15th International Heat Pipe Conference (15th IHPC)
Clemson, USA, April 25-30, 2010

Intermediate Temperature Fluids for Heat Pipes and Loop Heat Pipes

William G. Anderson, John R. Hartenstine, David B. Sarraf, and Calin Tarau
Advanced Cooling Technologies, Inc.
1046 New Holland Ave.
Lancaster, PA 17601 U.S.A.
717-295-6104, Bill.Anderson@1-act.com
717-295-6112, John.Hartenstine@1-act.com
717-295-6059, Dave.Sarraf@1-act.com
717-295- 6066, Calin.Tarau@1-act.com

ABSTRACT

Potential working fluids for heat pipes and loop heat pipes include water, organic fluids, elements, and halides. The paper surveys life tests 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. Life tests have been conducted with 19 different organic working fluids. Three sets of organic fluids stand out as good intermediate temperature fluids: (1) Diphenyl, Diphenyl Oxide, and Eutectic Diphenyl/Diphenyl Oxide, (2) Naphthalene, and (3) Toluene. While fluorinating organic compounds are believed to make them more stable, this has not yet been demonstrated during heat pipe life tests. Finally several halides are suitable for temperatures up to 673 K (400°C). Life tests at temperatures up to 400°C were conducted with titanium and three corrosion resistant superalloys, and six different working fluids: $AlBr_3$, $GaCl_3$, $SnCl_4$, $TiCl_4$, $TiBr_4$, and eutectic diphenyl/diphenyl oxide. (Therminol VP-1/Dowtherm A). Ongoing life tests with superalloys/$TiCl_4$ and $AlBr_3$ have been running for 28,000 hours. Ongoing life tests with up to 45,000 hours demonstrate that titanium/water and Monel/water heat pipes can be used at temperatures up to 550 K (277°C).

KEY WORDS*:* Heat pipe, loop heat pipe, compatible working fluids, life tests, water, halides, organic working fluids

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 725 K (170 to 450°C), including space nuclear power system radiators, fuel cells, and high temperature electronics cooling. At temperatures above 725 K (450°C), 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 (450°C), 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 (150°C). More recently, it has been shown that water can be used with titanium or Monel envelopes at temperatures up to 550 K (277°C) (Anderson et al., 2006). While water heat pipes can operate at temperatures up to 570 K (300°C), their effectiveness starts to drop off above 500 K (227°C), due to the decrease in the surface tension.

2. LIFE TEST 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.

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

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

2.1 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.

Additives have been used successfully with mercury to wet coarse, wicks; however, 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; see Anderson, Rosenfeld, Angirasa, and Mi (2004) for details.

Sulfur, Sulfur-Iodine, and Mercury life tests are summarized in Table 1. Mercury is compatible with 347 SS based on 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.

2.2 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, in a heat pipe, will decrease the circulation of the working fluid 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 therefore 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.

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

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

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 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.

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.

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

	Compatible	Incompatible
Naphthalene/Aluminum	488 K (215°C)/27,750 hrs./6061 Al (Saaski 1977, 1980)	
Naphthalene/Mild Steel	543 K (270°C)/~26,000 hrs./ST 35 & 13CrMo44. (Groll, 1982, 1987, 1989)	
Naphthalene/Stainless Steel	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) 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** 653 K (380°C)/Alloy 20 (Vasil'ev et al. , 1988)	
Naphthalene/Cu-Ni	593 K (320°C)/~9,000 hrs/CuNi10Fe (Groll, 1982, 1987, 1989)	
Naphthalene/Titanium	593 K (320°C)/~9,000 hrs/CP-Ti (Groll, 1982, 1987, 1989) 593 K (320°C)/~3,000 hours/Ti (Vasil'ev et al. , 1988)	
O-Terphenyl/Al		580 K (307°C)/672 hrs./6061 Al (Saaski 1977, 1980)
O-Terphenyl/Mild Steel	545 K (272°C)/27,750 hrs/Al-178 (Saaski 1977, 1980) stable NCG	
O-Terphenyl/SS	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)	
Decafluorobiphenyl/SS	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)	
Toluene/Aluminum	410 K (137°C)/23,120 hrs./6061 Al (Saaski 1977, 1980)	
Toluene/Mild Steel	523 K (250°C)/~26,000 hrs./ ST 35 & 13CrMo44 (Groll, 1982, 1987, 1989)	
Toluene/Stainless Steel	523 K (250°C)/~26,000 hrs./316 SS (Groll, 1982, 1987, 1989)	
Toluene/Copper Nickel	553 K (280°C)/9,000 hrs./CuNi10Fe (Groll, 1982, 1987, 1989)	
Toluene/Titanium	523 K (250°C) 9,000 hrs. (Groll, 1982, 1987, 1989)	
1-Fluoronaphthalene/ Aluminum	493 K (220°C)/13,380 hrs./6061 Al Some NCG (Saaski 1977, 1980)	
1-Fluoronaphthalene/ Mild Steel	530 K (257°C)/26,370 hrs./A178 (Saaski 1977, 1980)	
1-Fluoronaphthalene/ Stainless Steel		530 K (257°C)/26,370 hrs./304 SS (Saaski 1977, 1980)
N-Octane/Mild Steel	503 K (230°C)/~40,000 hrs./ST 35 (Groll, 1982, 1987, 1989)	

	Compatible	Incompatible
N-Octane/Stainless	523 K (250°C)/~40,000 hrs./321 SS (Groll, 1982, 1987, 1989)	
Dowtherm E	493 K (220°C)/~40,000 hrs./ST 35 (Groll, 1982, 1987, 1989)	
Octafluoronaphthalene/ Aluminum	482 K (209°C)/13,400 hrs./6061 Al Some NCG (Saaski 1977, 1980)	
Octafluoronaphthalene/ Mild Steel		488 K (215°C)/13,400 hrs./A178 Some NCG (Saaski 1977, 1980)
Quinoline/SS		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)
Monochloronaphthalene /Stainless Steel		560 K 287°C/642 hrs./A178 (Saaski 1977, 1980)
Formyl-piperidine/SS		553 K (280°C)/15 hrs/304 SS/ (Kenney/ Feldman)
P-Terphenyl/SS		723 K (450°C)/<96 hrs/304 SS (Kenney, 1978)
ortho- and meta-terphenyl/Mild Steel		673 K (400°C)/~9,000 hrs./13CrMo44 (Groll, 1982, 1987, 1989) 593 K (320°C)/~9,000 hrs./13CrMo44 (Groll, 1982, 1987, 1989)
ortho- and meta-terphenyl/SS		673 K (400°C)/~9,000 hrs./316L SS (Groll, 1982, 1987, 1989) 623 K (350°C)/~9,000 hrs./316L SS (Groll, 1982, 1987, 1989)
diphenyl, ortho- and meta-terphenyl/Mild Steel		673 K (400°C)/~9,000 hrs./13CrMo44 (Groll, 1982, 1987, 1989) 623 K (350°C)/~9,000 hrs./13CrMo44 (Groll, 1982, 1987, 1989)
diphenyl, ortho- and meta-terphenyl/SS		673 K (400°C)/~9,000 hrs./316L SS (Groll, 1982, 1987, 1989) 623 K (350°C)/~9,000 hrs./316L SS (Groll, 1982, 1987, 1989)
Perfluoro-1,3-5-triphenylbenzene		Severe thermal decomposition at 573 K (300°C) (Grzyll, 1991, 1994, 1995)

When using diphenyl, diphenyl oxide, or diphenyl/diphenyl oxide at temperatures over 673 K (400°C), non-condensable gas is generated in a relatively short time period; see Table 2. The exact period depends on 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 300 and 400°C, these fluids are generally suitable, for short duration tests near 400 C, and long duration tests near 300 C (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 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).

Table 4. Halide Life Test Summary.

	Compatible	Incompatible
$TiBr_4$/Titanium		653K (380°C)/20,040 hours/CP-Ti (Anderson et al., 2007)
$TiCl_4$/Mild Steel	432 K (159°C)/28,540 hrs./A-178 Steel (Saaski 1977, 1980)	
$TiCl_4$/Superalloy	573K (300°C)/27.792 hrs./Hastelloy (Anderson et al., 2007)	
$TiCl_4$/Titanium	500 K (227°C)/4,019 hrs./CP-Ti (Locci, 2005)	
$TiCl_4$/Aluminum		438 K (165°C)/2,500 hrs./Al-6061 (Saaski 1977, 1980)
$SnCl_4$/Aluminum		432 K (159°C)/--- hrs./Al-6061 (Saaski 1977, 1980)
$SnCl_4$/Mild Steel	429 K (156°C)/27,7500 hrs./A-178 Steel (Saaski 1977, 1980)	
$SnCl_4$/Superalloy		553K (280°C)/20,160 hrs./Hastelloy (Anderson et al., 2007)
$AlBr_3$/Superalloy	673K (400°C)/27,936 hrs./Hastelloy (Anderson et al., 2007)	
$AlBr_3$/Aluminum		500 K (227°C)/4,290 hrs./Al-5052 (Locci, 2005) 500 K (227°C)/1,100 hrs./Al-6061 (Locci, 2005)
$AlBr_3$/Titanium		500 K (227°C)/1,100 hrs./CP2-Ti (Locci, 2005)
$GaCl_3$/Superalloy		633K (360°C)/--- hrs./Hastelloy C-22 (Anderson et al., 2007)
$GaCl_3$/Titanium		613K (340°C)/16,632 hrs./CP-Ti (Anderson et al., 2007)
$SbCl_3$/Aluminum		500 K (227°C)/--- hrs./Al-6061 (Saaski 1977, 1980)
$SbCl_3$/Mild Steel		476 K (203°C)/5,000 hrs./A-178 Steel (Saaski 1977, 1980)
$SbBr_3$/Aluminum		500 K (227°C)/5,000 hrs./Al-6061 (Locci)

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 include:

- 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 stainless for short term tests at 380°C.

While fluorinated compounds are believed to be more stable than the same compound with out 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.

Table 5. Halide Life Test Pipes – Operating Hours as of April 15, 2010.

	CP-Ti	C22	C2000	B3
$AlBr_3$	–	400°C (673K) 27,936 hrs Low Gas	400°C (673K) 27,936 hrs Low Gas	400°C (673K) ~11,000 hrs (Fail)[1]
$GaCl_3$	340°C (613K) 19,632 hrs High Gas Shut down	Fail	Fail	Fail
$SnCl_4$	–	280°C (553K) 20,160 hrs High Gas Shut down	280°C (553K) 20,160 hrs High Gas Shut down	280°C (553K) 20,160 hrs High Gas Shut down
$TiCl_4$	–	300°C (553K) 27,792 hrs Low Gas	300°C (553K) 27,792 hrs Low Gas	300°C (553K) 27,792 hrs Low Gas
$TiBr_4$	380°C (653K) 20,040 hrs High Gas Shut down	–	–	–

[1] $AlBr_3$/B3 pipe was severely overheated at 300 hours testing

2.3 Halides

A halide is a compound of the type MX, where M may be another element or organic compound, and X may be 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 $AlBr_3$/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).

3. 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 are shown in Table 5. 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).

Operating temperatures for the heat pipes are shown in Table 7. The operating temperature was 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 $GaCl_3$/superalloy 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 $AlBr_3$ failed after roughly 11,000 hours, 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 $GaCl_3$/Titanium, $TiBr_4$/titanium, and $SnCl_4$/ superalloy 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 later analysis.

The $TiCl_4$/superalloy and $AlBr_3$/superalloy pipes continue to run without any problem. These pipes have currently been running for 3.2 years. The $AlBr_3$ pipes are of particular interest, since they are running at 673 K (400°C). This is close to the temperature at which cesium starts to work.

4. TITANIUM/WATER AND MONEL/WATER LIFE TESTS

Titanium, titanium alloys, Monel 400, and Monel K500 have higher yield strength and lower density than copper. As discussed below, 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
- Ti CP-2 Heat Pipe, with Sintered CP Titanium Wick
- Ti CP-2 Heat Pipe, with Integral Grooves
- 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
- Ti Grade 9 cylinder (3% Aluminum, 2.5% Vanadium) with CP Titanium Screen
- Monel K500 Heat Pipe, with Monel 400 Screen
- Monel K500 Heat Pipe, with Sintered Monel 400 Wick
- Monel 400 Heat Pipe, with Monel 400 Screen
- 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.

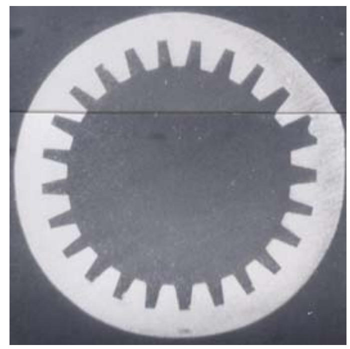

Figure 1. Titanium Heat Pipe with Integral Grooves for spacecraft thermal control.

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 Ni3(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.

Table 2 shows the different life test pipes on test. Note that the heat pipe operating temperature is dropped to 340 K (70°C), before the measurements. With this much lower temperature and vapor pressure, the non-condensable gas expands, making it easier to detect. The Monel pipes have shown almost no signs of gas generation. The titanium heat pipes all generated gas initially. This was believed to be a result of a passivation process that produced titanium oxide on the surface of the heat pipe.

Table 6. Titanium-Water and Titanium-Monel Life Test Pipes – Operating Hours as of April 15, 2010
.

Quantity	Wall Material	Wick	Operating Temperature	Operating Hours 15 Apr 2010
4	Monel K 500	200x200 Monel 400 Screen 0.064 mm wire	550 & 500 K	45,336 hours
4	CP-2 Ti	150x150CP-Ti Screen 0.069 mm wire	550 & 500 K	45,336 hours
2	CP-2 Ti	Sintered Titanium -35+60 Mesh CP-2	550 K	36,941 hours
1[2]	CP-2 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	1,996/34,256 hours
1	CP-2 Ti	Integral Grooves	550 K	27,400 hours
2	CP-2 Ti 21 S Foil Inside	100 x100 CP-Ti Screen 0.05 mm wire	550 K	36,941 hours
2	Grade 5 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	36,941 hours
2	Grade 7 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	36,941 hours
2	Grade 9 Ti	100 x100 CP-Ti Screen 0.05 mm wire	550 K	32,784 hours
2	Monel 400	120x120 Monel 400 Screen 0.05 mm wire	550K	32,280 hours
2	Monel K 500	120x120 Monel 400 Screen 0.05 mm wire	550K	31,440 hours
2	Monel 400	-100+170 Mesh Monel 400 Powder	550K	30,192 hours
2	Monel K 500	-100+170 Mesh Monel 400 Powder	550K	30,552 hours

[2] One pipe removed after 1996 hours and sent to NASA

The gas was removed from all of these pipes by heating to about 115°C and venting them. The thermocouples were monitored to verify that the non-condensable gas has been forced out of the condenser by the pressure difference. The heat pipe fill tube is then resealed. Since they have been resealed, they have all generated a small amount of gas.

Some of the CP-Ti and Monel pipes currently have operated for 5.2 years. Life test pipes with titanium alloys are also underway, with operating times of up to 4.2 years. Titanium/water and Monel/water heat pipes can be considered a mature or mainstream technology that is ready for widespread application.

5. CONCLUSIONS

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 agressive.

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

The halides appear to be suitable for temperatures up to 673 K (400°C), and possibly at higher temperatures. Long term life tests are ongoing with $TiBr_4$/titanium at 653K (380°C), and with $AlBr_3$/Superalloys at 673K (400°C). 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. However, property data for the halides is incomplete.

Water life test pipes have been fabricated with commercially pure (CP) titanium, Monel K-500, Monel 400, and various titanium alloys, and run at 500 K (227°C) and 550 K (277°C). Heat pipes have been tested with screen and sintered powder wicks, as well as grooved CP-Ti pipes. Some of the CP-Ti and Monel pipes currently have operated for 5.2 years. These pipes continue to operate successfully, with a small amount of gas generation in the CP-Ti pipes. Life test pipes with titanium alloys are also underway, with operating times of up to 4.2 years. Titanium/water and Monel/water heat pipes can be considered a mature or mainstream technology that is ready for widespread application.

ACKNOWLEDGEMENT

The water and halide life tests were sponsored by NASA Glenn Research Center under Contracts NNC05TA36T, and NNC06CA74C. Duane Beach and then David Ellis were the Technical Monitors. Chris Stover and Rod McClellan were the technicians on the program. We would like to thank Cheryl Bowman and Ivan Locci of NASA Glenn Research Center for helpful discussions about the fluids and materials. We would also like to thank Laurie Anderson, Al Basiulis, Claus Busse, Don Ernst, Manfred Groll, Larry Grzyll, and Bob Reid for their generous help in locating and supplying references.

REFERENCES

1. Anderson, W. G., "Intermediate Temperature Fluids for Heat Pipes and LHPs," W.G. Anderson, Proceedings of the 2007 IECEC, AIAA, St. Louis, MO, June 25-27, 2007a.
2. Anderson, W.G., Bonner, R.W., Dussinger, P.M., Hartenstine, J.R., Sarraf, D.B., and Locci, I.E., "Intermediate Temperature Fluids Life Tests – Experiments" Proceedings of the 2007 IECEC, AIAA, St. Louis, MO, June 25-27, 2007b.
3. Anderson, W.G., Dussinger, P.M., Bonner, R.W., and Sarraf, D.B., "High Temperature Titanium-Water and Monel-Water Heat Pipes," Proceedings of the 2006 IECEC, AIAA, San Diego, CA, June 26-29, 2006a.
4. Anderson, W.G., Dussinger, P.M., and Sarraf, D.B., "High Temperature Water Heat Pipe Life Tests," STAIF 2006, pp. 100-107, American Institute of Physics, Melville, New York, 2006b.
5. Anderson, W.G., "Evaluation of Heat Pipes in the Temperature Range of 450 to 700 K," STAIF 2005, Albuquerque, NM, February 13-17, 2005.
6. Anderson, W.G., Rosenfeld, J.R., Angirasa, D., and Mi, Y., "The Evaluation of Heat Pipe Working Fluids In The Temperature Range of 450 to 750 K," Proceedings, STAIF-2004, pp. 20-27, Albuquerque, NM, February 8-12, 2004.
7. Anderson, W.G., "Sodium-Potassium (NaK) Heat Pipe," Heat Pipes and Capillary Pumped Loops, Ed. A Faghri, A. J. Juhasz, and T. Mahefky, ASME HTD, 236, pp. 47-53, 29th National Heat Transfer Conference, Atlanta, Georgia, August 1993.
8. Basiulis, A., and Prager, R. C., "Compatibility and reliability of heat pipe materials," AIAA-1975-660, 10th AIAA Thermophysics Conference, Denver, Colo., May 27-29, 1975
9. Basiulis, A., and Fuller, M., "Operating Characteristics and Long Term Capabilities of Organic Fluid Heat Pipes," AIAA No. 71-408, AIAA 6th Thermophysics Conference, 1971.
10. Devarakonda, A. and Anderson, W.G., "Thermo-Physical Properties of Intermediate Temperature Heat Pipe Fluids," STAIF 2005, Albuquerque, NM, February 13-17, 2005. NASA Report NASA/CR—2005-213582, available from the NASA Glenn Technical Reports Server, http://gltrs.grc.nasa.gov/.
11. Devarakonda, A., and Olminsky, J.E., "An Evaluation of Halides and Other Substances as Potential Heat Pipe Fluids," Proceedings of the 2004 IECEC, Providence, RI, August 16-19, 2004.

12. Deverall, J.E., “Mercury as a Heat Pipe Fluid,” ASME Paper 70-HT/Spt-8, American Society of Mechanical Engineers, 1970.
13. Eastman, Y., personal communication, 2007
14. Ernst, D.M., personal communication, 2006.
15. Groll, M., Brost, O., Heine, D., and Spendel, T., “Heat Transfer, Vapor-Liquid Flow Interaction and Materials Compatibility in Two-Phase Thermosyphons,” CEC Contractors Meeting, Heat Exchangers – Heat Recovery, Brussels, June 10, 1982.
16. Groll, M., Brost, O., and Roesler, S., “Development of High Performance Closed Two-Phase Thermosyphons as Heat Transfer Components for Heat Recovery from Hot Waste Gases,” EG-Status Seminar, Brussels, October, 1987.
17. Groll, M., “Heat Pipe Research and Development in Western Europe”, Heat Recovery Systems and CHP (Combined Heat & Power), 9(1), pp. 19-66, 1989.
18. Grzyll, L.R., Ramos, C., and Back, D.D., “Density, Viscosity, and Surface Tension of Liquid Quinoline, Naphthalene, Biphenyl, Decafluorobiphenyl, and 1,2-Diphenylbenzene from 300 to 400°C,” J. Chem. Eng. Data, Vol. 41, pp. 446-450 1996.
19. Grzyll, L.R., Back, D.D., Ramos, C., and Samad, N.A., “Characterization and Testing of Novel Two-Phase Working Fluids for Spacecraft Thermal Management Systems Operating Between 300°C and 400°C,” Final Report to Phillips Laboratory, Kirtland Air Force Base, No. PL-TR-95-1089, 1995.
20. Grzyll, L.R., Back, D.D., Ramos, C., and Samad, N.A., “Characterization and Testing of Novel Two-Phase Working Fluids for Spacecraft Thermal Management Systems Operating Between 300°C and 400°C,” Proceedings of the 1st Annual Spacecraft Thermal Control Symposium, Albuquerque, NM 1994.
21. Grzyll, L.R., “Heat Pipe Working Fluids for Thermal Control of the Sodium/Sulfur Battery,” Proceedings of the 26th Intersociety Energy Conversion Engineering Conference, Vol. 3, pp. 390-394, American Nuclear Society, La Grange, Illinois, 1991.
22. Hartenstine, J.R., personal communication, 2007.
23. Heine, D., Groll, M., and Brost, O., “Chemical Compatibility and Thermal Stability of Heat Pipe Working Fluids for the Temperature Range 200 °C to 400 °C,” 8th ChiSA Congress, Prague, September 3-7, 1984.
24. Jaworskie, D., personal communication, April 5, 2007.
25. Kenney, D.D., and Feldman, K.T., “Heat Pipe Life Tests at Temperatures up to 400°C,” Proceedings of the 13th Intersociety Energy Conversion Engineering Conference, pp. 1056-1059, San Diego, CA, Aug. 20-25, 1978.
26. Locci, I.E., Devarakonda, A., Copeland, E.H., and Olminsky, J.K., “Analytical and Experimental Thermo-Chemical Compatibility Study of Potential Heat Pipe Materials,” Proceedings of the 2005 IECEC, San Francisco, CA, August 15-18, 2005.
27. Los Alamos Scientific Laboratory, “Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending April 30, 1970, Part 1,” Report No. LA-4446-MS, pp, 2-5, May, 1970.
28. Los Alamos Scientific Laboratory, “Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending January 31, 1968, Part 1,” Report No. LA-3881-MS, pg. 4, February, 1968a.
29. Los Alamos Scientific Laboratory, “Quarterly Status Report on the Space Electric Power R&D Program for the Period Ending April 30, 1968, Part 1,” Report No. LA-3941-MS, pg. 2, May, 1968b.
30. Lundberg, L.B., Merrigan, M., Prenger, F.C., and Dunwoody, W., “Sulphur Heat Pipes,” Energy Technology, Los Alamos Scientific Laboratory, LA 8797-PR, October-December 1980, pp. 69-70.
31. Polasek, F., and Stulc, P., “Heat Pipe for the Temperature Range from 200 to 600°C,” Proc., Second International Heat Pipe Conference, Bologna, Italy, 2, pg. 711, 1976.
32. Polasek, F., “Heat Pipe Research and Development in East European Countries,” 6th International Heat Pipe Conference (1987), Heat Recovery Systems and CHP, *9(1)*, pp. 3-17, 1989.
33. Reid, R.S., Merrigan, M.A., and Sena, J. T., “Review of Liquid Metal Heat Pipe Work at Los Alamos,” 8th Symposium on Space Nuclear Power Systems, Albuquerque, NM, January 6-10, 1991.
34. Saaski, E.W., and Owzarski, P.C., “Two-Phase Working Fluids for the Temperature Range 50°

to 350°C," Sigma Research, Inc., Final Report, Contract NAS3-20222, NASA Lewis Research Center, June 1977a.
35. Saaski, E.W., and Tower, L., "Two-Phase working fluids for the temperature range 100-350°C," American Institute of Aeronautics and Astronautics, 12th Thermophysics Conference, Albuquerque, NM, June 27-29, 1977b.
36. Saaski, E.W., and Hartl, J.H., "Two-Phase Working Fluids for the Temperature Range 50 to 350°C," Sigma Research, Inc., Phase II Final Report, Contract NAS3-21202, NASA Lewis Research Center, March, 1980.
37. Tarau, C., Sarraf, D.B., Locci, I.E., and Anderson, W.G., "Intermediate Temperature Fluids Life Tests – Theory," Proceedings, STAIF 2007, Albuquerque, NM, February 11-15, 2007.
38. Timrot, D.L., Serednitskaya, M.A., Medveditskov, A.N., and Traktueva, S.A., "Thermophysical Properties of a Sulfur-Iodine Binary System as a Promising Heat Transfer Medium for Heat Pipes," Journal of Heat Recovery Systems (now Applied Thermal Engineering), Vol. 1(4), pp. 309-314, 1981.
39. Vasil'ev, L.L., Volokhov, G.M., Gigevich, A.S., and. Rabetskii, M.I, "Heat Pipes Based on Naphthalene," Journal of Engineering Physics and Thermophysics, Vol. 54, No. 6, pp. 623-626, 1988.