Intermediate Temperature Heat Pipe Life Tests

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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, fuel cells, geothermal power, waste heat recovery systems, and high temperature electronics cooling. Titanium/water and Monel/water heat pipes are suitable for temperatures up to 550 K, based on life tests that have been running for over 54,000 hours (6.1 years). At higher temperatures, organic or halide working fluids can be used. Long term life tests (currently 50,000 hours or 5.7 years) show that Titanium/$TiBr_4$ at 653 K, and Superalloys/$AlBr_3$ at 673 K are compatible. These results are confirmed by optical and electron microscopy, and working fluids analysis on heat pipes chosen for destructive examination.

KEY WORDS*:* Intermediate Temperature Heat Pipe Life Tests, Halide Life Tests, Water Life Tests

1. 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). A survey of previous life tests on intermediate temperature working fluids can be found in Anderson (2007) and Anderson et al. (2010).

2. EXPERIMENTAL PROCEDURE

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, titanium alloys, Monel 400, and Monel K500 heat pipes. The test setup for the titanium/water and Monel/water life tests are discussed in Anderson et al. (2006). As of April 2012, several of the titanium and Monel heat pipes have been on test for over 54,000 hours (6.1 years). Periodically, the temperature is lowered to 343 K, and the pipes are tested for Non-Condensable Gas (NCG). A small amount of NCG has been noted in these pipes at 70°C, but the gas cannot be detected at the operating temperature of 550 K.

At temperatures above 550 K, the surface tension of water is so low, and the vapor pressure is so high, that water is no longer an attractive fluid. At higher temperatures, 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.

Table 1. Heat Pipes Selected for Evaluation.

Heat Pipe	Envelope	Wick	Working Fluid	Life Test Hours
6	Hastelloy B-3	None	$SnCl_4$	20,160
7	Hastelloy C-22	80 x 80 C22 Screen	$SnCl_4$	20,160
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

In some cases, a non-organic working fluid is desirable, particularly for nuclear fission space power, where the radioactivity with organic working fluids can generate gas. Since 2006, we have been conducting life tests at temperatures up to 673 K with titanium and three corrosion resistant superalloys (Hastelloy B-3, Hastelloy C-22, and Hastelloy C-2000), and five different halides working fluids: $AlBr_3$, $GaCl_3$, $SnCl_4$, $TiCl_4$, $TiBr_4$. The selection criteria were discussed in Anderson et al. (2007). Based on these life tests, two of the halides appear to be suitable for temperatures up to 673 K, and possibly at higher temperatures. Long term life tests are ongoing with $TiBr_4$/titanium at 653 K, and with $AlBr_3$/Superalloys at 673 K. As of April 2012, the $AlBr_3$ and $TiBr_4$ tests have been running for 50,000 hours (5.7 years).

In late 2010, several of the heat pipes were selected for destructive investigation (Table 1). One of each pair of water life test pipes was selected. The $GaCl_3$ and $SnCl_4$ pipes were known to be non-compatible, since they generated large amounts of NCG. The heat pipes containing halides were neutralized using an appropriate liquid. The neutralized fluid and water from the heat pipes using water as a working fluid were collected for chemical analysis.

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-micron silica and examined using optical and scanning electron (SEM) microscopes.

3. RESULTS AND DISCUSSION

3.1 Microscopic Examination of Cross-sections

3.1.1 Titanium-Water Heat Pipes

Analysis of the 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 micron thick. There were some indications in the backscattered electron (BSE) SEM images of changes in the structure of the Ti and Ti alloys as shown by the lighter bands near the surface in Figure 1. Energy dispersive spectroscopy (EDS) did not indicate that the layers changed composition, and they may be related to either a change of the Ti from the α phase to the β phase or changes in grain structure from pickup of interstitial elements such as O.

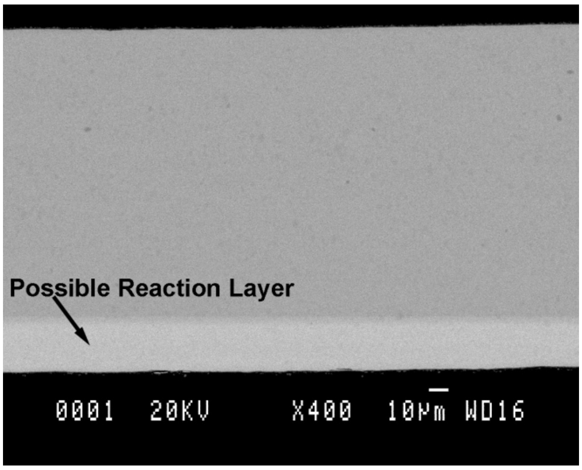

Figure 1. BSE Image of Timetal 21-S Strip Incorporated in Heat Pipe 124

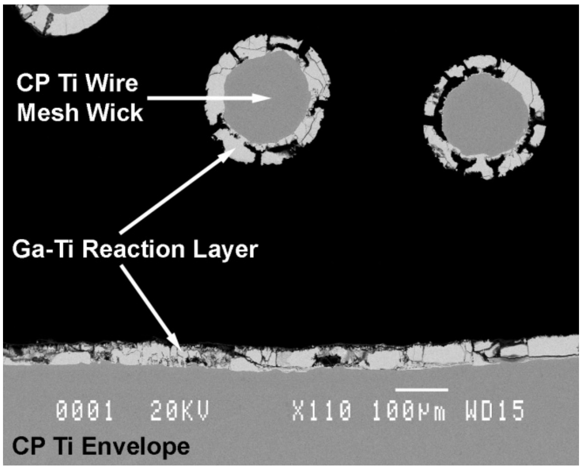

Figure 2. BSE Image of Heat Pipe 10 Envelope and Wire Mesh Wick

3.1.2 Titanium-Halide Heat Pipes

The two titanium halide heat pipes examined had very different responses. CP Ti with a $TiBr_4$ working fluid had minimal corrosion. There was evidence similar to the layers shown in Figure 1 of some potential change in the outer 10 microns.

CP Ti with $GaCl_3$ working fluid underwent extensive corrosion as seen in Figure 2. EDS analysis indicated that the corrosion layer was a Ga-29.7 wt.% Ti alloy. Examination of the Ga-Ti phase diagram (NPL, 2012) led to the conclusion that a mixture of Ga_2Ti, Ga_3Ti and Ga(l) was probably formed. Given the extensive nature of the voids, particularly on the wires, it was hypothesized that considerable Ga(l) was present in the voids and lost during neutralization. The remaining Ga_2Ti reaction layer also exhibited evidence of brittle fracture during polishing.

3.1.3 Monel-Water Heat Pipes

Figure 3 shows an optical micrograph of the envelope and wick for Heat Pipe 136, one of the Monel-water heat pipes that underwent the most change. The formation of a dark subsurface layer and bright nodules were observed in the Monel 400 wick using BSE imaging. This was typical of all Monel 400 surfaces examined to varying but large degrees. EDS spot analysis revealed that the surface nodules were nearly pure Cu, and the dark layer was a Cu-depleted zone. Most likely, there was a phase change from α to $\alpha_1 + \alpha_2$ (ASMI, 1992) followed by diffusion of the Cu to the surface during exposure.

The Monel K500 does not show similar changes. Close examination of the envelope reveals, at most, a very thin corrosion layer. Most likely, the layer was an oxide, but it was sufficiently thin to prevent definitive identification through EDS. Apparently, the composition of the Monel K500 stabilizes the α phase.

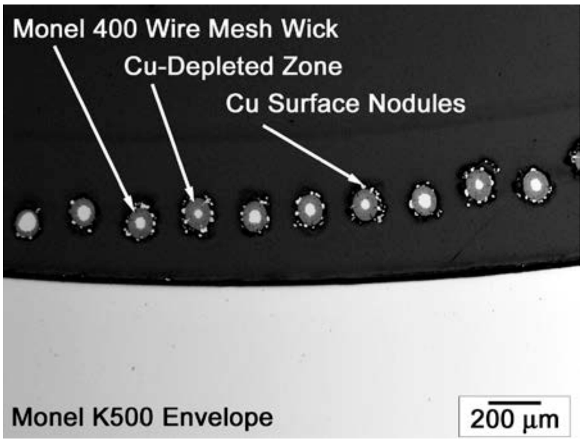

Figure 3. Optical Bright Field Micrograph of Heat Pipe 136 Envelope and Wire Mesh Wick

3.1.4 Hastelloy Superalloy-Halide Heat Pipes

Table 1 lists the five corrosion-resistant Hastelloy C-series superalloy heat pipes that were examined. As shown in Figure 4, the $SnCl_4$ working fluid caused considerable roughening of the C-22 envelope, up to 20-micron deep cracks in the substrate, a porous 10-micron thick corrosion layer and a thin, discontinuous Mo-W-Sn reaction layer beneath the corrosion product.

Table 2. Contaminants Found In Working Fluids (weight percent).

Heat Pipe	Working Fluid	Life Test Hours	Al	Co	Cr	Cu	Fe	Mn	Mo	Ni	Ti	V	W
6	$SnCl_4$	20,160	0.007		0.38		0.038	0.012	0.79	1.78			0.02
7	$SnCl_4$	20,160	0.006	0.001	0.11		0.004	0.057	0.083	0.027			
8	$SnCl_4$	20,160	0.005	0.007	0.7	0.022	0.018	0.003	0.31	0.83			0.001
9	$GaCl_3$	20,040									1.2		
10	$GaCl_3$	20,040									1.2		
153	$TiCl_4$	28,560			0.006		0.027			0.003			
157	$AlBr_3$	28,704			0.013		0.018			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		
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		<0.000005	<0.000005		<0.000005	<0.000005	<0.000005		<0.000005	<0.000005		

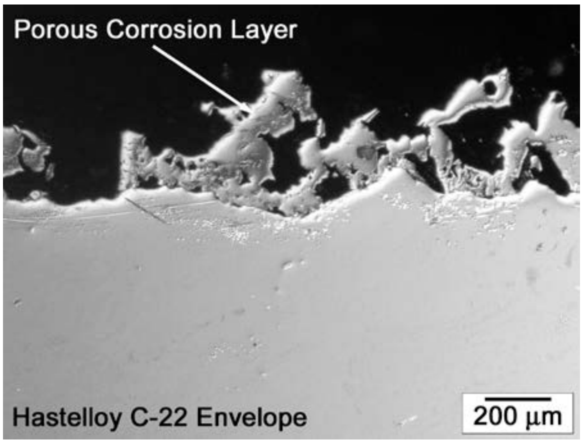

Figure 4. Differential Interference Contrast Optical Micrograph of Heat Pipe 7 C-22 Envelope

Hastelloy C-2000 also underwent extensive reaction with the $SnCl_4$ working fluid as shown in Figure 5. A 200-micron thick reaction layer identified as Ni_3Sn_2 with about 9 wt.% Cl was observed. In addition to the Ni-Sn-Cl reaction layer, Mo-Cl particles were observed at the reaction layer/substrate interface during X-ray mapping.

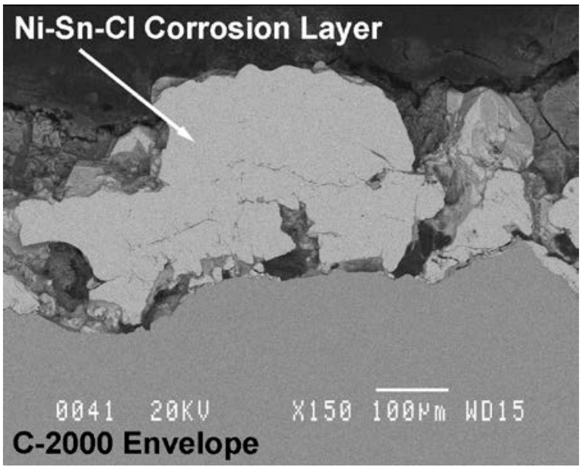

Figure 5. BSE Image of Heat Pipe 8 C-2000 Envelope Showing Thick Ni-Sn-Cl Reaction Layer

Heat Pipes 153 and 157 showed good promise. Hastelloy C-2000 underwent little corrosion when used with $TiCl_4$ working fluid. A 1 to 2 micron thick Ni-33 wt.% Ti-18 wt.% Mo-18 wt.%Cr-4 wt.% Cu-2 wt.% Cl corrosion layer was observed on the surface. BSE images showed that there might be a 0.5-micron thick region beneath the corrosion layer that was depleted in heavy elements.

Hastelloy C-22 exhibited a dual corrosion layer when tested with $AlBr_3$ working fluid as shown in Figure 6. The total thickness was 5 to 10 microns. 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. Based upon these analyses, it appears that $AlBr_3$ can slowly react with the C-22 over thousands of hours to form a relatively thin corrosion layer.

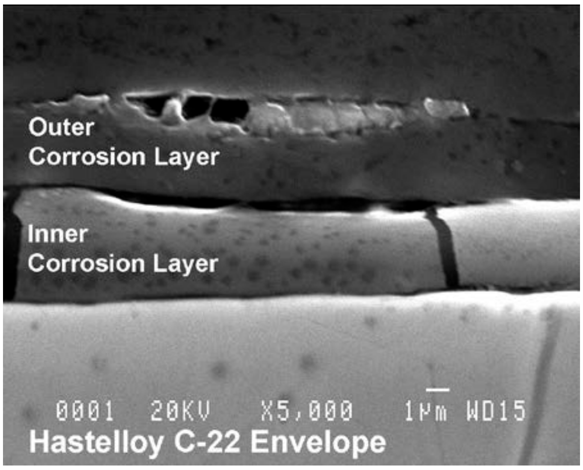

Figure 6. SE Image of Heat Pipe 157 C-22 Envelope Showing Two Corrosion Layers

3.2.5 Analysis of Working Fluids

Table **2** contains the results of the chemical analysis of the working fluids. As a standard, deionized water exposed to Ti and Monel 500 (DI Water Std.) was analyzed as well. Only the elements present in the envelope and wick alloys are listed. Minimal pickup of metals was observed for the heat pipes using water as a working fluid.

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 layer, exhibited low total metal contents between 300 and 350 ppm. Heat Pipes 7 and 8, which used $SnCl_4$, suffered more working fluid contamination with Cr being the major metal present. The relative amounts seem to be consistent with the qualitative levels of attack observed. $GaCl_3$ was clearly the most aggressive working fluid when paired with Ti, which is consistent with the microscopic observations. 1.2 wt.% Ti was present in the working fluid of Heat Pipes 9 and 10. Since titanium was the only metal in both the envelope and fluid, no fluid analysis was made for Heat Pipe 4 (CP-Ti/$TiBr_3$), 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. Note that this pipe developed a leak in the first few hours after it was put on life test.

3. CONCLUSIONS

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 over 54,000 hours (6.1 years) as of April 2012. 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 micron 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.

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/$TiCl4_4$ at 573 K (300°C), and Superalloys/$AlBr_3$ at 673K (400°C) are compatible. As of April 2012, the $AlBr_3$ and $TiCl_4$ tests have been running for 50,000 hours (5.7 years).

Hastelloy C-2000 underwent little corrosion when used with $TiCl_4$ working fluid, with the formation of only a 1-2 micron thick corrosion layer. Hastelloy C-22 exhibited a 5-10 micron thick dual corrosion layer when tested with $AlBr_3$ 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.

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