# INVESTIGATION OF SPONTANEOUS IGNITION IN A 100 N HTP/HTPB HYBRID ROCKET ENGINE

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### <u>Abstract</u>

The aim of the work is the research on self-ignition phenomena in a hybrid rocket engine. The engine uses 98 % hydrogen peroxide as oxidizer and HTPB (Hydroxyl-Terminated Poly-Butadiene) as fuel. The condition, that is essential to initiate self-ignition in this system, is the application of a catalytic reactor, which enables the decomposition process of liquid hydrogen peroxide into the mixture of steam and oxygen with the temperature 800 - 950 deg C. The research has been based on the use of different catalyst materials as well as various configurations of catalyst beds. During the research (hot tests) the following parameters are collected: pressure and temperature at the end of the catalyst bed and the thrust of the engine. The evaluation of the ignition delay (that is counted from the start of the HTP flow) is made on the basis of the chamber pressure as well as on the video recording of the fire test.

<u>Keywords</u>: hydrogen peroxide, HTPB, catalyst, decomposition, spontaneous ignitron, hybrid rocket engine.

### 1. INTRODUCTION

The aim of the development program, of which the work presented in the paper is a part, is to create a low-cost, simple, self ignitable, restartable and stable working hybrid rocket engine. Another important aspect of the project is to use environmentally-friendly, easy to store and handle liquid oxidizer in the form of 98 %+ HTP (High Test Peroxide). All these features are desired for the future rocket propulsion systems – both, transportation and in-space applications. Highly concentrated hydrogen peroxide is currently regarded worldwide as a promising propellant, alternative to toxic hydrazine and its derivatives. As a matter of fact, the subject of alternative fuels for aerospace industry is a part of key activities led at the Institute of Aviation both recently and in the past [4]. By the term "stability" the authors mean the minimum pressure and thrust oscillation amplitude. Combustion instability, apart from the low thrust-to-weight ratio, is one of the few disadvantages of such kind of rocket propulsion system. According to [10], a well-designed hybrid rocket engine should limit combustion roughness to 2 - 3 % of mean chamber pressure.

In order to verify the main assumptions, a small 100 N hybrid rocket engine was designed and built. It uses pure HTPB as fuel and 98 %+ ultra pure grade of hydrogen peroxide (HTP) as oxidizer. Fuel grains are casted and cured in-house, using forms made of PTFE. Ultra-pure 98 % HTP is prepared in the Propellant Laboratory of the Institute of Aviation, by means of its own laboratory method [6, 11]. The engine is designed as re-usable, with replaceable paper-phenolic thermal insulation, inside of which fuel grains are placed. The rocket grade hydrogen peroxide is delivered by the pressure feed system, fully made of stainless steel and sealed with PTFE seals.

The purpose of the research, presented in the paper, is to determine the optimum configuration of the catalyst bed, that makes the hybrid engine both fast response (by means of the ignition delay time) and high-performance. The important objective is to find the optimum volume as well as the length-to-diameter ratio of the catalyst chamber. This optimum geometry of the reactor (which might be different for different catalyst materials) ensures both: the minimum heat loss through the walls of the chamber and the maximum decomposition performance (that is needed for reaching the minimum ignition delay time). The intention is also to select the best – possible to purchase or manufacture *in situ* – catalyst materials.

## 2. ENGINE DESIGN

### 2.1. Assumptions

The engine has been originally designed for 100 N of thrust, that, according to computations, should be reached for chamber pressure of 1 MPa. Using the CEA code [2], the optimum oxidizer-to-fuel mass flow ratio was computed. For HTPB/98 % HTP the optimum (for maximum specific impulse) O/F is equal to 5.5. The theoretical specific impulse for the expansion ratio of  $p_2/p_1 = 10$  equals to 212 s. Assuming the overall effectiveness of the engine at the level of 80 %, Isp taken for the design process is 170 s. The propellant mass flow rate for 100 N engine is 59 g/s, where 9.1 g/s is the flow of fuel and 49.9 g/s for oxidizer.

### 2.2. Catalyst reactor design

The catalyst bed is a critical subsystem of a self-ignitable hybrid rocket engine utilizing HTP. Fast, self-ignition conditions might be met only, if sufficient portion of heat is delivered from hot HTP decomposition products into the solid fuel grain (through the surface of the inner port). Complete decomposition of 98 % HTP theoretically gives 2.8 MJ/kg of energy, that, heats up the products up to 937 °C [2]. According to the available data [1], the temperature of thermo-decomposition of HTPB (that is the condition of flame occurrence) is over 380 °C. The occurrence of self-ignition phenomena for this configuration is a matter of time. However, it is a quite complex structure.

The heat of 98 %+ HTP decomposition products is used for reaching the pyrolysis conditions of HTPB fuel and then for ignition of HTPB pyrolysis products. In order to minimize the ignition delay time, the efficient HTP decomposition system is required. Complete decomposition of HTP is generally not difficult to reach, when the catalyst bed is pre-heated. However, the cold start is a state of a transient flow, with increasing both temperature and pressure. Catalytic reactions are both temperature and pressure dependent [7]. The optimum operation of a reactor is thus reached after some time. This period of time depends on various factors such as: catalyst and its structure (pellets diameter and their effective area, corresponding to the porosity), chamber design (length and diameter), HTP flow rate and inlet temperature.

The minimum catalyst chamber volume, necessary to reach complete decomposition of HTP with a certain catalyst, is difficult to estimate without experimental data. Taking into consideration the need for investigational evaluation of the catalyst performance, the chamber has been designed in a modular manner. The casing has been divided into two 100 mm segments (Fig. 1a). A single 200 mm casing has been designed as well (Fig. 1a, b). This approach

makes possible to use 100 mm or 200 mm chamber, that helps to assess preliminarily performance of the catalyst bed.



Fig. 1. Hybrid rocket engine: (a) elements, (b) final assembly with 200 mm catalyst chamber [9]

The elements of the catalyst bed that have been prepared for the initial tests are presented in Fig. 1. For the further research, other configurations have also been designed and manufactured. Fig. 2 a) presents the 100 mm chamber with a cooling jacket (double-tube structure), in which HTP acts as a cooling fluid. The flow of the coolant is reversed – it is injected into the catalyst bed directly from the jacket. Single-tube configurations are presented in Fig. 2 b) and c), 120 mm and 50 mm respectively. All manufactured configurations are 35 mm in diameter.



(a) (b) (c) Fig. 2. Catalyst reactors: (a) 100 mm fluid-cooled, (b) 120 mm single tube, (c) 50 mm single tube [9]

# 2.3. Combustion chamber and nozzle design

The geometry of the combustion chamber is determined by the geometry of the solid fuel grain. In order to achieve suitable dimensions of the fuel grain, the regression rate law (Form 1) has been used to simulate work parameters of the hybrid engine [8, 12]. The assumption was that the fuel is a regular ring-shaped grain. As a result, the following dimensions have been obtained: inner and outer diameter equal 30 mm and 50 mm respectively and the length is 160 mm. Additional space of 70 mm in length has been added for the post-combustion chamber. The pre-chamber is not required for the chosen configuration as the oxidizer is both vaporized and heated at the inlet to the combustion chamber. The overall length of the combustion chamber is 230 mm.

$$\dot{r} = aG^n x^m \tag{1}$$

in which: *r* – regression rate, *G* – oxidizer mass flux, *x* – axial position, *a*, *n*, *m* – ballistic coefficients.

The graphite nozzle has been designed for the expansion ratio  $p_2/p_1 = 10$ . That gives the nozzle area ratio of  $A_e/A_t = 2,4$ . Both, convergent and divergent parts of the nozzle are conical, the angle of the divergent part equals to 30°. The throat diameters is 11,4 mm. Design formulas, used for the design, are taken from [2].

# 3. CATALYSTS

The steel-ceramic-platinum wire mesh catalyst made by Swedish Catator is one of the few commercially available heterogeneous catalysts that are suitable for 98 % HTP decomposition [5]. The catalyst has been successfully tested (Fig. 3). It is made of a stainless steel wire screen, 12 x 12 mesh. The metal screens were plasma coated with a quite solid and high strength ceramic coating. The bond between the plasma coating and the steel is strong enough to withstand harsh environment of decomposition products of 98 %+ HTP. During the tests it was noticed that the wash coating has a strong bond to the plasma-coated ceramic, but the porous layer still tends to wear off over time at usage.



Fig. 3. Wire mesh catalyst, manufactured by Catator [Authors, 2013]

Manganese oxides  $(Mn_xO_y)$  some of them doped with other transition metal oxides and promoted with lanthanide element) on alpha and gamma alumina pellets catalysts provide higher contact surfaces than described above platinum-grid catalyst. This is mostly due to the fact that their surface area is highly developed – at least several tens of square meters per gram (Fig. 4). This means that they may require only a fraction of residence time for sufficient HTP decomposition compared to platinum-grid catalysts.



Fig. 4. Single Mn<sub>x</sub>O<sub>y</sub> catalyst pellet of 3 mm diameter (left, 50x magnification) and its surface structure (right, 250x magnification) [9]

Relatively small catalyst volumes may be needed – making the catalytic system based on manganese oxides catalyst very cost effective. Also, the inlet high concentration of HTP does not affect the amount of the catalyst required or the design of the whole system. The manganese oxides on alumina pellets catalysts ( $Mn_xO_y/Al_2O_3$ ) used in these tests were typically available in a few millimetres diameters coated pellets, to minimize static pressure drop across the catalyst bed.

The tests have been carried out with the use of  $Mn_xO_y$  – based alumina and silica-alumina pellet catalysts, prepared by the impregnation method from the KMnO<sub>4</sub> solution, calcined at 600 °C or 700 °C for 2 hours. Some of these catalysts have been doped with Fe, Co and Cr oxides, additionally promoted with Sm. The final content of the active oxides phase was in quite broad range due to the different nature of the support pellets. The highest value reached was nearly 15 % of the total catalyst mass.

## 4. HOT TEST RESULTS

All the experiments have been performed with the use of different configurations of the catalyst beds as well as various catalyst materials. Geometry and composition of the fuel grain are similar. Inlet conditions are mainly the same for all tests (except for the case, in which the same material is tested four times, and the inlet pressure changes). For the most cases the tank pressure is 1.2 MPa (Fig. 6 - 10), for some it is 1.4 MPa (Fig. 11 - 14). The initial temperature of HTP is between 10 and 15 °C. Every test consumes 600 cm<sup>3</sup> (860 g) of oxidizer. All experiments were performed in the Rocket Engine Test Facility of the Institute of Aviation. The test stand, previously used for jet engines, has been adapted to conduct research on all types of chemical rocket propulsion [3]. In Fig. 5 a screenshot from the one of the test is presented. Fig. 6 - 14 show the results of the fire tests. Each test is recorded with a video camera. The following parameters are measured: thrust of the engine, pressure and temperature at the end of the catalyst bed. The pressure sensor, which is applied for tests, is Keller PA-21PY with the range 0 ÷ 40 bar. The thermocouple is manufactured by the Polish company Czaki. Its measurement range is up to 1100 °C. However, due to the hardware that is used with this thermocouple, there is a noticeable delay in data acquisition, with respect to the time. Thus, the temperature profile should be considered as approximate - it has not been presented in this paper.



Fig. 5. The hybrid rocket engine during the fire test [9]

The test result with 200 mm chamber and wire-mesh catalyst manufactured by Catator, shows, that the catalyst bed reaches the constant and stable work relatively very quickly – after 0.3 s. It can be easily noticed on the thrust line (Fig. 6). The pressure and the temperature

profiles were not recorded during this experiment. The phase, during which the engine keeps constant thrust due to the HTP decomposition, lasts 3 seconds. Then the solid fuel ignition occurs. The overall ignition delay is then 3.3 s.



Fig. 6. Thrust of the engine with 200 mm chamber, steel-ceramic-platinum wire mesh catalyst [9]

Fig. 7 presents the results of the test with the same catalyst as previously. However, 100 mm catalyst chamber, with the cooling jacket, was used instead. It can be noticed from both, thrust and pressure diagram, that the use of a shorter catalyst reactor makes the catalyst heating phase completely different. Pressure and thrust rise slowly, until the ignition of the fuel grain occurs. The overall ignition delay time (3.2 second) is comparable to the previous configuration. However, the slow pressure rise indicates that the fluid, which flows inside the chamber, heats up slowly (opposite to the previous example). It means that, for the catalyst used in this experiment, the bed is too short.



Fig. 7. 100 mm chamber with a cooling jacket, steel-ceramic-platinum wire mesh catalyst: (a) thrust, (b) pressure [9]

The next experiment, the result of which is presented in Fig. 8, was performed with 200 mm catalyst reactor. Half of its length was filled with steel-ceramic-platinum mesh catalyst and the other one with pellet oxide catalyst, which had been prepared in-house. This material is based on  $Al_2O_3$  carrier and impregnated with doped manganese oxides. The arrangement of the catalyst pack is as follows: three different zones (pellets of different shapes and structure), filled with this material, separated by wire meshes.

The engine reached 50 % of its nominal thrust instantly and, after this period, a constant catalyst bed heating could be observed. The ignition occurred 2.5 second after the main valve opening. Instability of pressure and thrust could be noticed at the beginning of the combustion process (which lasts for 1.5 second). After this period the engine works stable, with slightly progressive characteristic of the thrust.



Fig. 8. 200 mm chamber, 50 % wire mesh catalyst, 50 %  $Mn_xO_y$  pellets: (a) thrust, (b) pressure [9]

Fig. 9 presents the result of the test obtained with 120 mm chamber, which was filled in 80 % with the pellet catalyst. The active phase of the pellet catalyst consists of  $Mn_xO_y$  doped with Fe (the mass of  $Fe_xO_y$  constitutes less than 10 % of the total active phase mass). The significant improvement of performance can be observed with reference to the previous experiment. Ignition delay time was reduced to 1.5 second. The heating phase of the catalyst bed was relatively very rapid. The initial peak of the chamber pressure could be the effect of using porous catalyst carrier. The combustion phase was very stable. Considering the error of the pressure sensor, that could be observed before and after the test (without loading), the total pressure amplitude measured at the end of the catalyst bed is estimated at 5 %.

Following the improvement of the engine performance, due to the use of  $Al_2O_3/Mn_xO_y$  catalyst, the 50 mm long catalyst bed have been chosen for further experiments. The new batch of pellet catalyst, supported on porous spherical-shaped pellets (3 - 4 mm in diameter), was used. It was doped with cobalt and samarium oxides. The result of this experiment is presented in Fig. 10.



(a) thrust, (b) pressure [9]

It can be easily noticed, that the catalyst bed heating phase is barely visible. Both: thrust and pressure course rise quickly up to the nominal value, without the characteristic change in the buildup rate. According to the video recording, the ignition delay time was estimated as 0.4 s. The reduction of instability (of both; the chamber pressure and the thrust), with respect to the previous experiments, can be observed.



Fig. 10. 50 mm chamber, 5 % wire mesh catalyst, 95 % Mn<sub>x</sub>O<sub>y</sub> + Co + Sm pellets: (a) thrust, (b) pressure [9]

The next fire test was performed with the use the same catalyst bed. The tank pressure was raised to 1.4 MPa. Thus, the chamber pressure and thrust increased as well (Fig. 11). It can be noticed that a short catalyst heating phase occurred. The ignition delay time was 0.5 s. The thrust was steady (120 N), with the exception of the initial and final combustion phases.



Fig. 11. 50 mm chamber, 5 % wire mesh catalyst, 95 %  $Mn_xO_y$  + Co + Sm pellets: (a) thrust, (b) pressure [9]

Fig. 12 presents the result of the following fire test, in which the same catalyst bed was used. The feed pressure was set at 1.4 MPa. The fuel grain was lengthened by 12 % (from 160 mm to 180 mm). The pressure peak (and thrust – as the consequence) at the start could be observed from its record. After this initial period, the instantaneous ignition of the fuel grain occurred (the delay time was 0.25 s). The thrust record was steady (130 N). The increase of thrust and chamber pressure at the end of the combustion phase (that follows all the tests) is connected

to the simultaneous flow of HTP and gaseous nitrogen, which is used for the tank pressurization (the engine works until the whole amount oxidizer outflows from the tank). It was observed that when the oxidizer flow is cut-off earlier, such increase does not appear).



Fig. 12. 50 mm chamber, 5 % wire mesh catalyst, 95 % Mn<sub>x</sub>O<sub>y</sub> + Co + Sm pellets: (a) thrust, (b) pressure [9]

One more experiment was performed with the same catalyst bed – the result is presented in Fig. 13. The 160 mm long fuel grain was used. Similarly to the previous experiment, the pressure peak appeared at the start of the test. The ignition delay time was 0.5 s.



Fig. 13. 50 mm chamber, 5 % wire mesh catalyst, 95 %  $Mn_xO_y$  + Co + Sm pellets: (a) thrust, (b) pressure [Authors, 2013]

The last fire test, result of which is presented in Fig. 14, utilizes the 50 mm chamber with the same type of the catalyst that was used in four previous experiments. Although the carrier and the active phase were the same, it was a different batch of the catalyst. The difference was in the amount of the active phase and the additives. It influenced significantly the performance of the engine. The ignition delay time was 0.7 s.



Fig. 14. 50 mm chamber, 5 % wire mesh catalyst, 95 % Mn<sub>x</sub>O<sub>y</sub> + Co + Sm pellets: (a) thrust, (b) pressure [Authors, 2013]

### 5. CONCLUSIONS

The paper presents the research on the thermal self ignition in a hybrid rocket engine. The ignition is caused by the flow of the hot oxygen-steam mixture through the inner port of the solid fuel grain. Hot gases are produced in the catalyst chamber – by decomposition of 98 % hydrogen peroxide. Results of seven fire tests are presented in the paper. Different configurations of the catalyst chambers, as well as different catalysts, have been applied. The ignition delay time in a hybrid rocket engine depends mainly on the catalyst used and its quantity in the reactor. According to the results, collected during the test campaign, relatively short ignition delay time (below 0.5 s) is reached with the use of manganese oxide alumina-supported catalysts. The application of the wire mesh supported platinum catalyst gives much longer ignition delay time, even with four times longer catalyst pack (than in the case of alumina-pellet catalyst). The conclusion is that  $Mn_xO_y/Al_2O_3$  catalyst is more active (with respect to hydrogen peroxide) than steel-ceramic-platinum wire mesh catalyst. What is more, pressure and thrust oscillations are lower, as the  $H_2O_2$  decomposition process is better.

Four experiments, which were performed with the same catalyst bed, gave somewhat different results, by means of pressure and thrust at the initial phase of engine operation. Different character of the catalyst heating might correspond to the washing out of the active phase from the carrier that is a case for some of the catalysts. The phenomena is strongly connected to the impact of superheated steam that together with hot gaseous oxygen acts as very efficient eluant fluid. Another possibility is that some changes occurred in the catalyst structure (caused by the vary increase of the temperature and pressure as well). Further research on this phenomena will be continued.

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# BADANIA SAMOZAPŁONU W RAKIETOWYM SILNIKU HYBRYDOWYM O CIĄGU 100 N NA HTP/HTPB

## **Streszczenie**

Celem pracy jest badanie zjawiska samozapłonu stałego paliwa w rakietowym silniku hybrydowym. Silnik jest zasilanym 98 % nadtlenkiem wodoru (utleniaczem) oraz HTPB (paliwem). Warunkiem, koniecznym do zainicjowania samozapłonu, jest w tym przypadku zastosowanie reaktora katalitycznego, który umożliwia rozkład ciekłego nadtlenku wodoru na mieszaninę pary wodnej i tlenu o temperaturze 800 - 950 °C. Badania zostały oparte o wykorzystanie różnych katalizatorów (materiałów nośnika i fazy aktywnej) oraz różnych konfiguracji reaktorów katalitycznych. Podczas badań – gorących testów – rejestrowane są: ciśnienie oraz temperatura na granicy komory katalitycznej i komory spalania, a także siła ciągu silnika rakietowego. Ocena czasu wystąpienia zapłonu (liczona od momentu uruchomienia przepływu HTP) jest dokonywana na podstawie zapisu przebiegu ciśnienia w komorze oraz rejestracji video.

<u>Słowa kluczowe</u>: nadtlenek wodoru, HTPB, katalizator, rozkład katalityczny, zapłon samoczynny, rakietowy silnik hybrydowy.