Compatibility Studies of Hydrogen Peroxide and a New Hypergolic Fuel Blend

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ABSTRACT

Several preliminary materials compatibility studies have been conducted to determine the practicality of a new hypergolic fuel system. Hypergolic fuel ignites spontaneously as the oxidizer decomposes and releases energy in the presence of the fuel. The bipropellant system tested consists of high-test hydrogen peroxide (HTP) and a liquid fuel blend consisting of a hydrocarbon fuel, an ignition enhancer and a transition metal catalyst.

In order for further testing of the new fuel blend to take place, some basic materials compatibility and HTP decomposition studies must be accomplished. The thermal decomposition rate of HTP was tested using gas evolution and isothermal microcalorimetry (IMC). Materials were analyzed for compatibility with hydrogen peroxide including a study of the affect welding has on stainless steel elemental composition and its relation to HTP decomposition. Compatibility studies of valve materials in the fuel blend were performed to determine the corrosion resistance of the materials.

The thermal decomposition rate of 90% HTP was found to be in a range of $3x10^{-6}$ s^{-1} and $5x10^{-6}$ s^{-1}. Using an isothermal microcalorimeter, it was found that two of the metal samples were not compatible with 98% HTP and are not recommended for use as structural materials. The two other samples did not show any signs of premature decomposition of 98% HTP or any visible corrosion, therefore they are recommended for repeatable contact with HTP.

Composition changes in stainless steel due to welding were apparent from an X-ray Fluorescence (XRF) analysis. A correlation between and enhancement in Cr and Cu in the weld bead and an increase in the HTP decomposition rate in the IMC is supported by the data produced from the XRF.

The corrosion resistance analysis of the soft goods valve materials in the fuel blend yielded only one sample with visible damage. The one sample tested in the pure ignition enhancer solution, thought to be the most corrosive constituent of the fuel blend, at 150 °F showed an increase in thickness and mass, due to swelling of the material. The material is not recommended for use with the fuel blend.

1.0 INTRODUCTION

High-test hydrogen peroxide (HTP) and a hypergolic fuel blend consisting of a hydrocarbon fuel, an ignition enhancer and a transition metal complex catalyst have potential for being used as the constituents in a new hypergolic fuel system. Hypergolic fuel is of particular interest because it ignites spontaneously when it comes in contact with an oxidizer. This makes a more reliable propulsion system, since it eliminates the need for an ignition system, increasing system reliability. Another advantage of a liquid hypergolic system that is not feasible with a solid propellant system is it can be throttled for mid-course corrections. In the past, hypergolic propulsion systems have relied on hydrazine derivative fuels, a highly toxic carcinogen with costly handling procedures. In an effort to avoid these costs, new hypergolic fuels are being developed (6).

HTP is an extremely powerful oxidizer, with concentrations ranging from 70% to 98% hydrogen peroxide, which releases energy and oxygen used for propulsion as it decomposes. Interest in high concentrations of hydrogen peroxide has grown due to the fact it decomposes into water and oxygen. These are two components that are a necessity for everyday life and are not harmful towards the environment. The decomposition reaction is represented by the following chemical equation 1:

$$2\,H_2O_2\,(l) \rightarrow 2\,H_2O\,(l) + O_2\,(g) + \text{Heat} \qquad (1)$$

There are two types of reactions that can occur with HTP: heterogeneous and homogeneous. A heterogeneous reaction is a chemical reaction in which the reactants are components of different phases, in which one or more reactants undergo chemical changes at an interface on the surface of a solid catalyst (4). An example of a heterogeneous reaction is metal reacting with hydrogen peroxide. A homogeneous reaction is a chemical reaction that occurs in a single phase. The chemical changes that take place are solely dependent on the nature of the interactions of the reacting substances (5). An example of a homogenous catalyst is a fuel blend (8).

In order to have an efficient hypergolic fuel system, the HTP must not decompose prematurely, structural materials must not corrode when exposed to the fuel blend, a significant amount of precipitate from the fuel blend must not be formed and an optimization of the percentages of constituents in the fuel blend must be achieved.

1.1 HTP Decomposition

HTP decomposition may be caused by a material incompatibility. Therefore, tests have been conducted to determine the decomposition rate of HTP when exposed to various temperatures and types of metal. Material test methods are used as a starting point in evaluating materials compatible with highly concentrated hydrogen peroxide for use in space launch vehicles (2). The materials are placed in classes, which are determined by the effect hydrogen peroxide has on the material and the effect the material has on hydrogen peroxide. The effect of the material on the peroxide is determined by the rate of decomposition and the stability of hydrogen peroxide. The effect of hydrogen peroxide on the material was determined by observing if corrosion of metals occurred by liquid or vapor and if any signs of hardening, swelling, blistering, deterioration, and dissolving were noticed (3). Any of these signs occurring over a certain length of time determined which class the material was placed in. The materials placed in class 1 are materials considered to be satisfactory for unrestricted use. Storage containers for hydrogen peroxide would be an example of materials considered class 1. Class 2 materials are satisfactory for repeated short-time contact not exceeding four hours at 72°C or one week at 22°C. Valves and pumps used to transfer hydrogen peroxide are examples of this class. Materials used for only short-time contact (not exceeding one minute at 72°C and one hour at 22°C) are classified as class 3 materials. Class 4 materials are materials that are not recommended for use with hydrogen peroxide (3).

Organic fluids such as acetone, alcohols, and hydrocarbon fuels and lubricants are incompatible with HTP. Acetone will form explosive crystals upon evaporation. Also, the following metals, their alloys, and inorganic compounds are incompatible: cadmium, chromium, copper, iron, iron oxide (rust), lead, magnesium, mercury, nickel, platinum, potassium permanganate, silver, titanium, tungsten, and zinc. Common polymers such as nylon and Plexiglas® are known to be incompatible with HTP. Some materials considered to be safe with HTP are glass, pure aluminum, aluminum 5254, aluminum 1060, Teflon®, polypropylene, and stainless steel 300 series (9).

An isothermal microcalorimeter and a gas evolution apparatus were used to determine the thermal decomposition rate of 90% and 98% HTP. The IMC was further used to determine the active oxygen loss of 98% peroxide when it came in contact with the four materials. Tests of these materials were done at room temperature, 25 °C, and later at 60 °C.

The gas evolution apparatus consists of an oven, Teflon tubing, vials, and burets (7). The set up is shown in Figure 1.

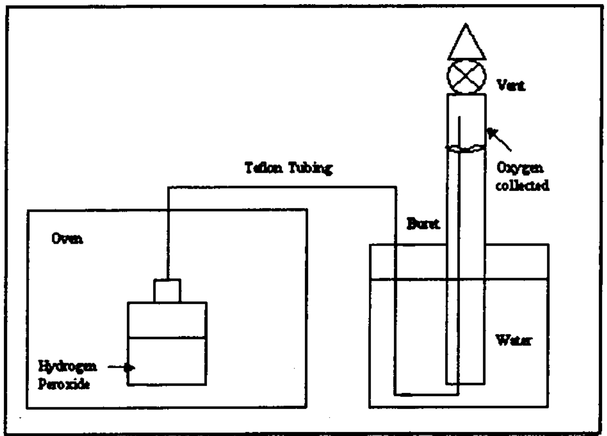


Figure 1. An example of one set up of the Gas Evolution Apparatus.

The concept is to heat the hydrogen peroxide to a desired temperature, and the oxygen released from its decomposition will push out the water of the burets. The displacement of the water shown in the burets may be plotted on a graph. If everything is working correctly the graph of volume displacement vs. time should be linear. Using the volume displacement, the concentration of HTP in the vials is calculated and plotted on a graph. The slope of the graph is the rate of decomposition for hydrogen peroxide in units of reciprocal seconds.

1.2 Effects of Welding on HTP Decomposition

The effect welding has on the chemical composition of the surface of two specific types of stainless steel, CRES 304L and CRES 316L, may also play a role in the decomposition of HTP. Localized changes in the chemical composition could have adverse implications in the corrosion properties of the steel (1). Increases in the elements Cr and Cu would cause the steel to become more reactive with the HTP.

X-ray fluorescence (XRF) can be used to determine the surface composition of the weld bead, the heat-affected zone (HAZ) and non-welded samples of stainless steel. XRF contains a source X-ray that irradiates the sample in the sample chamber, exciting inner shell electrons. As outer shell electrons lower to fill the empty inner shells, fluorescent X-rays are emitted towards the detector. The detected fluorescent X-rays vary by type and count rate depending on the elemental composition of the sample. Thus, an analysis of the composition can be obtained for various samples.

Previous studies of how welding changes the chemical composition of the welded area have been conducted using an electron microscope and have resulted in findings that localized regions of elemental enrichment and depletion exist in CRES 304L and 316L (1).

1.3 SSC Materials Compatibility

Another important investigation of the practicality of the hypergolic fuel blend involves a test of the corrosion resistance of soft goods structural materials. One sample was an O-ring and the remaining materials are being tested for use as other valve components. After exposure to the fuel blend, the initial and final diameters, thicknesses and masses of the samples are compared to draw conclusions about the reliability of these materials in conjunction with the fuel blend. A hardness test is also utilized to further detect corrosion the fuel blend may have caused.

2.0 EXPERIMENTAL METHODS

2.1 HTP Decomposition using IMC

The IMC has been used to test 98% hydrogen peroxide. It contains a hot bath that will keep the peroxide at a certain temperature. The peroxide is poured into a vial and is placed inside the water bath to thermally equilibrate. The vial is then moved to a measurement cell. There are three cells that can be used to test the peroxide; one cell was used as a reference cell, which would contain water. The peroxide is left in the calorimeter for several hours at the desired temperature. The calorimeter then records if any reaction has occurred by monitoring if there is a flow of energy into or out of the cell. The information is then saved on a disk and is placed into a macro program in Excel that was designed by another intern, Genne Nwosisi. The program converts the information from text form to spreadsheet form so that the results can be viewed as a table and graph in the Excel program. The IMC could also test to determine if the peroxide would react with certain materials. After measuring the background energy of the peroxide alone the vial is removed and opened. The sample is then submerged in the peroxide and placed into the calorimeter. The vials are equilibrated before being placed in the measurement cells. If a reaction occurs the calorimeter detects it by a difference in the energy from the results gathered with the peroxide alone with the results gathered when the sample was placed in the peroxide. With the information, the percentage of active oxygen loss (AOL%) can be calculated.

The materials had to be cleaned before they could be tested with 98% HTP. The materials were cleaned to remove any metals and/or organic compounds that could react with HTP. To reduce decomposition of HTP it is necessary to passivate the materials. Passivation consists of treating metals with acids or other chemicals to form an oxide film on the surface of the metal. This film is like a painted surface that serves as a layer between hydrogen peroxide and the metal. This oxide film can prevent attack by the hydrogen peroxide on the metal and reduce the effect of the metal on the decomposition rate of the hydrogen peroxide; as long as the film holds the metal will not corrode (9). In order to test for a worst-case scenario, passivation of the metal samples was not performed for these tests. Therefore, once the samples were cleaned, they were tested with 98% hydrogen peroxide. The following table shows the steps taken when working with the IMC:

Table 1. Procedure used for the IMC

Step	Procedure
1	Filled the vials with 20 mL of 98% HTP, recorded the mass, and capped vials.
2	Placed the vials in the measurement cells and observed the energy release of 98% HTP at 25 °C for approx. one week.
3	Removed the vials, opened the vials, placed the sample (approx. 1.5 x 0.4 x 1/16 in.) in each vial, and recapped.
4	Placed the vials in the measurement cells and observed the energy release of the material submerged in 98% HTP for at 25 °C for approx. one week.

The dimensions of the samples were approximately 1.5 x 0.4 x 1/16 inches so that they would fit in the vials and to keep the surface-to-volume ratio constant due to %AOL dependency.

2.2 HTP Decomposition using Gas Evolution

The initial step was to find a procedure that works for the set up. To do the tests, 35% hydrogen peroxide was used instead of 90% because it is not as hazardous to work with, but still safety precautions must be taken. First, the apparatus was set up and numbered one through six. Before any experiments can begin, all glassware that was going to come in contact with the hydrogen peroxide was cleaned and passivated. Passivation consisted of treating the glass surface with a base and an acid to form an oxide film on the surface of the glassware to reduce the attack of hydrogen peroxide on the glassware and the decomposition rate of the peroxide itself (3). The glassware was passivated first by being soaked in 10% NaOH for 1 hour. It was rinsed, dried and then soaked in 70% HNO_3 for 1 hour, rinsed, and dried. Then finally the glassware was soaked in 30% H_2O_2 for 24 hours, rinsed several times with deionized water, and left to air dry. Next, the burets were filled with water and left for a day to determine if there was a closed seal with the stopcock at the end. The burets were leaking so Silicon grease was used on the stopcocks to create a seal. To assure that no oxygen would be lost between the vial and the Teflon tubing, the vials were capped with the tubing and placed under water. Argon gas was then purged through the other end of the tube to see if bubbles formed where the tubing and the vials met. No bubbles had formed. All the vials were weighed before any hydrogen peroxide was added in them. After many trials and errors to assure the apparatus was working properly with the 35% hydrogen peroxide, a procedure was created for testing the decomposition rate of 90% HTP at 60 °C. The procedure using the gas evolution apparatus was as follows:

Table 2. The procedure used for the gas evolution apparatus.

Step	Procedure
1	Oven was set for desired temperature, 60 °C
2	The Teflon tubing was placed in the burets to where the opening of the tubing was just above the water
3	Burets were filled to a mark somewhere between 0.0 mL and 1.0 mL
4	Filled and weighed vials with 50 mL of 90% hydrogen peroxide
5	Once the oven reached the desired temperature, the vials were capped and placed in the oven
6	The water displacements of the burets were taken every half an hour for a time period of one week.

2.3 XRF to Determine Elemental Composition of Welded Stainless Steel Samples

An X-ray fluorescence (XRF) system was used to quantitatively analyze the elements present in CRES 304L and CRES 316L samples. Sample coupons approximately 0.5" x 1.0" x 0.1" of both types of austenitic stainless steel were welded using tungsten inert gas welding with no filler material. One of each type of welded sample was then oxidized. The weld beads were then separated from the coupons using a diamond-tipped saw, so that the weld bead could be independently analyzed from the HAZ next to the weld bead.

The XRF system consists of an X-ray source, a sample chamber and a detector. The system is controlled by a personal computer that allows the analyses to be optimally calibrated for different types of samples. For the stainless steel samples that were analyzed, the Zc Pd Thick setting was used along with the pure elemental reference spectra and AISI 304 and AISI 316 standards.

Initially, the XRF was set up to run a qualitative analysis, to determine the major elements present in the samples. A method file was set up that analyzed all of the samples using both the AISI 304 and AISI 316 standards. The method file was later modified to two separate method files, where only the 304 or 316 standards were used respectively with the 304 and 316 samples.

2.4 SSC Materials Compatibility Tests

The material compatibility of the soft goods materials in the experimental hypergolic fuel blend and pure ignition enhancer substance was tested by observing changes in size, mass and flexibility. Each of the samples had an initial approximate diameter of 1 in. and thickness of 0.6 in. Four compatibility tests were conducted on each of the five samples: fuel blend at ambient temperature, fuel blend at 150 F, pure ignition enhancer at ambient temperature, and pure ignition enhancer at 150 F. Using a micrometer, the diameter and thickness measurements were obtained for the samples prior to testing. The masses were also obtained for each sample. The samples were placed in 100mL beakers containing 20mL of the fuel blend or pure ignition enhancer substance. The beakers were placed in a constant temperature bath for 1 week. The samples were extracted from the beakers, rinsed with isopropyl alcohol, and allowed to dry before final diameter, thickness and mass measurements were obtained for each sample. The flexibility of the samples was compared to samples that had not been exposed to the fuel substances. Hardness tests were performed on the samples.

Table 3. The procedure used for materials compatibility testing.

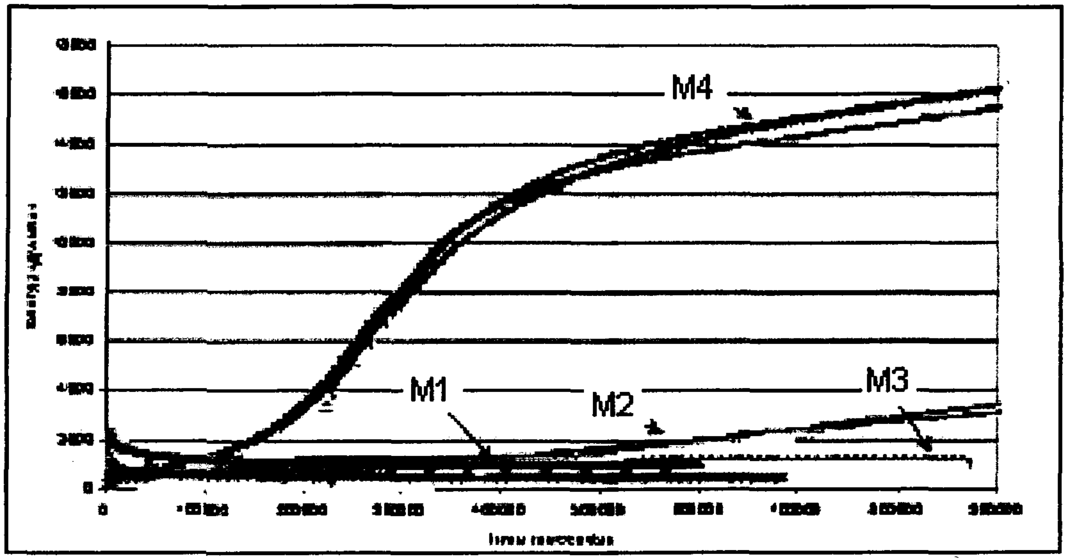
Step	Procedure
1	Diameter, thickness and mass recorded for each of the samples
2	Samples were placed in 100mL beakers with 20mL of fuel blend
3	Beakers were placed in a constant temperature bath for 1 week
4	Filled and weighed vials with 50 mL of 90% hydrogen peroxide
5	Samples were removed and rinsed with iso-propyl alcohol and allowed to dry
6	Diameter, thickness and mass recorded for each of the samples

3.0 RESULTS AND DISCUSSION

3.1 IMC

The data were saved on a disk and put into an Excel Macro program created to compare the background data and the sample data to determine if a reaction occurred. The data were viewed as a graph that showed energy (microwatts) vs. time (seconds). The peak energy of HTP released over a period of time was compared with the peak energy released by the sample submerged in HTP to determine the difference. The peak values were placed in a spreadsheet where the sample reaction rate (1/s), energy of decomposition (microwatt/gram), and the AOL%/week could be calculated. The results are viewed as follows:

Chart 1. This shows a comparison of energy released of all four materials after they have been in exposed to 98% HTP at 25 °C.



From the chart, it is apparent that the M2 and the M4 continued to increase without reaching a constant value. Obviously, the M4 had a greater effect on the peroxide then the M2. The energy released continued to increase indicating the rapid decomposition of HTP due to a reaction with the metal. The M2 does not have as significant of an effect on HTP as the M4. This could be due to the fact that there was an oxide layer on the surface of the metal and once the HTP broke through it, the metal started to decompose the HTP and never came to a constant value. Therefore, both M2 and M4 are not recommended for the apparatus desired. On the other hand, both the M1 and M3 did not show any signs of increase decomposition of HTP, therefore they are recommended for repeated contact with 98% HTP.

3.2 Gas Evolution

The volume displacement of the buret is plotted over the period of time the data was collected. The linearity of the graph shows that the apparatus was working properly.

Chart 2. This graph is of the displacement of water over a period of a week.

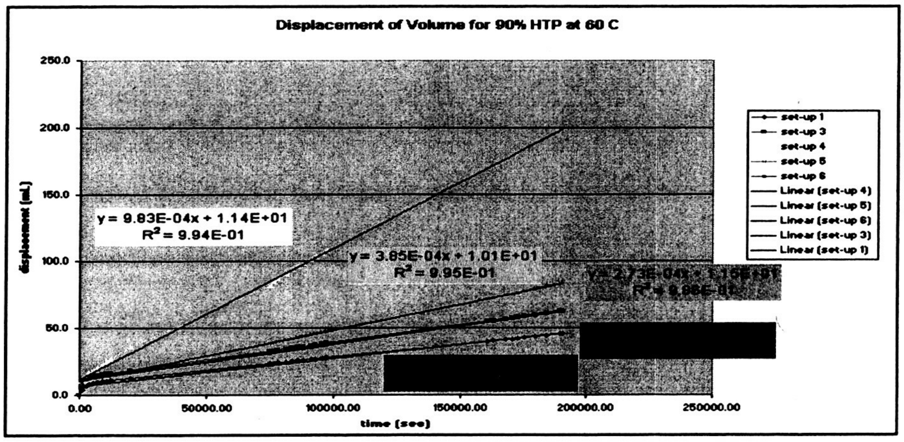


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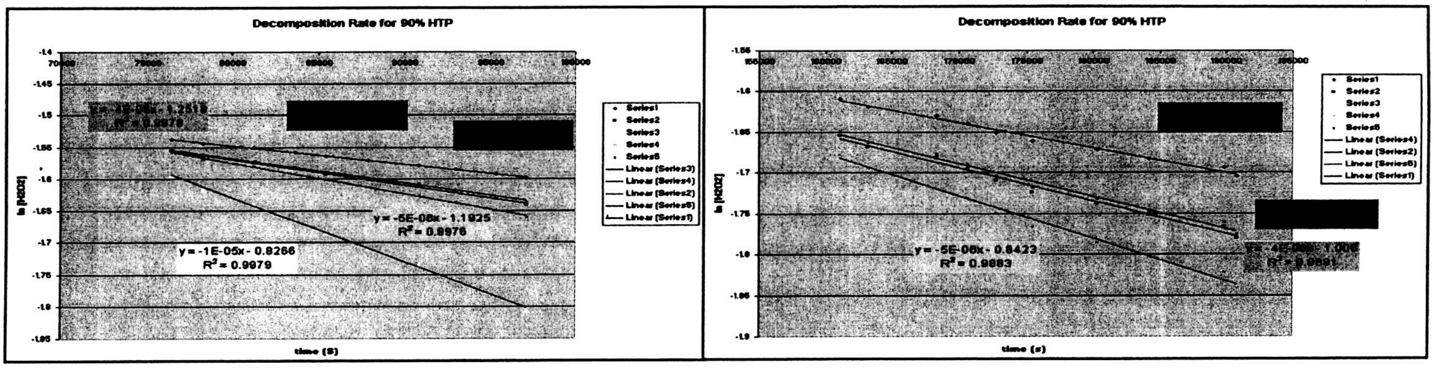
Chart 2 demonstrates the volume of oxygen released over a period of time. The Ideal Gas Law was used to calculate the number of moles of oxygen. Then concentration of the HTP was calculated by using stoichiometry. The log of the concentration of HTP was graphed against time in seconds. The slope of the line for each set up is the decomposition rate of 90% HTP in reciprocal seconds.

Table 4. This is an example of the spreadsheet used to calculate the concentration of HTP.

set-up:	1	3	4	5	6
time increment (sec):	900.00				
volume of oxygen (mL):	3.5	9.5	10.2	9.3	9.4
volume of oxygen (L):	0.0035	0.0095	0.0102	0.0093	0.0094
atmospheric pressure (mb):	998		*atm:*	0.9881	
room temperature (F):	77		*Kelvins:*	298.50	
moles of oxygen:	0.0001412	0.0003832	0.0004115	0.0003752	0.00037919
initial moles of H2O2:	1.8095238	1.8095238	1.8095238	1.8095238	1.80952381
moles of H2O2 reacted:	0.0002824	0.0007665	0.0008229	0.0007503	0.00075839
moles of H2O2 remaining:	1.8092414	1.8087574	1.8087009	1.8087735	1.80876542
volume of hydrogen peroxide:	41.845396	41.8342	41.832894	41.834573	41.8343869
volume of water (L):	5.088E-06	1.381E-05	1.483E-05	1.352E-05	1.3666E-05
new volume (L):	8.1546087	8.1658135	8.1671208	8.16544	8.16562679
concentration of H2O2 (mol/L)	0.2218673	0.2215036	0.2214613	0.2215157	0.22150969
ln [H2O2]:	-1.505676	-1.507316	-1.5075076	-1.507262	-1.507289

Charts 3a, b. These charts show the decomposition rate of 90% HTP. The graph on the left (a) shows readings from the first half of the week. The graph on the right (b) shows the readings from the second half of the week

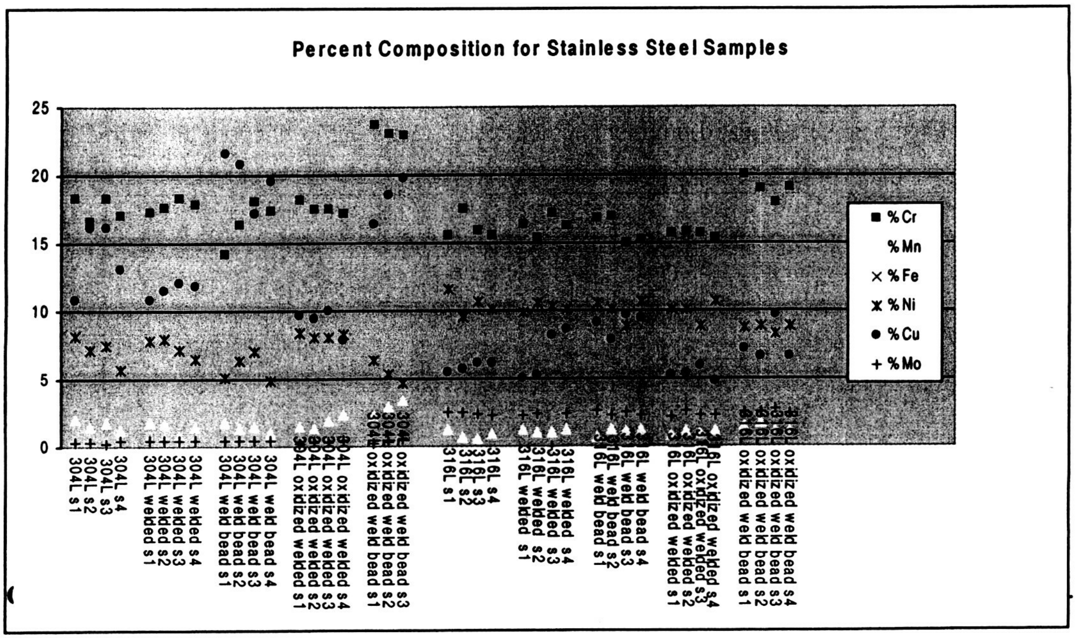


According to the slopes of the lines, the decomposition rate of 90% HTP ranged from 3 x 10^{-6} 1/s and 5 x 10^{-6} 1/s. The data for set up three was not taken into consideration for the final results. It had such a high decomposition rate that the data could not be taken throughout the whole week. This could be due to the fact that the vial was contaminated.

3.3 XFR Welded Samples Analysis

Welding stainless steel used for structural material may lead to HTP decomposition, due to an increase in the amount of Cr and Cu in localized areas of the welded material. The results of the XRF analysis show that there is an increase of Cr and Cu in the weld beads.

Chart 4. This chart shows each analysis of each sample. The sample position was changed for each run, which cause the percent compositions to vary for a given sample. An increase in Cr can be seen for the oxidized weld beads in each type of metal, and an increase in Cu can be seen for each of the weld beads, although it is more pronounced for CRES 304L.



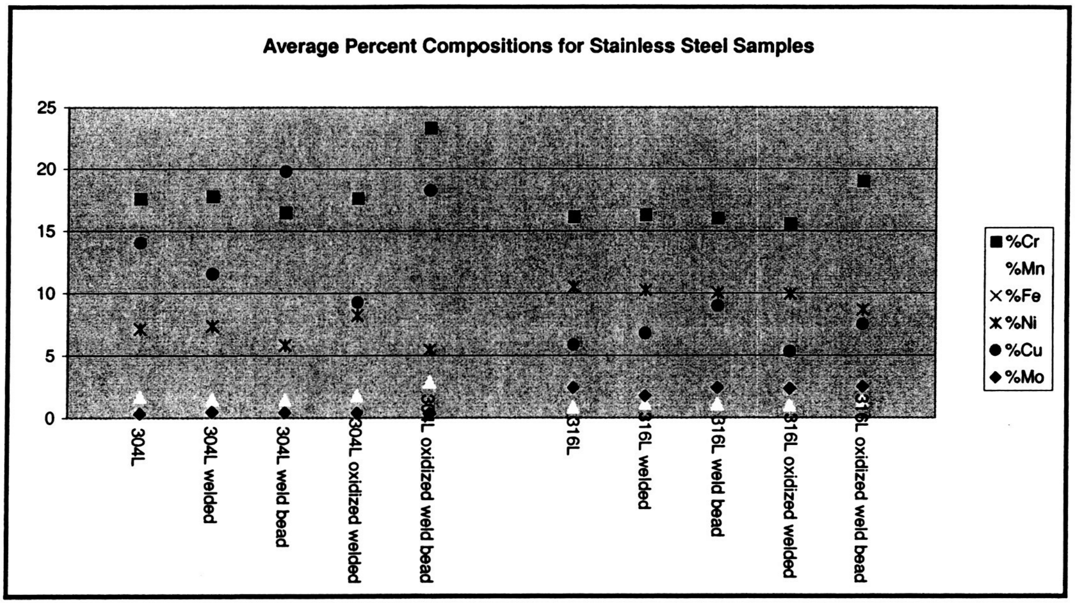
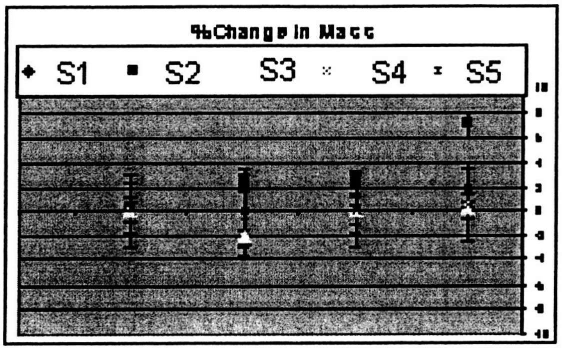


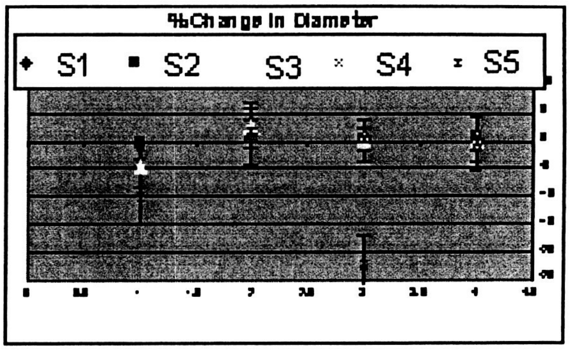
Table 5. Data from the IMC shows that there is an increase in HTP decomposition for the welded sample, especially the samples that were welded without purge gas. The CRES 304L samples show a greater increase in HTP decomposition, which supports the findings from the XRF of greater Cr and Cr regions.

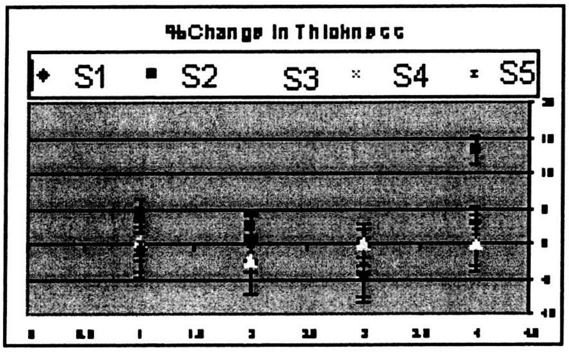
MATERIAL	MAX % AOL/wk (90% confidence)
CRES 304L	27.87 ± 1.56
CRES 304L welded	68.20 ± 11.46
CRES 304L welded (no purge gas)	94.47 ± 3.06
CRES 316L	5.87 ± 0.52
CRES 316L welded	34.03 ± 10.04
CRES 316L welded (no purge gas)	36.13 ± 4.08

3.4 SSC Materials Compatibility

The percent changes in mass, thickness, and diameter are shown in Charts 6 a-c for the different test conditions. The error bars were obtained from the standard deviation of the initial measurements for the type of material assuming that all of the initial samples had the same dimensions. The only significant variance was the S5 sample in pure ignition enhancer at 150° F. This sample showed visible corrosion and had an increase in mass and thickness, but no significant change in diameter. The inconsistency of the S4 percent change in diameter value in the ignition enhancer at ambient temperatures is attributed to the irregular shape of the S4 samples. Thus, the diameter measurements were inconclusive.







Charts 6 a,b,c. The percent change is shown for each sample type in four different fuel blend conditions. In each of the three charts, from left to right the conditions are: fuel blend at ambient, fuel blend at 150F, ignition enhancer at ambient, ignition enhancer at 150F.

A hardness test was also performed on the samples. The results from this test show that there was little variation in the hardness of the samples for the different test conditions.

Table 6. The hardness test results show little variation for any of the materials.

	Control	Enhancer 150	150F	Ambient	Enhancer Ambie
S1	61	62	62	62	60
S2	90	85	88	89	87
S3	84	87	84	87	87
S4	87	88	88	86	86

CONCLUSIONS

Out of the four materials tested with 98% HTP at 60 °C, both the M2 and the M4 are not recommended for the apparatus desired. On the other hand, both the M1 and M3 did not show any signs of increase decomposition of HTP, therefore they are recommended for repeated contact with 98% HTP. Also, according to the gas evolution apparatus, the decomposition rate of 90% HTP at 60 °C ranged from 3×10^{-6} 1/s and 5×10^{-6} 1/s.

Welding causes composition changes in the steel, which can lead to HTP decomposition. The increases in Cr and Cu in the CRES 304L and CRES 316L samples are present in the same types of samples that had the higher HTP decomposition rates in the IMC. The oxidized weld beads were the samples that showed the greatest Cr and Cu enhancement.

From materials compatibility studies of materials in the fuel blend, S2 is not recommended for use. However, S1, S3, S4 and S5 are recommended for use as valve materials with this fuel system.

Future testing of this hypergolic fuel could include precipitate testing and ignition delay testing. The precipitate of the fuel blend is an important factor when considering the practicality of the experimental fuel system. Precipitate studies are used to determine the tendency of the catalyst to precipitate out of the fuel blend, creating potential for disturbing the consistency of concentration and clogging the fuel system. A high-speed centrifuge and an analytical balance can be used to determine the amount of precipitate formed for a given blend of hypergolic fuel. Ignition delay testing is used to quantitatively determine the effectiveness of the fuel blend as a fast-reacting chemical propellant.

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