Alkali Metal Heat Pipe Life Issues

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Abstract – One approach to space fission power system design is predicated on the use of alkali metal heat pipes, either as radiator elements, thermal management components, or as part of the core primary heat-transfer system. This synopsis characterizes long-life core heat pipes. References are included where more detailed information can be found. Specifics shown here are for demonstrational purposes and do not necessarily reflect current Project Prometheus point designs.

I. INTRODUCTION

Alkali metal heat pipes are among the best-understood and tested components for first generation space fission reactors. A heat pipe reactor core self-starts and provides passive heat removal during all mission phases (startup, operation, shutdown, decay, and restart) with no freeze/thaw concerns. Heat pipes are reliable and arrays of them act as redundant coolant channels that minimize core temperature gradients and the consequences of individual failure. A heat pipe core can drive independent power conversion systems providing in-depth reliability. Heat pipes simplify reactor integration providing a well-defined generic interface to Brayton, thermoelectric, or Stirling energy converters. They offer numerous temperature and power design options and are modular allowing access for resistance heated hydraulic test before fueling.

Alkali metal heat pipes have been shown capable of multi-year operation and tolerant to many times the fast neutron fluence of expected core designs (Ranken, 1987). Alkali metal heat pipes have been successfully tested aboard the Space Shuttle Endeavor. Results correlated well with existing models indicating that the first flight heat pipe core would certainly operate as intended (Dickenson, 1997). A large body of data documents the excellent compatibility of stainless steels and refractory metals such as Nb or Mo with vaporizing alkali metals such as K, Na, or Li at temperatures up to 1300°C, (Romano, 1968), (Merrigan, 1994). With UN fuel, a properly built and integrated refractory metal heat pipe core with an alkali metal working fluid could produce megawatt levels of power for >12 years at >1000°C.

II. CURRENT STATE OF THE ART

Since their invention 40 years ago, heat pipes with fluids such as ammonia and water have found wide use on earth and in space. The original heat pipe embodiment was intended expressly for space nuclear reactor energy conversion (Grover, 1964). A heat pipe cooled reactor was the original baseline for the SP-100 program (Klein, 1984). Alkali metal heat pipes still occupy a niche associated mostly with space nuclear power systems. Despite the stunted US space reactor effort, R&D projects during the 1970s and 1980s sponsored by the DOE, NASA, and the US Air Force brought alkali metal heat pipes to a level of understanding and technical maturity unusual for space reactor components, (Merrigan, 1984, 1994). Impurity induced corrosion was identified as the only significant life-limiting factor (DeVan, 1984), (Lundberg, 1987). If heat pipe container and working fluid impurities can be kept low, and care is taken in fabrication, and isolation is achieved from external contamination sources, long operating life is practically assured. These conditions can be met with industry standard practice (e.g., ASTM C997-83, C1051-85, G68-80).

A heat pipe based primary heat transfer system consists of an array of ~100 heat pipes capable of transferring megawatt levels of thermal power from a reactor core to an energy converter. For most space exploration missions envisioned, core heat pipes in a space power reactor must work reliably and continuously for >12 years at >1000°C. These goals may be reached with established technology. Low-budget R&D alkali metal heat pipes have worked in fast neutron environments (Ranken, 1987), aboard the Space Shuttle (Dickenson, 1997), and with Nb-1%Zr containers (Sena, 1990). Anomalies for such pipes are rare and the few that do occur stem from quality control.
problems (such as weld failure) or impurities introduced from external sources that can cause evaporator hot spots or corrosion. Together, alkali metal heat pipe tests reflect years of real operation. Long-term test data is limited and necessarily tied to the period of typical government project cycles rather than any intrinsic life limit. Organizations seldom have incentive to continue meaningful tests (of any sort) after programs (such as SP-100) are canceled. Existing processing procedures have allowed alkali metal heat pipes to operate reliably for \( > 10^4 \) hours. Operation \( > 10^5 \) hours (~12 years) is practically assured with similar procedures given proper design.

There are ways to perform accelerated tests to experimentally investigate heat pipe lifetimes. Tests at elevated temperature and evaporator power density can accelerate potential life limiting mechanisms (Lundberg, 1984). Accelerated heat pipe tests can show, well before a mission is launched, that design and processing procedures meet mission performance and life requirements.

Core heat pipes will require more careful construction than has been previously possible in low budget research and development programs. Fortunately, industry standard fabrication techniques and quality control measures that were impractical in low budget R&D programs become practical and affordable for a space flight qualified system. Necessary requirements for successful integration of long life heat pipes into a core include:

1) Select compatible material combinations for the fuel, reflector, heat pipe container and working fluid.
2) Isolate the heat pipe material system from mobile non-metallic and metallic impurity sources during manufacture, assembly, and operation.
3) Measure impurity concentrations in the working fluid, structure, and surroundings during fabrication and (especially) final assembly.
4) Establish tests to conclusively demonstrate that the end product is of sufficient quality to meet operational and life requirements.

### III. OBJECT

The remainder of this paper describes a 2-MWt-core heat pipe (with some material system options) and shows methods to achieve and measure purity in completed Na heat pipes. Approaches to build a space qualified heat pipe core array and to confirm a life expectancy of \( > 12 \) years are shown.

### IV. HEAT PIPE OPERATING PRINCIPLE

Basic heat pipe operating principles are covered in several well-written textbooks (Petersen, 1993), (Silverstein, 1994), (Faghir, 1995). A heat pipe consists of a container enclosing a capillary structure, or wick, saturated with a fluid, and a space with the fluid’s vapor. Zones of heat addition and removal (by conduction through the tube wall) are called the evaporator and condenser sections, respectively, and are shown in Figure 1a. The alkali metal vaporizes in the evaporator at the wick surface and moves toward the condenser by a pressure gradient formed by capillary forces (note: boiling does not occur in properly-designed alkali metal heat pipes). Condensate returns to the evaporator by the pumping action of the wick. Using round numbers, a core heat pipe system might look something like that shown in Table 1.

![Figure 1a. Cutaway showing a generic heat pipe. Dashed lines bound the evaporator (left) and the condenser.](image)

#### Table 1. Generic 2 MWt Core Heat Pipe

| Operating Lifetime: \( > 12 \) years |
| Reactor Core Power: \( \sim 2 \) MWt |
| Vapor Temperature at Evaporator Exit: \( 1000^\circ \text{C} \) |
| Containment Material: Nb-1\%Zr (ASTM Type 3) |
| Working Fluid: Purified Reactor Grade Sodium |
| Core Configuration: Modular |
| Number of Heat Pipe Modules: 100 |
| Power Per Heat Pipe: 20 kWt |
| Overall Length: 1.5 m |
| Heated Length: 75 cm |
| Adiabatic Length: 0 cm |
| Cooled Length: 75 cm |
| Heat Pipe OD: 1.60 cm |
| Heat Pipe ID: 1.42 cm |
| Wall Thickness: 0.89 mm |
| Heat Pipe Length to Inside Diameter Ratio: 106 |

The wick forms menisci between the condensate and the vapor permitting a capillary potential to develop between the two. High performance wicks also have a low impedance condensate return passage. For capillary continuity, the wick is closed on the end nearest the evaporator. The wick normally remains open at the condenser end of the heat pipe to ease its fill with working fluid. As the pipe warms its working fluid expands. At the operating temperature excess liquid (typically \( 5\% \) of charge) seals the condenser end of the wick ensuring capillary continuity. Two wick geometries appear especially attractive for a high power density heat pipe core: an annular gap and an artery wick. Cross sections of each are depicted in Figure 1b.
There are several ways to build these wicks. Traditional alkali metal heat pipe wicks are formed from wire mesh screen (Merrigan, 1983). Mesh wick properties are shown in Table 2. Screen layers are wrapped around a mandrel and placed in a sheath. The sheath assembly is drawn to size and screen layers diffusion bonded at high temperature. The mandrel and sheath are then chemically dissolved. General Electric produced refractory metal foil wicks for the SP-100 program (Merrigan, 1994). These wicks performed well in test (Sena, 1993). Acceptable refractory metal foil wicks might be made from electroforming, chemical milling, or laser screen technologies. Foil wicks may be less inclined to introduce or retain impurities. Forming wicks from filtration cloth is yet another option.

Table 2. Generic 2-MWt Core Heat Pipe

<table>
<thead>
<tr>
<th>Wick Geometry: Annular Gap</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mesh Wire Count: 400 wires/inch [1]</td>
</tr>
<tr>
<td>Number of Screen Layers: 7</td>
</tr>
<tr>
<td>Annulus Thickness: 0.06 cm</td>
</tr>
<tr>
<td>Distribution Screen Thickness: 0.04 cm</td>
</tr>
<tr>
<td>Wire Diameter: 25 microns</td>
</tr>
<tr>
<td>Effective Pore Radius: 16 μm [2]</td>
</tr>
<tr>
<td>Wick Surface Porosity: 0.67</td>
</tr>
<tr>
<td>Thermal Conductivity of Wick: 0.675 W/cm-K</td>
</tr>
<tr>
<td>Nucleation Site Radius: 1 μm</td>
</tr>
<tr>
<td>Vapor Passage Area: 1.16 cm²</td>
</tr>
<tr>
<td>Total Liquid Flow Area: 0.27 cm²</td>
</tr>
<tr>
<td>Vapor to Liquid Area Ratio: 4.3</td>
</tr>
<tr>
<td>Condensate Return Velocity: 51 cm/s</td>
</tr>
<tr>
<td>Evaporator Radial Heat Flux: 53 W/cm²</td>
</tr>
<tr>
<td>Axial Heat Flux At Evaporator Exit: 10 kW/cm²</td>
</tr>
</tbody>
</table>


A modular core consists of an array of fuel and heat pipe elements. Each heat pipe is bonded to one or more fuel tubes. Monolithic cores are also possible with heat pipe evaporators embedded (brazed or integral) in a solid block. In either a modular or monolithic design, heat pipe condensers may be coupled to heat exchangers located just outside of the core. Heat pipes may also be routed to a heat exchanger or to thermoelectric elements on the opposite side of the shield. Heat pipes can be bent in different directions to accommodate design needs. Alkali metal heat pipes with multiple bends have been built and successfully tested. Instead of pipe bends miters might be effectively used to join angular wick and wall segments.

Specifications for a 2-MWt-core Na heat pipe are summarized in Table 2. Figure 2 plots axial heat transfer rate versus evaporator exit temperature (labeled “T-EE” in Figure 1a) predicted by HTPipe (Woloshun, 1988). Similar methods can approximate transient operation (Reid, 2002). The design point is well within the predicted operating limits. These core heat pipes use a conventional wire mesh wick yet have ample margin to accommodate core flux peaking and the failure of an adjacent heat pipe. The performance envelope in Figure 2 shows that the same heat pipe can be operated with similar margins at higher power and temperature (arrow). This trend continues to ~1400 K.

Heat pipe start up and restart issues (freeze-thaw) have been well characterized experimentally. El-Genk (2002) summarizes many of these efforts. Heat pipe frozen start up and restart physics have been closely examined in numerous computational models e.g., (Cao, 1993a, b), (Hall, 1994), and (Tournier, 1996, 2001, 2002). Since September 2000, SAFE-30 and SAFE-100 heat pipes at NASA MSFC and JPL have undergone hundreds of restarts without a single problem (Van Dyke, 2001). Reliability of this sort is the norm for properly designed and integrated alkali metal heat pipes.
V. ALKALI-REFRACTORY METAL LIFE ISSUES

This section discusses some life issues associated with candidate alkali-refractory metal material systems. A striking advantage of the heat pipe core approach is the numerous temperature and power configurations offered. For instance, a single Nb-1%Zr heat pipe design can work with K, Na, or Li allowing high power density operating options from 800 K to 1600 K.

Life issues are well established for alkali-refractory metal systems and are summarized in several textbooks (Addison, 1984), (Berry, 1971), (Borgstedt, 1987) and discussed in articles e.g., (Hoffman, 1968) and (King, 2001). When a working fluid condenses it is pure and free of impurities compared with the condenser wall. Nonmetallic impurities such as oxygen and carbon diffuse from the condenser wall and into the working fluid. These impurities are carried toward the evaporator where they concentrate. Impurities can precipitate and clog the wick, form low melting point eutectics with the container, or form ternary compounds with the container and working fluid.

If any of the elements in the wick or wall are soluble in the working fluid they can dissolve and move to the evaporator end of the pipe. The heat pipe structure must be sufficiently insoluble to avoid this problem entirely. In the absence of non-metallic impurities, the solubility of refractory metals in alkali metals is typically <<100 ppm (by weight).

Solubility increases in the presence of impurities when ternary compounds form with the working fluid and containment. Impurity corrosion rate in Na heat pipes is proportional to accumulation of elements such as O, Si, and C in the heat pipe evaporator. As working fluid flows into the evaporator it vaporizes, concentrating the impurities and makes corrosion rate somewhat dependent on mass fluence. The radial heat flux applied to the evaporator is $\dot{G}_r = \dot{M} \left( \frac{\pi d L_e}{6} \right)$, the mass flux through the evaporator is a function of the radial heat flux $G = \dot{M}_r h_f$, and the mass fluence through the evaporator $M' = G r$.

Mass diffusion transfers impurities from the heat pipe structure to the working fluid. The Arrhenius equation relates impurity diffusion rates to heat pipe temperature. Data can be Arrhenius normalized for heat pipe tests conducted away from the operating temperature by:

$$\alpha(T) = \exp\left\{ (\Delta H/k) \left( \frac{1}{T_0} - \frac{1}{T} \right) \right\},$$

where $k$ is Boltzmann's constant, $T_0$ is the operating temperature, $T$ is the heat pipe test temperature and $\Delta H$ is the activation energy. Testing on the order of 100°C over the design temperature can greatly accelerate the Arrhenius-governed diffusion rate for Nb-1%Zr heat pipes.

Mass fluence can be accelerated by applying power along a shortened heat pipe evaporator length. The Nb-1%Zr/Na heat pipe shown in Table 2 has a 1273 K nominal operating temperature, 0.75-m long evaporator, and a 12-year operating life. As a guideline, the equivalent of 12 years mass fluence, Arrhenius diffusion, and radiation fluence can be compressed into ~3 years by applying power over a 0.2-m section and testing at ~1400 K in an enhanced fast neutron environment.

V.A. Nb-1%Zr/Na System

The solubility ranges for niobium in vacuum-distilled and filtered sodium over the temperature range 800°C to 1300°C are 0.25 to 250 ppm (Kovacina and Miller, 1964). Despite measurable solubility of one of its constituents, the alloy Nb-1%Zr shows good resistance to solubility-induced attack in purified Na. The corrosion resistance of niobium alloys such as Nb-1%Zr and C-103 results from Zr, Ti, and Hf substitution elements that scavenge oxygen from solid solution and precipitate it as ZrO2, TiO, or HfO2. Figure 3 shows the relative thermodynamic stability of ZrO2 and TiO compared with niobium and sodium compounds containing oxygen (Chase, 1985) and (Natesan, 1983). Keeping impurities in the niobium lattice at a low level with additives such as Zr inhibits corrosion and reduces embrittlement effects from low-level contaminants. It should be remembered that such Ellingham plots are useful guides but are not always completely reliable indicators of element distributions (DiStefano, 1964).

![Figure 3. Partial molar free energies of Nb/Na/O compounds.](image-url)

Perkins (1977) reported oxygen diffusion rates in niobium and niobium-zirconium alloys as a function of temperature. The values given by Perkins can be used to
estimate oxygen transfer rates within an Nb-1\%Zr heat pipe system. Ignoring chemical partitions between compounds and diffusion through ternary oxide scales, the time dependent oxygen distribution in the condenser and evaporator can be approximated by a coupled system of diffusion equations. The time to deplete oxygen from the condenser of an Nb-1\%Zr heat pipe at 1273 K and transport it to the evaporator is order of days.

Nb-1\%Zr has been extensively tested in phase change sodium systems and has shown excellent corrosion resistance. Nb-1\%Zr tested in sodium that had been purified in Zr at 650°C for 250 hours showed only slight sodium attack at the liquid vapor interface after 6,000 hours exposure at 1150°C (Romano, 1966). In another study (Romano, 1968) Nb-1\%Zr specimens with oxygen levels ranging from 2250 ppm to 6125 ppm were tested in capsules containing hot trapped refluxing sodium (<10 ppm) for 500 hours at 1095°C. Micrographs and depths of corrosion of the 1200°C annealed specimens at various initial oxygen concentration are shown in Figure 4. Nb-1\%Zr alloy specimens annealed at 1200°C with <3375 ppm O initially (stoichiometric with respect to zirconium) showed little weight loss or evidence of corrosion. Intergranular corrosion occurred only when the initial oxygen content of precipitation heat-treated Nb-1\%Zr exceeded 3375 ppm.

Table 3 compares oxygen concentrations for various Nb-1\%Zr systems. Based on the photographic evidence of Figure 4, noticeable intergranular attack of Nb-1\%Zr did not occur until the material exceeded 3375 ppm O.

Table 3. Initial oxygen concentrations Nb-1\%Zr

<table>
<thead>
<tr>
<th>Oxygen Concentration</th>
<th>Characteristic</th>
</tr>
</thead>
<tbody>
<tr>
<td>6125 ppm O</td>
<td>-0.002&quot; Nb-1%Zr after 500 hrs in Na 1095°C</td>
</tr>
<tr>
<td>3375 ppm O</td>
<td>O stoichiometry for Zr in Nb-1%Zr</td>
</tr>
<tr>
<td>2250 ppm O</td>
<td>-0.000&quot; Nb-1%Zr after 500 hrs in Na 1095°C</td>
</tr>
<tr>
<td>150 ppm O</td>
<td>O concentration reactor grade Nb-1%Zr</td>
</tr>
<tr>
<td>60 ppm O</td>
<td>O concentration high purity Nb-1%Zr [1]</td>
</tr>
<tr>
<td>&lt;10 ppm O</td>
<td>O concentration possible in purified Nb [2]</td>
</tr>
<tr>
<td>&lt;1 ppm O</td>
<td>O concentration possible in purified Na [3]</td>
</tr>
</tbody>
</table>


A first order estimate of long-term corrosion of the Nb-1\%Zr/Na system defined in Table 2 can be made with a series of very conservative assumptions. First ignore the observations that stoichiometric concentration is needed to start corrosion in Nb-1\%Zr and that corrosion rate decreases with exposure time (see ASTM G68-80). Instead, assume corrosion rate is linear with oxygen concentration and exposure time. Assume also that impurity isolation can be achieved between the heat pipe and surroundings by material selection, getters, impurity depletion, and geometric features (such as an isolating gas gap).

Linear ratios of \((\text{corrosion depth}) / (\text{exposure time})\) initial Nb-1\%Zr oxygen concentration product) are used to estimate long-term corrosion depth. Romano (1968) observed a 0.002" penetration after Nb-1\%Zr with an initial concentration of 4,400 ppm O was exposed to refluxing sodium for 500 hours at 1095°C. Bounding estimates are made. An upper bound estimate uses commercially purified Nb-1\%Zr with 60 ppm O. A lower bound estimate uses getter purified Nb-1\%Zr initially with 10 ppm O. A final estimate assumes that the 3375-ppm O threshold (stoichiometry) is not reached and no corrosion occurs. Calculation results are summarized and compared to a typical heat pipe wall thickness in Table 4.

Table 4. Nb-1\%Zr/Na corrosion depth estimates at 1095°C

<table>
<thead>
<tr>
<th>Time (hours)</th>
<th>Initial O (ppm)</th>
<th>Depth (inches)</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>500</td>
<td>4,400 ppm O</td>
<td>0.0020</td>
<td>Romano (1968)</td>
</tr>
<tr>
<td>105,120</td>
<td>~1,000 ppm O</td>
<td>-0.0000</td>
<td>Threshold unreached</td>
</tr>
<tr>
<td>105,120</td>
<td>10 ppm</td>
<td>0.0019</td>
<td>Lower bound</td>
</tr>
<tr>
<td>105,120</td>
<td>60 ppm</td>
<td>0.0112</td>
<td>Upper bound</td>
</tr>
<tr>
<td>-</td>
<td>-</td>
<td>0.0350</td>
<td>Wall thickness</td>
</tr>
</tbody>
</table>

*Initial sodium purity <10 ppm O

The assumptions used here tend to over-predict corrosion rate. The upper bound corrosion depth estimate predicts no Nb-1\%Zr wall penetration after 12 years operation at 1095°C. Using penetration of un-irradiated material as a failure criteria these estimates suggest ample margin for >30 years to penetration. Long term (>10,000 years).
hour) sodium corrosion data is currently under review and additional details in the literature will be provided in the future. Corrosion rate usually decreases with exposure time (ASTM G68-80) so long-term extrapolation from 500-hour data is conservative.

V.B. LCAC Mo/Na System

Figure 5 plots partial molar free energies of various Mo/Na/O compounds versus temperature. This data suggests that Na₂O does not transfer oxygen to metallic molybdenum in the form of MoO₂. Sodium molybdate is a more stable reaction product.

A molybdenum sodium heat pipe operated for 45,039 hours at 1391 K and 24 W/cm² (Lundberg, 1987). This test ended for lack of support. This heat pipe was made from low carbon arc-cast molybdenum that was 99.97% pure with less than 50-ppm carbon and 15 ppm oxygen. It is significant that this heat pipe operated near 1400 K, over 100°C higher than the design shown in Table 1. This suggests long life potential for heat pipes made from materials that have low levels of interstitial oxygen. Many molybdenum alloys are corrosion resistant in pure sodium.

TZM showed -0.01 mm grain boundary attack at welds in the vapor region of capsules containing sodium for 6271 hours at 1315°C (Berry, 1971).

Mo/Na systems life tested with UO₂ fuel met with mixed success. Although stoichiometric UO₂ is more stable than molybdenum, small deviations from stoichiometry can reverse this. Post-test examinations showed oxygen-depleted fuel suggesting oxygen transport from non-stoichiometric regions of the fuel (Lundberg, 1984).

A thermal chemical simulation of the Nb/K system with typical contaminant levels found no life limiting corrosion after 7 years of operation at 875 K (Feber and Merrigan, 1987). Based on the Nb-1%Zr/K life test data and modeling, it appears that Nb/K heat pipes have good potential for long life. Exposures of Nb-1%Zr up to 10,000 hours in refluxing potassium at temperatures to 1150°C showed virtually no attack (Romano, 1966). In the absence of oxygen alloys of niobium showed little evidence of corrosion mass transfer in refluxing potassium for up to 10,000 hours at 1200°C and 5,000 hours at 1300°C (DeVan, 1966). Potassium rapidly depletes oxygen from commercially pure niobium above 600°C. This observation opposes what would be expected from the Ellingham diagram for Nb/K (see Figure 7) suggesting ternary compound formation (no data found yet).

Glass (1999) gave results for the start up of an Nb-1%Zr/K heat pipe. Sena (1990) reported results for eight potassium filled Nb-1%Zr heat pipes that were tested for 7,000 to 14,000 hours in the 850-950 K range. One of these pipes developed a small evaporator leak at 13,000 hours that did not affect the operation of the heat pipe and was not detected by the vacuum system monitor. Tests on the other heat pipes concluded with no apparent problems. It is believed that Zr in an Nb-1%Zr centering wire came into contact with the quartz tube during test. Figure 6 shows a wire-quartz contact point on the failed heat pipe surface after 13,000 hours of test. Zirconium being more stable than quartz partitioned O, causing O to diffuse from the quartz to the condenser wall (see Figure 7). Oxygen from the quartz appears to have migrated to the heat pipe and saturated evaporator zirconium.

Figure 5. Partial molar free energies of Mo/Na/O compounds.

Figure 6. Example of contact point between Nb-1%Zr/K life test heat pipe and quartz enclosure.
**V.D. Refractory Metal Lithium Systems**

Only brief mention of refractory metal lithium system issues is given here. A summary of pre-1980 lithium heat pipe work is found in (DeVan, 1984). For reactor applications lithium heat pipes show exceptional performance at >1400 K. An axial power density of 23 kW/cm² at 1500 K was demonstrated with a Mo-Li heat pipe (Merrigan, 1986). Nb-1%Zr/Li and Mo/Li heat pipes have been tested on numerous occasions for instance (Merrigan, 1983) and (Reid, 1999). Lundberg (1984) presented data on a MO/Li heat pipe that was tested for 25,216 hours at 1700 K before the evaporator perforated. Failure of the molybdenum was attributed to grain boundary attack from nickel impurity. Operation for this length of time at >200°C above any designs considered to date suggests good potential for lifetimes exceeding 100,000 hours. Control of initial impurities in the container and fluid (especially Ni, O, N, and C) is essential for long life operation.

Figures 8 and 9 show partial molar free energies of oxygen and nitrogen compounds associated with some lithium refractory metal systems. Values for N compounds above 1200 K are extrapolations of free energy and solubility fits given in (Natesan, 1983). Lithium effectively getters O from Mo and Nb alloys but transfers N and C to refractory alloys. The solubility of N in Li is quite large.

Li₃N (melting point 1086 K) is unstable in Mo >1400 K and has been observed to contribute to molybdenum corrosion. As previously stated, residual nickel has also been found to play a role in Mo-Li corrosion (Lundberg, 1984).

Hafnium foil disks located at the evaporator end of the heat pipe have been used to getter nitrogen in molybdenum-lithium systems. Figure 9 indicates that zirconium and titanium also getter nitrogen from lithium. Combinations of these materials might provide margin in the event of the breach of a Re UN fuel liner.

Merrigan (1983) summarized steps taken to control contamination sources within a 2-m long Mo-Li artery heat pipe designed to operate at 15 kW and 1500 K. Chemical
characterization of heat pipe materials during fabrication and assembly, material cleaning during pre-processing, in-situ cleaning during heat pipe fill by hot and cold trapping, and gettering during operation were used. Tests were satisfactorily conducted on this heat pipe near the design point for >100 hours. No problems were apparent during this test series.

VI. METHODS TO ASSURE LONG LIFE

In this section, techniques to minimize and to precisely measure impurities in the heat pipe working fluid and structure are discussed. Proper isolation of the heat pipe from non-metallic impurity sources and the use getters will significantly increase the likelihood of indefinite heat pipe operating life.

VI.A. Start with Proper Material Selection

Nb-1%Zr was extensively used in this paper as an example of a material that has reactor heritage and demonstrated compatibility in refluxing sodium systems. Other refractory materials, such as TZM, or LCAC molybdenum with getters could work as well with proper fabrication techniques. Use of container and wick materials that contain materials such as Zr, Hf, and Ti greatly inhibits corrosion in sodium. Niobium based alloys that contain these elements include:

- **Nb-1%Zr:** Nominal Composition (wt%) 98.5 Nb min, 0.8 to 1.2 Zr. Composition limits 0.0100 C max, 0.0150 O max, 0.0300 N max, 0.0020 H max, 0.01 Hf max, 0.005 Fe max, 0.005 Mo max, 0.005 Ni max, 0.005 Si max, 0.1 Ta max, 0.03 W max. Nb-1%Zr is easily fabricated and available.

- **C-103:** Nominal Composition (wt%) 89 Nb-10 Hf-I Ti. Composition limits 0.01 C max, 9 to 11 Hf, 7 to 1.3 Ti, 0.7 Zr max, 0.0300 O, 0.0300 N max, 0.020 H max, 0.5 W max, 0.5 Ta max, Bal Nb. C-103 is easily fabricated and available.

- **C-129Y:** Nominal Composition (wt%) 80 Nb-10 W-10 Hf-0.1 Y. Composition limits 9 to 11 W, 9 to 11 Hf, 0.05 to 0.3 Y, 0.5 Ta, 0.5 Zr max, 0.015 C max, 0.025 O max, 0.015 N max, 0.0015 H max, Bal Nb. Tougher to fabricate and weld than C-103 and maintains significant strength to 1650°C. Not now in production. It would take some time to obtain useful quantities of C-129Y. C-129Y is more difficult to fabricate than Nb-1%Zr or C-103.

Standards for reactor grade Nb-1%Zr are contained in ASTM B391-03, B392-99, B393-99, B394-99. Note that ASTM B392-99 applies to wire with diameter greater than 0.02 inches. Since <0.01-inch diameter wire is required for fine-mesh screen B 392-99 does not strictly apply.

Use of UN contained within a Re diffusion barrier minimizes nitrogen contamination from the fuel. In addition liberal use of Zr, Hf, or Ti foils, films, or wires within the heat pipe or on the outside of the heat pipe can protect the system from external sources of contamination providing almost unlimited life margin.

VI.B. Deplete Impurities from Wall and Wick

For long heat pipe life the container and working fluid must be sufficiently free of non-metallic impurities to prevent the initiation of corrosion. Processing procedures have evolved to keep non-metallic impurities in the wick and wall within acceptable levels. These processing procedures have the side benefit of eliminating outgas products. All parts that are to be exposed to working fluid must be chemically cleaned and vacuum fired above the core operating temperature in an ultra clean furnace. Examples of the chemical cleaning and welding steps for niobium alloys are contained in the ASM Handbook and (Lessmann, 1984).

Heat pipe wicks, particularly those made from sintered powder or felt have large surface to volume ratios. Care must be taken to ensure that surface and bulk contaminates are kept low in these structures. Once materials enter the fabrication process it is a good idea to avoid exposing them to atmosphere. Inert gas carriers can be built for transfer of articles between dry boxes or vacuum systems.

Kim (1997) used titanium gettering to deplete oxygen from reactor grade Nb. A temporary Ti or Zr layer can be deposited on to niobium by vacuum sputtering or chemical vapor deposition. The assembly can then heated to a temperature (based on diffusion rates) and for a time sufficient to deplete the wall of impurities but insufficient to cause significant diffusion between the tube lattice and the getter. If desired, the layer can be removed by chemical or mechanical means. Similar approaches are found in the US patent literature (Wurm, 1978) and (Fisher, 1991). Adsorbed oxygen on refractory metal surfaces or in solution can be reduced with Ca, Ba, or molten Li immersed in getters. A heat pipe built using molten Li gettering is reported by (Merrigan, 1983).

The total impurity concentration of C, O, H, and N in Nb and Nb-1%Zr can be measured using glow discharge mass spectrometry or the residual resistance ratio value. This ratio is the dc resistivity of the metal at room temperature to the resistivity at 4 K (in the non-superconducting state). Coupon samples should be removed from the heat pipe wall and wick at each step during fabrication as part of the quality control process. Periodic chemical analysis should be used to cross check these impurity measurements.
VI.C Isolate Condenser from External Impurity Sources

Surrounding structures in contact with the heat pipe must contain low impurity levels of or be isolated from the heat pipe (particularly the condenser). Fortunately, there are ways to maintain thermal contact between the core, heat pipe, and heat exchanger under these restrictions.

Figure 10 suggests two possible methods to isolate an Nb-1%Zr heat pipe condenser from external impurity sources. The method depicted on the top shows the heat pipe condenser bonded to heat exchanger surface with a "fat" braze of a getter material such as a V or Ti alloy. Alternatively, the heat exchanger material itself could consist of a heavily gettered alloy such as C-103. The method depicted on the bottom shows a gap filled with a conducting gas such as He that isolates the heat pipe condenser surface from heat exchanger impurities. The weld region forms a single contact point further isolated from the core and heat exchanger with getters. Fuel tube to heat pipe bonds by hot isostatic pressing of Nb-1%Zr have been recently demonstrated for modular cores. Rhenium sleeves can isolate nitrogen in UN fuel from the rest of the system.

![Image](image.png)

**Figure 10. Methods to isolate a heat pipe condenser from external sources of impurities.**

VI.D Minimize Mobile Oxygen in Working Fluid

Techniques have been developed to control sodium purity to the demonstrated detection limit (~0.1 ppm O). These techniques can be incorporated into a flow loop connected to a heat pipe sodium distillation apparatus (ASTM G 68-80) and (Lundberg, 1980). Specifications for reactor grade sodium are contained in ASTM C 1051-85. Electrolytic reactor grade sodium stock can be procured such as DuPont NIAPURE brand. Reactor grade sodium is among the cleanest source stocks available. It contains trace impurities of P, I, Cl, S, Si, and B. Corrosion mechanisms for these elements have not been established and are likely unimportant. Cold trapping and distillation should reduce these elements to negligible levels.

Raw NIAPURE supplied to Los Alamos in early 2000 contained 105(+30) ppm O (determined by neutron activation analysis). Passing the raw sodium stock through a ~10 micron filter at 120°C cleans it to ~10 ppm O level. The filtered sodium can be introduced into a loop containing hot and cold traps as well as impurity measuring devices. A port at one end of the flow loop might be configured to allow sodium transfer to a distillation apparatus.

High purity alkali metal working fluid can be introduced into the heat pipe by vacuum distillation or directly transferred from the loop. A distillation unit should be made from stainless steel or nickel. Distillation of sodium at ~350ºC-400ºC removes common impurities to negligible levels except potassium. Table 5 lists approximate impurity concentrations after typical purification steps.

<table>
<thead>
<tr>
<th>Step</th>
<th>Amount in Na, ppm O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Obtain Reactor Grade Na</td>
<td>100.00</td>
</tr>
<tr>
<td>Cold Filter Na (15 μ, 120ºC)</td>
<td>~10.00</td>
</tr>
<tr>
<td>Cold/Hot Trap Na</td>
<td>1.00</td>
</tr>
<tr>
<td>Vacuum Distill Na</td>
<td>~0.10</td>
</tr>
<tr>
<td>Getter Trap Na (theoretical)</td>
<td>&lt;0.01</td>
</tr>
</tbody>
</table>

VI.E Measure Sodium Impurity Concentration

Impurity concentrations in sodium can be measured by a number of industry standard techniques discussed in ASTM C 997-83. Methods for pre-fill (in-loop) purity monitoring of a representative sodium sample include:

a. Plug meter for coarse impurity measurement with range 5 to 100-ppm O.

b. Electrochemical meter with range 1 to 30-ppm O.

c. Vanadium wire oxygen activity technique with range ~0.1 to 20 ppm O.

d. Distillation of Na from a crucible. Residual oxygen in the crucible is measured by analytical techniques with range 1-100 ppm O.

Slightly different methods apply for the measurement of carbon and hydrogen in sodium. The cost versus benefit of measuring for these elements as well as others should be carefully examined.

The vanadium wire technique was successfully used for sodium loops in the EBR-II program (Smith, 1972). A vanadium wire present in the heat pipe during processing appears suitable to characterize oxygen levels in fully assembled and filled heat pipe modules. A method has been devised to remove the wire from the sodium during closure with negligible impurity introduction. The validity of this technique can be independently confirmed by analysis of sample heat pipe modules.
Figure 11 depicts one possible sequence of steps. Sodium is introduced into the heat pipe by transfer through a fill stem containing a 0.01-inch diameter x 1.2-inch long vanadium wire (step “A”). The heat pipe is then closed and wetted over a 48-hour period at the core design temperature. The heat pipe is inverted and heated to immerse the vanadium wire in sodium. The assembly is then brought to ~750°C for 4 to 24 hours to reach an equilibrium oxygen concentration between the sodium and the wire.

The heat pipe is cooled to room temperature and the fill stem containing the vanadium wire is severed from the heat pipe inside an argon dry box (step “C”). The wire and a sodium sample are extracted from the fill stem for analysis. A cap is attached to the heat pipe using an electron beam welder connected to the dry box (step “D”). Since the vanadium wire further purifies the sodium during equilibration some correction will be necessary to establish the true oxygen concentration in the working fluid before and after equilibration.

**VI.E Incorporate Getters as Part of the Material System**

Non-soluble getters remove interstitial impurities from heat pipe materials during processing as well as slow diffusion rates and trap impurities during operation. Group IVB elements such as Ti, Zr, and Hf are virtually insoluble in sodium. They form low free energy compounds with corrosion inducing impurities. Exposing these materials to sodium removes these impurities. The rate-determining mechanism associated with oxygen gettering is solid-state diffusion of oxygen through the material. To work properly it is important that these materials have a large active surface area that is well distributed through the heat pipe, especially near the evaporator. Alloys with Ti-Zr combinations have enhanced gettering rates over pure group IVB elements. Getters may be considered for use in either niobium or molybdenum systems.
VII. QUALITY CONTROL PRACTICES

To date, most alkali metal heat pipes have been built in small quantities in research and development environments. Production and R&D reliabilities necessarily differ, the former far exceeds the latter. R&D prototypes fail more frequently than production systems as new processes are established. When failure sources become known, corrective action enhances prototype reliability. As investment is made for successively larger lot sizes quality control practices improve and reliability increases.

Lewis (1996) summarizes general engineering reliability practices. A “bathtub” shaped curve describes time-dependent failure rate of component life. Early in life variability due to manufacturing process defects can result in high component failure rate. Juvenile failures might result from weld failures, and corrosion or de-wetting from excess non-metallic impurities not removed by processing and fill. Failure rate remains small and roughly constant during the normal operating period if working fluid and structure impurity concentrations are controlled during processing. Steps that should maximize heat pipe reliability and life for cores containing tens to hundreds of heat pipes (Reid, 2003) include:

a. Build each flight heat pipe module under a strict quality control program. Use procedures during all phases of heat pipe manufacture to minimize non-metallic impurities in the working fluid, wick, and wall.

b. Identify sub-standard material batches and monitor material and weld defects ultrasonically and by radiography.

c. Measure impurity levels in wall, wick, and working fluid samples as a part of the fabrication quality control process.

d. Perform accelerated life tests on sample modules at elevated temperature, mass flux, and various impurity levels to determine corrosion sensitivity. Shorter accelerated tests may be possible by operating the heat pipe nearer its operating limit.

e. Conduct destructive chemical and metallographic examinations on sample modules during and after accelerated life test for evidence of corrosive attack and to determine impurity distributions. Compare measured impurity distributions to thermal chemical model predictions.

f. Conduct acceptance tests on each flight module to identify fill errors and to weed out defects. A 6 to 8 month acceptance test should be sufficient. If schedule becomes tight acceptance tests might be accelerated in a similar fashion to life tests.

g. Assay completed flight heat pipe as a quality control check to ensure nothing went wrong during the heat pipe fill and close out process.

h. Perform post-acceptance destructive evaluation on a representative sample of modules.

VIII. CONCLUSIONS

Heat pipe core point designs are well within heat pipe axial and radial steady state performance limits. Alkali metal heat pipe operation has been demonstrated in micro gravity with good agreement with established design models. With proper cleaning and out gassing procedures and good fabrication and welding methods as part of an effective quality control program heat pipe defects can be reduced to negligible levels.

Alkali metal heat pipes have successfully operated for several years under reactor prototypic conditions without contaminate getters. Incorporation of impurity getters is a worthwhile investment to provide an extra level of assurance for a flight system. The life times achieved in the EBR-II reactor indicate that corrosion rates in alkali metal heat pipe systems are not noticeably influenced by fast neutron irradiation.

Accelerated tests of refractory metal heat pipe assemblies under core prototypic conditions can answer, compatibility and materials related life questions. The redundant nature of a heat pipe cooled reactor core provides in-depth reliability. The heat pipes themselves are reliable and the consequences of individual heat pipe failure are small.

Heat pipe technology for primary core cooling has the benefit of simplicity, maturity, and reliability necessary for further consideration in supporting NASA's future space exploration needs.

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