

# EXECUTIVE SUMMARY REPORT

ESA Contract No. 400118443/16/NL/PS

*This report summarizes the research on the long-term storage potential and material compatibility of hydrogen peroxide (HTP) with components used in satellite propulsion systems. Conducted under ESA contract No. 400118443/16/NL/PS, this study evaluated the effects of various HTP concentrations on metal and polymer materials, with a focus on chemical stability and safety.*

*This report makes a significant contribution to understanding the safety and long-term stability of materials exposed to HTP, which has broad applications in future propulsion technologies for the space industry. The findings support the identification of materials that enhance the safety and efficiency of propulsion systems, offering valuable insights that could strengthen the position of the Polish space sector in the global arena.*

## **Acknowledgments:**

*We extend our sincere appreciation to the Technical Officer overseeing the project. His professionalism and dedication were instrumental in achieving the project's goals, greatly contributing to advancements in Polish space technology.*

**Project: Hydrogen Peroxide Storability/ Compatibility Verification**

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**ABSTRACT:** The research conducted under Contract No. 400118443/16/NL/PS aimed to assess the long-term storage, compatibility, and safety of concentrated hydrogen peroxide (HTP) in satellite propulsion systems. Various materials were tested, including metals and polymers, at different concentrations of HTP. The results allowed for the classification of materials based on their compatibility with HTP and confirmed the chemical stability and safety of using hydrogen peroxide in these systems.

## 1. INTRODUCTION

Concentrated hydrogen peroxide aka HTP (High Test Peroxide) is a promising monopropellant as well as oxidizer in the green rocket fuel sector, therefore the purpose of presented research was characterize and evaluate the long-term storage, compatibility and safety of high-purity hydrogen peroxide in contact with satellite propulsion system materials. Metallic materials, such as tanks, valve bodies and pipes, and non-metallic materials such as valve seats and seals, used in in propulsion equipment were selected for testing. The scope of research included samples such as aluminum alloys, welded aluminum alloys, polymers ( e.g. PETE, EPDM, FEP), and different types of stainless steels.

The testing procedures and methods were appropriate to the actual space materials, material condition and fuel grade. In order to make a complete assessment, testing of various properties was carried out at different concentrations of hydrogen peroxide to determine their sensitivity to concentration. Four types of hydrogen peroxide were tested: 90% hydrogen peroxide ES grade (extra stabilized), 98% hydrogen peroxide ES grade (extra stabilized), 90% hydrogen peroxide HP grade (high purity) and 98% hydrogen peroxide HP grade (high purity).

The characterization effort was address to assess propellant decomposition, leaching of material (tank and valve) into the propellant, material corrosion, degradation of the material's physical properties (e.g. tensile strength, tear test, hardness test), susceptibility to stress corrosion cracking. Additionally static stability of the propellant (e.g. influence of impurities and/or additives to the hydrogen peroxide, risk of catalytic converter poisoning) and dynamic fuel stability (e.g. influence of handling agitation, vibration testing, other environmental conditions (e.g. radiation environment) were evaluated.

## 2. MATERIALS AND METHODS

Hydrogen peroxide of high concentration – High Test Peroxide (HTP) – was tested with regard to its safety, physical properties and compatibility with selected materials.

Safety testing included: Sympathetic detonation, Electrical spark sensitiveness, Impact sensitiveness, Vibration and mixing influence, Contact angle, Surface tension, Gas absorption, Radiation testing.

Compatibility of hydrogen peroxide with selected aluminum and steel alloys as well as polymers was verified under immersion testing at different conditions. Afterwards change in mechanical properties of the materials was determined. Concentration loss and physicochemical properties of hydrogen peroxide after contact with material was determined.

Testing of metal properties included: Static tensile strength, Tear test, Hardness test, Crack propagation, Corrosion testing and surface imaging, Stress corrosion cracking.

Testing of polymer properties included: Static tensile strength, Tear test, Hardness test, Dilatometry, Thermo-gravimetric analysis (TGA), Thermo-mechanical analysis (TMA, Dynamic thermo-mechanical analysis (DTMA).

Four types of hydrogen peroxide were produced by Jakusz Sp. Z o.o. in order to perform research:

- 90% hydrogen peroxide ES grade (extra stabilized)
- 98% hydrogen peroxide ES grade (extra stabilized)
- 90% hydrogen peroxide HP grade (high purity)

- 98% hydrogen peroxide HP grade (high purity)

Each batch was stabilized using sodium nitrate, sodium pyrophosphate and sodium stannate. Because HTP Grade ES turned out to be very unstable, amount of stabilizers was increased.

Quality control of each batch was performed which included:

Physicochemical analysis and concentration (by JST), Stability test (by JST), Anions determination (by Institute of New Chemical Syntheses Inorganic Chemistry Division in Gliwice (IChN)), Metals determination (by Spark-Lab), Ammonium ion determination (by Spark-Lab), TOC determination (by Spark-Lab), Evaporation residue determination (by JST).

### 3. SAFETY AND HANDLING TESTING

#### 3.1. Sympathetic detonation

Sympathetic detonation testing was performed by Institute of Industrial Organic Chemistry according to PN-EN 13631-11:2005-04 standard. Method is based on trial to initiate detonation of acceptor charge, containing sample, by detonation of donor charge.

No detonation was observed for neither of tested types of hydrogen peroxide (90ES, 90HP, 98ES, 98HP).

#### 3.2. Electrical spark sensitivity

Electrical spark sensitivity was performed by Institute of Industrial Organic Chemistry according to internal research procedure. Method is based on carrying out series of capacitor discharges of specific capacity. On one steel electrode sample is placed and the second electrode is moved towards plate electrode. Initiation of explosive reaction indicates that the substance is sensitive to electrical spark of certain energy.

No explosive reaction was observed for neither of tested types of hydrogen peroxide (90ES, 90HP, 98ES, 98HP). Applied energy was 4,2 J and 13,5 J.

#### 3.3. Impact sensitivity

Impact sensitivity testing was performed by Institute of Industrial Organic Chemistry according to PN-EN 13631-4: 2004-02 standard. Method is based on evaluating impact energy (set in [J]) of a hammer of specific mass from specified height under which HTP could start explosive transformation. First test usually is performed for 10J of impact energy. Depending on the result, next impact will have more or less energy depending on established procedure. The test is performed until impact sensitiveness is determined ie. the lowest energy required to ignite hydrogen peroxide evaluated in a series of six tests where at least one result is positive. Energy required to ignite hydrogen peroxide is calculated using the following formula:

$$E = 10 \times m \times h \quad \text{Eq.1}$$

E – hammer impact energy, [J]

10 – approximate gravity constant [m/s<sup>2</sup>],

m – hammer mass, [kg]

h – hammer drop height, [m]

No explosive reaction was observed for neither of tested types of hydrogen peroxide (90ES, 90HP, 98ES, 98HP). Maximum applied energy was 50 J.

#### 3.4. Friction sensitivity

Friction sensitivity testing (Koenen'a method) was performed by Institute of Industrial Organic Chemistry according to PN-EN 13631-3: 2006-12 standard. Method is based on determination of friction stimulus, which initiates explosive reaction of tested sample. Test is performed using Peters friction tester. Approximately 5 mg of HTP is placed on a ceramic plate. Ceramic peg is placed on the sample and the plate is moved from side to side, which exerts force on the sample. Test is considered positive if explosion reaction is observed.

No explosive reaction was observed for neither of tested types of hydrogen peroxide (90ES,

90HP, 98ES, 98HP). Maximum applied force was 360 N.

### 3.5. Vibration and mixing influence

Vibration and mixing influence testing was performed by Jakusz.

Influence of vibration on HTP (high test peroxide) was tested by measuring concentration loss and visual observation. Test was conducted at frequency 100 Hz and 15 G amplitude during different time at 8-10 hours and 24 hours cycles using vibration table. Tests were conducted in air conditioned room at constant temperature 17-17,5°C.

Vibration at tested conditions does not increase decomposition of hydrogen peroxide. No pressure build-up was observed in the test tubes in case of HP Grade HTP. When opening test tubes with Grade ES HTP audible noise of gas evolving was noticed. But this effect occurs also in containers with Grade ES HTP which were not influenced by vibration. Another visible effect during testing was foaming and bubbling, which stopped shortly after turning off vibrating table. The same effect was observed at initial testing using water instead of hydrogen peroxide. Based on the observations and measurement results it can be assumed that vibrations up to 100 Hz frequency does not induce uncontrolled decomposition of hydrogen peroxide.

Influence of mixing on HTP (high test peroxide) was determined by measuring concentration loss (assay by density measurement) of hydrogen peroxide due to decomposition and visual observations. Test was conducted at three different stirring speed values: 2000, 4000 and 6000 rpm using propeller type mechanical stirrer. Sample of tested liquid was collected every 2 hours. Full test at given stirring speed took 8 hours for each type of hydrogen peroxide. Test was conducted in air conditioned room at constant set temperature 20°C.

Mixing hydrogen peroxide significantly speeds up decomposition rate of hydrogen peroxide, but it does not cause violent or uncontrolled decomposition. However, depending on the conditions hydrogen peroxide loses from 0,25% to 0,5% of its concentration per hour of intense mixing. It is not clear whether increase of temperature during mixing or mixing itself causes decomposition.

### 3.8. Gas absorption

Gas solubility in hydrogen peroxide was performed by Lodz University of Technology. Nitrogen and helium solubility has been measured in the following, concentrated solutions of hydrogen peroxide:

- 90 % ES (extra stabilized)
- 90 % HP (high purity)
- 98 % ES
- 98 % HP

Measurement of gas pressure decrease during isothermal gas absorption in liquid has been applied as a measurement technique. Measurements were done using reactor calorimeter CPA (Chemical Process Analyzer – ChemiSens) at temperatures: 5, 10, 20°C. Results of solubility measurement has been expressed as Henry's law constant and solubility coefficient determining volume of gas dissolved in given volume of liquid [m<sup>3</sup>/m<sup>3</sup>] (Table 1 and 2).

Table 1 Solubility of nitrogen in hydrogen peroxide

| Solubility of nitrogen |       |  |  |
|------------------------|-------|--|--|
| Sample                 | Temp. | Henry's Law constant<br>[MPa m <sup>3</sup> /kmol] | Solubility coefficient<br>[Nm <sup>3</sup> gas /m <sup>3</sup> solution] |
|                        | [°C]  |  |  |
| 90 ES                  | 5     | 406  | 0.0506   |
|                        | 10    | 457  | 0.0452   |
|                        | 15    | 531  | 0.0385   |
| 98 ES                  | 5     | 393  | 0.0522   |
|                        | 10    | 426  | 0.0485   |
|                        | 15    | 616  | 0.0333   |
| 90 HP                  | 5     | 360  | 0.0575   |
|                        | 10    | 443  | 0.0461   |
|                        | 15    | 539  | 0.0380   |

|       |    |     |        |
|-------|----|-----|--------|
| 98 HP | 5  | 354 | 0.0586 |
|       | 10 | 415 | 0.0499 |
|       | 15 | 428 | 0.0486 |

Solubility of nitrogen in four types of tested liquid decreases (Henry's law constant increases) with the increase of temperature. Obtained values of Henry's law constant at low temperatures (higher gas solubility) shows lower scatter than values obtained during measurements at 20°C. It was observed that „older” solutions i.e. portion of given liquid poured into the CPA reactor from 1L storage container, kept at refrigerator, as the last ones, showed lower absorption capacity than “fresh” solutions. In other words, “older” solutions, which had longer and more frequent contact with air showed other properties than “fresh” solutions, which indicates its instability.

Table 2 Solubility of helium

| Solubility of helium |       |                            |  |
|----------------------|-------|----------------------------|--|
| Sample               | Temp. | Henry's Law constant       | Solubility coefficient                         |
|                      | [°C]  | [MPa m <sup>3</sup> /kmol] | [Nm <sup>3</sup> gas /m <sup>3</sup> solution] |
| 90 ES                | 5     | 676                        | 0.0312   |
|                      | 10    | 730                        | 0.0298   |
|                      | 15    | 769                        | 0.0278   |
| 98 ES                | 5     | 728                        | 0.0292   |
|                      | 10    | 897                        | 0.0237   |
|                      | 15    | 899                        | 0.0236   |
| 90 HP                | 5     | 783                        | 0.0270   |
|                      | 10    | 853                        | 0.0242   |
|                      | 15    | 945                        | 0.0218   |
| 98 HP                | 5     | 916                        | 0.0183   |
|                      | 10    | 956                        | 0.0178   |
|                      | 15    | 1183                       | 0.0180   |

Solubility of helium in four types of tested liquid decreases (Henry's law constant increases) with the increase of temperature. Correlation between helium solubility and temperature is lower than for nitrogen in analogical solutions. Solubility of helium in HTP solutions is lower (higher Henry's law constant) than in ES solutions. Helium solubility lowers along with increase of ES solution's concentration (higher Henry's law constants). Helium solubility lowers with increase of HP solution's concentration (higher Henry's law constants). It was observed that ES solutions are less stable than HP solutions, which caused high scatter in solubility measurement at the same conditions (for example for 98% ES at 10°C). Moreover, atypical behaviour of ES solution was observed during much of experiments of saturating it with helium: total gas pressure dropped, and raised as a result of increase of rotational speed of the stirrer. In order to calculate equilibrium gas solubility in liquid, minimal pressure value was taken, according to adopted measurement procedure. It is worth mentioning, that above phenomenon did not occur during measurement with HP solutions – after physicochemical equilibrium was reached, further increase of rotational speed of stirrer did not cause distortion of this state, i.e. pressure was kept at a constant level.

### 3.9. Radiation testing

Radiation testing was performed by Institute of Nuclear Chemistry and Technology in Warsaw. Cobalt gamma radiation source GC 5000 (made in India) was used for irradiation of samples. Applied dose rate was from 3.4 to 3.5 kGy/h. Evolution of oxygen due to decomposition of hydrogen peroxide was determined using two methods: weighting (%Active Oxygen Loss) and chromatographic.

98% hydrogen peroxide regardless amount of contaminants, remains stable up to dosage level 1.5 kGy. When it comes to 90% hydrogen peroxide, higher purity grade (HP) shows

better stability up to 1.5 kGy regardless concentration.  
Higher purity grade 90% and 98% hydrogen peroxide shows much better stability in a range of doses 2 – 5 kGy than contaminated (ES) 90% and 98% hydrogen peroxide.

Volume of O<sub>2</sub> released during irradiation was measured using gas chromatography technique. Efficiency of radiolytical decomposition of H<sub>2</sub>O<sub>2</sub> within tested range is approximately proportional to amount of absorbed dose of radiation.

Good correlation with gas chromatography and mass methodology was obtained. In general, mass results are slightly lower than those obtained by gas chromatography. Perhaps this is due to a water loss after opening the flasks before weighing. In Tab. 3 comparison of test results for both methodologies is presented. Expressed as [ml/kg\*Gy]

Table 32. Comparison of results of two methodologies – volume of released oxygen per mass per dose

| Type of hydrogen peroxide | Average value VO <sub>2</sub> – mass methodology | Average value VO <sub>2</sub> – GC methodology |
|---------------------------|--|--|
| 98HP                      | 0.07   | 0.12   |
| 98ES                      | 0.15   | 0.16   |
| 90HP                      | 0.10   | 0.12   |
| 90ES                      | 0.35   | 0.32   |

## 4. IMMERSION TESTING

### 4.1. Sample passivation and preparation

Prior to immersion testing samples were cut in proper shape depending on the test, cleaned and passivated using methods according to NASA/TM-2004-213151 procedure.

### 4.2. Screening immersion testing

Screening immersion testing at ambient temperature was conducted for 2 hours in order to preliminary evaluate compatibility of different materials with hydrogen peroxide. Based on visual observation the least compatible materials were excluded from further testing. Signs of decomposition of hydrogen peroxide are bubbling, gas evolution or change of appearance of the material.

22 different materials (including welded aluminum) and 8 combinations of materials previously passivated were tested:

- polymers: FEP, PTFE, EPDM white, Karlez 1050LF, Karlez 6380, Viton
- aluminum: 1100, 2219, 6061, 6068 (additional welded sample for each)
- stainless steel: 15-5PH, 321, A286, 301, 304, 304L, 316L, 347, 430

As a result it was observed that for 90% and 98% grade HP hydrogen peroxide:

Aluminum alloys 1100, 6061 and 6082 did not show incompatibility. In case of alloy 2219 little bubbling on the surface of the material was observed.

All polymers except for FKM (Viton) did not show incompatibility (no gas bubbles observed). Stainless steel alloys showed fairly good compatibility, except for alloy 347 (90% and 98% HP) and 15-5PH, 321 (98% HP), where signs of decomposition of hydrogen peroxide were observed.

None of the materials caused violent decomposition of hydrogen peroxide. Only FKM (Viton) was incompatible showing large amount of gas bubbles formed on the surface of the material, therefore it was excluded from further testing.

No signs of corrosion or brittleness were observed on any of the samples.

### 4.3. Immersion testing at 66°C

Compatibility of 21 materials with hydrogen peroxide was tested during immersion at 66°C for 1 week and for 12 weeks. Concentration of hydrogen peroxide samples was measured at the beginning and at the end of immersion period. In the next step stability of remaining liquid (material samples taken out) was measured according to MIL-PRF-16005F method. Comparison of these two types of measurements within immersion testing is presented in Tab. 3. Size of sample and volume of liquid corresponds surface area to liquid volume ratio equal to 0,13 cm<sup>-1</sup>.

Table 4. Comparison of measurements within immersion testing at 66°C.

|                            | <b>AOL 66°C seasoning</b>                           | <b>Stability Test</b>   |
|----------------------------|---|---|
| <b>Test item</b>           | material + liquid                                   | seasoned liquid<br>no material                                |
| <b>Temperature</b>         | 66°C  | 100°C   |
| <b>Time</b>                | 1 week / 12 weeks                                   | 24 hours  |
| <b>Result</b>              | concentration loss                                  | stability   |
| <b>Decomposition cause</b> | direct contact with material and proceeding elution | permanent contamination due to material elution to the liquid |

The following materials were subjected to testing:

- aluminum alloy 1100
- welded aluminum alloy 1100
- aluminum alloy 2219
- welded aluminum alloy 2219
- aluminum alloy 6061
- welded aluminum alloy 6061
- aluminum alloy 6082
- PTFE
- FEP
- EPDM white
- Kalrez 1050LF
- Kalrez 6380
- stainless steel 15-5PH
- stainless steel A286
- stainless steel 301
- stainless steel 304
- stainless steel 304L
- stainless steel 316L
- stainless steel 430
- stainless steel 347
- stainless steel 321

Samples of materials were immersed in four types of hydrogen peroxide HTP class (High Test Peroxide): 90% Grade ES, 98% Grade ES, 90% Grade HP, 98% Grade HP.

The best compatibility was observed between HP grades of hydrogen peroxide and aluminum alloys (1100, 6061 and 6082) as well as polymers (FEP, PTFE, Kalrez 1050LF and Kalrez 6380). ES grades of hydrogen peroxide in general showed worse stability due to additional contamination (chlorides and sulphates) and higher content of stabilizers did not counteract decomposition of hydrogen peroxide.

Short term contact with stainless steel (especially SS301 and SS304 alloys) may be considered as concentration loss and stability worsening is not that significant. But prolonged contact



(longer than couple of weeks) especially at increased temperature, should be avoided, as material elutes to the liquid, decreasing its stability. Even if hydrogen peroxide is no longer in contact with the material, it can get permanently contaminated and as a result less stable.

#### 4.4. Immersion testing at 100°C

Compatibility of 11 materials with hydrogen peroxide was verified during immersion testing at 100°C for 1 day and for 12 days. Initially aluminum alloy 2219 was introduced into testing but because of its incompatibility it was rejected from testing due to safety reasons. Volume of liquid corresponds to surface area to liquid volume ratio equal to 0,13 cm<sup>-1</sup>. After immersion period samples of materials were dried and sent to subcontractors for material properties testing and samples of hydrogen peroxide were collected for quality control analyzes.

The following materials were subjected to testing:

- aluminum alloy 1100
- welded aluminum alloy 1100
- aluminum alloy 2219 - excluded
- welded aluminum alloy 2219 - excluded
- aluminum alloy 6061
- welded aluminum alloy 6061
- aluminum alloy 6082
- PTFE
- FEP
- Karlez 1050LF
- Karlez 6380

Samples of materials were immersed in two types of hydrogen peroxide HTP class (High Test Peroxide: 90% Grade HP, 98% Grade HP).

The best compatibility was observed between 90% and 98% hydrogen peroxide for polymers (FEP, PTFE) as concentration of hydrogen peroxide did not decrease significantly and stability remained below 2%. Karlez 1050LF and Kalrez 6380 are compatible for short term contact and do not cause stability decrease. After prolonged contact stability remains at proper level but significant concentration loss is observed for Karlez 1050LF. Good compatibility was observed between 98% hydrogen peroxide and aluminum alloys (Al1100, Al6061 and Al6082). But aluminum alloys increase decomposition rate of 90% hydrogen peroxide as well as decrease stability after contact, probably due to elution of metals to liquid.

Apart from concentration loss and stability testing of hydrogen peroxide collected after immersion period, following physicochemical analyzes were performed: Anions determination, Ammonium ion determination, Evaporation residue determination, TOC (Total Organic Content) determination, Metal determination.

Content of anions did not increase in most cases. Only slight increase of nitrates was observed, because nitric acid is used for passivation of the material prior to contact with hydrogen peroxide, its trace residues may remain in the material. Afterwards it may be eluted into the liquid during immersion period. Leaching of nitrates was higher for rubbers – Kalrez 1050LF and especially Kalrez 6380, in which case level of anion exceeded allowable level.

Content of ammonium ion did not increase due to immersion of the material in hydrogen peroxide above allowable level according to MIL-PRF-16005F requirements.

Evaporation residue did not change significantly for most samples, differences were in a range of measurement error ( $\pm 2$  mg/kg). The amount of leached out non-volatiles (such as

aluminum) from the materials is too low to increase considerably evaporation residue. In case of Kalrez 1050LF there is noticeable trend, that amount of evaporation residue increases along with duration of seasoning, but does not reach the limit. Whereas Kalrez 6380 dissolves so considerably, that amount of evaporation residue exceeds allowable level (20 mg/kg) even after seasoning for 24 hours.

Total organic carbon decreased for most samples. Probably organic substances have been oxidized by hydrogen peroxide during immersion at 100°C.

Aluminum has eluted from all of the materials but the acceptable level with accordance to MIL-PRF-16005F was not exceeded. An exception was Kalrez 6380, because elution of aluminum was significantly higher than for other materials. Moreover after 12 days seasoning, aluminum content in hydrogen peroxide was much higher than the limit. Also iron and titanium was leached from Kalrez 6380 seasoned for 12 days in 90HP. Whereas no other metals than aluminum were detected in other samples.

Tin, which is a stabilizer of hydrogen peroxide (added as sodium stannate) forms a colloid of stannic oxide, which adsorbs contaminants. Unfortunately it tends to precipitate from the solution, especially in presence of impurities. In case of all samples, detected tin content was lower than initial. This might or might not influence further stability of the hydrogen peroxide, but no correlation between tin content and stability of seasoned hydrogen peroxide was observed. General trend may be observed that final tin content was lower for 90HP than 98HP.

Significant change of physicochemical properties were observed in hydrogen peroxide seasoned with Kalrez 6380. Resulting liquid contained large amount of contaminants especially organic substances, which leached due to material decomposition. Slight increase of nitrates, evaporation residue and total organic carbon was observed also in Kalrez 1050LF, nevertheless hydrogen peroxide remained compliant with MIL-PRF-16005F standard.

Seasoning of FEP and PTFE did not change properties of hydrogen peroxide in a considerable manner. The main issue was decrease of tin content while seasoning in 90HP over 24 hours (FEP) and 12 days (PTFE) below required level.

Low contamination with aluminum was observed while seasoning aluminum alloys, but it remained below allowable limit. But on the other hand decrease of tin level was more noticeable than for polymers and rubber.

Specification of 90HP hydrogen peroxide remained compliant with MIL-PRF-16005F after seasoning at 100°C with below materials:

- Al1100 (24 hours only)
- FEP
- PTFE (24 hours only)
- Kalrez 1050LF
- Kalrez 6380 (24 hours only)

Specification of 98HP hydrogen peroxide remained compliant with MIL-PRF-16005F after seasoning at 100°C with below materials:

- Al1100 welded
- Al6061 (24 hours only)
- Al6061 welded
- Al6082
- FEP
- PTFE
- Kalrez 1050LF
- Kalrez 6380

#### 4.5. Immersion testing at ambient temperature

Compatibility of 10 materials with hydrogen peroxide was verified during immersion testing at ambient temperature for 1 year. Initially EPDM was introduced into testing but due to its incompatibility it was rejected, because of safety reasons. Volume of liquid corresponds to surface area to liquid volume ratio equal to 0,13 cm<sup>-1</sup>. After immersion period samples of hydrogen peroxide were collected for quality control analyzes. Concentration and weight of the samples was measured at the beginning and at the end of seasoning. After seasoning at ambient temperature, material sample was taken out from liquid and stability was measured. Remaining liquid was analyzed in terms of stability and physicochemical properties.

Table 5. Summary of performed tests

|                            | <b>AOL ambient seasoning</b>                        | <b>Stability Test</b>   |
|----------------------------|---|---|
| <b>Test item</b>           | material + liquid                                   | seasoned liquid<br>no material                                |
| <b>Temperature</b>         | ambient   | 100°C   |
| <b>Time</b>                | 1 year  | 24 hours  |
| <b>Result</b>              | concentration loss/%AOL                             | stability   |
| <b>Decomposition cause</b> | direct contact with material and proceeding elution | permanent contamination due to material elution to the liquid |

The following materials were subjected to testing:

- aluminum alloy 1100
- welded aluminum alloy 1100
- aluminum alloy 2219 - excluded
- welded aluminum alloy 2219 - excluded
- aluminum alloy 6061
- welded aluminum alloy 6061
- aluminum alloy 6082
- PTFE
- FEP
- Karlez 1050LF
- Karlez 6380

Samples of materials were immersed in two types of hydrogen peroxide HTP class (High Test Peroxide: 90% Grade HP, 98% Grade HP).

Concentration loss ambient/1 year, %AOL ambient / 1 year, Stability 100°C/24h were analyzes.

Results show that probably high elution of impurities occurs in case of Al2219 alloy, which results in highly instable liquid, especially 90HP. Higher concentrated hydrogen peroxide 98HP does not show such deterioration, which may indicate its lower corrosivity and better compatibility with aluminum materials.

There was high difference in concentration loss of hydrogen peroxide with immersed materials and reference sample, but much less significant for %AOL and stability. The reason for such phenomena is not known, but it is worth mentioning, that reference samples were tightly closed with glass stopper. On the other hand, samples with material were kept in flask

covered only with watchglass, therefore closure was not tight, which prevented pressure build up.

Seasoning of FEP and PTFE did not change properties of hydrogen peroxide in a considerable manner.

Low contamination with aluminum was observed while seasoning aluminum alloys, but it remained below allowable limit for all samples except Al2219 and Al2219 welded in 90HP. But on the other hand decrease of tin level was more noticeable than for polymers and rubber, especially for 90HP.

Specification of 90HP hydrogen peroxide remained compliant with MIL-PRF-16005F after seasoning for 1 year at ambient temperature with below materials:

- FEP
- PTFE

Specification of 98HP hydrogen peroxide remained compliant with MIL-PRF-16005F after seasoning for 1 year at ambient temperature with below materials:- Al1100

- Al1100 welded
- Al6061
- Al6061 welded
- Al6082
- FEP
- PTFE

## 5. METAL PROPERTIES TESTING

Prior to testing samples were immersed in 90HP and 98HP hydrogen peroxide for 12 days at 100°C.

### 5.1. Static tensile strength

Test was performed by Institute of Non-Ferrous Metals. The essence of test consists in subjecting a specimen of the examined material characterized by a specific shape and size to the effect of tension, recording at the same time the force and the change in the length of the specimen caused by this force. In this way, the basic properties of the material such as the tensile strength (Rm), yield strength (Rp), and elongation (A). In this test was used sheet specimens with gage length 50mm. Static tensile test was performed on Instron 5582 (max load 100kN). The test was performed according to Static Tensile test method B with two different displacement velocities (0,75mm/min and 5mm/min after crossing 2% of strain deformation). Strain deformation was measure by very accurate video extensometer.

In some cases it may be found that properties of the material decrease after contact with hydrogen peroxide, but the same effect was observed for samples heated to 100°C. Welded samples cracked beyond the weld. Based on the results, it was found that hydrogen peroxide does not influence static tensile strength of tested materials.

### 5.2. Tear test

Tear Test was performed on Instron 5582 by Institute of Non-Ferrous Metals. Changes in value of initiation and activation energy are insignificant and may be caused by increased temperature (100°C). The most important parameter - tear strength value did not change after immersion of sample in hydrogen peroxide, which indicates no influence on this parametr.

### 5.3. Hardness Test

Investigation was performed according to polish standard PN EN – ISO 6506-1:2006 „Standard Test Method for Brinell Hardness of Metallic Materials” by Institute of Non-Ferrous Metals using the Duramin E-2500. Based on the Hardness test results, it was found that there

were no important influences hydrogenperoxide (HTP) on tested materials and welds. The differences are within the measurement error (3%).

#### **5.4. Crack propagation**

Crack propagation testing was performed by Air Force Institute of Technology in Warsaw. "Laboratory test was carried out on the compact tension C(T) specimens according to Laboratory Test Procedure PB-5/31 and Standard Test Method ASTM E647-15. The Constant Amplitude (CA) fatigue crack growth rate tests carried out for the C(T) test specimens manufactured from four different aluminum alloys allowed estimation of the crack growth rate curves.

#### **5.5. Corrosion testing and surface imaging**

Testing was carried out by the Institute of Non-Ferrous Metals in Skawina according to NACE TM0169/G31-12a. Samples were subjected to observation under the microscope as well as use of scanning electron microscopy and the x-ray analysis of chemical composition in the microareas on the surface and the cross-sections of metallographic microsections. Application of scanning electron microscopy with chemical analysis of microareas (EDS method) enabled determining products of corrosion of alloys exposed to concentrated H<sub>2</sub>O<sub>2</sub>.

Based on the research carried out, it was found:

- aluminum alloy 1100 before the conditioning in H<sub>2</sub>O<sub>2</sub>, a strongly defective surface with numerous defects was observed. Moreover, the occurrence of initial stages of layer corrosion was found as grains of propagating metal layers were observed at the grain boundaries,
- aluminum alloy 6061 before the conditioning in H<sub>2</sub>O<sub>2</sub> contains a few corrosion pits on the surface. There were slight defects in the form of fine pits mainly in the areas where there were particles,
- aluminum alloy 6082 before the conditioning in H<sub>2</sub>O<sub>2</sub> was free of defects. It was observed few and very small pitting sizes that may have come from surface preparation to testing,
- in most of the analyzed materials pits were observed. The amount and size of pits in most samples was dependent on the number and size of AlSiFe phases,
- the sample no 150 (6082 90HP) was completely covered by oxide. In some places small defects could be distinguished on the sample, which could indicate pitting corrosion,
- in samples no 149 (6082 90HP), 159 (6061 90HP), 166, 168 (6061 98HP), 169 (6082 98HP) and 179 (6061 98HP) apart from the round pits, grain boundaries were visible. The deepest grain boundaries were visible in samples 168 and 169. As in the other samples in which pits occurred, their amount was associated with the presence of AlSiFe phases,
- the average mass loss of aluminum alloys in 98% H<sub>2</sub>O<sub>2</sub> concentration for all analyzed aluminum alloys is almost twice as high as the 90% concentration,
- the average mass loss for 6061 and 6082 aluminum alloys is lower for both H<sub>2</sub>O<sub>2</sub> concentrations (90 and 98%) compared to the 1100 alloy,
- all observed pits and surface defects are in the surface layer and do not affect the quality of the materials.

#### **5.6. Stress corrosion cracking**

Stress corrosion cracking test was carried out by Institute of Non-Ferrous Metals according to the standard ECSS-Q-ST37C. Tests were performed under immersion in 90% and 98% H<sub>2</sub>O<sub>2</sub> (instead of solution of NaCl) over thirty day exposure period.

Based on results of the testing, it was found that hydrogen peroxide (HTP) and the applied tension does not affect both the metal sheets and welded sheets. Defects occurring at the joints between the weld and the sheets material do not arise as a result of the corrosion environment but the result of the welding process.

The results of strength properties of metal sheets and welded samples with non-stress corrosion. The samples were broken in the weld zone and border with the metal sheet.

## **6. POLYMER PROPERTIES TESTING**

Prior to testing samples FEP and PTFE were immersed in 90HP and 98HP hydrogen peroxide for 1 day and 12 days at 100°C.

### 6.1. Static tensile strength

Research was carried out by Institute of Engineering of Polymer Materials and Dyes Department of Elastomers and Rubber Technology in Piastów according to PN-ISO 37:2007 standard. The tensile strength value of FEP after exposure in hydrogen peroxide is unchanged compared to reference samples. Differences in the measured value are within limits of measurement error of this test method. Tensile strength of PTFE increased after immersion in hydrogen peroxide.

### 6.2. Tear Test

Research was carried out by Institute of Engineering of Polymer Materials and Dyes Department of Elastomers and Rubber Technology in Piastów according to PN-ISO 34-1:2007 standard. The value of tear strength of FEP and PTFE after exposure in hydrogen peroxide is unchanged compared to reference samples. Differences in the measured value are within limits of measurement error of this test method.

### 6.3. Hardness Test

Research was carried out by Institute of Engineering of Polymer Materials and Dyes Department of Elastomers and Rubber Technology in Piastów according to PN-ISO 868:2005 standard. The value of hardness of FEP and PTFE after exposure in hydrogen peroxide is unchanged compared to reference samples. Differences in the measured value are within limits of measurement error of this test method.

### 6.4. Cyclic Fatigue Compression Test

Research was carried out by Institute of Engineering of Polymer Materials and Dyes Department of Elastomers and Rubber Technology in Piastów according to internal method. Significant decrease in force value in the samples of FEP and PTFE after exposure to hydrogen peroxide was observed.

### 6.5. Dilatometry

Dilatometry analysis was conducted on Q400 EM TA Instruments apparatus by Warsaw University of Technology, Physics Faculty. Cylindrical samples were placed on a quartz measuring table and its dimension change in dependence of temperature was measured using quartz expansion probe. During measurements temperature was measured by adjacent to the quartz table thermocouple. Samples were heated with 5°C/min rate and contact pressure force of probe was equal to 0,2 N. During the measurement samples stayed at atmosphere of pure (99,999%) helium flowing through furnace chamber at flowrate 50 ml/min.

Analysis of FEP after immersion in 90HP and 98HP did not show any changes compared to reference samples.

In case of PTFE samples, slight increase of thermal expansion coefficient was observed. Moreover samples after immersion in 98HP did not exhibit contraction while heating from 40°C to 150°C which occurred for reference samples.

Kalrez 1050LF after contact with hydrogen peroxide shows contraction at 200 - 270°C, which was not observed in reference samples.

Thermal expansion coefficient lowered for samples of Kalrez 6380 after contact with hydrogen peroxide. Moreover its softening temperature lowered 20°C after immersion in 98HP and about 50°C after immersion in 90HP.

### 6.6. Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) were conducted on Q600 TA Instruments apparatus by Warsaw University of Technology, Physics Faculty. Samples of mass 10 to 30 mg were placed in a ceramic test crucible and heated with rate 10°C/min from ambient temperature up to final temperature of decomposition process. During the measurement samples stayed at

atmosphere of pure (99,999%) argon flowing through furnace chamber at flowrate 100 ml/min.

In case of samples of FEP and PTFE there was no changes observed between samples exposed to hydrogen peroxide and reference.

Final mass after heating to 600°C Kalrez 1050LF of exposed samples lowered from 15% of initial mass to 12%. The opposite effect was observed for Kalrez 6380, where final mass increased from 4% to 8%. Moreover decomposition process was more rapid than for reference samples.

#### **6.7. Thermo-Mechanical analysis (TMA)**

Thermomechanical analyzes (TMA) were conducted using Q400 EM TA Instruments apparatus, by Warsaw University of Technology, Physics Faculty. Samples were placed in quartz element for 3-point bending measurements (blade distance 5,08 mm) and measurement was conducted by a flexural probe. During measurements temperature was measured by adjacent to the quartz table thermocouple. Samples were heated with 5°C/min rate and contact pressure force of probe was equal to 0,2 N. During the measurement samples stayed at atmosphere of pure (99,999%) helium flowing through furnace chamber at flow rate of 50 ml/min.

In case of samples of FEP and PTFE there was no changes observed between samples exposed to hydrogen peroxide and reference.

Samples of Kalrez 1050LF and Kalrez 6380 after exposure to hydrogen peroxide were more prone to three-point bending than the reference samples at elevated temperatures.

#### **6.8. Dynamic Thermo-Mechanical analysis (DTMA)**

Dynamic Thermomechanical Analysis (DTMA) tests were conducted using the Q600 TA Instruments apparatus by Warsaw University of Technology, Physics Faculty. Samples were placed in a special aluminum element dedicated to 3-point bending dynamic measurements – low friction aluminum fixture (distance 10,16 mm) and measurement was conducted by a quartz flexural probe. Shape of FEP and PTFE samples was tile of 11 mm length, 3 mm width and 1 mm thickness, Kalrez 1050LF and Kalrez 6380 samples were a cylinder of 1 mm diameter and 11 mm length. During measurements temperature was measured by adjacent to the quartz table thermocouple. Samples were heated with 5°C/min rate and contact pressure force of probe between blades was equal to 0,2 N. For FEP and PTFE samples modulation force of 0,1 N amplitude and 0,5 Hz frequency as applied. For Kalrez 1050LF and Kalrez 6380 samples modulation force of 0,5N and 1Hz was applied. During the measurement samples stayed at atmosphere of pure (99,999%) helium flowing through furnace chamber at the flow rate of 50 ml/min.

No significant changes were observed for any material except Kalrez 1050LF, where contraction of sample occurred.

### **7. CATALYST LIFE**

Catalyst life testing was performed by Institute of Aviation. Aim of the research was to investigate influence of impurities in hydrogen peroxide, resulted from seasoning with material, on catalyst lifetime. Thruster-like catalyst bed configuration with propellant injector and outlet nozzle was applied for hot tests. Tested catalysts were commercial platinum supported on  $\gamma$ -alumina and developed by Institute of Aviation manganese oxide supported on  $\alpha$ -alumina. In first stage initial testing was performed in order to select catalyst for main campaign. Main test was performed in order to evaluate influence of type of propellant on catalyst lifetime. Selected catalyst for main campaign was 5% Pt supported on  $\gamma$ -alumina, provided by Elemental Microanalysis. Test duration was 530 s and total throughput of hydrogen peroxide amounted to 5,3 kg. Analysis of catalyst bed temperature and efficiency of characteristic velocity allowed to determine influence of impurities and stabilizers on catalyst lifetime. Tested types of hydrogen peroxide were:

- 90ES, 90HP, 98ES, 98HP

- 90HP seasoned with Al1100 (12 days, 100°C)
- 98HP seasoned with Al1100 (12 days, 100°C)
- 90HP seasoned with Al6061 (12 days, 100°C)
- 98HP seasoned with Al6061 (12 days, 100°C)

It is concluded that some level of random distribution of results occurs for all tests, but repeatability was in general high (especially for 98HP and 98ES) between tests of the same initial parameters. General logic between concentration of hydrogen peroxide and test results was noticed. Platinum catalyst was proved to be more active than the one based on manganese oxides. Due to the fact that differences of tests results are low, influence of stabilizers or impurities on catalyst lifetime cannot be clearly determined. Larger amount of propellant would be needed in order to investigate catalyst lifetime for different types of propellant. Based on test results it is indicated, that the lifetime reduction occurs (from highest to lowest influence) for the following types of propellant: Al110, Al6061, ES, HP.

## 8. CONCLUSION

### 8.1 Hydrogen peroxide safety

Based on results of testing it may be stated that hydrogen peroxide does not show unstable or explosive properties. Vibrations up to 100Hz does not cause decomposition of hydrogen peroxide but foaming may be observed but mixing causes slow decomposition, which is proportional to stirring speed. Gamma radiation causes oxygen evolution equal to 0,16 – 0,32 ml/kg\*kGy. Solubility of helium and nitrogen in hydrogen peroxide as well as its contact angle and surface tension was measured

### 8.2 Material compatibility

Change in mechanical properties of materials due to immersion in hydrogen peroxide was not significant or within measurement error. Influence of hydrogen peroxide on tested materials is determined as low at tested conditions (12 days, 100°C). Aluminum alloys did not show changes in mechanical properties. EPDM showed incompatibility after prolonged contact (2–3 weeks at room temperature) due to swelling and its following decomposition. Based on thermomechanical testing Kalrez 1050LF and Kalrez 6380 it may be concluded, that hydrogen peroxide changes properties such as susceptibility to bending, thermal expansion coefficient, increase of final mass of decomposition and contraction at certain temperatures. Moreover sticky substance evolved from the material after heating, which may indicate decomposition of the material. Bleaching after immersion in HTP was also observed. FEP and PTFE did not show changes in mechanical properties except for small increase of thermal coefficient for PTFE.

## 9. CLASSIFICATION OF MATERIALS

Classification of materials based on Active Oxygen Loss Testing according to NASA Report [1] "Fire, Explosion, Compatibility and Safety Hazards of Hydrogen Peroxide", NASA/TM-2004-213151, is shown in Tab. 5.

**Class 1** – Materials satisfactory for Unrestricted Use – suitable for long term contact, typical for storage. Stability after 66°C/1 weeks should not be worse than 95% and concentration loss higher than 5%.

**Class 2** – Materials satisfactory for Repeated Short-Term Contact – suitable for transient contact prior to storage or limited contact prior to use. Such contact should not exceed 4h at 72°C or 1 week at 22°C. Typical for valves, pumps in transfer lines or feed tanks. Stability after 66°C/1 weeks should not be worse than 95%.

**Class 3** – Materials that Should be Used Only for Short-Time Contact – to be used only if materials of Class 1 and Class 2 are not sufficient. Repeated contact is possible, but each single period should not exceed 1 minute at 71°C or 1h at 22°C prior to immediate use, because contamination may cause hydrogen peroxide unsuitable to storage. Hydrogen peroxide after contact with material of Class 3 consumed or disposed.



**Class 4** – Materials Not Recommended for Use – materials which cause excessive decomposition of hydrogen peroxide even on short-time contact, are attacked or deteriorated on contact, yield corrosion or deterioration products which cause excessive decomposition of hydrogen peroxide or form impact-sensitive materials.

Table 6. Classification of materials

| <b>Compatibility of 90% HP Grade hydrogen peroxide:</b>            |                |
|--|----------------|
| Aluminum alloys: 1100, 6061, 6082                                  | <b>Class 1</b> |
| Aluminum alloys: 2219  | <b>Class 2</b> |
| Welded aluminum alloys: 1100, 6061                                 | <b>Class 1</b> |
| Welded aluminum alloys: 2219                                       | <b>Class 3</b> |
| Polymer: PTFE, FEP   | <b>Class 1</b> |
| Polymer: Karlez1050LF, Karlez 6380                                 | <b>Class 2</b> |
| Polymer EPDM   | <b>Class 4</b> |
| Stainless steel: 15-5PH, A286, 301, 304, 304L, 316L, 430, 347, 321 | <b>Class 3</b> |
| <b>Compatibility of 98% HP Grade hydrogen peroxide:</b>            |                |
| Aluminum alloys: 1100, 6061, 6082                                  | <b>Class 1</b> |
| Aluminum alloys: 2219  | <b>Class 3</b> |
| Welded aluminum alloys: 1100                                       | <b>Class 1</b> |
| Welded aluminum alloys: 2219, 6061                                 | <b>Class 3</b> |
| Polymer: PTFE, FEP   | <b>Class 1</b> |
| Polymer: Karlez1050LF, Karlez 6380                                 | <b>Class 2</b> |
| Polymer EPDM   | <b>Class 4</b> |
| Stainless steel: 15-5PH, A286, 301, 304, 304L, 316L, 430, 347, 321 | <b>Class 3</b> |
| <b>Compatibility of 90% ES Grade hydrogen peroxide:</b>            |                |
| Aluminum alloys: 1100, 2219, 6061, 6082                            | <b>Class 3</b> |
| Welded aluminum alloys: 1100, 2219, 6061                           | <b>Class 3</b> |
| Polymer: PTFE, FEP   | <b>Class 1</b> |
| Polymer: Karlez1050LF, Karlez 6380                                 | <b>Class 2</b> |
| Polymer: EPDM  | <b>Class 4</b> |
| Stainless steel: 15-5PH, A286, 301, 304, 304L, 316L, 430, 347, 321 | <b>Class 3</b> |
| <b>Compatibility of 98% ES Grade hydrogen peroxide:</b>            |                |
| Aluminum alloys: 1100, 6061, 6082 – Class 2                        |                |
| Aluminum alloys: 2219  | <b>Class 3</b> |
| Welded aluminum alloys: 1100                                       | <b>Class 2</b> |
| Welded aluminum alloys: 2219, 6061                                 | <b>Class 3</b> |
| Polymer: PTFE, FEP   | <b>Class 1</b> |

|  |                |
|--|----------------|
| Polymer: Karlez1050LF, Karlez 6380                                 | <b>Class 2</b> |
| Polymer: EPDM  | <b>Class 4</b> |
| Stainless steel: 15-5PH, A286, 301, 304, 304L, 316L, 430, 347, 321 | <b>Class 3</b> |

## 10. ADDITIONAL FINDINGS: MATERIAL COMPATIBILITY AND IMMERSION TESTING

As an extension of the primary research, further immersion tests and mechanical property assessments were conducted to evaluate the effects of high-concentration hydrogen peroxide (HTP) on a broader range of metals and polymers.

This phase, executed by the Jakusz Spacotech specifically tested various materials under high-stress conditions to determine long-term stability and suitability in HTP-rich environments. In the initial stage, various materials, including aluminum, titanium, and stainless steel plates, along with composite samples, were subjected to screening immersion testing in 98% HTP. Based on results, proposed material for further testing were as follow:

Table 7. Proposed material for long term test program.

| No. | Material Type | Primery Material                            | Secondary Material |
|-----|---------------|---|--------------------|
| 1   | Monolithic    | Diffusion bonded aluminium A6061; process 2 | -                  |
| 2   | Polymer       | EPDM/FEP (Compound 1)                       | -                  |
| 3   | Fibre         | Fibre sic                                   | -                  |
| 4   | Composite     | Al 1050                                     | Al 6061            |
| 5   | Monolithic    | Al 1050                                     | Al 6061            |
| 6   | Monolithic    | Al 1050                                     | Al 6082            |
| 7   | Monolithic    | Al 1050                                     | Al 2219            |
| 8   | Monolithic    | Al 1050                                     | Al 1050            |
| 9   | Monolithic    | Al 1050                                     | Ti - 64            |

The samples were passivated and cleansed of impurities to minimize extraneous reactions. After chosing the materilas with the potenentially highest compatibility, focuse on long-term immersion tests. Testing included exposure at different temperatures (ambient, 66°C, and 100°C) and durations, enabling the evaluation of each material's stability and the rate of HTP concentration loss.

The study used analytical methods to measure metal elution and conducted tensile strength tests post-immersion to assess any changes in the material's mechanical properties

Key findings include:

- ✓ Aluminum Alloys: Al1050 as primary exhibited exceptional stability in static tensile strength, with minimal degradation, underscoring its suitability for applications exposed to high-concentration oxidizers.
- ✓ Polymer Compatibility: EPDM/FEP polymers demonstrated high resilience, maintaining structural integrity under extended immersion, making them potential candidates for flexible components like seals in HTP systems.
- ✓ Material Instability in Stainless Steel: Stainless steel showed significant reactivity and is strongly discouraged in HTP applications due to its rapid decomposition under testing conditions. SS materials were rejected after screening immersion test.

These additional tests support the identification of optimal materials for aerospace and industrial applications, emphasizing aluminum alloys and select polymers as primary candidates due to their proven chemical and thermal stability.

## 11. ABBREVIATIONS AND ACRONYMS

HTP - high test peroxide

AOL - Active Oxygen Loss  
TOC – Total Organic Carbon  
ES grade - extra stabilized grade  
HP grade - high purity grade  
TGA - Thermogravimetric analysis  
TMA - Thermo-Mechanical analysis  
DTMA - Dynamic Thermo-Mechanical analysis

### **11.1. Sample References**

1. NASA Report "Fire, Explosion, Compatibility and Safety Hazards of Hydrogen Peroxide"