Aluminum-Scandium Alloys: Material Characterization, Friction Stir Welding, and Compatibility With Hydrogen Peroxide

(MSFC Center Director's Discretionary Fund Final Report, Project No. 04–13)

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LIST OF ACRONYMS AND SYMBOLS

Al	aluminum
AOL	active oxygen loss
CDDF	Center Director's Discretionary Fund
Cr	chromium
Cu	copper
FSW	friction stir welding
HAZ	heat-affected zone
HNO_3	nitric acid
H_2O_2	hydrogen peroxide
Hyper-X	Hypersonic-X
IMC	isothermal microcalorimeter
ISTAR	integrated system test of an air-breathing rocket
Mg	magnesium
Mn	manganese
MSFC	Marshall Space Flight Center
NaOH	sodium hydroxide
O_2	oxygen
RBCC	rocket-based combined cycle
Sc	scandium
Ti	titanium
TM	Technical Memorandum
Zn	zinc
Zr	zirconium

NOMENCLATURE

C initial fraction H_2O_2

C_1 initial concentration (H_2O_2 stability)

d_1 density at initial temperature

W_1 initial net weight (active oxygen loss); initial flask weight (H_2O_2 stability)

W_2 final net weight (active oxygen loss); final flask weight (H_2O_2 stability)

TECHNICAL MEMORANDUM

**ALUMINUM-SCANDIUM ALLOYS: MATERIAL CHARACTERIZATION,
FRICTION STIR WELDING, AND COMPATIBILITY WITH HYDROGEN PEROXIDE**
(MSFC Center Director's Discretionary Fund Final Report, Project No. 04–13)

1. INTRODUCTION

This Technical Memorandum (TM) describes the development of high-strength aluminum (Al) alloys for fuel tanks and air frame applications that are chemically compatible with hydrogen peroxide (H_2O_2) propellant. These new high-strength alloys could represent an enabling material technology for the development of NASA's next generation of Hypersonic-X (Hyper-X) vehicles, where flight weight reduction is a critical requirement. These Hyper-X vehicles are being studied as air-breathing hypersonic research vehicles that feature a lifting body configuration with a rocket based combined cycle (RBCC) propulsion system.[1] This task supports three main Engineering Directorate's thrust areas at Marshall Space Flight Center (MSFC): (1) Advanced cryogenic tank area for materials' compatibility with H_2O_2 systems, including metallic liners and permeation barriers, (2) advanced structures and materials area for lightweight and high specific strength Al alloys, and (3) advanced manufacturing in the space transportation area for welding of advanced Al alloys compatible with H_2O_2 propellant.

As shown in figure 1, this work directly supports the product line for NASA's Hyper-X flight test air-breathing vehicles such as the X–43B, which is an integrated system test of an air-breathing rocket (ISTAR) flight demonstrator.[2] The flight-type ground test engine sized specifically for the X–43B uses JP–7 fuel and 90 percent concentrated liquid H_2O_2 as an oxidizer. When highly concentrated H_2O_2 is allowed to decompose in the combustion chamber, the H_2O_2 rapidly decomposes into steam (water) and oxygen (O_2) and releases a large amount of heat that can be used as an oxidizer in a bipropellant mode, for thrust in monopropellant mode, and as the working fluid for pressurization in a turbine drive. Currently, conventional 1060 and 5254 materials are the baseline Al alloys for H_2O_2 fuel tanks and integral structures, but their yield strengths are relatively low at 17 ksi. Being non-heat-treatable alloys, these materials may not be suitable for advanced lightweight tanks or airframes due to low specific strengths.
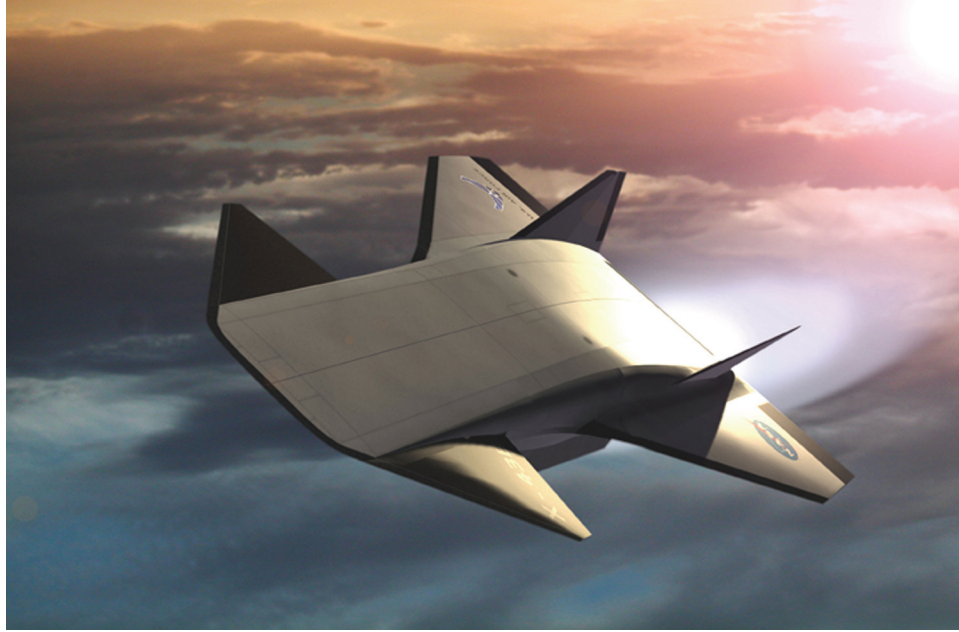
Figure 1. Artist rendering of the air breathing X–43B Hyper-X vehicle.

1.1 Hydrogen Peroxide Impact on Design of Propulsion Systems and Storage Tanks

H_2O_2 has a water-like appearance but is very chemically active as an oxidizer, and is highly soluble in water at all proportions; the usual commercial forms are from 3 to 30 percent aqueous solution. For rocket propulsion applications, high concentrations of H_2O_2 solutions of >70 percent are used since they are stable at room temperature. When properly contained in chemically compatible vessels, H_2O_2 has a very weak decomposition rate. A concentration of 90 percent grade H_2O_2 was selected for the X–43B vehicle, with an active O_2 content of 42.3 percent. Table 1 shows some selected physical properties for highly concentrated H_2O_2. When highly concentrated H_2O_2 comes in contact with incompatible materials like heavy metals or various organic compounds, or mixes with certain impurities, it produces O_2 gas and decomposition heat. The decomposition rate increases about 2.2 times for each 10 °C increase in temperature from 20 to 100 °C. Table 2 shows the effect of heat on the decomposition rate. At near room temperature, the rate of decomposition is ≈1 percent per year. This rate increases exponentially to 1 percent per week if the temperature is allowed to rise to 66 °C (151 °F), and could result in a rapid pressure increase in a closed system.

Indications of H_2O_2 decomposition are: pressure buildup by activation of pressure relief valves, visible rapid bubbling, temperature increase, gas or steam evolution, and possible explosion when pressure relief is inadequate. Pressure buildup will occur very rapidly in a closed system and excessive pressure buildup can result in ruptured storage tanks, fuel tanks, or feed lines. Using H_2O_2 could have an impact on propulsion system and storage tank designs. Figure 2 shows the X–43B vehicle configuration layout for the H_2O_2 tank and the JP–7 fuel tank.

Table 1. Selected physical properties of highly concentrated H_2O_2.

Properties	H_2O_2 Concentration (wt. %)		
	70%	90%	99%
Active oxygen (%)	32.9	42.3	46.59
Specific gravity @ 25 °C	1.28	1.38	1.43
Boiling point (°C)	125.4	141.2	149
Freezing point (°C)	−40.3	−11.5	−1.5
Flash point (°C)	None	82–85	73.9
Autoignition in air (°C)	316	169	122
Vapor pressure (mmHg)	10.1	5	2.8

Table 2. Effect of heat on decomposition rate of H_2O_2.

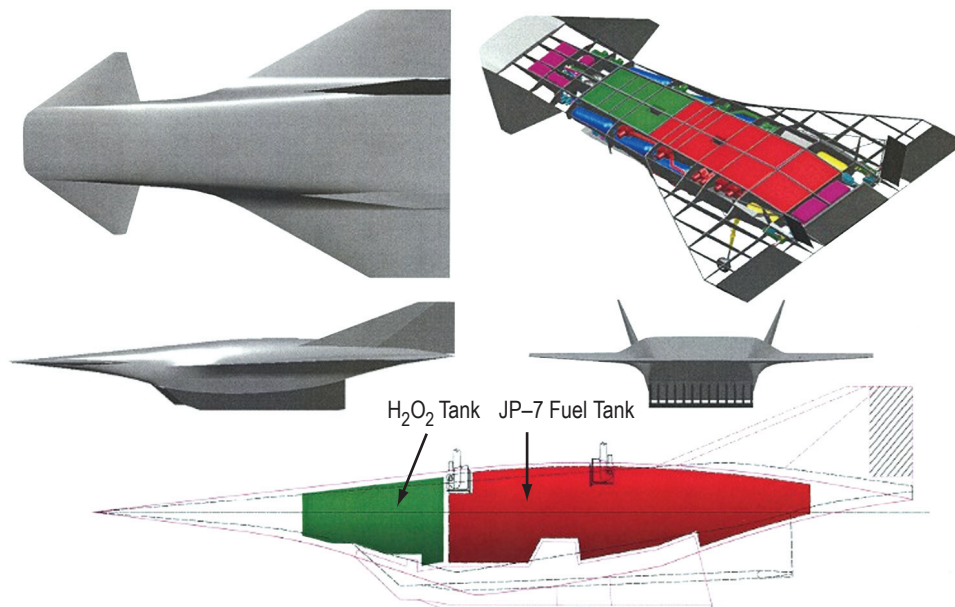| Effect of Heat on Decomposition		
Temperature		Rate of Decomposition
°C	°F	
22	72	1% per year
66	151	1% per week
103	218	2% per day

Figure 2. X–43B configuration layout for the H_2O_2 and JP–7 fuel tank.

Using any H_2O_2 system would affect the design of certain types of Hyper-X vehicle's propulsion system, such as onboard fuel tanks, valves, and pumps, which must be pacified to enhance chemical compatibility. All valves must be compatible and have relief capability with no isolated segments in the feed system to trap any H_2O_2. H_2O_2 is not an intrinsically hazardous fluid, but it must be contained in a pacification system that is well engineered to remove excessive heat and pressure from the storage and feed system due to decomposition and high-temperature effects. Pacification of storage tanks and feed lines is usually accomplished through various acid baths and exposure to varying degrees of H_2O_2. An H_2O_2-based system will impact overall system complexity because of feed system architecture.

1.2 Classifications for Material Compatibility With Hydrogen Peroxide

According to FMC corporation's technical data from *Bulletin 104*, materials should be classified into categories based on their contemplated types of use. Therefore, all materials are not required to be suitable for indefinite storage, because in applications requiring only short-time contact with H_2O_2, materials with a lesser degree of compatibility can be employed. To facilitate the material selection on this basis, FMC has developed four categories of material classifications as follows:[3]

• Class 1: Unlimited use materials—can be used for long-term contact with H_2O_2 as storage containers.

• Class 2: Repeated use in short-time contact materials—can be used for either transient contact with the H_2O_2 prior to storage or limited contact prior to use. Such contact is not to exceed 4 hr at 72 °C (160 °F) or 1 wk at 22 °C (70 °F)—typically used for valves and pumps in transfer lines and feed tanks.

• Class 3: Short-time contact materials—can be used for repeated contact, but no single contact period should exceed 1 min at 72 °C (160 °F), or 1 hr at 22 °C (70 °F), because sufficient contamination of the H_2O_2 may render it unsuitable for storage. Many class 3 materials indicate satisfactory service at room temperature. However, the material should be pretested prior to use.

• Class 4: Not recommended materials—will cause excessive decomposition of H_2O_2 even on short-time contact and yield corrosion or deterioration on products to form an impact-sensitive mixture with concentrated H_2O_2.

2. EXPERIMENTAL

2.1 Alloy Development

The current baseline material recommended for long-term storage of highly concentrated class 1 (>90 percent) H_2O_2, is 5254 Al alloy, which is wrought and non-heat-treatable Al-magnesium (Mg) alloy. The 5000 series Al-Mg alloys are good candidate materials for structural applications due to their low density, reasonably good weld ability, and excellent corrosion resistance. However, similar to most Al-Mg systems, the 5254 alloy does not have adequate specific strength and density for advanced aerospace applications. Table 3 shows the compatibility assessment of selected alloys for H_2O_2 storage tank applications. As a general rule, the higher strength alloys tend to be incompatible with H_2O_2 for long-term storage applications.

Table 3. Compatibility assessment of selected alloys for H_2O_2 storage tanks.

Current Materials for Tankage	Temper	Yield Strength (ksi)	H_2O_2 Compatibility Class
1060	H14	13	1
1100	H14	17	1
2024	T6	57	3
Al–Li 2195	T81	66	2
2219	T81	50	3
5254	H112	17	1
6061	T6	40	2
7075	T6	73	4

The initial goal was to develop a new experimental Al-Mg-based alloy that has the same class 1 compatibility rating with H_2O_2, but with a significant improvement in yield strength by a factor of 2–3 times more than 5254. This strategy was designed to modify the chemistry of 5254 alloy by adding scandium (Sc) and zirconium (Zr), which was not perceived to act as catalysts to decompose liquid H_2O_2. Fine Al_3Sc precipitates are known to be coherent with the Al matrix and are expected to contribute to the alloy strength through dislocation-particle interactions.[4] Scandium also effectively increases the recrystallization temperature for Al. However, simultaneous addition of Sc and Zr has been shown to synergistically promote much higher strengths than either Sc or Zr additions produce alone.[5,6] Figure 3 shows the synergistic strengthening effect of Sc and Zr additions for Al.
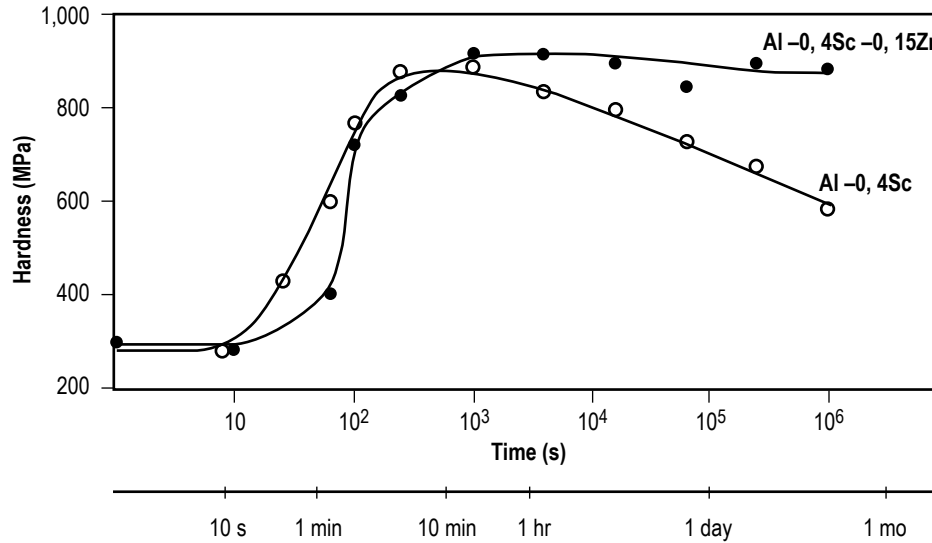
Figure 3. Synergistic strengthening effect of Sc and Zr additions.

Analyses for the catalytic effects of Sc, Zr, copper (Cu), zinc (Zn), manganese (Mn), titanium (Ti), and Mg additions to the Al matrix have shown that these elements may yield only a minor effect on the rate of decomposition for H_2O_2 if they are added in relatively small amounts without surpassing their maximum solid solution in the Al matrix composition. Such elements have been added to several experimental Al-Mg alloys in appropriate amounts. Ingots were cast and rolled into thin sheet metals and test coupons were machined from sheet metals for long-term exposure and mechanical properties testing with H_2O_2 (fig. 4). In addition, the ability to weld the new alloys using friction stir welding (FSW) has also been explored.

Figure 4. Typical microstructures for experimental alloys: (a) cast
and (b) cold rolled at × 200 magnification.

2.2 Candidate Materials

Table 4 shows the chemical compositions of four candidate Al-Mg-Sc-bearing alloys, including the 5254 alloy as a baseline reference material. The rationale for selecting these candidate materials is explained as follows:

• Successfully developed a new experimental Al-Mg-chromium- (Cr-) based alloy named RX 5000 by adding Sc and Zr.

• Strengthened RX 5000 by Mg atoms in solid solution and by cold working in the H112 conditions, which increased yield strength to 42 ksi—nearly 2.5 times stronger than the 17 ksi yield strength of conventional 5254 alloy.

• RX 5000 is completely compatible with class 1, 90 percent H_2O_2.

Table 4. Chemical compositions (wt. %) of candidate alloys.

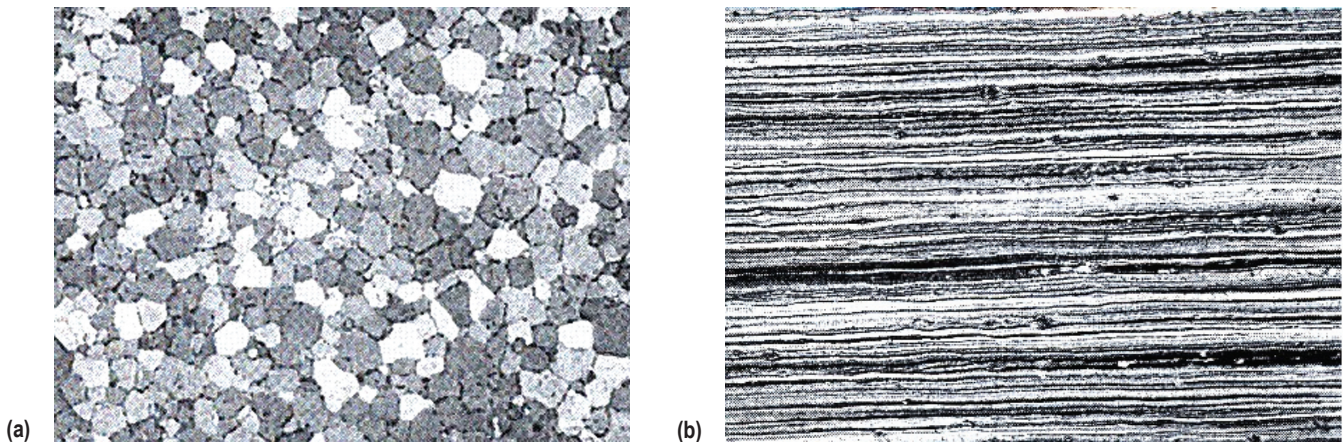| Alloy	Chemical Composition (wt. %)										
	Mg	Zn	Cr	Mn	Cu	Sc	Zr	Ti	Si	Fe	Al
5254	3.5	0.2	0.25	0.01	0.05	–	–	0.05	0.25	0.2	Balance
RX 5000	5.1	0.01	0.35	0.01	0.01	0.26	0.12	0.01	0.08	0.08	Balance
C557	4.02	0.015	–	0.62	0.003	0.24	0.096	0.023	0.062	0.095	Balance
7X0X	2.16	5.13	–	0.19	–	0.13	0.19	0.03	–	–	Balance
7X11	2.12	5.27	–	0.19	0.29	0.14	0.21	0.04	–	–	Balance

A search for an existing alloy with similar composition to RX 5000 in commercial production yielded the C557 alloy, which is Al-Mg-Sc-Zr with a slightly higher Mn content than the RX 5000 alloy, without Cr. The chemistry of the C577 rolled sheet is very similar to the Russian alloy 1535 (table 4). The C557 alloy was produced by Alcoa® for commercial scale ingots with the H116 heat treatment to ensure good stress corrosion resistance. Several C577 test panels were later received from Alcoa for FSW and H_2O_2 compatibility testing at MSFC.

The characterization of Sc-bearing Al-Mg alloy C577 for aerospace applications was previously documented in the literature for strength-toughness behaviors.[7] As previously stated, the C557 is also completely compatible with class 1, 90 percent H_2O_2.

Because RX 5000 and C557 alloys are non-heat-treatable, further attempts to increase strength would be limited without further development and/or characterization of heat-treatable Al-Mg-Sc-Zr alloys with the additions of either Zn and/or Cu. Surface Treatment Technologies has characterized two heat-treatable alloys named 7X0X and 7X11 (T. Langan, Presentation at Technical Interchange Meeting, 2003). These two heat treatable alloys are completely compatible with class 1, 90 percent H_2O_2.

3. RESULTS AND DISCUSSION

3.1 Mechanical Properties

Table 5 shows comparisons of typical mechanical properties of four candidate Al-Mg-Zr-Sc-bearing alloys, and displays the properties for conventional 5254 alloy as the baseline reference material.[8] 7X0X and 7X11 wrought, heat-treatable alloys are more superior in tensile and yield strengths than the 5254 alloy by a large margin; followed by a moderate increase in strength from non-heat-treatable alloys, C557 and RX 5000. An important point of reference is that the yield strength and apparent fracture toughness values of C577 are within 10 percent of established values for 2024-T3 sheet.[7]

Table 5. Typical mechanical properties of candidate alloys.

Candidate Alloy	Temper	Yield Strength (ksi)	Tensile Strength (ksi)	Elongation (%)	Hardness (HRB)
7X11	T4	62.6	83.3	14	78.4
	T6	82.3	86.8	9.1	88
	T7	66	72	8.6	83.3
7X0X	T4	61.8	80.4	12.2	78.3
	T6	78.5	84.1	8.2	86.7
	T7	65.6	70.7	10	81.9
C557	H116	47.5	62	12	66
RX 5000	H112	42	55	15	63
	0	40	52	17	60
5254	H112	17	35	25	63
	0	17	35	27	58

On the other hand, C557 and RX 5000 are inherently more corrosion resistant than 7X0X and 7X11 because they do not contain secondary strengthening precipitates from Mg, Zn, and Cu.

Comparisons between 7X0X and 7X11 show a minor increase in strength for the 7X11 alloy, due to the addition of Cu. With the T6 condition, the 7X11's yield strength is ≈4.8 times higher than the conventional 5254. Since yield and tensile strength are important basic material design properties, figure 5 shows the yield and tensile strengths of all four candidate materials as compared to 5254 baseline alloy.

3.2 Chemical Compatibility With Hydrogen Peroxide

FMC personnel conducted chemical compatibility tests for each type of alloy at 66 °C for 7 days in a water bath with controlled temperature, and each set of coupons ran in duplication. The approximate surface area of each coupon is ≈1.75 in^2. About 86 mL of H_2O_2 was used for each run, which made the surface area-to-H_2O_2 volume ratio roughly equal 0.33 in^{-1}. This coupon was immersed in 0.25 percent sodium hydroxide (NaOH) for 20 min and then immersed in 45 percent nitric acid (HNO_3) for 1 hr,

and finally conditioned in 35 percent H_2O_2 for 24 hr before the compatibility test in 90 percent H_2O_2. Appendix A shows the procedure used by FMC for determining the compatibility of materials with H_2O_2 by immersion in liquid and vapor at controlled temperatures. Appendix B shows the procedure for determining the stability of H_2O_2. Figure 6(a) shows test coupons immersed in a typical test vial that contains 90 percent H_2O_2. Figure 6(b) shows the isothermal microcalorimeter (IMC), which is another method used to determine decomposition rate. The IMC determines the difference between the background thermal energy of the H_2O_2 before and after the sample is added in the vial. The IMC was used at MSFC to determine the active oxygen loss (AOL) percent of the H_2O_2.
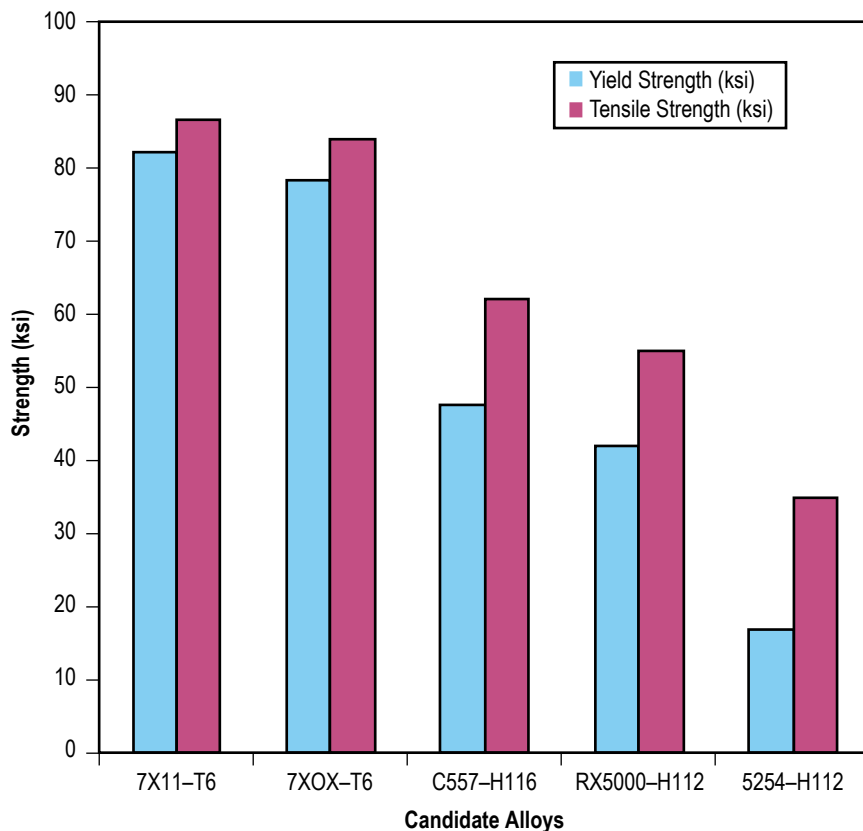
Figure 5. Strengths of candidate materials compared to 5254 baseline alloy.

The results of the H_2O_2 compatibility test are shown in table 6, which was performed using procedures outlined by FMC in appendices A and B. Four types of Al-Mg-Sc-bearing alloys were tested, plus a fifth type of sample C577 that was taken from an FSW joint. For each type of material, two coupons were required to conduct the test. A blank control test was also run in parallel for each type of alloy for monitoring the system contributions to the results. Changes in weight and concentration of the H_2O_2 solution were measured in the presence of coupons to calculate relative AOL percent. In table 6, the ΔAOL percent represents the measured difference between the sample's AOL and the blank's AOL, which displays the true effect of the coupon on H_2O_2 decomposition.

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Figure 6. (a) Vial contains 90 percent grade H_2O_2 and test coupons, and (b) isothermal microcalorimeter is used to determine the H_2O_2 decomposition rate.

Table 6. H_2O_2 compatibility assessment of current class 1 materials.

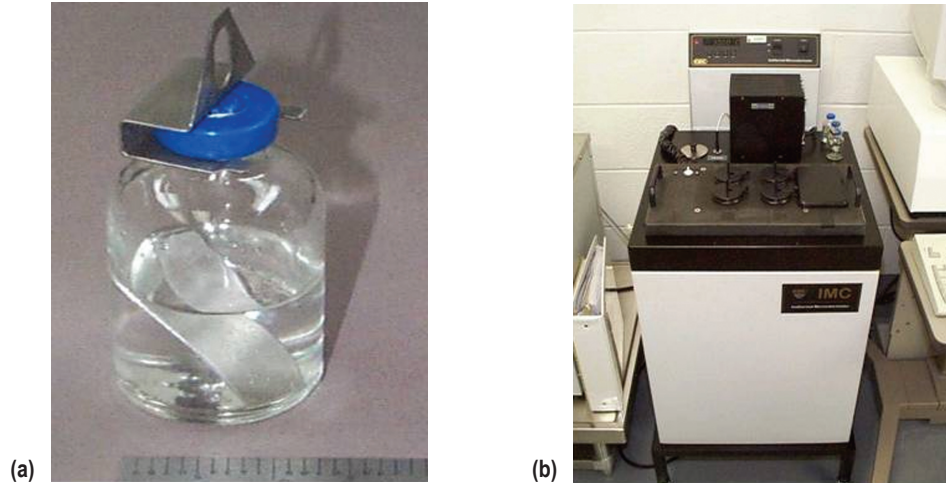| Run No.	Coupon ID	H_2O_2 Start Weight (g)	H_2O_2 Weight Loss (g)	Final Assay (%)	AOL (g)	AOL (%)	ΔAOL (%)	Coupon Weight Loss (g)	Stability (%)
G0380–138–1	Blank–1	119.48	0.036	90.21	0.097	0.192	–	–	99.65
G0380–138–2	Blank–2	119.85	0.034	90.21	0.097	0.19	–	–	
G0380–138–3	RX 5000–1	119.71	0.053	90.2	0.115	0.226	0.035	0	99.16
G0380–138–4	RX 5000–2	119.99	−0.214	89.9	0.166	0.326	0.135	0	
G0380–138–5	C557–1	119.64	0.444	89.87	0.464	0.913	0.722	0.0001	98.24
G0380–138–6	C557–2	120.44	0.427	89.9	0.438	0.856	0.665	0.0002	
G0380–146–1	Blank–3	120.82	0.156	89.9	0.268	0.524	–	–	99.79
G0380–146–2	Blank–4	122.34	0.058	89.94	0.209	0.403	–	–	
G0380–146–3	7X0X–1	119.29	0.073	90.05	0.151	0.297	−0.248	−0.0001	99.51
G0380–146–4	7X0X–2	119.88	−0.009	89.91	0.196	0.384	−0.161	−0.0001	
G0376–20–1	Blank–5	119.7	−0.5	90.23	0.113	0.222	–	–	99.62
G0376–20–2	Blank–6	120	0	90.67	0.08	0.157	–	–	
G0376–20–3	7X11–1	119.4	0.1	90.58	0.173	0.34	0.151	−0.0003	99.29
G0376–20–4	7X11–2	121.1	0.1	90.58	0.175	0.339	0.15	−0.0004	
G0376–20–5	FSW C557–1	120.9	0.1	90.6	0.165	0.319	0.13	−0.0003	99.57
G0376–20–6	FSW C557–2	119.6	0	90.56	0.141	0.277	0.088	−0.0003	

Each candidate material has shown a stability value that is similar to the value from a blank test run >99.6 percent, which did not have any coupons in them. This is the baseline for the test results because H_2O_2 solutions may be slightly different from batch to batch, depending on the containers and varying temperature control from test to test. Blank samples served as references to eliminate all the noises. The results indicate that all candidate materials can be classified as class 1. FSW samples were also extracted from the welding surface area and tested for compatibility. The data also showed that an FSW joint has a stability of class 1, 99.57 percent. These data confirm the notion that FSW has

a potential for welding Al-Sc-bearing alloys with minimal material contamination that could affect the compatibility with H_2O_2 in the weld joint area.

3.3 Friction Stir Welding

FSW is a relatively new joining process that was originally developed and patented by the Welding Institute of Cambridge, England.[9] Since 1993, FSW has been studied and demonstrated by researchers for Al alloys and metal matrix composites.[10] FSW can be best described in conventional terms as a combination of extrusion and forging of metals at elevated temperatures. This process is considered a solid state process and does not require the need for gas shielding or filler metals. FSW consists of a rotating, nonconsumable pin tool that is slowly plunged into the bond line until the pin tool's shoulder is in intimate contact with the work piece. As the tool rotates and moves forward along the bond line, the material on the bond line begins to heat and is forced to flow around the rotating tip to consolidate on the pin tool's backside. This heat source is developed mainly because of local friction and plastic deformation while keeping the pin tool's shoulder in intimate contact with the work piece at all times. Figure 7 shows FSW and the pin tool's geometric design.

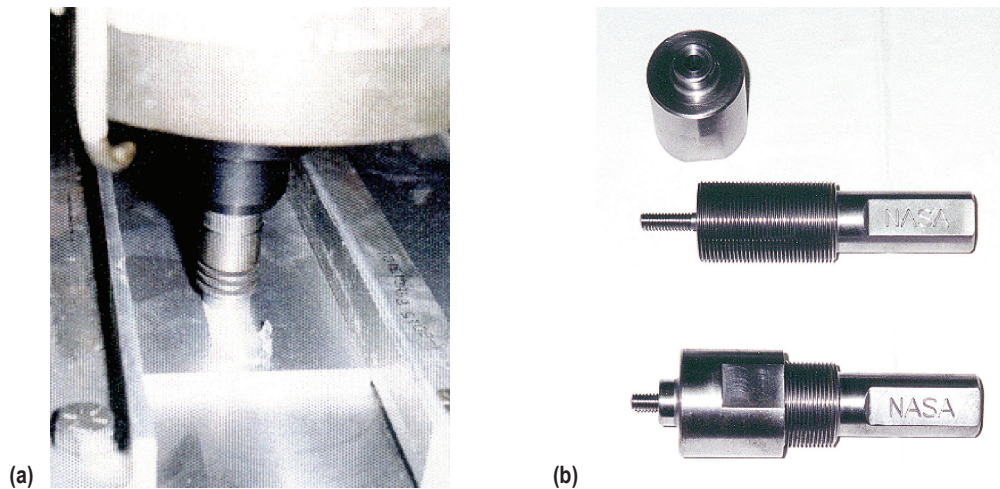(a)   (b)

Figure 7. (a) FSW and (b) the pin tool's geometric design.

Interestingly, FSW has the potential for welding Al-Sc alloys because the processing temperature occurs well below the metal's melting point, thereby eliminating the undesirable chemical reactions, which could reduce material contaminations that may affect the compatibility with H_2O_2 in the weld joint area. Figure 8 shows a cross-sectional view of an FSW joint for C557 alloy. The hardness measurement was taken across the crown side of the weld zone as shown in figure 9. The yield and tensile strengths were slightly reduced in the heat-affected zone (HAZ) by overheating from the FSW process. However, the FSW strength efficiency of >90 percent is excellent when compared with relatively high-temperature fusion joining processes such as arc welding. Table 7 shows the FSW tensile strength and joint efficiency for candidate Al-Mg-Sc-bearing alloys. Because the FSW process does not require gas shielding or filler metals, welding joints have less contamination than arc welding (table 8).
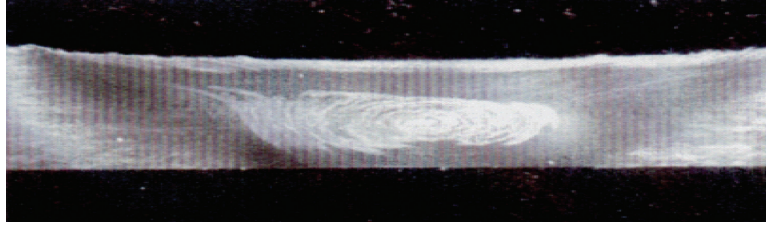
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Figure 8. Cross-sectional view of an FSW joint for C557 alloy.
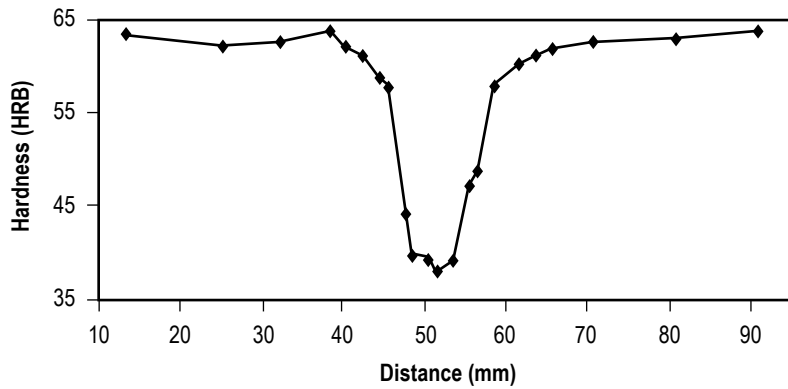
Figure 9. HAZ across an FSW joint for C557 alloy.

Table 7. FSW tensile strength and joint efficiency.

Candidate Alloy	Temper	Weld Joint Ultimate Tensile Strength (ksi)	Joint Efficiency (Weld Ultimate Tensile Strength/Base Metal Ultimate Tensile Strength) (%)
7X11	T7	66.2	91.9
7X0X	T7	63.6	90
C557	H116	53.4	86
RX 5000	H112	48.6	88.3

Table 8. FSW results in low contamination at the welding joint.

Alloy	H_2O_2 Stability After 7 Days @ 66 °C (%)	Compatibility Classification
No sample	99.65	Reference
FSW C557 joint	99.57	Class 1

4. SUMMARY AND CONCLUSIONS

The purpose of this work was to develop a new Al-Mg-based alloy that has the same class 1 compatibility rating with H_2O_2, similar to conventional 5254 alloy, but with a significant improvement in yield strength. Several Al-Mg-Sc-based alloys have been successfully developed and/or characterized and tested for H_2O_2 compatibility. Analysis for the catalytic effects of Sc, Zr, Cu, Zn, Mn, Ti, and Mg additions to the Al matrix has shown that these elements may yield only a minor effect on the decompose rate of H_2O_2, provided that they are added in relatively small amounts, without surpassing their maximum solid solution in the Al matrix.

The preliminary test data indicate that all of these alloys are chemically stable (inert) when exposed to 90 percent H_2O_2. With the T6 condition, the 7X11 heat-treatable alloy's yield strength is about 4.8 times higher than the conventional 5254 yield strength, while maintaining excellent H_2O_2 compatibility similar to the class 1 5254 alloy. Moreover, test data show that these alloys can be welded successfully using FSW.

APPENDIX A—PROCEDURE FOR DETERMINING THE COMPATIBILITY OF MATERIALS WITH HYDROGEN PEROXIDE BY IMMERSION IN LIQUID AND VAPOR AT CONTROLLED TEMPERATURES

A.1 Introduction

Compatibility testing and evaluation of materials with H_2O_2 is conducted by immersing material samples either in H_2O_2 for 4 wk at 30 °C (86 °F), if it cannot be tested at higher temperature, or 1 wk at 66 °C (150.8 °F). The percent loss of active O_2 is calculated from the decreased weight of oxide concentration and the decreased weight of the system. The AOL, together with an appraisal of the condition of the tested material, is used to govern the selection and recommendation of suitable materials for use with H_2O_2.

A.2 Sample Size

A 3 in × 0.5 in × 0.0625 in sample strip is normally used to provide an evaluation of both liquid and vapor phase attack. Surface-to-volume ratio is calculated for the wetted area only. A 1.5 in × 0.5 in × 0.0625 in sample strip will be used only for the material tested under a continuously wetted condition. A volume of 74 mL of liquid H_2O_2 will be used for this test, which yields an apparent sample surface area of 1 in^2 of test surface to 42.8 mL of H_2O_2. This approximates the 0.33 in^2/in^3 surface-to-volume ratio (0.33 in^{-1}) of the wetted surface of a standard drum containing 250 lb of H_2O_2. If it is necessary to test a sample that is not the standard 3 in × 0.5 in × 0.0625 in wetted surface, retaining this apparent surface-to-volume ratio will help interpret the results. For liquids, a 5-mL sample is used; for greases, a sample of ≈5 g is smeared on the inside of the test flask.

A.3 Apparatus

The following elements are used in the compatibility testing process:

- 125 mL pyrex kjeldahl flask
- 110 °C drying oven
- 66 °C (150.8 °F) at ±0.5 °C water bath
- 30 °C (86 °F) at ±0.5 °C water bath
- Aluminum foil to cover flasks
- Distilled water with conductivity of <2 μmho cm.

A.4 Cleaning and Passivation

A.4.1 Glassware

Prior to use, all glassware is immersed in a 10 percent NaOH solution for 1 hr at room temperature, rinsed with distilled water, immersed in a 10 percent HNO_3 solution for a minimum of 3 hr,

and finally rinsed with distilled water. The mouths of the flasks are then covered with clean aluminum foil and the flasks are dried in a 110 °C oven.

A.4.2 Metals

Aluminum samples are scrubbed with a warm detergent solution and then immersed at room temperature in N/15 NaOH for 15–20 min. Then they are washed and immersed for 45 min to 1 hr at room temperature in 45 percent HNO_3, and finally washed with distilled water, then pretreated with 35 percent H_2O_2 at 20–22 °C (68–72 °F) for 8–24 hr. Stainless steel samples are scrubbed with trichlorethylene to remove grease, rinsed with water, allowed to drip dry, immersed in 70 percent HNO_3 for 4–5 hr at room temperature, washed with clean, potable water, and finally washed with distilled water and pretreated with 35 percent H_2O_2. Special treatments for stainless steels that have not responded well to the above passivation technique are detailed in the FMC's *Bulletin 104*.

A.4.3 Plastics

Plastics and elastomer samples are thoroughly scrubbed in 0.5 percent solution of Nacconal, a synthetic detergent, rinsed with distilled water, exposed to a 10 percent HNO_3 water solution at 20–22 °C for 1 hr, then pretreated with 35 percent H_2O_2 at 20–22 °C (68–72 °F) for 8–24 hr.

A.4.4 Samples Handling

During the final rinsing, gloves or tongs should be used so samples are not touched with bare fingers. It is convenient to wash strips on a pyrex funnel as a handling medium, taking care to wash all areas. The strip is dried between two sheets of filter paper at room temperature or in a 50 °C oven and then placed in a test flask, which is immediately covered with aluminum foil.

A.5 Sample Screening

Prior to quantitative testing, a new or untried material is immersed, after chemical pretreatment, in 75 mL of H_2O_2 at room temperature for 24 hr. Violent decomposition, combustion, solution, or dimensional changes are watched for. If no unusual action occurs, the sample is then subjected to a screening at 66 °C (150.8 °F) for 24 hr.

A.6 Test Procedure

The prepared screened sample is placed in a passivated 100-mL kjeldahl flask, which is rinsed with a small volume of H_2O_2. The weight of the flask is measured to ±0.1 g. H_2O_2 of known strength (75 mL) is added to the flask, the Al covering replaced, and the flask again weighed. The initial weight of H_2O_2 solution is the difference in the two weights.

The flasks are placed in a constant temperature water bath for 1 wk at 66 °C (150.8 °F) and 4 wk at 30 °C (86 °F). The flask and its contents are then removed and cooled to room temperature, weighed, and a sample of the H_2O_2 withdrawn to determine concentration. These tests are run in duplication.

A.7 Results

The percent of AOL is calculated as follows:

$$\text{Percent AOL} = (W_1 - W_2)/(CW_1 \times 0.470) \times 100 \text{ percent} \ , \tag{1}$$

where

W_1 = initial net weight
W_2 = final net weight
C = initial fraction H_2O_2.

Qualitative observations are also made and recorded concerning such effects as discoloration of the H_2O_2 and apparent changes in the physical properties of the test material. Physical property changes include:

• For metals—any corrosion, staining, or surface change during or after test.

• For plastics—any blistering, swelling, distortion, changes in flexibility, color, transparency, or tear resistance.

A.8 Stability Test

Following the compatibility test, a sample of the remaining H_2O_2 is removed and subjected to a stability test at 100 °C (212 °F) for 24 hr, according to the procedure presented in appendix B.

APPENDIX B—PROCEDURE FOR DETERMINING HYDROGEN PEROXIDE STABILITY

B.1 Introduction

This test is done to determine the storage stability of H_2O_2, and runs for 24 hr at 100 °C (212 °F). For materials evaluation, this test is run on the remaining H_2O_2 after the compatibility tests.

B.2 Apparatus

The following elements are used in the stability determining process:

- Constant temperature baths at 100 °C (212 °F) ±1 °C.
- Thermometer zero to 150 °C (302 °F) ±1 °C.
- 50-mL volumetric flask with fused 4-in neck extension.
- Distilled or deionized water and aluminum foil to cap the mouth of the flask.
- Laboratory balance.

B.3 Preparation of Test Flask

The flasks are filled with 10 percent NaOH solution for 1 hr at room temperature, rinsed with tap water and refilled with 35 percent H_2SO_4 for 3 hr at room temperature. The flasks are thoroughly rinsed with distilled water and filled with H_2O_2 for 4–6 hr. The H_2O_2 is poured out and the flasks are capped with aluminum foil without any rinsing.

B.4 Test Procedure for Concentration Above 35 Percent Hydrogen Peroxide

Measure exactly 50 mL of H_2O_2 of a known concentration into a cleaned and conditioned, special extended neck, 50-mL volumetric flask and weigh. Cap with glass cap or aluminum foil, immerse in the constant temperature bath at 100 °C (212 °F) and continue the test for 24 hr as above. After 24 hr, remove the flask from the bath and remove the cap and reweigh it on the balance used for the initial weighing. The difference is the AOL. The tests run in duplication. Normal stability of H_2O_2 is 98 to 99 percent. Samples having stability below 90 percent should be closely watched. If stability is below 80 percent, H_2O_2 should be dumped. Calculate the stability as follows:

$$\frac{50(d_1)(C_1)(0.47) - (W_1 - W_2) = \text{percent stability}}{50(d_1)(C_1)(0.47)} \; , \tag{2}$$

where

d_1 = density at initial temp
C_1 = initial concentration
W_1 = initial weight
W_2 = final flask weight.

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13. ABSTRACT *(Maximum 200 words)*

This Technical Memorandum describes the development of several high-strength aluminum (Al) alloys that are compatible with hydrogen peroxide (H_2O_2) propellant for NASA Hypersonic-X (Hyper-X) vehicles' fuel tanks and structures. The yield strengths for some of these Al-magnesium-based alloys are more than 3 times stronger than the conventional 5254-H112 Al alloy, while maintaining excellent H_2O_2 compatibility similar to class 1 5254 alloy. The alloy development strategy is to add scandium, zirconium, and other transitional metals with unique electrochemical properties, which will not act as catalysts, to decompose the highly concentrated 90 percent H_2O_2. Test coupons are machined from sheet metals for H_2O_2 long-term exposure testing and mechanical properties testing. In addition, the ability to weld the new alloys using friction stir welding has also been explored. The new high-strength alloys could represent an enabling material technology for Hyper-X vehicles, where flight weight reduction is a critical requirement.

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