than for LMP-103S. Additionally, a higher overall performance (I_{sp}) as was shown by the PRISMA mission can be achieved [95]. A third improvement compared to hydrazines is a higher volumetric specific impulse due to higher density leading to smaller tanks, and therefore reduced structural weight [95].
Aqueous ADN-based liquid propellants were investigated at M11 during the EU projects GRASP and RHEFORM. ADN-based monopropellants have major advantages, but also some limitations. The combustion temperature of LMP-103S, the most mature propellant blend, is $1630°C$. Thus it is much higher than the one of hydrazine, which is about 1000°C depending on the amount of ammonia dissociation [96]. In order to withstand these temperatures, combustion chambers from materials that are ITAR regulated are currently used. Cheaper and ITAR-free combustion chamber materials could be used, if the combustion temperature of the propellants is reduced. A second disadvantage is that the catalyst used to decompose and ignite the propellant blend requires pre-heating. The catalyst is currently electrically pre-heated to a temperature of about 350°C, which takes around 30 min before firing, to ensure decomposition of the propellant followed by sustained and complete combustion. Objective of the projects was the development of ignition systems that require less pre-heating energy. For this purpose, the possibility of improving the catalyst was researched and the use of thermal ignition was investigated.
It was found that without sufficient heat-feedback from the combustion process the activation, especially the vaporization of the water portion in the propellant, can not be realized and ignition will not sustain [42].



(a) Insufficient heat-feedback resulting in ADN-combustion extinguishing

(b) Flame-holding device leading to complete combustion - including copper-heat-feedback-material

Figure 25: Hot runs of thermal ignition systems with ADN-based propellants

6.6 Nanomaterials for Space Applications

Nanomaterials are a key technology for modern developments in many areas of everyday life. Thus, they are also potential carriers for advanced solutions in space travel. Due to their high surface to volume ratio, nanoparticles can be used in much lower proportion than the corresponding bulk material. Studies on the catalytic decomposition of metallic nanoparticles in hypergolic bipropellants with H_2O_2 as oxidizer have already provided encouraging results in this regard. [97, 98] In the DLR internal project NatAs 2022, further application areas of nanomaterials in space propellants are currently being investigated. One approach is the formation of so-called Heat Transfer Fluids (HTFs), whose properties are expected to be improved by carbon-based nanoparticles.

6.7 Microcapsules in Space Propellants

The latest research project of the Chemical Propellant Technology department is concerned with microencapsulation of fuels. Here, one of two (possibly incompatible) propellant components is to be encapsulated in organic polymer

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materials. The encapsulated component should then be homogeneously dispersed in the second one, so that positive properties of mono- and bipropellants are combined. Figure 26 shows recently synthesized microcapsules filled with n-heptane.

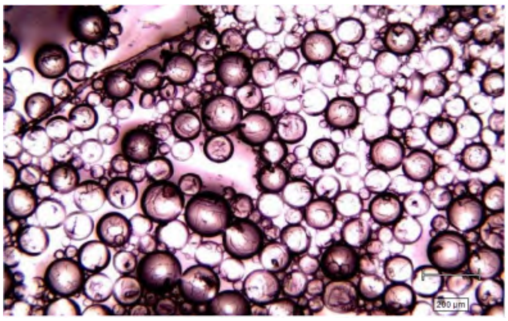

Figure 26: Microcapsules out of terephthaloyl chloride and hexamethylenediamine, filled with heptane.

7. Machine Learning Research at M11

The rapid progress in the development of machine learning algorithms leads to various applications of machine learning to solve real word engineering problems. Autonomous driving [99], autonomous robots [100], picture classification, speech recognition and the flight control of drones [101] are famous examples for the application of machine learning. Also, in the aerospace industry more applications seem conceivable [102]. So far the German Aerospace Center (DLR) in Lampoldshausen uses neural networks e.g. for the heat transfer prediction in rocket engine cooling channels [103] and fatigue life estimation [104]. Furthermore, the automatic detection of suitable precursors of combustion instabilities [105], highly efficient data analytic, computably inexpensive design studies [106] and the use of picture recognition and cluster analysis for evaluation of experimental data [107] are subject of investigations. In all this applications safety, repeatability, interpretability and certification of the machine learning method must be ensured, which is a current field of research.

At the M11 test facility the research focus in machine learning is on developing and testing a neural network controlled, throttleable, green propulsion system [50, 108]. In the last years several use cases for throttleable rocket engines became relevant. Obviously, the propulsive landing technology as demonstrated by SpaceX is one well-known use case [109]. But also lander applications for either robotic or manned missions to the moon, mars and beyond show the need for throttleable thrusters and a suitable control system thereof. At DLR's Institue of Space Propulsion in Lampoldshausen a reinforcement learning [110] based control approach is investigated [111]. The benefit in using reinforcement learning for engine control is that no linearized state space model for the controller development is needed. The controller is derived directly from the nonlinear simulation model and can therefore provide an optimal control for a wide range of system states, also for start up and shut down [111]. It is possible to react intelligently to changes in the system due to reuse or component wear. Furthermore it is possible to include side conditions in the control law to maximize for example the fatigue life of the combustion chamber, minimize the wall temperature at a given thrust level or maximize the efficiency during throttling. Due to safety and cost reasons first tests with the reinforcement learning control approach are conducted with a throttleable nitrogen cold gas thruster. A EcosimPro/ESPSS simulation of the test facility is used to train the neural network with simulated data [50]. After the training the network can be tested at the M11.5 test facility, where a dedicated python interface for the integration of neural networks in the test facility control program exists [108]. The network receives pressure and temperature sensor-data from the test facility as input value and calculates a valve position as output value. In this way a closed loop control is achieved. First experiments with the nitrogen cold gas thruster are ongoing (see Fig. 27). It is possible to regulate the thrust chamber pressure to different levels with the control inputs of the trained neural network. In the experiment shown in Fig. 27, the control starts at 11 s. It has to be remarked, that by now the used control valves are slow and therefore the settling time is slow (up to 2.3 s). In a next step it is planned to expand this control method to a 22 N nitrous oxide/ethane combustion chamber. With the use of faster control valves and the demonstration at a more complex system (Bipropellant nitrous oxide/ethane) the advantages of this approach will be demonstrated.

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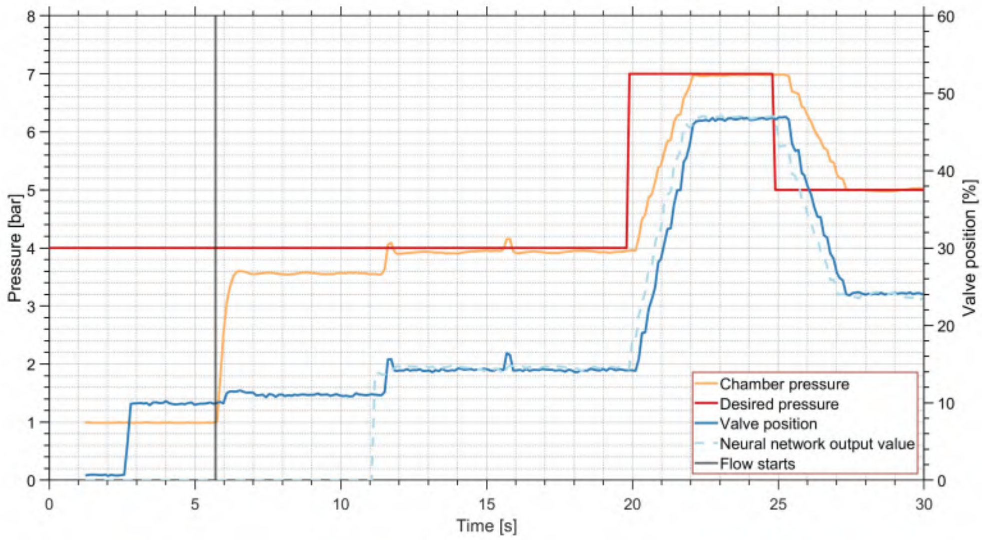

Figure 27: Experimental pressure and valve data for a neural network controlled nitrogen cold gas thruster [108]

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