Intermediate Temperature Fluids Life Tests – Theory

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Abstract. There are a number of different applications that could use heat pipes or loop heat pipes (LHPs) in the intermediate temperature range of 450 to 750 K, including space nuclear power system radiators, and high temperature electronics cooling. Potential working fluids include organic fluids, elements, and halides, with halides being the least understood, with only a few life tests conducted. Potential envelope materials for halide working fluids include pure aluminum, aluminum alloys, commercially pure (CP) titanium, titanium alloys, and corrosion resistant superalloys. Life tests were conducted with three halides (AlBr₃, SbBr₃, and TiCl₄) and water in three different envelopes: two aluminum alloys (Al-5052, Al-6061) and CP-2 titanium. The AlBr₃ attacked the grain boundaries in the aluminum envelopes, and formed TiAl compounds in the titanium. The SbBr₃ was incompatible with the only envelope material that it was tested with, Al-6061. TiCl₄ and water were both compatible with CP2-titanium. A theoretical model was developed that uses electromotive force differences to predict the compatibility of halide working fluids with envelope materials. This theory predicts that iron, nickel, and molybdenum are good envelope materials, while aluminum and titanium halides are good working fluids. The model is in good agreement with results from previous life tests, as well as the current life tests.

Keywords: Heat pipe life tests, intermediate temperature heat pipes, halide working fluids, space radiator systems, high temperature electronics cooling. **PACS:** 44.30.+v, 44.35.+c.

INTRODUCTION

NASA is interested in Brayton cycle converters for nuclear space power system (Siamidis, 2006;, Siamidis and Mason, 2006) A radiator is required to dissipate the waste heat generated during the thermal-to-electric conversion process. A pumped sodium-potassium (NaK) or water secondary loop is used to transfer waste heat from the power converters to the heat pipe radiator. The radiator panel consists of a series of heat pipes located between two high conductivity fins. The heat pipes transfer the heat to the fins, which radiate the waste heat to space.

Depending on the system design, some of these heat pipes may need to operate in the intermediate temperature range of 450 K to 750 K. There are currently no heat pipe working fluids that are suitable from roughly 500 to 700 K. One promising family of fluids is the halides. A halide is a compound of the type MX, where M may be an element or organic compound, and X may be fluorine, chlorine, bromine, iodine, or astatine. Some of the halides have properties which appear to be suitable for use as intermediate temperature heat pipe fluids. This paper will develop a theoretical method for calculating halide compatibility, and compare predictions with experimental results.

INTERMEDIATE TEMPERATURE FLUIDS

The intermediate temperature region is generally defined as 450 to 750 K. At temperatures above 700-725 K, alkali metal heat pipes start to become effective. As the temperature is lowered, the vapor pressure and vapor density of the alkali metals are decreased. Below about 725 K, the vapor density is so low that the vapor sonic velocity limits

the heat transfer. The heat pipe (or LHP) vapor velocity becomes too large to be practical for alkali metals in the intermediate temperature range.

Historically, water was used at temperatures up to about 425 K. More recently, it has been shown that water can be used with titanium or Monel envelopes at temperatures up to 550 K (Anderson et al., 2006). While water heat pipes can operate at temperatures up to 550 K, their effectiveness starts to drop off above 500 K, due to the decrease in the surface tension. Since alkali metal heat pipes are effective above 700 K, and water heat pipes are effective at temperatures below 500 K, we have concentrated in this work on working fluids in the 500 to 700 K range.

A number of researchers have suggested that halides could be used as working fluids in the intermediate temperature range (Saaski and Owarski, 1977; Saaski and Hartl, 1980; Anderson et al., 2004; Devarakonda and Olminsky, 2004; Devarakonda, Anderson, and Beach, 2005; and Locci et al., 2005). Life tests must be conducted before these fluids can be reliably used in heat pipes for long time periods. This paper will review current and previous halide life tests, describe a theoretical model to predict heat pipe/halide compatibility, and compare predictions with experiments.

Previous Life Tests

The only previous life tests with halides that we are aware of were conducted by Locci et al. (2005) and Saaski and his co-workers (Saaski and Owarski, 1977; Saaski and Hartl, 1980). The results of Locci et al. are described in the next section. Saaski and his co-workers life-tested the halides SbCl₃, SnCl₄, and TiCl₄ with aluminum 6061 and mild steel envelopes, for periods of up to 3 years. 6061 aluminum was chosen because it is commonly used in grooved aluminum heat pipes for spacecraft applications. The aluminum heat pipes had a single wrap of 100-mesh Al-1100 screen. The mild steel envelopes had a single wrap of 200-mesh 304 stainless steel screen. The fluids were tested slightly above their normal boiling point. The differences between the evaporator/adiabatic, and adiabatic/condenser thermocouples were monitored. A large difference in the evaporator/adiabatic thermocouples generally indicated problems with clogging of the evaporator wick. A large difference between the adiabatic/condenser thermocouples indicated non-condensable gas generation.

The results are shown in Table 1. All 3 halides were incompatible with aluminum. Gross corrosion of the evaporator and evaporator wick was observed with $SbCl_3$ and $SnCl_4$ in aluminum. $SnCl_4$ and $TiCl_4$ were compatible with mild steel (and stainless steel), with the life tests running roughly 3 years. The $SbCl_3$ reacted with the stainless steel wick and generated significant quantities of gas.

Working Fluid	Envelope	Operating Time (hrs.)	Operating Temp. (K)	ΔT _{Evap Adiab} (K)	ΔT _{Adiab/Cond} (K)	Comments
SnCl ₄	Al 6061		432			Incompatible
SnCl ₄	A-178 Steel	27750	429	4	9.7	Stable Operation
						Sudden Burnout/Wick
TiCl ₄	Al 6061	2500	438	6	7.2	Corrosion
TiCl ₄	A-178 Steel	28540	432	4.1	1.7	Stable Operation
SbCl ₃	Al 6061		500			Incompatible
						Incompatible - Rising
SbCl ₃	A-178 Steel	5000	476	6.3 to 169	62	ΔT_{Evap} Adiab

TABLE 1. Halide Life Test Data (Saaski and Hartl, 1980).

Halide Life Tests

More recently, tests were conducted at NASA Glenn with three halides and water in three different envelopes: two aluminum alloys (Al-5052, Al-6061) and commercially pure grade 2 titanium (CP-2 Ti). The wall materials were chosen because of their availability and wide use in space applications. All of the heat pipes were operated as pool boilers, and did not contain a wick. The pool level with no power varied from 4.8 to 6.5 cm. The NASA life tests are summarized in Table 2.

Envelope	Al-6061	Al-5052	CP2-Ti
Fluid	(0.8-1.2 Mg, 0.4-0.8 Si)	(2.2-2.8 Mg, 0.25 max Si)	
	1,100 hrs.	4,290 hrs.	1,100 hrs.
	Intergranular Corrosion	Failed	Secondary Products – TiAl
AlBr ₃	$\Delta T = 100 \text{ K}$	$\Delta T = 70 \text{ K}$	$\Delta T = 90 \text{ K}$
	5,000 hrs.		
	Wall Thickness Change		
SbBr ₃	$\Delta T = 90 \text{ K}$	—	—
			4,019 hrs Stable
TiCl ₄	Not Suitable (Saaski)	Probably Not Suitable	$\Delta T = -25 \text{ K}$
Water	Not Suitable	Not Suitable	8,000 Hours - Ongoing

TABLE 2. NASA Glenn Life Tests with Halides and Water. All Tests Conducted at 500 K.



FIGURE 1. Schematic of Experimental Pipe, Location of Thermocouples and Heating Devices.

Figure 1 shows a schematic of the experimental test set-up. The power was varied to maintain TC1, located below the heater, at 500 K. Temperature differences, TC1 – TC3, are also shown in Table 2. They were measured with the heat pipe operating at 500 K. Selection criteria for the halides, as well as procedures for fabrication and testing, are given in Locci et al. (2005). They also reported on two of the life tests, which were stopped after 1,100 hours, the AlBr₃/Al-6061 and AlBr₃/CP-Ti heat pipes. Internal pipe surfaces and metallographically polished cross sections were observed by optical and field emission scanning electron microscopy (FESEM). Chemical and x-ray analyses were performed on the products and layers that formed on the inner diameter of the capsule.

Neither of these systems was found to be compatible. The AlBr₃ reacted heavily with the titanium wall, becoming discolored. Energy dispersive spectroscopy (EDS), chemical and x-ray analysis performed on the loose layers found in the reacted Ti (CP2) envelope indicated the formation of TiAl and complex Al-O-Br regions dispersed in the original Ti envelope. In the AlBr₃/Al-6061 system, the AlBr₃ attacked the grain boundaries, and intergranular corrosion had initiated which could eventually lead to envelope failure. The AlBr₃/Al-5052 system was also incompatible, failing after 4,290 hours with a leak to the environment. As shown in Figure 2, the Al-5052 pipe failed and the AlBr3 has leaked out from a location above the heating block. The wall thickness in the failed section was significantly reduced, from 1.30 mm (0.051 in.) to 0.58 mm (0.023 in.), and the chemical reaction had significantly embrittled the alloy.

The TiCl₄/Titanium system was run for 4,019 hours, with no problems observed. The heat pipe was then sectioned and analyzed. As shown in Figure 3, no indication of titanium wall damage was noticed at any of the three locations observed. The wall thickness was also unchanged after the exposure to TiCl₄.



FIGURE 2. (a) Testing Station Showing the Al-5052 Pipe Failure after 4290 Hours of Exposure to $AlBr_3$ at 500K; (b) Perforation Observed in a Section Above the Heating Block. FESEM Images of (c) Failure Site and (d) Observed Embrittled Region.

Finally, SbBr₃ was tested at 500 K with an Al-6061 envelope. The test was stopped after 5,000 hours, and then the pipe was sectioned and analyzed. The wall thickness at the bottom of the pipe was reduced from 1.58 mm (0.062 in.) to 1.32 mm (0.052 in.), indicating that the system is incompatible. A cross section view of the bottom section of the Al-6061 pipe after the 5000 hour exposure to SbBr3 is presented in Figure 4. Although, the Al-5052/SbBr₃ interaction does not appear as aggressive as the one observed for AlBr₃ (Locci et al., 2005), ragged envelope walls and the formation of cavities are clearly visible in the sample cross section.

HALIDE COMPATIBILITY

As discussed above, in some cases the halides will react with the heat pipe envelope walls or wick. The rest of the paper will describe a method, first proposed by Saaski and Owarski (1977), to evaluate halide compatibility; then the predictions will be compared with the experimental results to date. The method calculates the electromotive force difference, EMF, which predicts the probability of reaction between a halide and a metal envelope (or wick).



FIGURE 3. FESEM Images Showing No Damage to the Ti (CP2) Internal Pipe Surfaces after 4019 Hours of Exposure to TiCl₄ at 500 K, (a) Bottom Section under Liquid Interaction (b) Middle and (c) Top sections under the Effect of Condensed Vapor.



FIGURE 4. FESEM Cross Section Image Showing the Al-6061 Envelope Internal Rugged Surface and the Formation of Cavities After the 5,000 hour Life Test with SbBr₃ at 500 K

Theoretical Background

If an inorganic molten halide $M_b X_c$ is in contact with a metallic wall (M_a) , the following double displacement reaction may occur depending on the relative chemical activities of the halide working fluid versus the halide that results from the metal in the envelope wall:

$$fM_a + gM_bX_c \leftrightarrow fM_aX_{cp} + gM_b . \tag{1}$$

The free energy change corresponding to this reaction is:

$$\Delta G = \Delta G^{o} + RT \ln \left[\frac{(a_{M_{a}} X_{cp})^{f} (a_{M_{b}})^{g}}{(a_{M_{a}})^{f} (a_{M_{b}} X_{c})^{g}} \right].$$
(2)

The corresponding electromotive force difference (or the corrosion potential) for this reaction is:

$$\Delta E = \Delta E^{o} - \frac{RT}{nF} \ln \left[\frac{(a_{M_{a}} X_{cp})^{f} (a_{M_{b}})^{g}}{(a_{M_{a}})^{f} (a_{M_{b}} X_{c})^{g}} \right].$$
(3)

where ΔE^0 , the electromotive force difference when all reactants are at unit chemical activity, is given by:

$$\Delta E^{o} = \frac{-\Delta G^{o}}{nF} \ . \tag{4}$$

In order to appropriately define chemical activities a physical model is needed. Such a model is presented in Saaski and Owzarsky (1977), where a hypothetical corrosion cell is presented for a metal/metal halide combination similar to equation (1). The corrosion cell consists of the following reactants and reaction products:

$$M_a, M_a X_{cp}, M_b X_c, M_b$$

where the first two, M_a , M_aX_{cp} represent one of the electrodes while the other two, M_bX_c , M_b , represent the other electrode. In other words, each electrode consists by the pure metal and its halide. The electrodes are coupled by

ionic conduction through the reactant halide. Under the assumption that solubility of the two reactants in the metal reaction product together with the solubility of the initial halide (working fluid) in the resultant halide (of the envelope material) can be neglected, the chemical activity of the constituents becomes unity. In this case

$$\Delta E = \Delta E^{\circ} \quad \text{and} \quad \Delta G = \Delta G^{\circ}. \text{ Indeed, the logarithmic term in (3), } \quad \frac{RT}{nF} \ln \left[\frac{(a_{M_a} X_{cp})^f (a_{M_b})^g}{(a_{M_b} X_c)^g} \right], \text{ was}$$

demonstrated (Saaski and Owzarsky, 1977) to be very small in comparison with the standard electromotive force difference, ΔE^0 , hence, the error introduced by the just mentioned assumption is negligible.



FIGURE 5. Good Working Fluids (From a Compatibility Standpoint) Have High Decomposition Potentials, While Halides/Salts of Good Envelope Materials Have Low Decomposition Potentials.

FIGURE 6. Decomposition Potential for Various Halides.

With the above assumptions, the electromotive force difference of the reaction between the working fluid and envelope can be directly calculated as the standard electromotive force difference of the reaction. The standard electromotive force difference or the potential difference, ΔE^0 , is the difference between the decomposition potentials of the two halides, the metal envelope halide, $M_a X_{cp}$ and the working fluid, $M_b X_c$:

$$\Delta E^{o} = E_{P_{-M_{a}X_{cr}}}(T) - E_{P_{-M_{b}X_{c}}}(T)$$
⁽⁵⁾

The standard EMF difference, ΔE^0 is the decomposition potential of the envelope minus the decomposition potential of the fluid. If the standard EMF difference, ΔE^0 , is positive, then the reaction can proceed spontaneously and the wall will react chemically. When the standard EMF difference is negative, the probability of spontaneous reaction decreases significantly. This gives the following working fluid/envelope material selection criterion: The envelope material halide should have a lower decomposition potential than the working fluid halide. This is shown in Figure 5. AlCl₃ and TiCl₄ have a high decomposition potential, so they are good working fluids. Molybdenum and iron have a low decomposition potential, so should be good envelope materials.

Electromotive Force Difference

In order to find out the decomposition potentials of the selected halide working fluid/envelope material couples a literature survey was carried out. Since the data (Delimarskyi and Markov, 1961; Janz, 1967; Saaski and Owarski, 1977) was mostly available for chlorides, and relatively poor for bromides and iodides, especially for the halides of the selected envelope metals, the compatibility study focused on chlorides. The limited data for bromides and halides suggest that they behave in a similar fashion, so the conclusions for the chlorides should be valid for the corresponding bromides and iodides. Based on the literature survey results, the decomposition potentials for the halides are shown in Figure 6. In general, the decomposition potential information was found for two or three data points (temperatures) only. The plot and calculations use a linear interpolation of these data points as a function of temperature.



FIGURE 7. Electromotive Force Difference for Potential Halide/ Envelope Material Reactions.

differences with Mo and Fe, hence have a low probability of spontaneous reaction. The results for iron suggest that carbon steel is a relatively stable envelope material for almost all the halides. Nickel, which is a major component in stainless steels and superalloys, shows a moderate lack of stability with bismuth trichloride, antimony trichloride and tin tetrachloride. However, it might be stable with the other halides including tin dichloride. Titanium has a higher tendency for corrosion, especially in the presence of antimony tribromide, bismuth trichloride, antimony trichloride and tin tetrachloride.

decomposition The potentials in Figure 6 were used to calculate electromotive force differences. Potential envelope materials include aluminum, aluminum alloys, titanium, titanium alloys, carbon steel, stainless steels, and the superalloys. The dominant metallic components for these envelopes include Ti, Ni, Fe, Cr, Mo and Al. The following halides were examined: aluminum chloride, aluminum bromide, antimony chloride, antimony bromide, bismuth gallium chloride, chloride, lead chloride, magnesium chloride, tin dichloride, tin tetrachloride, zinc chloride and zirconium chloride.

The electromotive force differences are shown in Figure 7 and Table 3. Values above zero are unstable. The calculations used a temperature of 400° C (673 K), linearly interpolating the available data. As shown in Figure 6, the slopes of all of the halides are similar, so the potential difference is only a weak function of temperature.

Aluminum is the least suitable envelope material for our halides, with the exception of Mg and Zr. From an EMF standpoint, the best envelope material would be molybdenum, followed by iron. All of the halides have strong negative potential It should be noted that these results give a rough guide to the behavior of the halides, particularly when the envelope material is an alloy. The calculations above assume that the pure materials are in contact. In some cases, the alloy may have a stronger affinity for a component than the halide, preventing it from being dissolved.

	Ti	Ni	Cr	Мо	Fe	Al
SbBr ₃	0.83					
BiCl ₃	0.653	0.293	0.633	-0.397	0.053	1
SbCl ₃	0.6	0.26	0.6	-0.43	0.02	0.97
SnCl ₄	0.42	0.08	0.4	-0.63	-0.18	0.773
SnCl ₂	0.177	-0.213	0.127	-0.9	-0.453	0.5
PbCl ₂	0.12	-0.22	0.12	-0.91	-0.46	0.49
AlCl ₃	0.11	-0.71	-0.25	-1.4	-0.57	
GaCl ₃	0.07	-0.27	0.07	-0.96	-0.51	0.44
$ZnCl_2$	-0.18	-0.52	-0.18	-1.21	-0.76	0.19
$ZrCl_4$	-0.6	-0.94	-0.6	-1.63	-1.18	-0.23
MgCl ₂	-1.28	-1.62	-1.28	-2.31	-1.86	-0.91
TiCl ₄		-0.34	0	-1.03	-0.58	0.37

TABLE 3. Electromotive Force Difference, Volts.

Comparison of Theory with Life Tests

Table 4 compares the theoretical predictions with the existing halide life test data, and shows very good agreement. As discussed above, Saaski and his co-workers tested three halides, titanium tetrachloride, tin tetrachloride and antimony trichloride with aluminum and mild steel envelopes. The steel heat pipes had a stainless steel wick. As predicted, gross corrosion of the evaporator and evaporator wick was observed with SbCl₃ and SnCl₄ in aluminum envelopes. TiCl₄ was also incompatible with aluminum. This is expected, since the decomposition potential of the aluminum halides are significantly higher than all other halides.

Halide	6061 Aluminum	Mild Steel	304 SS Screen (Ni)	Titanium
AlBr ₃	6061 Incomp./500 K ¹			
	5052 Failed/4,290			No, Ti/Al compounds ¹
	hrs./500 K			(different mechanism)
	Partially Agree (attacked			AlCl ₃ is slightly
	grains)			unstable
SbCl ₃	Incomp./500 K ²	Incomp./5,000 hrs./476 K ²	Incomp./5,000 hrs./476 K ²	
	Agree	reacted with SS Wick	Agree	
SbBr ₃				Incomp./5000
				hours/500 K
				Agree
SnCl ₄	Incomp./432 K ²	27,750 hrs./429 K ²	27,750 hrs./429 K ²	
	Agree	Agree	Agree	
TiCl ₄	Incomp./438 K ²	28,540 hrs./432 K ²	28,540 hrs./432 K ²	4,019 hrs./500 K
	Agree	Agree	Agree	Agree

TABLE 4. Comparison of Halide Life Tests and Predictions.

¹Locci et al., 2005. ²Saaski and Hartl, 1980.

Life tests in carbon steel/stainless steel were more stable, as predicted. The exception was antimony chloride, which reacted with the stainless steel wick and generated significant amounts of gas. Referring to Figure 7, the reaction with stainless steel wick was expected since chromium, a component of more than 10% weight in stainless steel composition, forms halides with relatively high decomposition potentials. Moreover, among all the halides reacting with Fe, SbCl₃ shows the highest potential difference, very slightly positive.

Comparison of the theory with the NASA Glenn tests also shows good agreement. As predicted, titanium and $TiCl_4$ are compatible, while titanium and $SbBr_3$ are incompatible. No predictions were made for $AlBr_3$ in titanium; however, Table 3 shows that $AlCl_3$ in titanium is slightly unstable (with $TiCl_2$, not $TiCl_4$), and the bromides should behave in a fashion similar to the chlorides. The life tests found that TiAl formed, which is not predicted by the current procedure. The theory predicts that $AlBr_3$ should be compatible with pure aluminum. As discussed above, the $AlBr_3$ attacked the grain boundaries in the aluminum alloys; clearly the reactivity of alloying additions in commercial alloys requires closer consideration.

CONCLUSIONS

Life tests were conducted at NASA Glenn with three halides (AlBr₃, SbBr₃, and TiCl₄) and water in three different envelopes: two aluminum alloys (Al-5052, Al-6061) and CP-2 titanium. The AlBr₃ attacked the grain boundaries in the aluminum envelopes, and formed TiAl compounds in the titanium. The SbBr₃ was incompatible with the only pipe that it was tested with, Al-6061. Finally, TiCl₄ and water were both compatible with CP2-titanium.

A theoretical model based on electromotive force differences has been developed to predict the compatibility of halide working fluids with envelope materials. The envelope material halide should have a lower decomposition potential than the working fluid halide. AlCl₃ and TiCl₄ have a high decomposition potential, so should be good working fluids. Molybdenum and iron have a low decomposition potential, so should be good envelope materials.

The method almost always predicted the compatibility of halide life tests. For example, it successfully predicted that $TiCl_4$ was incompatible with aluminum and was compatible with mild steel. The only two cases without full agreement were (1) AlBr₃ and aluminum, where the AlBr₃ attacked the alloying materials at the grain boundaries, and (2) AlBr₃ and titanium, where the method predicted incompatibility, but not that TiAl compounds would form.

NOMENCLATURE

a_P^h	= chemical activity of constituent P with h as multiplier
<i>f</i> , <i>g</i>	= molecules multiplier resulted from balancing the reaction equation
M_a	= metallic element (envelope material)
M_b	= metallic element (reaction product)
$M_b X_c$	= halide of metal M_b (working fluid)
$M_{\rm a}X_{cp}$	= halide of metal M_a (reaction product)
E_P	= decomposition potential (V)
F	= Faraday (96.59 kJ/V mol)
n	= number of electrons transferred in the balance equation
Т	= absolute temperature (K)
EMF	= electromotive force (V)
FESEM	= field emission scanning electron microscopy
$E_{P_{-}M_{x}X_{y}}(T)$	= decomposition potential of the metallic halide $M_x X_y$ at the reaction temperature T (V)
ΔΕ	= electromotive force difference (V)
ΔE^0	= standard electromotive force difference (V)
ΔG	= free-energy change (J/mol)
ΔG^0	= standard free-energy change (J/mol)

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