Hydrogen Peroxide Material Compatibility Investigation

Sebastian Schuh, Tobias Bartok*, Robert-Jan Koopmans*, Carsten Scharlemann***
**FOTEC Forschungs- und Technologietransfer GmbH, Wiener Neustadt, Austria, Email: schuh@fotec.at*
***University of Applied Sciences Wiener Neustadt, Austria*

Abstract

Due to its potential use as monopropellant as well as oxidiser for bipropellant systems, hydrogen peroxide is again in the focus of several research groups. One of the major challenges prior to extended use for propulsive means is the questions of material compatibility of hydrogen peroxide in general and with standard materials used in space applications in particular. Although a significant number of material compatibility investigations have been performed throughout the years, most of the results are outdated or poorly documented and even, when comparing results from different groups, contradictory. The present paper details the experimental efforts conducted at FOTEC to investigate the compatibility of hydrogen peroxide with various materials at different temperatures. The recently finalized first test series with different types of stainless steels and synthetic materials shows the high accuracy of the test facility.

1. Nomenclatures

AOL	Active oxygen loss
EPDM	Ethylene propylene diene monomer
FEP	Fluorinated ethylene propylene
HTP	High Test Peroxide
PTFE	Polytetrafluoroethylene
PVDF	Polyvinylidene fluoride
SEM	Secondary electron microscope

2. Introduction

Highly concentrated hydrogen peroxide is investigated as propellant for space propulsion applications. Its main benefits compared with other monopropellants (e.g. hydrazine) or other oxidisers (e.g.: MON) are its low toxicity, easy handling and, most importantly, its existing [1-3]. A variety of scientific groups currently investigate different propulsion systems based on highly concentrated hydrogen peroxide [4-16]. Despite its regular use on launch systems and its past use in space (mission duration over several years), its long term storage capability and therefore its general material compatibility remains for many people one of the most urgent questions to be answered [17-19]. Premature decomposition of the stored propellant in the tank or in the feed system (both in general are non-vented systems) would lead to pressure increase. Hydrogen peroxide compatibility has been investigated regarding its compatibility since the early 1930s, but lack of set-up description, incomparability of test setups and investigated probes often led to inconclusive or even contradictory results. This paper presents the effort of establishing an experimental facility for systematic investigation of compatibility of highly concentrated hydrogen peroxide with various materials. In the following chapter, an overview and discussion of past efforts is given. The section afterwards describes the experimental setup and its key features. Accuracy of the measurements are presented and discussed in the subsequent section, followed by a section dedicated to the detailed description of the material sample preparation. This is followed by a presentation of the first compatibility results conducted with various types of stainless steels and synthetic materials, obtained with the test facility.

3. Overview and discussion of previous studies

Ref. 20 reports an early investigation of hydrogen peroxide storability, using unstabilised hydrogen peroxide with a concentration of 98% by weight. Stored at temperatures of 20°C in 4 litre, non-vented vessels, a pressure increase of less than 1 bar over a duration of 4 weeks is reported. Unfortunately, no detailed information on ullage tank volume, material of the vessels and many other relevant information is given.

An early comprehensive survey on compatibility studies existing to this date is reported in [21], summarising results from various investigations. This includes H_2O_2 compatibility data for aluminium and aluminium alloys, stainless steel alloys, selected metals, plastics and rubbers, lubricants, ceramics and refractory materials. In addition, storability improvements as a function of improved production processes are reported, comparing data for 90% weight concentrated H_2O_2 from 1947, 1953 and 1965, showing AOL rates to decrease from 1% (1947) to 0.02% (1965) per year for storage at approximately 30°C. However, no detailed information about the hydrogen peroxide or the test conditions are given, other than stating that the improvement is anticipated to be caused by improved purity in hydrogen peroxide manufacturing, improved container material selection and surface treatment and the development of more effective stabiliser techniques.

Although these studies, along with others [22, 23], comprise important early information on the principal ability of long term storage of hydrogen peroxide, these results should be treated with caution, as manufacturing methods and quality for hydrogen peroxide and storage materials have changed significantly over the past 40 years. This is especially true for improved hydrogen peroxide stabiliser mixtures and material surface treatment methods.

A more recent investigation has been conducted by Ventura [24], reporting compatibility data for extreme storage conditions at ambient outdoor conditions in Texas in a vented container. He reports on decomposition of less than 0.4% for 90% weight concentrated hydrogen peroxide. A reference container with equal initial concentrated hydrogen peroxide, stored at 5°C is stated to show virtually no decomposition over the duration of 17 years. Again, no detailed information on test procedures and test conditions, such as detailed information on the investigated propellant, (e.g. nature of stabilisers), nor concerning the storage tanks, such as tank materials used, is given. In addition, tests in vented storage tanks as used in this study are deemed a rather unrealistic simulation of actual in space storage conditions.

Whithead [25] published a thorough study of long term storability of hydrogen peroxide. He investigated variable tank sizes manufactured out of anodised and non-anodised Aluminium 6061-T6, polycarbonate and PVDF. This investigation is of particular interest since viable detailed information concerning surface to volume ratios and ullage volumes are given. In addition, this study was performed for non-vented storage conditions. Due to safety reasons, only pressures below 20 bar were investigated. Whitehead reports the safety limit be reached in the worst case (Al 6061-T6 uncoated) within 3 to 4 weeks, whereas tank pressure stabilised for aluminium tanks with PVDF liner at approximately 8 bar after 25 weeks. However, Whitehead explicitly states that unstabilised hydrogen peroxide was used for all investigations and no dedicated cleaning or passivation procedures were conducted before testing. In addition, the study’s focus on small satellites led the investigation of rather small tanks, leading to large, unfortunate ratios of wetted tank surface to volume. All these factors lead to the results published being some kind of a worst case scenario for long term storability of hydrogen peroxide as employed in typical mission scenarios.

In space experience of hydrogen peroxide storability is available from the 1960's space missions, Syncom II, Syncom III, and the Early Bird satellite [26]. All of them used a monopropellant hydrogen peroxide propulsion system. In particular Early Bird showed in orbit a lifetime of 3 years but was estimated to have a useful life of 5 years. The Russian spacecraft Soyuz, operating since 1967, uses eight 10 N hydrogen peroxide monopropellant thruster for attitude control. No accidents due to hydrogen peroxide on-board are known. Iarochenko and Dedic [27] from the Russian Scientific Center (RSC) point out that with the presently used stabiliser mixture the decomposition of hydrogen peroxide is reduced to 0.01% per year. Unfortunately, neither for the early space mission nor for the present Russian system detailed information about tank material, tank surface treatment, hydrogen peroxide stabilisers etc. are available.

It is obvious from the above discussion that for most published studies, a severe lack of information concerning vital parameters about the propellant investigated as well as concerning the tank materials persist and test conditions makes them less valuable for other researchers.

4. Classification of storage materials

An often employed scheme, used to classify materials concerning their compatibility with hydrogen peroxide, distinguishes four classes of materials [21]. Though these classes are arbitrary, they provide a standard throughout most of literature for rating the compatibility of materials with hydrogen peroxide. The definitions stated are reproduced from Ref. 21:

- **Class 1: Materials Satisfactory for Unrestricted Use with H202.**
  Such service includes long-time contact with the H_20_2. Typical use is for storage containers.
- **Class 2: Materials Satisfactory for Repeated Short-Time Contact with H_20_2.**
  Such materials are used for transient contact with the H_20_2 prior to storage of the H_20_2, or limited contact with the H_20_2 prior to use. Such contact is not to exceed 4 hours at 72 °C or 1 week at 22 °C. Typical uses are for valves and pumps in H_20_2 transfer lines and feed tanks.

- **Class 3: Materials which should be used only for short-time contact with H_20_2.**
  These materials should be used only where neither a Class 1 nor Class 2 material would suffice. These materials can be used for repeated contact, but a single use period should not exceed 1 minute at 72°C or 1 hour at 22°C. An example of a Class 3 application is materials for use in a flow system. The hydrogen peroxide should be consumed in the application or disposed of after the test because contamination of hydrogen peroxide solutions with Class 3 material is usually sufficient to render it unsuitable for storage. Many Class 3 materials indicate satisfactory room temperature service. However, the material should be checked prior to use.
- **Class 4: Materials not recommended for use with H_20_2.**
  These materials
  - cause excessive decomposition of H_20_2 even on short-time contact,
  - are attacked or deteriorate on contact,
  - yield corrosion or deterioration products which cause excessive decomposition of H_20_2 on subsequent contact, or
  - form impact sensitive mixtures with concentrated H_20_2.

One can see that these classifications leave a lot of room for interpretation for the authors, especially when it comes to different requirements of a H_20_2 application. There are no clear distinctive requirements a material has to fulfil in order to classify as ‘Class 1’ material. This makes it almost impossible to use the classifications found in literature to determine whether a certain material can be used for a H_20_2 based propulsion system. Figure 1 shows about 300 AOL results found in literature, coloured by the classification the authors have chosen. The data presented comprises of results from Refs. 17, 20, 21, 22, 24, 28, 29, 30. To enhance comparability, AOL results have been normalised to AOL% per week values. In order to avoid large numbers of overlapping markers, results for 30°C and 66°C have been split to ±2°C for display purposes only to enhance visibility, as indicated.

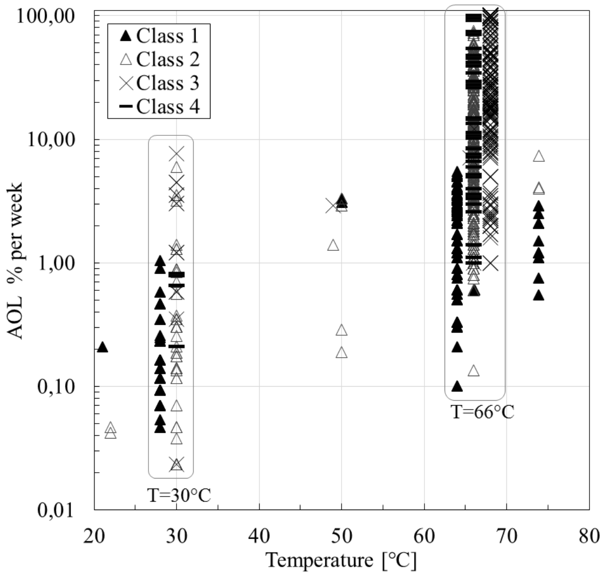

Figure 1: Contradictory classification found in literature

The lack of stringent classification definition as well as the problem in comparability of different studies, mainly caused by lack of available information, leads to largely inconclusive and contradictory results as evident in Figure 1. This becomes evident when observing that for identical temperature, material class categorisations largely overlap or even show reduced AOL values for materials that were categorised in higher classes, and vice versa.

The lack of comparability, contradicting results and largely outdated data necessitates a systematic investigation of hydrogen peroxide material compatibility. In addition, such an effort accounts for the anticipated increased in hydrogen peroxide storability due to improved stabiliser composition as well as production quality, a thorough investigation of material compatibility specifically dedicated to identify the impact of storage tank material and surface properties is needed. Such an investigation, able to identify high-grade hydrogen peroxide compatibility for different materials typically used in commercial chemical propulsion systems is described hereafter.

5. Experimental setup

The basic idea behind the design of the experimental setup is the determination of the amount of evolved oxygen due to the decomposition of HTP on the surface of the material sample. The requirements to the test facility are therefore the integration of a precise volume measurement, the recording of all parameters influencing the volumetric measurement (temperature, pressure) and the application of tightness tested connecting systems and materials to achieve the aspired resolution. A schematic of the test facility is shown in Figure 2. The major components are listed in Table 1.

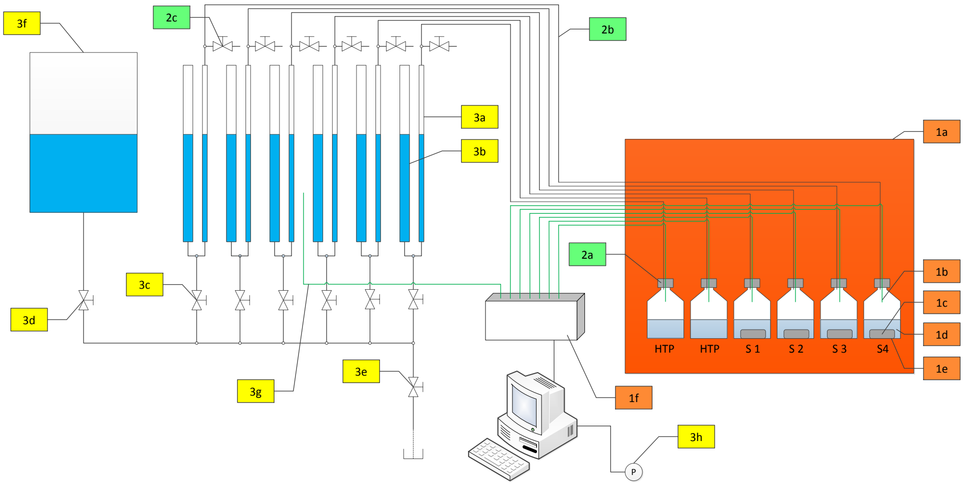

Figure 2: Schematic of test setup

Table 1: Key components of test facility

	No	Component
Heating system	1a	Thermal chamber
	1b	Thermocouple
	1c	Material sample
	1d	Test fluid
	1e	Sample flask
	1f	Thermocouple input module
Gas connection	2a	Connection gas piping/sample flask (PTFE screw cap)
	2b	Gas piping
	2c	Gas vent for venting of measurement setup
Volume measurement setup	3a	Pipette
	3b	Water reservoir
	3c	Water refill vent
	3d	Water tank vent
	3e	Water vent for emptying of measurement setup
	3f	Water tank
	3g	Thermocouple
	3h	Atmospheric pressure sensor

The test setup can be divided into two main parts: the thermal chamber, where the test flasks (containing the material samples immersed in HTP) are located and the measurement unit for the determination of the evolving oxygen volume.

Part 1 – Thermal chamber:
The temperature inside the thermal chamber can be varied between 5 and 50°C, allowing investigation of the influence of temperature on the decomposition rate. The test samples are placed in 25 ml Pyrex flasks, which are partly filled with HTP. To ensure that all test flasks are on the same temperature and the temperatures show a satisfying stability ($\Delta T < 0.1$ K between start and end of testing), every flask is connected to an individual thermocouple, recording the temperatures during the entire test run. For measuring the oxygen evolution, the flasks are connected to the volume measurement unit (part 2) by stainless steel pipes, where PTFE-plugs are used to connect the pipes to the test flasks.

Part 2 – Volume measurement unit:
The key components of the volume measurement unit are water filled pipettes and water reservoirs which are connected to each other (comparable to a water level gauge). As oxygen evolves due to the decomposition of HTP, the increasing volume leads to a displacement of water in the pipettes. Therefore, the volume of the collected oxygen gas is directly measured using the scale on the pipettes.
The test setup allows for six individual samples to be investigated in parallel. However, it was decided to investigate four identical samples of one material only per test run, in order to provide statistical relevant results. The two remaining test flasks are used to determine the influence of the decomposition of HTP at the glass surface of the Pyrex flask, the bulk decomposition and the evaporation of water and HTP during the measurement. Therefore, the amount of HTP is chosen in a way to ensure the wetted surface inside the flasks to be equal to the flasks filled with material samples.

To investigate the tightness of the test facility, a series of tightness tests were performed. For such testing, the test facility was pressurised (leading to a higher water level in water reservoirs compared to the water level in the pipettes), followed by monitoring the water level in the pipettes over time. A change of the gas volume in the test facility may be attributed to several reasons: expansion/contraction due to temperature change, expansion/contraction due to pressure change or a leakage. To be able to consider the influence of a change of the temperature and the pressure, thermocouples for measuring the room temperature and the temperature inside every flask, as well as a sensor for measuring the ambient air pressure are implemented into the test facility. To further increase the accuracy, the evaporation of water in the volume measurement equipment is included in the analysis. Using the ideal gas equation, the theoretical change of volume is then calculated. In order to do so, all gas volumes inside the test facility (pipes, flasks, pipettes, connecting pieces, etc.) have to be well known. By comparing the calculated values with the measured volume changes, a maximal deviation rate is determined. Tightness tests without material samples showed a maximal deviation rate of 0.004 ml/h.

6. Measurements, correction and accuracy

AOL measurements are performed at different temperatures to investigate its impact on the decomposition rate. The test campaign starts at a thermal chamber temperature of 5°C, followed by a stepwise increase of 15°C until reaching 50°C. After every single compatibility test at a given temperature, the measured gas volumes are corrected by taking into account the change of the ambient temperature and pressure, as well as the change of the temperatures inside the flasks and the amount of evaporated water in the volume measurement unit. By comparing the corrected gas volumes of systems with and without material sample, the AOL rate induced by the decomposition of HTP on the material sample surface only can be determined. As a final step, the reduction of the HTP concentration during the test is calculated to determine the starting conditions for the next compatibility test. It has to be mentioned that beside the decomposition of HTP at the material sample surface, the bulk decomposition, decomposition on the Pyrex glass surface, etc. are considered for the correction procedure.

7. Sample preparation and test procedure

Materials typically encountered in propulsion system components have been selected for compatibility investigations. The materials tested in this effort are stainless steels 15-5 PH, 304L, 347 and 430 and the synthetic materials PTFE (virgin), FEP (virgin) and EPDM. For all but one material, an edge free design of the material samples (see Figure 3) was established. Stainless steel and PTFE samples have been fabricated in-house with a rotating machine. To avoid any contamination during the fabrication process, the cutting inserts were changed after every material change. As a final processing step the stainless steel samples were polished with a Schotch-Brite 07447 handpad before they were wiped with ethanol. The FEP samples were delivered by Holscot in the desired sample shape already. EPDM was available as sheet material only. For this reason a disc-shaped sample geometry was manufactured by means of water jet cutting. Besides PTFE cylinders also PTFE discs were manufactures with the turning lathe. By comparing tests with PTFE cylinders and PTFE discs, it was possible to deduce the influence of the sample geometry on the AOL.

Figure 3: Left: cylindrical stainless steel sample; middle: cylindrical PTFE sample; right: disc-shape PTFE sample

The sample properties (shown in Table 2) were selected such that the surface to volume ratio for all compatibility tests was fixed at 1.1 cm^{-1}.

Table 2: Sample properties

Sample shape	Cylindrical	Disc (PTFE)	Disc (EPDM)
Height [mm]	21.0	2.0	0.4
Diameter [mm]	13.5	24.6	25.7
Rounding radius [mm]	2.0	0	0
Surface to volume ratio [cm^{-1}]	1.1	1.1	1.1

For cleaning the test samples, an extensive procedure was applied to exclude any influences of contamination on the decomposition of HTP. The stainless steel probes were cleaned with special lowlinting wipes in combination with demineralised water, acetone and isopropanol, in a first step. The chemicals were chosen in that way to cover a spectrum of polarity indices as large as possible. After mechanical cleaning, the stainless steel samples were placed in an ultrasonic bath. All synthetic material samples were mechanically cleaned only with lowlinting wipes in combination with demineralised water and ethanol. Synthetic material sample cleaning with an ultrasonic bath was skipped to avoid the risk of diffusion of the solvent into the synthetic material. All samples were dried with nitrogen gas in between every single cleaning step. After the cleaning procedure the samples were gauged (geometric parameters and mass) and investigated with an optical microscope (all samples) and a secondary electron microscope (only metallic samples and PTFE). Subsequently the samples were cleaned once again (metallic samples with ultrasonic bath, synthetic material samples mechanically with lowlinting wipes) to remove possible contamination during the measurements. Finally, all samples were inserted into Pyrex flasks, which were then filled with HTP. Stabilised Evonik Propulse^{TM} 875 HTP from a single batch with actual weight concentration of 87.73% complying with manufacturer specifications regarding purity and stabiliser composition, was used for all tests presented hereafter. After 24 hours at room temperature, the HTP was replaced by fresh HTP before the compatibility test was started. The measurements started at the lowest investigated temperature. For this the thermal chamber was cooled to 5°C and measurements started as soon as a stable temperature was reached. To achieve an as high as possible accuracy, the full measuring range of the pipettes was exploited thereby collecting the largest possible oxygen volume. Depending on the material's compatibility with HTP, the test periods varied between 15 min and several days. After a decomposition test was finalised, the temperature was increased by 15°C. Measurements continued as soon as the temperature was stable again. The highest investigated temperature was 50°C. After finalising a test series, the flasks with the material samples immersed in HTP were stored in the fridge. After one month the flasks were removed from the fridge, the HTP was replaced by fresh HTP and the compatibility test at four different temperatures was performed again. When all compatibility tests were completed the test samples were weighed again and investigated with an optical and secondary electron microscope.

8. Results

Figure 4 to Figure 7 show results for the four investigated stainless steels using the facility described above. It should be noted that all four investigated stainless steels were chosen based on their frequent occurrence in standard commercial propulsion system components only and not based on any anticipation regarding good compatibility characteristics.

The results show the expected exponential dependency of decomposition on temperature (straight line in the semi-logarithmic diagram), as well as ability of the facility to measure low %AOL values as indicated by the error bars.

Note that the resulting error is smallest for small temperatures and increases for higher temperatures, although the logarithmic scale would lead to believe otherwise. It is worth mentioning that the decomposition of hydrogen peroxide on the flask wall is typically found to be in the range from approximately 0.03 %AOL per week at 5°C to 2.5 %AOL per week at 50 °C.

On the right side of Figure 4 to Figure 7 a comparison of the SEM pictures of the sample surfaces before and after the whole test series is shown. Note, that in Figure 7 the AOL value at 5°C was extrapolated by using the measurement results at 20, 35 and 50°C. The error bars indicate the maximal extrapolation error. All but 304L revealed the occurrence of dark spots after the whole test series. Especially the spots on the 15-5 PH samples were easily noticeable as they have diameters of some tenths of a µm. To investigate if the diameter and/or number of the dots increases when increasing the storage time, the 15-5 PH samples were immersed in HTP again and stored at room temperature for 10 days. Afterwards the same location of the sample surface (45 x 35 µm) was scanned again with the SEM, followed by a comparison of the pictures. As can be seen in Figure 8 no noticeable increase in diameter of the spots was determinable, however, a slight increase in number of spots was observed. They are indicated in the picture with white arrows. It is expected that these spots are an indication of pitting, but a more detailed investigation is necessary to determine the exact cause.

A comparison of the measured AOL values with literature is difficult due to lack of information on precise test conditions. For example, Cefic [29] defines 304L as a class 1 material, but no information concerning AOL value, material sample shape, surface to volume ratio or temperature are specified. Tests with 304L shown by McCormick [20] with HTP (Becco) with a concentration of 90% showed an AOL of 58 % per week at 66.11 °C, but again important information like the surface to volume ratio is missing. In the hydrogen peroxide handbook of the U.S. Air Force [21] stainless steel 430 is defined as a class 4 material with an AOL of 74.4 % per week at 66°C. Considering Figure 5 and Figure 6, it is obvious that stainless steel 430 and 304L have a similar behaviour concerning their compatibility with HTP. But as mentioned before, the material 304L is defined as class 1 material by Cefic [29], 430 as class 4 material by [21]. These examples clearly show that the current classification of materials should be treated with extreme care as difference in test conditions can lead to entirely different results.

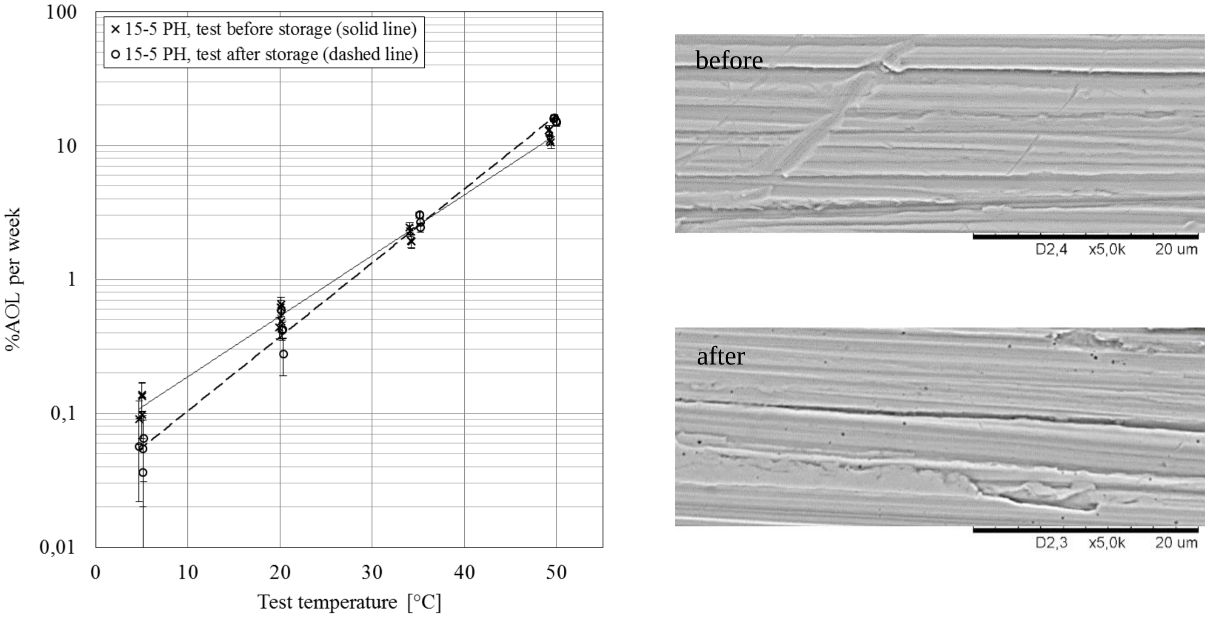

Figure 4: Left: AOL % per week for 15-5PH before and after storage; right: SEM pictures before and after the whole test series.

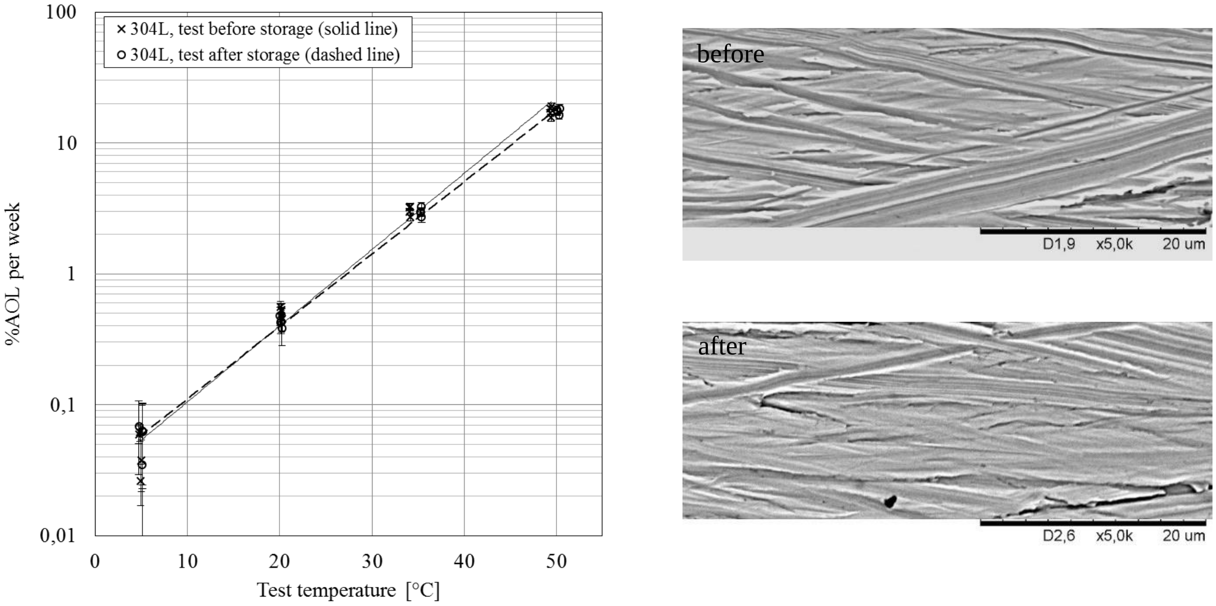

Figure 5: Left: AOL % per week for 304L before and after storage; right: SEM pictures before and after the whole test series.

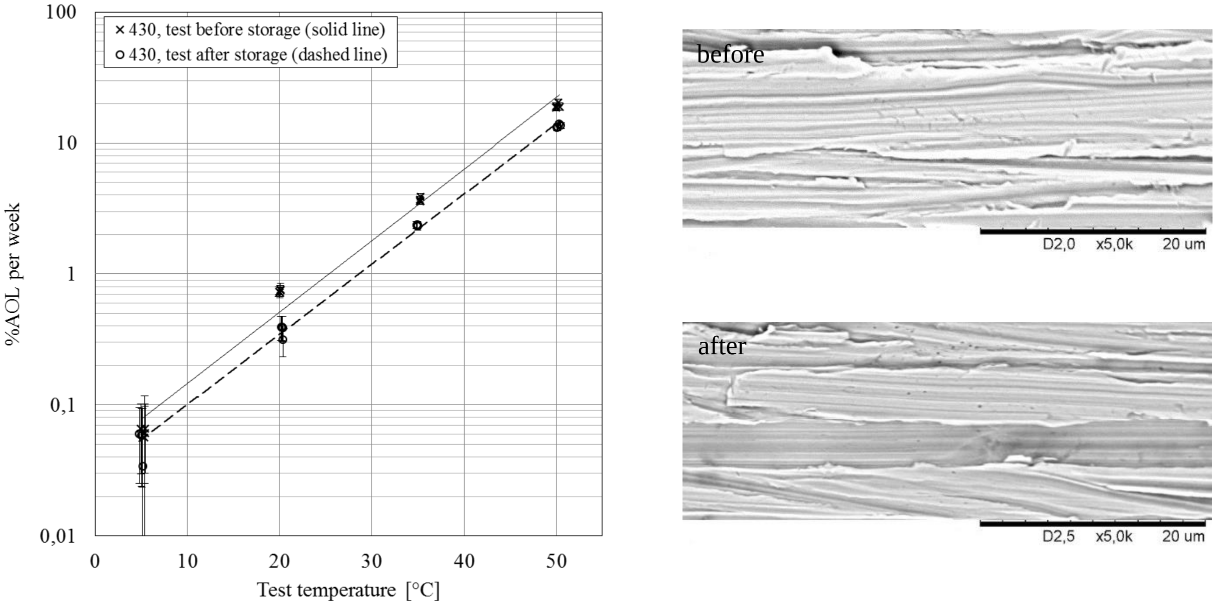

Figure 6: Left: AOL % per week for 430 before and after storage; right: SEM pictures before and after the whole test series.

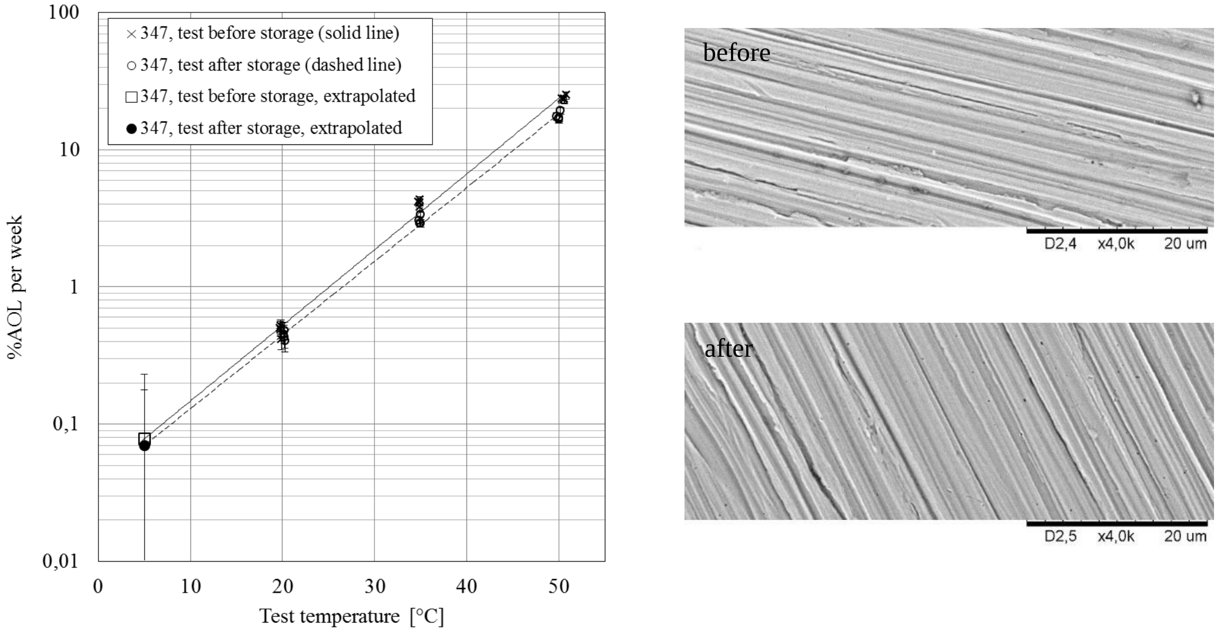

Figure 7: Left: AOL % per week for 347 before and after storage; right: SEM pictures before and after the whole test series.

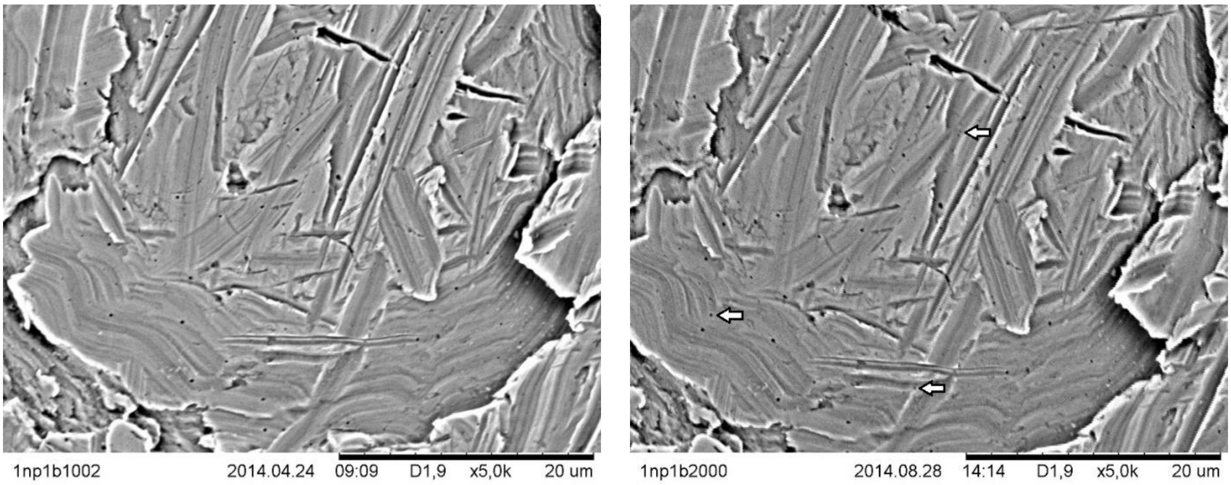

Figure 8: Left: SEM picture of 15.5 PH sample surface after storage in HTP for 1 month in the fridge; right: SEM picture of 15-5 PH sample surface after storage in HTP for 1 month in the fridge and 10 days at room temperature. Newly formed dots are marked with white arrows.

Besides metals, a number of synthetic materials were investigated. These were virgin PTFE, virgin FEP and EPDM. PTFE and FEP samples were manufactured in the preferred cylindrical shape. Due to the fact that EMPD was only available as a rubber sheet, disc-shaped samples were used for the tests instead. To be able to compare the results between tests with cylindrical shaped and disc-shaped samples, a reference measurement was performed. For this PTFE samples were manufactured in both cylindrical and disc shape, where the parameters were chosen in such a way that the S/V ratio was kept the same during tests. As can be seen in Figure 9 the difference in shape has almost no influence on the test result. This result shows that the assumption that different geometries of the same synthetic material does not influence the AOL result is reasonable.

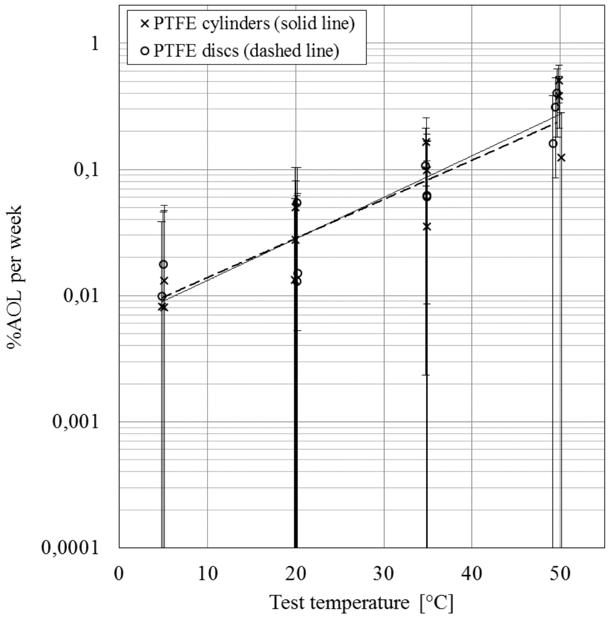

Figure 9: Comparison of compatibility test results performed with PTFE using cylindrical and disc-shaped samples.

Figure 10 and Figure *11* show the results of the investigated synthetic materials. Due to the risk of contamination of the SEM as a consequence of outgassing of the test samples made of FEP and EPDM, the SEM investigation was skipped for these materials. In comparison to the investigated metals, tests with synthetic materials resulted in AOL values up to two order of magnitude lower, indicating a better compatibility with hydrogen peroxide. For better comparability all temperature dependent AOL values are plotted in one diagram on the left side of Figure 12. For sake of completeness the results of the investigated metals are shown as well. In this diagram the distinction of the two material groups is easy to see. To signify the difference in AOL values at 35 and 50°C the results are plotted on a linear scale as well, see the right of Figure 12.

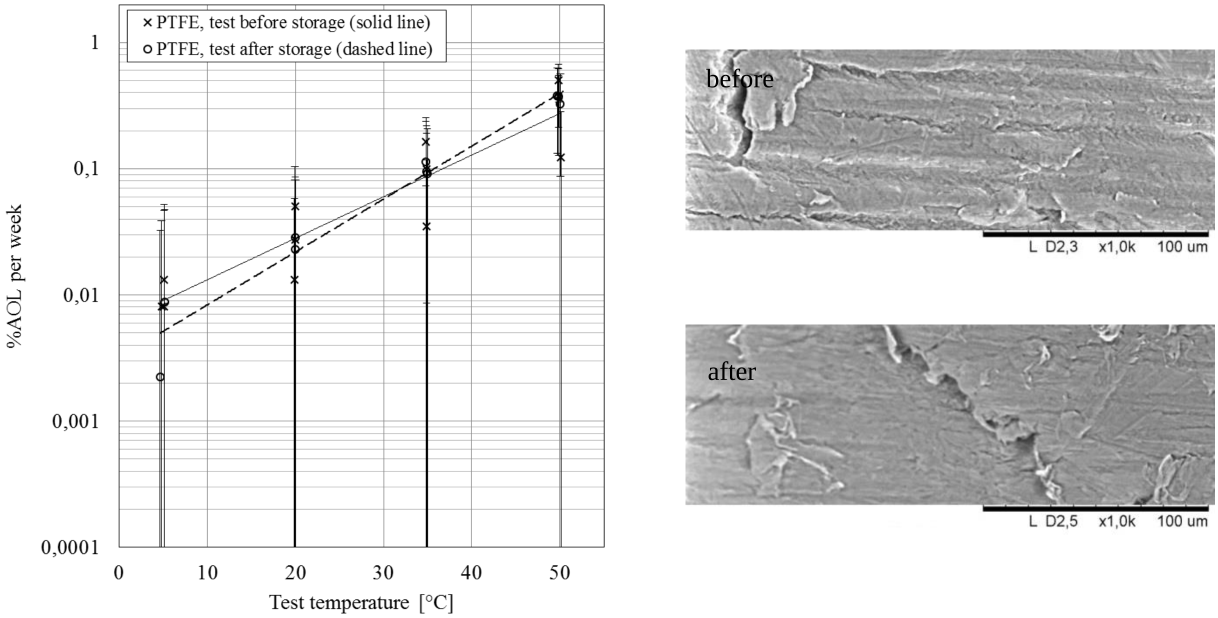

Figure 10: Left: AOL % per week for PTFE before and after storage; right: SEM pictures before and after the whole test series.

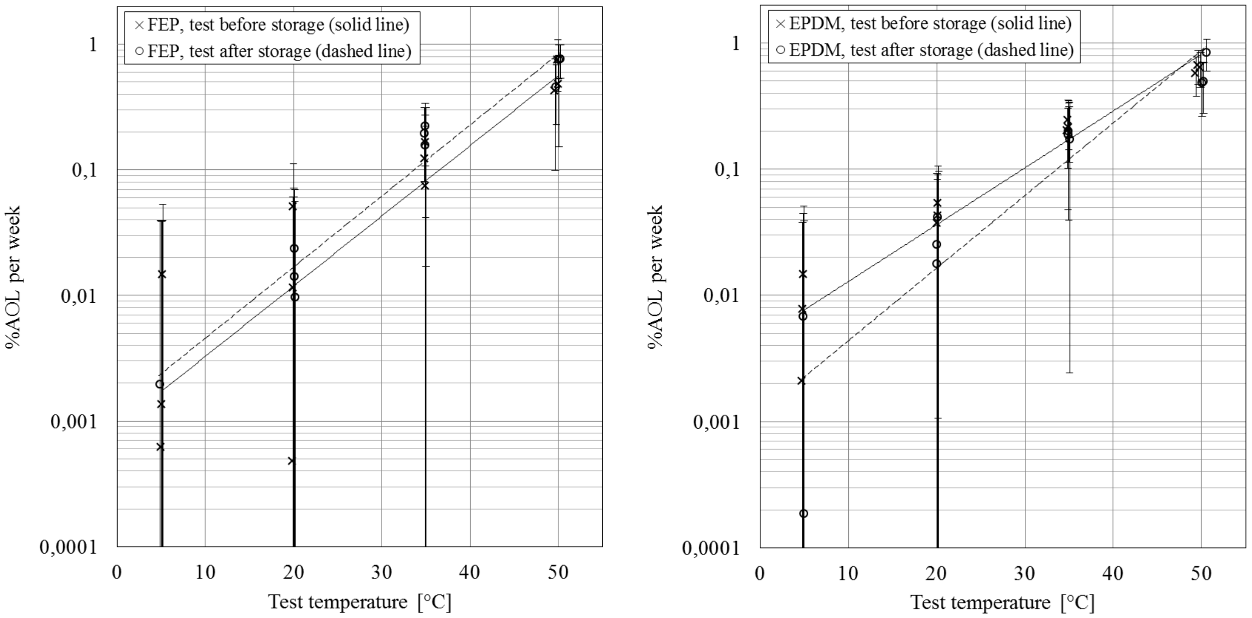

Figure 11: Left: AOL % per week for FEP before and after storage. Right: AOL % per week for EPDM before and after storage.

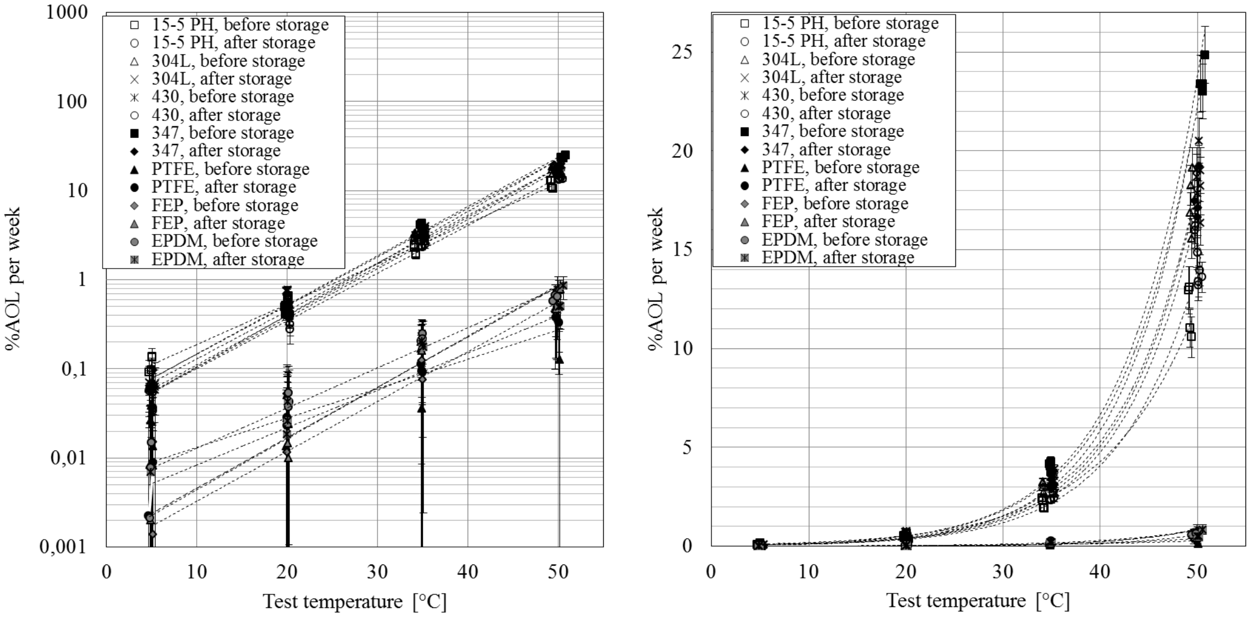

Figure 12: Left: comparison of AOL values of the investigated materials in a semi-logarithmic plot; right: comparison of AOL values of the investigated materials shown in a plot with linear scales.

9. Conclusion and future work

An experimental facility and procedure to investigate hydrogen peroxide material compatibility in a standardised manner has been presented. This effort is intended to contribute to the understanding of propellant storability. Key components of the facility are described and test conditions and procedures as well as material samples and sample preparation are given. Special effort has been put into describing the precise test conditions to guarantee reproducibility. The first temperature dependent set of results for four different stainless steels and three different synthetic materials have been presented. The results prove the facility's ability to investigate material compatibility, by reproducing expected decomposition dependency on temperature, as well as by experimentally proving the measurement error to be small, even for AOL values smaller than 0.1%AOL per week.

Future work will include a spectral analysis of the HTP to find out if any of the materials or components of the materials dissolve in HTP over time. Therefore the HTP that was exchanged after the storage of the material samples for one month, is stored in the fridge in Pyrex glass flasks. Additionally, also the HTP from the reference flasks will be investigated to be able to distinguish between elements already present in the HTP, such as stabilisers, and elements that originate from the test samples.

The decomposition of HTP is a kind of a chemical reaction that can be described with an Arrhenius equation

$$k = A_0 e^{-\frac{E_a}{RT}} \tag{1}$$

where k is the rate constant of the chemical reaction [mol m^{-2} s^{-1}], A_0 the frequency or pre-exponential factor [m/s], E_a the activation energy [J mol^{-1}], R the universal gas constant [J mol^{-1} K^{-1}] and T the absolute temperature [K].

From the obtained results the activation energy and frequency factor will be derived.

10. Acknowledgement

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