Intermediate Temperature Heat Pipe Life Tests and Analyses

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There are a number of 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, fuel cells, geothermal power, waste heat recovery systems, and high temperature electronics cooling. Since 2004, we have been conducting life tests at temperatures up to 550 K with water and Commercially Pure Titanium Grade 2 (CP-Ti), titanium alloys, Monel 400, and Monel K500 heat pipes. Since 2006, life tests have been conducted at temperatures up to 673 K with titanium and Hastelloy B-3, C-22, and C-2000 envelopes paired with AlBr₃, GaCl₃, SnCl₄, TiCl₄, and TiBr₄ halide working fluids. Recently, roughly half of these heat pipes were selected for destructive evaluation. The working fluids were analyzed, and sections of the heat pipes were examined to determine the type and amount of corrosion in the wicks and heat pipes. The results showed that Titanium/water and Monel/water heat pipes are suitable for temperatures up to 550 K. Analysis of titanium/water heat pipe crosssections using optical and electron microscopy revealed little if any corrosion even when observed at high magnifications. Copper depleted zones, as well as copper surface nodules formed on the Monel 400 screen wick, but not on the Monel K500 envelopes. An analysis of the water working fluids showed minimal pickup of metals. The long terms tests also established that Titanium/TiBr₄ at 653 K, and Hastelloy B-3, C-22 and C-2000/AlBr₃ at 673 K were compatible. Hastelloy C-2000 underwent little corrosion when used with TiCl₄ working fluid. Hastellov C-22 exhibited a 5-10 micrometer thick dual corrosion layer when tested with AlBr₃ working fluid. The results indicate that the tested envelope materials and working fluids can form viable material/working fluid combinations.

I. Introduction

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, fuel cells, geothermal power, waste heat recovery systems, and high temperature electronics cooling. The intermediate temperature region is generally defined as the temperature range between 450 and 750 K. At temperatures above 700-725 K, alkali metal (cesium) heat pipes start to become effective. Below about 725 K, the vapor density for cesium is so low that the vapor sonic velocity limits the heat transfer. 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, Dussinger, Bonner, and Sarraf, 2006).

II. Literature Survey

At present, there is no commonly accepted working fluid over the entire intermediate temperature range. Potential working fluids include elemental working fluids such as sulfur, organic compounds, and halides. This paper reviews many of the intermediate temperature heat pipe life tests conducted over the past 40 years, and then recommends suitable working fluid/envelope combinations. Anderson (2006) provides more detailed information on these tests.

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A. Elements – Sulfur, S/I, and Mercury

Pure sulfur is not suitable in the intermediate temperature range because of its high liquid viscosity, although it may be useful at higher temperatures. Sulfur has a unique temperature dependent polymerization property at 470 K, which increases its liquid viscosity peak to approximately 100 Pa-s. This is about three orders of magnitude higher than the maximum level for effective heat pipe operation. The addition of a small percentage of iodine reduces the viscosity to a level that may be acceptable for reasonable heat pipe operation (Polasek and Stulc, 1976, Timrot et al., 1981). A potential problem with both sulfur, and sulfur/iodine mixes is that they react strongly with many envelope materials.

There are several problems with mercury as a wetting fluid including:

- Toxicity
- · Difficulty in achieving good wetting of the wick and wall material without extensive corrosion
- High density, which translates into increased mass.
- Aggressive attack or solutioning of many metals, e.g., copper

Additives have been used successfully with mercury to wet coarse, wicks, but it appears to be very difficult to achieve wetting in finer pore wicks. Heat pipe tests with mercury in a sintered stainless steel wick failed because the mercury did not wet the stainless steel (Anderson, Rosenfeld, Angirasa, and Mi, 2004).

Sulfur, Sulfur-Iodine, and Mercury life tests are summarized in Table 1. Mercury is compatible with 347 SS based upon long term life tests. Sulfur is compatible with pure aluminum based on a short term life test, as is Sulfur-10% Iodine with 304 SS.

	Compatible	Incompatible
	Short Term	
	873 K (600°C)/~200 hrs./3003 Al	
Sulfur/Al	(Ernst, 2006)	
Sulfur/SS		773 K (500°C)/500 hrs. (Polasek, 1989)
	Short Term	973 K(700°C)/740 hrs. (Polasek, 1989)
	623 (350°C)/1,008 hrs./304 SS	893 K(620°C)/740 hrs. (Polasek, 1989)
Sulfur – 10% I/SS	(Anderson, 2004)	833 K(560°C)/8,000 hrs. (Polasek, 1989)
		623 (350°C)/1,028 hrs./5052 Al (Anderson,
Sulfur – 10% I/Al		2004)
		623 (350°C)/1,000 hrs./Ti-6Al-4V (Anderson,
		2004)
Sulfur – 10% I/Ti		523 (250°C)/24 hrs./CP-Ti (Anderson, 2004)
		623 (350°C)/950 hrs./Nb-1% Zr (Anderson,
Sulfur – 10% I /Nb		2004)
	603 (330°C)/10,000 hrs./347 SS	
Mercury/SS	(Deverall, 1971, Reid 1991)	

Table 1. Summary of Sulfur, Sulfur-Iodine, and Mercury Life Tests.

B. Organic Fluids

Life tests have been conducted with 19 different organic working fluids. Most of the suitable organic fluids are ring compounds. The reason for this was discussed by Saaski and Owzarski (1977) who pointed out that these types of compounds should be more stable than the long chain hydrocarbons. Saaski and Owzarski also pointed out replacing some (or all) of the hydrogen atoms with fluorine may make the compound more stable.

Potential problems with the organic working fluids include the possibility of polymerization and/or dissociation. Polymerized fluids generally undergo an increase in liquid viscosity, which will decrease the circulation of the working fluid in a heat pipe and therefore its heat transport capacity. Dissociation normally generates non-condensable gases (NCG), which over time will build up in the heat pipe condenser. The presence of NCG reduces the effective length of the heat pipe condenser and hence the area available for heat radiation. This will either cause the temperature to rise at a given power level or the power level to be decreased at a given temperature.

Typically, organic fluids develop problems more quickly as the temperature is increased. The maximum operating temperature for an organic fluid depends both on the operating temperature, and how long the heat pipe needs to operate. For example, Anderson et al. (2007b) tested 304 stainless steel heat pipes with Dowtherm A working fluid. Heat pipes operating at 723 K (450°C) gassed up in ~180 hours, while pipes operating at 673 K (400°C) took roughly 1,500 hours for NCG gas generation to start affecting their behavior.

	Compatible	Incompatible
	514 K (241°C)/8,000 hrs./6061 Al (Saaski	<u>^</u>
Diphenyl/Aluminum	1977, 1980)	
		673 K (400°C)/~9,000 hrs. (Groll,
		1982, 1987, 1989)
		598 K (325°C)/4,040 hrs. (Kenney,
	523 K (250°C)/~9,000 hrs. (Groll, 1982, 1987,	1978)
	1989)	526 K (253°C)/24,400 hrs. (Saaski
Diphenyl/Mild Steel	498 K (225°C)/6,174 hrs (Kenney, 1978)	1977, 1980)
Diphenyl/Black Iron	523 K (250°C)/7,158 hrs (Kenney, 1978)	
	Cycle 598 K to 653 K (325°C to 380°C)/5,520	
	hrs./ 316 SS (Grzyll, 1991, 1994, 1995)	
	623 K (350°C)/ 5,520 hrs./316 SS (Grzyll,	
	1991, 1994, 1995)	
	548 K (275°C)/6,174 hrs/304 SS (Kenney,	748 K (475°C)/72 hrs./304 SS (Kenney,
	1978)	1978)
	543 K (270°C)/~9.000 hrs./316L SS (Groll.	738 K (465°C)/100 hrs./304 SS
	1982, 1987, 1989)	(Kenney, 1978)
		695 K (422°C)/366 hrs./304 SS
	Short Term	(Kenney, 1978)
Diphenyl/Stainless	673 K (400°C)/1200 hrs./304 SS (Kenney/	673 K(400°C)/~9,000 hrs./316L SS
Steel	Feldman)	(Groll, 1982, 1987, 1989)
	Short Term	
Diphenyl Oxide/	573 K (300°C)/3200 hrs./347 SS & 304 SS	
Stainless Steel	(LASL, 1968a, 1968b, 1970)	
	543 K (270°C)/~40,000 hrs/ST 35 (Groll,	
Dowtherm A/Mild	1982, 1987, 1989)	573 K (300°C) /~40000 hrs./ST 35
Steel	523 K (250°C)/8383 hrs. (Kenney, 1978)	(Groll, 1982, 1987, 1989)
	573 K (300°C)/~40,000 hrs/321 SS (Groll,	
	1982, 1987, 1989)	723 K (450°C)/180 hrs./304 SS
	541 K (268°C)/24,500 hrs/304 SS (Kenney,	(Anderson et al. 2007)
	1978)	673 K (400°C)/1,770 hrs./304 SS
		(Anderson et al., 2007)
	Short Term	623 K (350°C)/~40,000 hrs/321 SS
	673 K (400°C)/1,200 hrs (Kenney, 1978)	(Groll, 1982, 1987, 1989)
Dowtherm A/	618 K (345°C)/1,000 hrs./304 SS (Anderson	473 K (200°C)/17,016 hrs.
Stainless Steel	et al., 2007)	(Basilius/Prager) (slow gas generation)
		473 K (200°C)/7,016 hrs/ (Basilius and
Dowtherm		Prager, 1975)
A/Copper		slow gas generation
Dowtherm A/		523 K (250°C)/~9,000 hrs./CuNi10Fe
Copper-Nickel		(Groll, 1982, 1987, 1989)
Dowtherm	543 K(270°C)/~9,000 hrs. (Groll, 1982, 1987,	406°C (680K)/~2,000 hrs (Anderson et
A/Titanium	1989)	al. 2007)

Table 2. Summary of Diphenyl, Diphenyl Oxide and Eutectic Diphenyl/Diphenyl Oxide Life Tests

	Compatible	Incompatible					
	488 K (215°C)/27,750 hrs./6061 Al						
Naphthalene/Aluminum	(Saaski 1977, 1980)						
1	543 K (270°C)/~26.000 hrs./ST 35 &						
Naphthalene/Mild Steel	13CrMo44 ⁶ . (Groll, 1982, 1987, 1989)						
I	Cycle 598 K to 653 K (325°C to						
	380°C)/5.520 hrs./ 316 SS (Grzvll, 1991.						
	1994, 1995)						
	623 K (350°C)/ 5,520 hrs./316 SS						
	(Grzyll, 1991, 1994, 1995)						
	593 K (320°C)/~9,000 hrs/316L SS /						
	(Groll, 1982, 1987, 1989)						
	593 K (320°C)/Alloy 20/~3,000 hours						
	(Vasil'ev et al., 1988)						
	Short Term						
	$(53 \text{ K} (380^{\circ}\text{C})/\text{Alloy }20 \text{ (Vasil'ev et al. ,})$,					
Naphthalene/Stainless Steel	1988)	/					
	593 K (320°C)/~9,000 hrs/CuNi10Fe ⁻						
Naphthalene/Cu-Ni	(Groll, 1982, 1987, 1989)						
	593 K (320°C)/~9,000 hrs/CP-11 (Groll,	,					
	1982, 1987, 1989						
Nanhthalana/Titanium	$595 \text{ K} (520^{\circ}\text{C})/~3,000 \text{ nours/11} (vasilev$						
	et al. , 1988)	590 K (207%)(72 hrs/60(1 A1 (Sasali					
O Tembeny $1/\Lambda 1$		$1077 \ 1080$) K (507 C)/072 IIIS./0001 AI (Saaski					
	545 K (272°C)/27 750 brs/A1 178 (Saaski	1977, 1980)					
O-Ternhenvl/Mild Steel	1977 - 1980) stable NCG						
0-replicity//wind Steel	$C_{\text{vole}} = 50\% K$ to $653 K (325\% C)$ to						
	$(320^{\circ}C)/5520$ hrs / 316 SS (Grzyll 1991						
	1994 1995)	,					
	$623 \text{ K} (350^{\circ}\text{C})/ 5.520 \text{ hrs}/316 \text{ SS}$						
O-Terphenyl/SS	(Grzvll, 1991, 1994, 1995)						
	Cycle 598 K to 653 K (325°C to						
	380°C)/5.520 hrs./ 316 SS (GrzvII, 1991.						
	1994, 1995)						
	623 K (350°C)/ 5,520 hrs./316 SS						
Decafluorobiphenyl/SS	(Grzyll, 1991, 1994, 1995)						
	410 K (137°C)/23,120 hrs./6061 Al						
Toluene/Aluminum	(Saaski 1977, 1980)						
	523 K (250°C)/~26,000 hrs./ ST 35 &						
Toluene/Mild Steel	13CrMo44 (Groll, 1982, 1987, 1989)						
	523 K (250°C)/~26,000 hrs./316 SS						
Toluene/Stainless Steel	(Groll, 1982, 1987, 1989)						
	553 K (280°C)/9,000 hrs./CuNi10Fe						
Toluene/Copper Nickel	(Groll, 1982, 1987, 1989)						
	523 K (250°C) 9,000 hrs. (Groll, 1982,						
Toluene/Titanium	1987, 1989)						
1-Fluoronaphthalene/	493 K (220°C)/13,380 hrs./6061 Al						
Aluminum	Some NCG (Saaski 1977, 1980)						

Table 3. Summary of Organic Fluid Life Tests Other Than Diphenyl and Diphenyl Oxide.

⁶ 13CrMo44 is a 1% Cr-1/2% Molybdenum Steel
 ⁷ Copper Nickel Alloy, resistant to corrosion in seawater

	Compatible		Incompatible
1-Fluoronaphthalene/	530 K (257°C)/26,370 hrs./A178 (Saas	ski	<u>^</u>
Mild Steel	1977, 1980)		
1-Fluoronaphthalene/			530 K (257°C)/26 370 hrs /304 SS (Saaski
Stainless Steel			1977 1980)
	$503 K (230^{\circ}C)/_{\odot}/10000 hrs/ST$	35	1777, 1900)
N. Ostana (Mild Staal	$(C_{roll} = 1082 \pm 1087 \pm 1080)$	55	
	(01011, 1982, 1987, 1989)		520 K (25700)/26 270 1 /204 88 (8 1)
1-Fluoronaphthalene/			$530 \text{ K} (257^{\circ}\text{C})/26,370 \text{ hrs}/304 \text{ SS} (Saaski$
Stainless Steel			1977, 1980)
	503 K (230°C)/~40,000 hrs./ST	35	
N-Octane/Mild Steel	(Groll, 1982, 1987, 1989)		
	523 K (250°C)/~40,000 hrs./321	SS	
N-Octane/Stainless	(Groll, 1982, 1987, 1989)		
	493 K (220°C)/~40.000 hrs./ST	35	
Dowtherm E	(Groll, 1982, 1987, 1989)		
Octafluoronanhthalene/	$482 \text{ K} (209^{\circ}\text{C})/13 400 \text{ hrs} /6061 \text{ A1}$		
	Some NCG (Saaski 1977 1980)		
Addining Additional Ad	Some ived (Saaski 1977, 1960)		489 K (2159C)/12 400 hm / 179
			460 K (213 C)/13,400 IIIS./A178
			Some NCG (Saaski 1977, 1980)
			Cycle 598 K to 653 K $(325^{\circ}C \text{ to } 380^{\circ}C)/5,520$
			hrs./ 316 SS (Grzyll, 1991, 1994, 1995)
			623 K (350°C)/ 5,520 hrs./316 SS (Grzyll,
Quinoline/SS			1991, 1994, 1995)
Monochloronaphthalene			560 K 287°C/642 hrs./A178 (Saaski 1977,
/Stainless Steel			1980)
			553 K (280°C)/15 hrs/304 SS/ (Kenney/
Formyl-piperidine/SS			Feldman)
			723 K (450°C)/<96 hrs/304 SS (Kenney,
P-Terphenyl/SS			1978)
			673 K (400°C)/~9 000 hrs /13CrMo44 (Groll
			1982 1987 1989)
ortho and mota			$503 K (320^{\circ}C)/.000 \text{ hrs} / 13 CrMo44 (Groll)$
termhanul/Mild Steel			$1092 \ 1097 \ 1090$
			(72, 1907, 1909)
			6/3 K (400°C)/~9,000 hrs./316L SS (Groll,
			1982, 1987, 1989)
ortho- and meta-			$623 \text{ K} (350^{\circ}\text{C})/~9,000 \text{ hrs./316L SS (Groll,}$
terphenyl/SS			1982, 1987, 1989)
			673 K (400°C)/~9,000 hrs./13CrMo44 (Groll,
			1982, 1987, 1989)
diphenyl, ortho- and meta-			623 K (350°C)/~9,000 hrs./13CrMo44 (Groll,
terphenyl/Mild Steel			1982, 1987, 1989)
			673 K (400°C)/~9,000 hrs./316L SS (Groll,
			1982, 1987, 1989)
diphenyl, ortho- and meta-			623 K (350°C)/~9,000 hrs./316L SS (Groll.
terphenyl/SS			1982, 1987, 1989)
Perfluoro-1.3-5-			Severe thermal decomposition at 573 K
triphenylbenzene			$(300^{\circ}C)$ (Grzvll, 1991, 1994, 1995)
	1		

. Table 3. (continued) Summary of Organic Fluid Life Tests Other Than Diphenyl and Diphenyl Oxide

The most commonly tested organic fluids have been diphenyl, diphenyl oxide, and a eutectic mixture of diphenyl/diphenyl oxide (Trade Names Dowtherm A, Therminol, and Diphyl). Eutectic diphenyl/diphenyl oxide is nearly an azeotrope (Basilius and Prager, 1975), so the liquid and vapor have almost the same composition. This avoids the problems encountered with other mixtures such as NaK, where fractional distillation can occur (Anderson, 1993). Life test results for these three fluids are summarized in Table 2.

When using diphenyl, diphenyl oxide, or diphenyl/diphenyl oxide at temperatures over 673 K (400°C), noncondensable gas is generated in a relatively short time periods shown in Table 2. The exact period depends upon the fluid and material and decreases as the temperature increases. For example, Kenney and Feldman found that their diphenyl pipes took less than 72 hours to gas up at 748 K (475°C), and 366 hours to gas up at 695 K (422°C). Between 573 and 673 K (300 and 400°C), these fluids are generally suitable, for short duration tests near 673 K, and long duration tests near 573 K. For example, Groll et al. found that 321 SS was compatible for ~40,000 hours at 573 K (300°C), but not at 623 K (350°C).

Life tests results for organic fluids other than diphenyl and diphenyl oxide are summarized in Table 3. Fluids have been ranked by the highest temperature for a compatible life test with any envelope material.

Since all of their life tests to date have been compatible, two fluids stand out in Table 3: toluene and naphthalene. Toluene was compatible with a copper-nickel alloy, CuNi10Fe, at 553 K (280°C), as well as with aluminum, mild steel, stainless steel, and titanium at lower temperatures. This is probably close to the maximum useful range of toluene, since the critical point of toluene is 592 K (319°C).

Water is generally a better working fluid, since it can also be used in this temperature range, and has a Merit number that is roughly 50 times higher than toluene. However, toluene has three advantages over water, which may make it a suitable choice for certain conditions. The advantages are:

- Compatibility with a larger number of envelope/wick materials
- Melting temperature of 178 K (-95°C) versus 273 K (0°C)
- Lower saturation pressure (e.g., 23.4 atm. at 550 K versus 60.4 atm. for water)

Naphthalene is compatible with stainless steel, copper-nickel, and titanium, based on long term life tests at 593 K (320° C) and above. It has also been shown to be compatible at lower temperatures with aluminum and mild steel. It was compatible with Alloy 20 stainless steel for short term tests at 380° C.

While fluorinated compounds have been theorized to be more stable than the same compound without fluorine, this has not been verified in life tests date. Gryzll, Back, Ramos, and Samad, (1994) found that Decafluorobyphenyl $(C_{12}F_{10})$ was less stable than Diphenyl $(C_{12}H_{10})$ under the same test conditions. Perfluoro-1,3,5-triphenylbenzene underwent severe thermal decomposition. Naphthalene was compatible with mild steel at 623 K (350°C) for 5,520 hours, while Monochloronaphthalene was found to be unsuitable after 642 hours at 560 K (287°C), and Octafluoronaphthalene had NCG gas generation at 488 K (215°C). Other stable, fluorinated life tests have been conducted at temperatures of 530 K (257°C) and below.

C. Halides

A halide is a compound of the type MX, where M may be another element or organic compound, and X may be any of the Group 17 elements: fluorine, chlorine, bromine, iodine, or astatine. Starting with Saaski and Owarski (1977), a number of researchers have suggested that halides are potential heat pipe fluids. They are attractive because they are more stable at high temperatures than organic working fluids, and because their Merit number peaks in the intermediate temperature range. Information on halide properties can be found in Anderson, Rosenfeld, Angirasa, and Mi (2004) and Devarakonda and Anderson (2005).

Saaski and Owzarsky (1977) proposed an electrochemical method to predict the compatibility of halide working fluids with envelope materials. Tarau, Sarraf, Locci and Anderson (2007) found that this procedure had good agreement with the halide life tests discussed above.

Halide life tests are summarized in Table 4. Some halides appear to be suitable for temperatures up to 673 K (400°C), and possibly at higher temperatures. Tests are ongoing with $TiBr_4/titanium$ at 653K (380°C), and with AlBr₃/Superalloys at 673K (400°C). Very long term life tests show that $TiCl_4$ and $SnCl_4$ are both compatible with mild steel. No tests to date with an aluminum envelope have been successful. This is due to the very high decomposition potential of aluminum when compared to other metals (Tarau, 2007).

	Compatible	Incompatible				
		653K (380°C)/20,040 hours/CP-Ti				
TiBr ₄ /Titanium		(current paper)				
	432 K (159°C)/28,540 hrs./A-178 Steel (Saask	i				
TiCl ₄ /Mild Steel	1977, 1980)					
	573K (300°C)/ 59,184 hrs./Hastelloy (curren	t				
TiCl ₄ /Superalloy	paper)					
TiCl ₄ /Titanium	500 K (227°C)/4,019 hrs./CP-Ti (Locci, 2005)					
		438 K (165°C)/2,500 hrs./Al-6061				
TiCl ₄ /Aluminum		(Saaski 1977, 1980)				
		432 K (159°C)/ hrs./Al-6061 (Saaski				
SnCl ₄ /Aluminum		1977, 1980)				
	429 K (156°C)/27,7500 hrs./A-178 Steel (Saask	i				
SnCl ₄ /Mild Steel	1977, 1980)					
		553K (280°C)/20,160 hrs./Hastelloy				
SnCl ₄ /Superalloy		(current paper)				
	673K (400°C)/ 58,992 hrs./Hastelloy (curren	t				
	paper)					
AlBr ₃ /Superalloy						
		500 K (227°C)/4,290 hrs./Al-5052				
		(Locci, 2005)				
		500 K (227°C)/1,100 hrs./Al-6061				
AlBr ₃ /Aluminum		(Locci, 2005)				
		500 K (227°C)/1,100 hrs./CP2-Ti				
AlBr ₃ /Titanium		(Locci, 2005)				
		633K (360°C)/ hrs./Hastelloy C-22				
GaCl ₃ /Superalloy		(current paper)				
		613K (340°C)/19,632 hrs./CP-Ti				
GaCl ₃ /Titanium		(current paper)				
		500 K (227°C)/ hrs./Al-6061 (Saaski				
SbCl ₃ /Aluminum		1977, 1980)				
		476 K (203°C)/5,000 hrs./A-178 Steel				
SbCl ₃ /Mild Steel		(Saaski 1977, 1980)				
		500 K (227°C)/5,000 hrs./Al-6061				
SbBr ₃ /Aluminum		(Locci)				

Table 4. Halide Life Test Summary.

III. Experimental Procedure

A. Water Life Tests

Titanium, titanium alloys, Monel 400, and Monel K500 have higher yield strength and lower density than copper. As discussed above, they have been shown to be compatible with water, hence can be used in thinner and lighter weight heat pipes than copper at a given operating temperature and working fluid vapor pressure. Anderson, Dussinger, Bonner, and Sarraf (2006) started a series of life tests with commercially pure (CP) titanium, titanium alloys, Monel 400, and Monel K-500. The life test results are updated below. The materials under test include:

- Ti CP-2 Heat Pipe, with CP Titanium Screen
- Monel K500 Heat Pipe, with Monel 400 Screen
- Ti Grade 5 Cylinder (6% Aluminum, 4% Vanadium), with CP Titanium Screen
- Ti Grade 7 Cylinder (0.2% Pd), with CP Titanium Screen
- Ti CP-2 Cylinder, with 21S foil and CP Titanium Screen (21S tubing was not available)
- Ti Grade 9 cylinder (3% Aluminum, 2.5% Vanadium) with CP Titanium Screen
- Ti CP-2 Heat Pipe, with Sintered Cylindrical Wick

- Ti CP-2 Heat Pipe, with Integral Grooves
- Monel 400 Heat Pipe, with Monel 400 Screen
- Monel K500 Heat Pipe, with sintered Monel 400 wick
- Monel 400 Heat Pipe, with sintered Monel 400 wick

The heat pipes with integral titanium grooves are intended for spacecraft thermal control. A typical cross-section is shown in Figure 1. Three integrally grooved heat pipes are currently on life test at NASA Glenn Research Center (Sanzi and Jaworske, 2011).



Figure 1. Titanium Heat Pipe with Integral Grooves for spacecraft thermal control.

Initial Quantity	Wall Material	Wick	Operating Temperature	Operating Hours May 6 2013
4	Monel K 500	200x200 Monel 400 Screen 0.064 mm wire	550 & 500 K	72,192 hours
4	CP-2 Ti	150x150CP-Ti Screen 0.069 mm wire	550 & 500 K	72,192 hours
2	CP-2 Ti	Sintered Titanium -35+60 Mesh CP-2	550 K	60,672 hours
2	CP-2 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	61,064 hours
1	CP-2 Ti	Integral Grooves	550 K	41,345 hours
2	CP-2 Ti 21 S Foil Inside	100 x100 CP-Ti Screen 0.05 mm wire	550 K	62,622 hours
2	Grade 5 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	69,845 hours
2	Grade 7 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	60,672 hours
2	Grade 9 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	60,072 hours
2	Monel 400	120x120 Monel 400 Screen 0.05 mm wire	550K	60,168 hours
2	Monel K 500	120x120 Monel 400 Screen 0.05 mm wire	550K	67,536 hours
2	Monel 400	-100+170 Mesh Monel 400 Powder	550K	58,824 hours
2	Monel K 500	-100+170 Mesh Monel 400 Powder	550K	57,792 hours

Table 5. Titanium-Water and Titanium-Monel Life Test Pipes – Operating Hours as of May 6, 2013.

Table 5 shows the different life test pipes on test. Monel 400 is a solid solution alloy with roughly 63% nickel and 30% copper. It is a single-phase alloy, since the copper and nickel are mutually soluble in all proportions. It can only be hardened by cold working. Monel K500 is a similar nickel-copper alloy, with the addition of small amounts of aluminum and titanium that give greater strength and hardness. The system is age-hardened by heating so that small particles of Ni_3 (Ti, Al) are precipitated throughout the matrix, increasing the strength of the material. The advantage of Monel K-500 is that the strength can be partially recovered after a wick is sintered inside.

B. Halide Life Tests

A series of halide life tests are ongoing at ACT. The selection criteria were discussed in Anderson et al. (2007). The fluid/envelope combinations tested and the operating conditions are shown in Table 6. The titanium pipes had a 50 x 50 mesh titanium screen wick, and the C-22 pipes had an 80 x 80 mesh C-22 wick. The other two types of pipes were bare. Note that all of the superalloy pipes had C-22 endcaps and fill tubes (due to availability).

The operating temperatures in Table 6 were set based on the vapor pressure, and the allowable stresses in each heat pipe as a function of temperature. During the life tests, the temperature of the evaporator and condenser for each heat pipe are monitored, to detect any problems. It is possible that oxygen can affect the outside of the titanium pipes during the test. To prevent this problem, the life tests are conducted inside a box that is purged with argon. During the life test, heat pipe temperatures are monitored to detect the formation of non-condensable gas.

The superalloy/GaCl₃ pipes all leaked at the pinchoff weld after roughly one week of operation at 360°C (633K). Note that all of the superalloy pipes used a C-22 fill tube, since that was more readily available. After roughly 11,000 hours, the Hastelloy B3 pipe with AlBr₃, apparently at a weld. Note that this failure is probably due to the pipe being severely overheated after the first 300 hours of testing when the heater shorted out. The maximum temperature is not known, however, it was sufficient to bubble the aluminum heater block. The titanium/GaCl₃, titanium/TiBr₄, and superalloy/SnCl₄ pipes all developed large amounts of non-condensable gas. After roughly 20,000 hours of operation, the pipes were taken off life test, and stored for the analyses discussed below.

One superalloy/TiCl₄ and one superalloy/AlBr₃ pipe continue to run without any problem (the others were taken down for destructive analysis). These pipes have currently been running for 57,000 hours (6.7 years). The AlBr₃ pipes is of particular interest, since it is running at 673 K (400°C). This is close to the temperature at which cesium starts to work.

Working Fluid	CP-Ti	C22	C2000	B3
		400°C (673K)		
AlBr		28,704 hrs	400°C (673K)	
AIDI ₃		Low Gas	58,992 hrs	400°C (673K)
	-	Fluid Analysis	Low Gas	$\sim 11,000 \text{ hrs (Fail)}^8$
	340°C (613K)			
GaCl ₃	19,632 hrs			
	Shipped to NASA	Fail	Fail	Fail
		280°C (553K)	280°C (553K)	280°C (553K)
SnCl_4		20,160 hrs	20,160 hrs	20,160 hrs
		High Gas	High Gas	High Gas
	CP-I1 400°C 28,7 Lo - Fluid 340°C (613K) 19,632 hrs 19,632 hrs 280°C Shipped to NASA 280°C 20, Hig - Fluid 300°C 28,5 Lo Fluid 300°C 28,5 Lo Fluid 380°C (653K) 20,040 hrs High Gas Fluid Analysis	Fluid Analysis	Fluid Analysis	Fluid Analysis
		300°C (573K)		
TiCl		28,560 hrs	300°C (573K)	300°C (573K)
11C14		Low Gas	59,184 hrs	58,200 hrs
	-	Fluid Analysis	Low Gas	Low Gas
	380°C (653K)			
T:D.	20,040 hrs			
11D14	High Gas			
	Fluid Analysis	—	—	-

Table 6. Current Halide Life Test Pipe Temperatures and Operating Times (May 6, 2013).

 $^{^{8}}$ AlBr₃/B3 pipe was severely overheated at 300 hours testing

C. Heat Pipe Sectioning and Analysis

In late 2010, several of the heat pipes were selected for destructive investigation, see Table 6. One of each pair of water life test pipes was selected, while the other one continued on life test. The GaCl₃ and SnCl₄ pipes were known to be non-compatible, since they generated large amounts of NCG. The heat pipes containing halides were neutralized with water to form a stable sediment. The sediment from the halides, and the water from the heat pipes were collected for chemical analysis. No fluids analysis was done on Pipe 4, CP-Ti/TiBr₄, since titanium is the only metal present in the fluid and envelope.

To examine the cross-sections to determine the type and amount of corrosion in the wicks and heat pipes, the heat pipes were cut in half, pressure infiltrated with epoxy and sectioned at a location approximately one-third of the way above the bottom of the heat pipe. The sections were polished through 0.05-micrometer silica and examined using optical and scanning electron (SEM) microscopes.

Heat				
Pipe	Envelope	Wick	Working Fluid	Life Test Hours
4	CP Ti	50 x 50 mesh CP-Ti Screen	TiBr ₄	20,040
6	Hastelloy B-3	None	SnCl ₄	20,160
7	Hastellov C 22	80 x 80 C22 Saman	SpCl	20.160
/		80 X 80 C22 Screen		20,100
8	Hastelloy C-2000	None	SnCl ₄	20,160
9	CP Ti	None	GaCl ₃	20,040
10	CP Ti	50x50 mesh CP-Ti	GaCl ₃	20,040
153	Hastelloy C-2000	None	TiCl ₄	28,560
157	Hastelloy C-22	80 x 80 C22 Screen	AlBr ₃	28,704
100	CP Ti	150x150 CP Ti Screen	Water	48,100
103	CP Ti	150x150 CP Ti Screen	Water	48,100
105	Monel K500	200x200 Monel 400 Screen	Water	48,100
107	Monel K500	200x200 Monel 400 Screen	Water	48,100
121	CP Ti	Sintered CP-2 -35+60 Mesh	Water	39,701
122	Ti Grade 7	100x100 CP Ti Screen	Water	39,701
123	Ti Grade 5	100x100 CP Ti Screen	Water	42,528
124	CP Ti with Timetal 21-S Strip	100x100 CP Ti Screen	Water	39,917
133	Monel K500	Monel 400 -100 +170 Sinter	Water	34,344
134	Monel 400	120x120 Monel 400 Screen	Water	35,040
135	Ti Grade 9	100x100 CP Ti Screen	Water	35,544
136	Monel	120x120 Monel 400 Screen	Water	34,992

 Table 7. Heat Pipes Selected for Destructive Evaluation.

IV. Results and Discussion

A. Examination of Cross-sections

1. Titanium-Water Heat Pipes

Analysis of the titanium/water heat pipe cross-sections using optical microscopy revealed little if any corrosion when observed at high magnifications. Even using differential interference contrast, it was difficult to find any corrosion layer. When any evidence of corrosion was observed, the layer was typically ~1 micrometer thick. SEM imaging and EDS analysis also did not indicate any substantial corrosion layer.

The Timetal-21S strip placed in Heat Pipe 124 had apparent differences between the surface and the center as shown in Figure 2. On the side that faced the envelope, the layer extends approximately 25 micrometers while on the side facing the center of the heat pipe the layer extends only about 10 micrometers. EDS analysis indicated that there may be depletion of Mo in the layers, but the difference between the center and layers was sufficiently small to be inconclusive.

Heat Pipes 103, 124 and perhaps 122 also had evidence of a layer on the surface of the wires of the wick and envelope. An example is shown in Figure 3. A layer approximately 5 to 10 microns thick that is not mottled like the core can be observed in BSE images. The layer does not have a detectable difference in chemical composition. It is hypothesized that there has been a change in the grains that has reduced the contrast between grains. This is not caused by thermal exposure since the core does not exhibit the same change. Instead, it is most likely caused by the introduction of interstitial atoms of O and H into the Ti at levels too low to detect by EDS.

It was noted in several cases such as shown in Figure 4 that the wires and, in some cases, envelopes had a very rough surface. It was not possible to compare these surfaces to unexposed samples made from the same stock, so it is not clear if the roughness was caused by drawing of the wire and tubing used. There does not appear to be any corrosion product in the crevices observed in the wires, so it is most likely that these are caused by drawing. Additional investigation is required to confirm this.



Figure 2. BSE Image of Timetal 21-S Strip in Heat Pipe 124 Showing Possible Reaction Layer.



Figure 3. BSE Image of CP Ti Mesh Wire Wick in Heat Pipe 124 (Arrow denotes possible reaction layer)



Figure 4. BSE Image of CP Ti Mesh Wire Wick in Heat Pipe 103 Highlighting Surface Roughness Of Wire.

2. Titanium-Halide Heat Pipes

The two titanium halide heat pipes examined had very different responses. Heat Pipe 4, which had CP Ti with a $TiBr_4$ working fluid, had minimal corrosion. Like Heat Pipes 103 and 124, there was evidence of some change at the surface as the BSE images showed the outer 10 micrometers had notably less mottling than the core.

Heat Pipe 10, which had a CP Ti with $GaCl_3$ working fluid, underwent extensive corrosion as seen in Figure 5. Extensive voids and cracking were observed in the corrosion layer. EDS analysis indicated that the corrosion layer was a Ga-29.7 wt.% Ti alloy. Examination of the Ga-Ti phase diagram (National Physical Laboratory, 2012) showed that the composition is similar to the Ga₂Ti phase. Immediately adjacent to the Ga₂Ti phase on the phase diagram is Ga(1) and Ga₃Ti. Given the extensive nature of the voids, particularly on the wires, the Ga(1) and Ga₃Ti(s) phases may have been present in these voids. During neutralization of the halide, the Ga(1) and any Ga₃Ti particles within it could have been physically removed, leaving behind the voids.

Some of the corrosion layer was observed to crack and chip during polishing. The fracture surfaces were indicative of a brittle failure mode. In combination with the extensive cracking of the corrosion layer, this seems to indicate that the corrosion layer is quite brittle.



Figure 5. BSE Image of Heat Pipe 10 (CP Ti-GaCl₃) Showing Ga-Ti Reaction Layer

3. Monel-Water Heat Pipes

Several water working fluid heat pipes made with Monel K500 (Ni-30Cu-2Fe-2.7Al-0.6Ti) envelopes and Monel 400 (Ni-31Cu-2.5Fe-2Mn) wicks were tested at varying times and temperatures. While the heat pipes performed well overall, there were some surprises when the heat pipe cross-sections were examined.

Figure 6 shows an optical micrograph of the envelope and wick for Heat Pipe 136, one of the heat pipes that underwent the most change. Extensive changes, including the formation of a dark subsurface layer and bright nodules, were observed in the Monel 400 wick. While the change was the most extensive for the Monel-Water heat pipes, the morphology was typical of all Monel 400 wire mesh and powder wicks as well. Heat Pipe 134, which has a Monel 400 envelope, shows a similar dark subsurface layer. It is much thinner, on the order of 1 to 8 micrometers thick, and variable in thickness.

The Monel K500 envelope does not show an extensive change. Close examination of the envelope reveals, at most, a thin (5-10 micrometer) corrosion layer. Most likely the layer was an oxide, but it was sufficiently thin to prevent definitive identification through EDS.

Figure 7 shows a detail of one of the Monel 400 wires from Heat Pipe 107 (Monel K500-Monel 400 wick-water). EDS spot analysis was conducted on the surface nodules, the dark subsurface layer and the matrix. The results showed that the surface nodules were essentially pure Cu, the subsurface layer was Cu-depleted, and the matrix even adjacent to the dark layers retained essentially the same composition as the bulk composition of Monel 400. Additional examination of this and other heat pipes with Monel 400 screens indicated that the dark layer could be between 2 wt.% and 23 wt.% Cu compared to 31 wt.% Cu for the bulk alloy.

Examination of the Cu-Ni phase diagram (ASM International, 1992) shows that while Cu and Ni form a solid solution, below about 627 K (354 °C) there is a decomposition of the α phase to $\alpha_1 + \alpha_2$ phases. This decomposition creates a Cu-rich and a Ni-rich phase. It appears that this is the driving force for the separation of the Cu and Ni that is observed. Apparently the activity gradient was such that the Cu-rich phase preferentially moved through diffusion to the surface. The addition of several alloying elements to Monel K500 appeared to stabilize the α phase and prevent this decomposition. While no large amounts of oxygen were observed, preferential oxidation of the Ni may have also played a role in the development of the observed morphology and phases.

Since both Monel 400 and Monel K500 are extensively used in steam plant operations, the literature was searched to find similar observations. So far, no such reference has been located. The literature search is continuing so that this phenomena can be better understood.



Figure 6. Optical Bright Field Micrograph of Heat Pipe 136 (Monel K500 Envelope, 120 Mesh Monel 400 Wick and Water Working Fluid)



Figure 7. BSE Image of Monel 400 Wire In Heat Pipe 107 (Monel K500-Monel 400 wick-Water).

4. Hastelloy C-Series Superalloy-Halide Heat Pipes

Four corrosion-resistant Hastelloy C-series superalloy heat pipes were examined. Hastelloy C-22 and C-2000 were paired with $SnCl_4$ as a working fluid in Heat Pipes 7 and 8 respectively. Heat Pipe 153 used a Hastelloy C-2000 envelope with a TiCl₄ working fluid, and Heat Pipe 157 paired Hastelloy C-22 with AlBr₃ as a working fluid.

Heat Pipe 7 exhibited considerable roughening of the surface (Figure 8) and surface cracks/crevices that extended about 20 micrometer into the envelope (Figure 9). As seen in Figure 10, a corrosion layer about 10 micrometers thick was observed on the surface of the envelope. This layer did not appear to be protective as it had extensive voids, but it did appear somewhat adherent.

BSE imaging of the interface revealed the presence of a lighter phase as seen in Figure 11. EDS spot analysis showed that the lighter phase was a Mo-W-Sn phase. It appears that the Sn from the $SnCl_4$ can react with the Mo and W of the Hastelloy C-22 under the heat pipe operating conditions.



Figure 8. Differential Interference Contrast Optical Micrograph of C-22 Envelope In Heat Pipe 7 Showing Roughening and Corrosion Layer (Hastelloy C-22-80 mesh Hastelloy C-22 Wick-SnCl₄)



Figure 9. SE Image of C-22 Envelope In Heat Pipe 7 Showing Typical Crevice (Hastelloy C-22-80 mesh Hastelloy C-22 Wick-SnCl₄)



Figure 10. SE Image of C-22 Envelope In Heat Pipe 7 (Hastelloy C-22-80 mesh Hastelloy C-22 Wick-SnCl₄)



Figure 11. BSE Image of C-22 Envelope In Heat Pipe 7 Showing Presence Of Lighter Mo-W-Sn Phase (Hastelloy C-22-80 mesh Hastelloy C-22 Wick-SnCl₄)

The Hastelloy C-2000 envelope of Heat Pipe 8 also underwent extensive reaction with the $SnCl_4$ working fluid. A 200 micrometer thick corrosion layer shown in Figure 12 was observed. The layer showed extensive cracking including cracking at the interface, which indicates it may not be adherent or has a large coefficient of thermal expansion mismatch with the Hastelloy C-2000 substrate. EDS analysis of the corrosion layer revealed that it was primarily Ni and Sn with some Cl. It appears to be Ni₃Sn₂ modified by reaction or incorporation of about 9 wt.% Cl and minor amounts of Cr, Cu, Fe and Mn from the Hastelloy C-2000. EDS analysis of the matrix immediately beneath the corrosion layer did not indicate any change in the composition from the bulk composition, indicating that there was no depleted zone beneath the corrosion layer.

In addition to the Ni-Sn-Cl corrosion layer, Mo-Cl particles were observed during X-ray mapping. These generally occurred at the interface between the corrosion layer and the substrate. It was not possible to perform an EDS analysis on the particles due to their small size relative to the excitation volume of the SEM beam, but the X-ray maps qualitatively indicated that considerably more Cl was present relative to the corrosion layer.



Figure 12. BSE Image of C-2000 Envelope In Heat Pipe 8 Showing Presence Of Thick Ni-Sn-Cl Reaction Layer. (Hastelloy C-2000-SnCl₄)

Heat Pipe 153 paired Hastelloy C-2000 with TiCl₄ as the working fluid. SEM observations such as the image shown in Figure 13 revealed a 1 to 2 micrometer thick corrosion layer on the surface. The thickness of the layer varied as the outer surface was not planar. EDS analysis showed the reaction layer consisted of Ni-33 wt.% Ti-18 wt.% Mo-18 wt.% Cr-4 wt.% Cu-2 wt.% Cl. The layer, while non-uniform does appear to be somewhat protective and adherent.

High magnification BSE images of the interface such as the one shown in Figure 14 showed the presence of a very thin (~0.5 micrometer) thick layer beneath the reaction layer that was darker than the surrounding matrix. The thinness of the layer prevented good EDS analysis, but the BSE indicates that there is a diffusion zone beneath the reaction layer that has lost the heavier elements such as Mo. This is consistent with the 18 wt.% Mo observed in the reaction layer.



Figure 13. SE Image of C-2000 Envelope In Heat Pipe 153 Showing Presence Of Reaction Layer (Hastelloy C-2000-TiCl₄)



Figure 14. SE Image of C-2000 Envelope In Heat Pipe 153 Showing Presence Of Reaction Layer (Hastelloy C-2000-TiCl₄)

Heat Pipe 157, which had a Hastelloy C-22 envelope and wick with AlBr₃ working fluid, exhibited a dual corrosion layer with a total thickness of 5 to 10 micrometers. The two corrosion layers were about equal in thickness, but there is variability due to a wavy interface between the two layers. EDS analysis of the two layers showed that the outer layer composition was Ni-11.5 wt.% Cr-11.9 wt.% Mo-3.6 wt.% Fe-9.4 wt.% W-0.6 wt.% Mn-1.7 wt.% Co-0.3 wt.% V-0.8 wt.% Si-9.5 wt.% Br. The inner corrosion layer composition was Ni-12.8 wt.% Cr-12.4 wt.% Mo-3.2 wt.% Fe-6.4 wt.% W-0.2 wt.% Mn-1.3 wt.% Co-0.3 wt.% V-21.9 wt.% Br. Spot EDS analysis immediately beneath the corrosion layer showed the presence of 1 wt.% Br, indicating that the Br may be diffusing into the metal substrate. Based upon these analyses, it appears that Br can react with the C-22, but it takes a considerable length of time to build up the corrosion layers., in this case 28,560 hours.

As shown in Figure 15, there is evidence of through-thickness cracking in the inner corrosion layer. This may be caused by a CTE mismatch between this layer and the substrate since there is no continuation of the crack into the outer layer and there is no evidence of any reaction on the surfaces of the cracks or development of corrosion product within the cracks. If this is the case, operating the heat pipes isothermally should result in no cracking and a relatively protective corrosion layer.



Figure 15. SE Image of C-22 Envelope In Heat Pipe 157 Showing Two Corrosion Layers (Hastelloy C-22-AlBr₃)

B. Chemical Analysis Of Working Fluids

Table 7 contains the results of the chemical analysis of the working fluids. Only the elements that could be present from dissolution of the metals are listed. Since the halides are reactive, they were chemically neutralized with water prior to chemical testing to allow safe testing of the working fluids. The stable sediment was then sent out for analysis.

The chemical analyses of the heat pipes that use water as a working revealed that there was some pickup of metal from the metals, most notably Cu for the Monel heat pipes. However, the levels were in the very low ppm range and represent very minimal contamination of the water. There was no evidence of corrosion that resulted in the movement of metal from the envelopes and wicks to the working fluid. For the Monel samples, this is consistent with the diffusion of Cu to the surface rather than a dissolution/precipitation process for creating the large Cu surface nodules.

The heat pipes that used halides as a working fluid showed more contamination of the working fluids. Heat pipes 153 and 157 which appeared to form a protective corrosion layer showed some of the lowest amounts of contamination. the total contamination was on the order of 300 to 350 ppm. While indicating some corrosion occurred, the amount is small and should be relatively insignificant.

All of the heat pipes with a high gas content showed had 1% or more or the envelope constituents dissolved in the halide. In comparison to Heat Pipes 153 and 157, Heat Pipes 7 and 8 which used $SnCl_4$ suffered considerably more contamination of the working fluids with Cr being the major metal present though traces of most constituents of the alloys used for the envelopes and wicks are observed. The relative amounts seem to be consistent with the levels of attack observed with Heat Pipe 8 undergoing much more attack and reaction than Heat Pipe 7.

Since titanium was the only metal in both the envelope and fluid, No fluid analysis was made for Heat Pipe 4 (CP-Ti/TiBr₃), which had little evidence of attack. The high level of Ti in the GaCl₃ for Heat Pipe 10 is consistent with the large amount of corrosion and possible Ti-containing particles in the working fluid. Recall that this pipe developed a leak in the first few hours after it was put on life test.

Lloot Dino	Working	Life Test	A1	Co	Cr	Cu	Гo	Ma	Ma	NI	T:	M	10/
Heat Pipe	Fiulu SnCL	H0015	AI	CO		Cu	re 0.020	0.012	IVIU 0.70	1 70	11	V	0.02
7	SnCL	20,100	0.007	0.001	0.30		0.038	0.012	0.79	0.027			0.02
, 0	SnCl	20,100	0.000	0.001	0.11	0.022	0.004	0.007	0.003	0.027			0.001
0	GaCL	20,100	0.005	0.007	0.7	0.022	0.016	0.003	0.31	0.03	1.2		0.001
7 10	GaCl.	20,040									1.2		
152	TiCL	20,040			0.006		0.027			0.003	1.2		
155	ΔIRr.	20,300			0.000		0.027			0.003			
137	AIDI 3	20,704			0.013		0.010			0.002			
100	Water	48,100									0.00013		
103	Water	48,100									0.000016		
105	Water	48,100	0.000007			0.0011	0.000031	0.00021		0.00056	0.000008		
107	Water	48,100	0.000005			0.0021	0.00002	0.0016		0.00041	0.000006		
121	Water	39,701									0.000018		
122	Water	39,701									0.000012		
123	Water	42,528	0.000011								0.000025		
124	Water	39,917									0 000037		
	Mator	07,717									0.000007		
133	Water	34,344	0.000007			0.000021							
134	Water	35,040		0.000005		0.00064	0.000015	0.00095		0.00011			
135	Water	35,544	0.000007								0.000062	0.000005	
Di Water STD	Water		<0.00005	<0.00005		<0.00005	<0.00005	<0.00005		< 0.000005	< 0.00005		

Table 8. Contaminants Found In Working Fluids (weight percent).

V. Conclusion

A survey was conducted for intermediate temperature life tests. Life tests have been conducted with 30 different intermediate temperature working fluids, and over 60 different working fluid/envelope combinations. Life tests have been run with three elemental working fluids: sulfur, sulfur-iodine mixtures, and mercury. Other fluids offer

benefits over these three liquids in this temperature range. Mercury is toxic, has a high density, and problems have been observed with getting the mercury to wet the heat pipe wick. Sulfur and Sulfur/Iodine have high viscosities, low thermal conductivities, and are chemically aggressive.

Life tests have been conducted with 19 different organic working fluids. As the temperature is increased, all of the organics start to decompose. Typically they generate non-condensable gas, and often the viscosity increases. At high enough temperatures, carbon deposits can be generated. The maximum operating temperature is a function of how much NCG can be tolerated, and the heat pipe operating lifetime. Three sets of organic fluids stand out as good intermediate temperature fluids:

- 1. Diphenyl, Diphenyl Oxide, and Eutectic Diphenyl/Diphenyl Oxide (Dowtherm A, Therminol VP, Diphyl)
- 2. Naphthalene
- 3. Toluene

A non-organic working fluid is desirable for nuclear fission space power and other applications where radioactivity can generate gas with organic working fluids. Long term life tests show that Superalloys/TiCl4₄ at 573 K (300°C), and Superalloys/AlBr₃ at 673K (400°C) are compatible. As of May 2013, the AlBr₃ and TiCl₄ tests have been running for over 59,000 hours (6.7 years).

Hastelloy C-2000 underwent little corrosion when used with TiCl4 working fluid, with the formation of only a 1-2 micrometer thick corrosion layer. Hastelloy C-22 exhibited a 5-10 micrometer thick dual corrosion layer when tested with AlBr3 working fluid. The working fluids of these two heat pipes exhibited total metal contents between 300 and 350 ppm. The results indicate that the tested envelope materials and working fluids can form viable material/working fluid combinations.

Titanium/water and Monel/water heat pipes are compatible at temperatures up to 550 K, based on ongoing life tests that have been running for up to 72,000 hours (8.2 years) as of May 2013. Analysis of titanium/water heat pipe cross-sections using optical and electron microscopy revealed little if any corrosion even when observed at high magnifications. When any evidence of corrosion was observed, the layer was typically around 1 micrometer thick. Copper depleted zones, as well as copper surface nodules formed on the Monel 400 screen wick. This was not observed on the Monel K500 envelopes. An analysis of the water working fluids showed minimal pickup of metals.

Acronyms

- BSE Backscatter Electron (Image)
- EDS Energy Dispersive Spectroscopy
- SE Secondary Electron (Image)

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