AN INVESTIGATION OF CORROSION IN LIQUID-METAL HEAT PIPES

Douglas R. Adkins, K. Scott Rawlinson, Charles E. Andraka, Steven K. Showalter,
James B. Moreno, Timothy A. Moss, and Patricia G. Cordiero
Sandia National Laboratories*
(505) 844-0611, dradkin@sandia.gov

ABSTRACT

Research is underway to develop a 75-kW heat pipe to transfer solar energy from the focus of a parabolic dish concentrator to the heater tubes of a Stirling engine. The high flux levels and high total power level encountered in this application have made it necessary to use a high-performance wick structure with fibers on the order of 4 to 8 microns in diameter. This fine wick structure is highly susceptible to corrosion damage and plugging, as dissolved contaminants plate out on the evaporator surface. Normal operation of the heat pipe also tends to concentrate contaminants in localized areas of the evaporator surface where heat fluxes are the highest. Sandia National Laboratories is conducting a systematic study to identify procedures that reduce corrosion and contamination problems in liquid-metal heat pipes. A series of heat pipes are being tested to explore different options for cleaning heat-pipe systems. Models are being developed to help understand the overall importance of operating parameters on the life of heat-pipe systems. In this paper, we present our efforts to reduce corrosion damage.

INTRODUCTION

The Department of Energy is sponsoring programs to commercialize solar-to-electric power-generating systems that use parabolic mirror solar concentrators coupled with a Stirling engine and generator. One of the major challenges of this program is to develop a solar receiver system to transfer energy from the focus of the concentrator to the working fluid of the engine. Flux levels near the focus can be on the order of 100 W/cm^2 , and depending on the quality of the concentrator, the flux distribution on the receiver can be very nonuniform. In earlier solar/Stirling systems, tubes transporting the engine's working fluid were directly heated with concentrated solar energy. This practice created large thermal gradients on the tubes and led to concerns about the life expectancy of direct-illumination receiver systems. The uneven flux profiles on the receiver also led to problems in achieving a balanced thermal input in multi-cylinder engines.

Heat-pipe solar receivers are being developed as an alternative to directly illuminated tube receiver systems. In a heat-pipe receiver system, a wick structure distributes sodium across a solar-heated dome, and thermal energy is removed from the dome as the sodium evaporates (see Fig. 1). Sodium vapor condenses on the heater tubes of the engine where the energy is transferred to the engine's helium working fluid. Condensed liquid then flows back to the wick-covered evaporator surface under the influence of gravity. Since the sodium is in a saturated state, temperatures within the receiver are uniform and, therefore, thermal stresses are minimized.

The concept behind a heat pipe receiver is straightforward, and recent tests by Andraka et al. [1996] demonstrated that a heat-pipe receiver system could improve the efficiency of a dish/Stirling system by 20% over a directly illuminated tube system. Despite the advantages of a heat pipe receiver, operating requirements on the wick structure have made the receiver development challenging. The wick is required to distribute liquid sodium across a receiver dome that is nominally 16 inches (41-cm) in diameter and to withstand

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peak flux levels approaching 100 W/cm^2. Often, it must operate in orientations where gravity opposes the flow of liquid in the wick.

The ideal wick would have a micron-sized pore structure for high capillary pumping capabilities, and it would be nearly 100 percent porous to minimize flow losses through the wick. Pores in the wick would also be graded so that vapor bubbles that may form near the heated surface could migrate to the wick surface and escape rather than remain in the wick and block the flow of liquid. This ideal wick could be envisioned as a cubic lattice made of infinitely fine wires that are separated by a few microns near the heated surface and open up to a few tens of microns towards the wick's free surface. Many of these ideal features can be found in felt metals that are commonly used in filter applications. Felt metals made of micron-sized wires can have effective pore radii in the 20 to 60 micron range while retaining porosities greater than 90% [Adkins, 1995]. Through fiber selection and selective compaction, it is also possible to create a graded pore structure to aid in the movement of vapor out of the wick. (Even without these measures, felt wicks have a distributed pore structure that provides for some venting of vapor.)

Mechanical instability and the susceptibility to corrosion are the major drawbacks to using a felt wick. Felt metals of interest for receiver wicks are made of wires 2 to 10 microns in diameter. These felts can support loads of only a few thousand Pascals before collapsing. At typical operating temperatures of 700 to 800°C, even the surface tension loads imposed by the sodium can crush the wick. Methods to support the wick and prevent crushing are being investigated, but the major focus of this paper is on corrosion. After only a few hours of operation, the fine wires of the wick can dissolve into the liquid-metal working fluid and lead to a failure of the receiver. Efforts to understand corrosion and dissolution processes in heat pipes and to determine procedures to reduce these problems are discussed here.
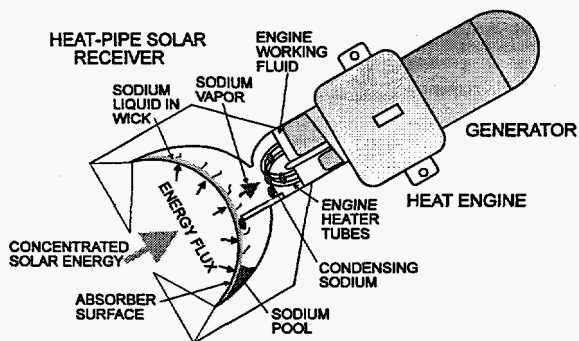

Figure 1. Operating schematic of a heat pipe solar receiver.

CORROSION MECHANISMS AND MITIGATION

Corrosion and compatibility issues in liquid-metal systems have been studied extensively in relation to nuclear power reactors, and reviews of this work are provided by Foust [1972], Barker and Wood [1974], and Natesan [1975]. A general finding from this work is that stainless steels and refractory metals have solubilities below a few ppm in pure liquid metals, even at elevated temperatures. Impurities such as oxygen, however, can drastically increase the dissolution rates in stainless steels [Barker and Wood, 1974]. It is hypothesized that the accelerated dissolution rates are a result of the formation of ternary compounds, such as $NaCrO_2$ or Na_4FeO_3, that are relatively soluble in liquid metals [Lundberg, 1987].

While research for liquid-metal reactors is applicable to heat pipe systems, there are unique differences. Typical sodium reactor systems were designed for operating temperatures below 600°C, whereas sodium heat pipe systems usually operate at temperatures above 700°C. In heat pipes, metals and contaminants are also concentrated in specific regions of the heat pipe as a result of the evaporation and condensation processes. Los Alamos National Laboratory has been somewhat successful in modeling the corrosion process in heat pipes, but limited thermochemical data and inadequate kinetic considerations hinder the predictive capability of the model [Merrigan and Feber, 1985].

The combined effects of an increased material solubility with oxygen levels and the action of the heat pipe to concentrate impurities, such as oxygen, creates a situation where the wick structure in the evaporator is extremely vulnerable to damage. Natesan's review of corrosion in liquid metal systems found that corrosion rates in liquid metals vary linearly with oxygen concentration. A 1-ppm oxygen concentration in sodium could cause corrosion rates on the order of 1 micron per year on stainless steel, and a 10-ppm concentration could cause a 10 micron per year rate under the same temperature and flow conditions. Oxygen can be introduced with the sodium, however, this is generally not a major source. At a typical sodium transfer temperature of 160°C, only 3 ppm of oxygen can be carried into solution. Most of the oxygen contamination is introduced into the system on the surface of the wick [Ivanovsky et al., 1995]. For a typical heat-pipe receiver system, oxides on the felt wick surface can raise the oxygen levels in the sodium to about 100 ppm by weight if steps are not taken to reduce the native oxide layer [Adkins, 1996].

Heat pipes are typically cleaned through flushing techniques or high-temperature vacuum bakeouts. Vacuum bakeouts will not be effective unless temperatures are high enough to reduce the native oxides. In processing sodium heat pipes, Los Alamos uses vacuum firing temperatures of 1400 K (1127°C), and it is suggested that this temperature is effective in removing oxygen from the system [Woloshun, 1997]. Mausteller, et al. [1967] indicate that carbon in stainless steels reduces oxides and forms CO at elevated temperatures. At

Sandia, it has been observed that CO is the dominant residual gas released from stainless steel systems that are vacuum fired at temperatures above 950°C. Even at firng temperatures of 1127°C, however, Woloshun [1997] indicates that vacuum baking can take on the order of a week.

Oxides can be reduced at lower temperature by providing a reactive atmosphere in the heat pipe. Hydrogen firing is traditionly used to reduce surface oxides, but this practice will introduce hydrogen into the systems that can be difficult to remove [O'Hanlon, 1980]. Another option is to use the reactive liquid metal itself to flush the system clean and transport contaminants to separate loops where they can be trapped. Mausteller, et al. [1967] provide a practical guide to cleaning liquid metal systems through flushing and trapping, and Stelman and Newcomb [1993] describe the use of such a system for cleaning heat pipes. In processing their system, Stelman and Newcomb vacuum baked the heat pipe at 427°C, soaked the interior of the vessel for 24 hours in sodium at 427°C, and then flushed the heat pipe for 48 hours with hot sodium. The hot soak and flushing is repeated at least three times to achieve oxygen concentrations below 10 ppm in the drained sodium.

Like vacuum baking, the flushing procedures described by Stelman and Newcomb took a week or more to complete. The process could be accelerated by increasing the flushing temperature and by ensuring that the process does not become diffusion limited. Areas of stagnant liquid will prevent the effective transport of oxygen from the surface and lengthen processing times. Rather than flushing liquid through the vessel, directly condensing the liquid on the surface will ensure that the liquid near the contaminated surface is pure, and diffusion distances are minimized. Small-scale tests were recently performed to determine the effectiveness of reflux cleaning. The following section describes these tests and compares the results between two cleaning processes.

BENCH-SCALE RECEIVER SYSTEM
Construction
A schematic of the bench-scale receiver system is illustrated in Fig. 2. The envelope of the receiver is made of a nickel-based super alloy (Haynes 230) which has been selected for full-scale receiver applications because of its high strength and air-side corrosion resistance at elevated operating temperatures. The wick material is a 316L stainless steel metal-fiber mat that is sold by Bekaert Fibre Technologies as Bekipor® WB 04/150, where 04 refers to the wire diameter (4 μm) and 150 refers to the mass of one square meter of the material (150 g/m²). Two of these vessels were constructed for the cleaning tests, and construction of the test vessels is described by Adkins et al. [1996].

One vessel was subjected to minimum processing to serve as a reference. It was vacuum baked at 700°C for four days before it was charged with 450 g of sodium. A fan-folded strip of zirconium was located in the bottom of the heat pipe in the pool area to act as a getter for oxygen. The second heat pipe was vacuum baked at 600°C for 72 hours, then flushed with liquid sodium through a distillation/condensation process referred to as reflux cleaning. A 450-g sodium charge was first loaded into a stainless steel vessel at the bottom of the heat pipe. This fill vessel was then heated to vaporize the sodium. Sodium vapor traveled into the test pipe, condensed on the interior surfaces at a temperature between 500 and 600°C, and then flowed back to the fill vessel. Reflux cleaning continued for approximately six hours before the final charge was completely distilled from the reflux/fill can to the test vessel. Zirconium was not used in the second vessel.

Operation
After the cleaning and filling, the heat pipes were mounted vertically in a test stand, and a 42-mm x 102-mm area was heated with an array of quartz lamps (see Fig. 2). The average flux into the heated area was approximately 60 W/cm². Temperatures were controlled by circulating air across an uncovered wall of the heat pipe with a thermostatically controlled fan. At the operating temperature of 750°C, the pool was approximately 64 mm (2.5") deep. The lamps were 51 mm (2") from the top of the receiver, so most of the condensation occurred on the side opposite the lamps. The liquid was required to flow about 444 mm (17.5") to reach the top of the heated area. The total power throughput was on the order of 2600 W based on calorimetry tests on an earlier system.
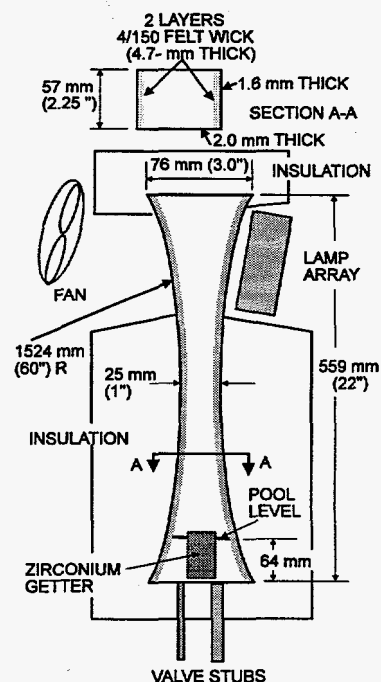

Figure 2. Schematic of the bench-scale receiver system.

The reference pipe was tested first. After operating 1995 hours, a hot spot developed on the surface near the top of the heated region. Sodium was distilled at 600°C from the heat pipe, and the vessel was milled open and sectioned. Near the top of the evaporator region, the wick was eaten through to the substrate, and a white residue was on the surface. About 1 cm below this hole in the wick, a thin stripe of copper appeared, and many other deposits could be observed in the wick structure with a microscope. A black deposit was present in the

Postmortem

The structure of the wick was examined using scanning electron microscopy, and energy dispersive spectroscopy (EDS) was used to determine the composition of corrosion products. Results from the reference pipe are shown in Fig. 3. Near the hole in the evaporator wick, there were oxide formations containing the primary constituents of stainless steel (see point B at the top of Fig. 3) as well as calcium (point A). Wick material extracted from the condenser section showed some dissolution and corrosion although the attack was less pronounced than was observed in the evaporator area. Sites containing both oxygen and aluminum were identified (see Region B in center photograph of Fig. 3); however, the general fiber surfaces exhibited the nominal composition of the as-received felt alloy. Black deposits in the pool area were found to have zirconium, nickel, and oxygen as the primary constituents. Nodules on the pool wick that are illustrated in the lower photograph of Fig. 3 were composed of the elemental constituents of stainless steel with a very high concentration of chromium. Chrome probably came out of solution in the pool because the liquid pool was about 200°C cooler than the vapor. Away from the chrome nodules, the composition of the pool wick material matched the standard constituents of 316L.

Results from the reflux-cleaned heat pipe are shown in Fig. 4. Deposits in the evaporator and the pool area that were prevalent in the base-line heat pipe were not present in the reflux-cleaned system. The wick material in the evaporator had a knobby and faceted appearance; however, weight measurements revealed that there was no loss of material. It is possible that the faceting is a result of recrystalization of the stainless steel fibers at elevated temperatures [Hlava et al., 1997]. Similar facets appeared in all regions of both of the tested heat pipes. Wick material from the condenser region was relatively clean. A few small deposits of sodium oxides were visible on the surface of the condenser. No copper deposits were found on the surface of the wick; however, a copper nugget was found embedded in the evaporator wick. Around the copper nugget, there were signs of corrosion damage in the 316L stainless steel wick material and the Haynes 230 substrate.

pool area, and a strong sulfur smell emanated from the heat pipe after it was opened. The wick in the evaporator was compacted to less than 1 mm thick.

The reflux-cleaned vessel operated for 1300 hours before a localized hot spot developed in the heated region. Like before, sodium was distilled from the vessel prior to sectioning. There was no sign of corrosion damage, but it was evident that the wick had compacted in the evaporator region.

DISCUSSION OF RESULTS

From the tests, it appears that the reflux cleaning was effective in removing the oxygen from the system. The material deposits and mass transfer that were apparent in the base-line heat pipe were not present in the reflux-cleaned system. Reflux cleaning, however, was not effective in removing copper from the system. The copper is an artifact left from the forming of the micron-sized wire, and it has been found on the surface of the 316L stainless steel felt materials obtained from Bekaert. The concentration of copper on the fiber was measured to be about 2 percent by weight.

The minimum time that is required to remove oxides from interior surfaces of the heat pipe through reflux cleaning is still unknown. This time will be a function of the reflux flow rate on the contaminated surface, the reflux temperature, and the reaction rate between the metal-oxides (chrome and iron) and the liquid sodium. It is assumed that the reaction rates are relatively fast, but there was no documented reference for this assumption. In earlier tests, it was observed that after about six hours of operation, condenser regions appeared to be relatively clean, but small regions of the evaporator were heavily contaminated. This was the basis for the six-hour reflux cleaning time chosen for this series of tests. It is likely that a longer reflux time is required to draw copper into solution, and this is the reason that copper remained in the system after the reflux cleaning.

While reflux cleaning appears to be an effective method of cleaning a heat pipe, there is a possibility that oxides can be reintroduced into the system during the process through some vapor transport mechanism. Ivanovsky, et al. [1995] indicate that distillation/condensation cleaning methods are ineffective because a "fine system of complex compounds is volatile." The precise meaning of this statement is unknown, but Ivanovsky et al. imply that the sublimation of compound colloids and the transfer of compound colloids by wet vapor droplets plays a major role in transporting oxygen in heat pipes. Mausteller et al. also suggest that oxygen can be transported back into the system as CO if the distillation process continues until the fill vessel is completely dry. Neither of these oxygen transport mechanisms appears to have hindered the reflux cleaning process in the present test.

The tests showed that corrosion damage could be avoided with reflux cleaning, but questions remain about how bench-scale results translate to full-scale systems. Corrosion rates are affected by operating temperatures, liquid flow rates,

and contaminant concentrations in the liquid metal. The 750°C operating temperature of the bench-scale system is typical of temperatures encountered in full-scale systems. Liquid flow rates are proportional to the thermal flux on the evaporator surface, and, at 60 W/cm^2, the bench-scale heat pipe was at the upper end of fluxes encountered in full-scale receiver systems. With respect to temperature and liquid transport rates, the bench-scale heat pipe is representative of a full-scale receiver.
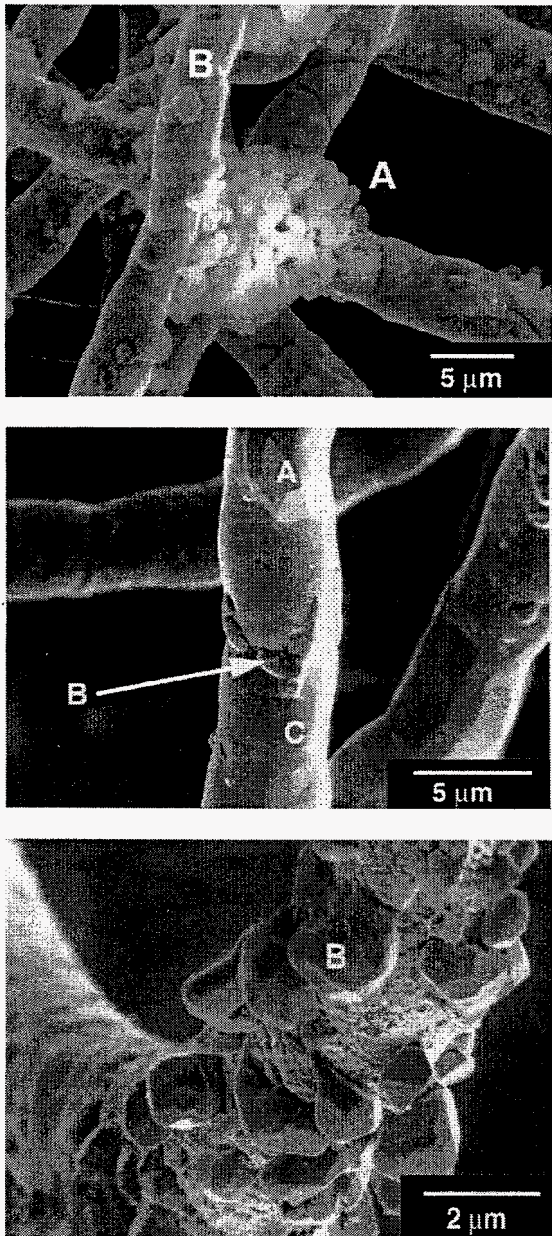

Figure 3. Scanning electron micrograph of the wick structure in reference heat pipe. (Top) Calcium-oxide formations in the evaporator region. (Middle) Relatively clean wick structure in the condenser. (Bottom) Chromium nodules on the wick structure in the pool area.
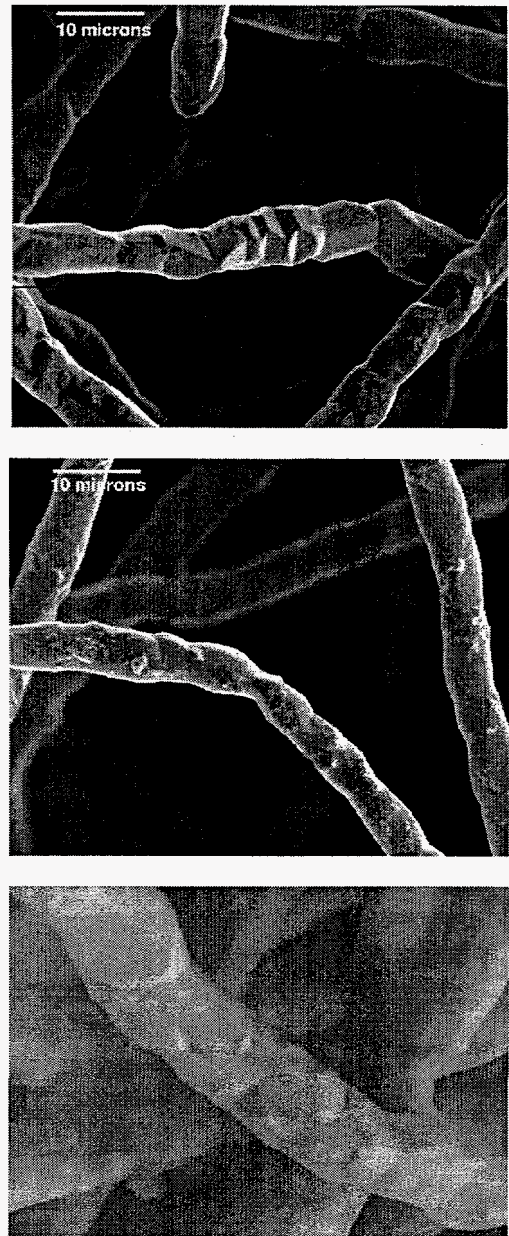

Figure 4. Scanning electron micrograph of the wick structure in the reflux-cleaned heat pipe. (Top) Evaporator region with no deposits. (Middle) Clean wick structure in the condenser. (Bottom) Pool area is free of chromium nodules on the wick.
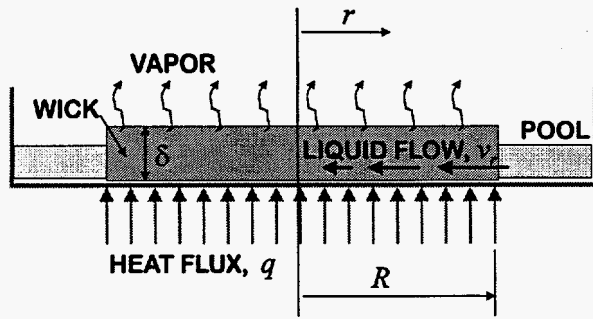
Figure 5. Two-dimensional liquid flow on a disk-shaped evaporator subjected to a uniform heat flux.

APPLYING BENCH-SCALE RESULTS TO FULL-SCALE RECEIVERS

In full-scale receiver systems there is a two-dimensional concentration of contaminants, but in the bench-scale system the concentration is more one-dimensional. The magnitude of contaminant concentration can be estimated by considering the simple disk-shaped evaporator system illustrated in Fig. 5. Here it is assumed that a pool of sodium around the edge of the disk feeds a wick covering the disk. As a uniform flux is applied to the disk, sodium evaporates from the wick, and liquid is drawn from the periphery to a stagnation point at the center. For this system, the superficial radial velocity of liquid flowing through the wick is

$$v_r(r) = -\frac{q\,r}{2\,\delta\,h_{fg}\,\rho_{Na}} \,, \qquad (1)$$

where h_{fg} and ρ_{Na} are respectively the latent heat of vaporization and density of the liquid sodium, and the other variables are described in Fig. 5. Diffusion limits the magnitude of contaminant concentration at the

stagnation point, and once steady-state conditions are achieved, the diffusion of a contaminant in sodium is described by the expression,

$$\rho_c\,v_r = \rho_{Na}\,D\,\frac{d}{dr}\left(\frac{\rho_c}{\rho_{Na}}\right) , \qquad (2)$$

where ρ_c is the density of the contaminant and D is the binary diffusion coefficient of the contaminant in sodium. For an initial contaminant concentration of ω_0, Equation 2 can be integrated to determine the final concentration distribution of the contaminant in the wick as

$$\omega_r = \frac{\rho_c}{\rho} = \omega_0\left(\frac{R^2}{\kappa}\right)\frac{exp\left(-r^2/\kappa\right)}{1 - exp\left(-R^2/\kappa\right)} \,, \qquad (3)$$

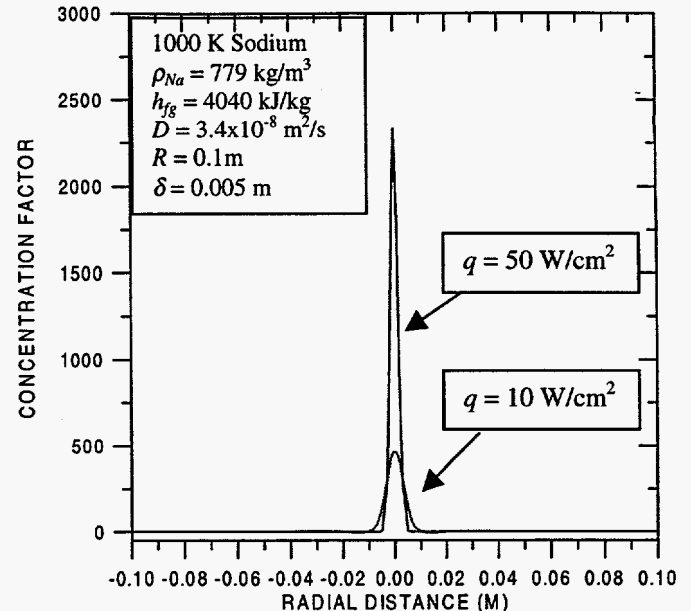where $\kappa = 4\,\delta\,D\,\rho_{Na}\,h_{fg}/q$.

Figure 6. Concentration effect of 2-D flow on a disk-shaped evaporator surface.

Figure 6 shows the oxide distribution predicted by Equation 3 for typical properties and dimensions encountered in a receiver system. A reference for diffusion coefficients of oxides in sodium was not available, so, for this example, the self diffusion coefficient was estimated from a correlation that Foust [1972] provides for liquid sodium up to 226°C. Predicted concentrations should be accurate at least to the order of magnitude. As Fig. 6 illustrates, the evaporative action of the heat pipe can cause contaminants to increase a thousand-fold above the initial concentration in the sodium, and the peak concentration is directly proportional to the heat flux. In general, the contaminants will be concentrated to a region roughly 2 centimeters in diameter for any evaporator surface where $R > 2\sqrt{\kappa}$. While these results are based on a disk-shaped evaporator, they are indicative of results that might be encountered in any wick system with two-dimensional concentration. On a full-scale hemispherical receiver, any localized high-flux region that creates a flow stagnation point will concentrate contaminants into a centimeter-sized area where maximum wick damage will occur. This estimated concentration area correlates well with observations on full-scale receivers.

The bench-scale receivers can more appropriately be represented as a system where a uniform flux is applied to a WxL rectangular surface where sodium is introduced along one edge of length W. An analysis similar to the one above shows that the concentration distribution is given by

$$\omega_x = \omega_0\left(\frac{4\,L}{\sqrt{2\pi\,\kappa}}\right)\frac{exp\left(-2\,x^2/\kappa\right)}{erf\left(2L/\sqrt{2\kappa}\right)} , \qquad (4)$$

where x is the distance from the top of the heated area, L is the length of the heated zone, and κ was defined in the previous example. For a 10-cm long heated zone subjected to a 60 W/cm^2 flux, the peak concentration factor is around 85 times the original contaminant concentration in the liquid metal. This indicates that the peak contaminant concentrations in a full-scale receiver system may be an order of magnitude higher than those encountered in the current bench-scale tests.

Corrosion problems appear to be eliminated with reflux cleaning, however, it is not clear that the bench-scale results can be fully translated to a full-scale system. Much higher contaminant concentrations will be encountered in full-scale systems as a result of two-dimensional concentrating effects.

CONCLUSIONS

Heat-pipe systems are being designed to transport solar-thermal energy from the focus of a parabolic mirror to the heater tubes of a Stirling engine. These heat-pipe solar-receiver systems are intended to operate for 90,000 hours with little or no maintenance. The lifetime of the heat-pipe systems is highly dependent on the purity of the sodium working fluid of the heat pipe. One of the major sources of contamination in the heat pipe is the native oxide layer on the interior surfaces of the heat pipe. The operating characteristics of a heat pipe will tend to concentrate damage in the evaporator section of the heat pipe. High heat flux levels and operating temperatures contribute to the rapid deterioration of the current systems. In full-scale receiver systems, contamination levels can be concentrated 1000-fold during normal operation of the receiver.

Two cleaning procedures were described in this paper. The 600°C vacuum bake out was not effective in removing oxygen from the test heat pipe. Oxygen-contaminated liquid sodium dissolved metals from the wick, and deposited them in the pool and the evaporator. Contaminants were concentrated in the evaporator where the wick developed a hole. There was no sign of corrosion damage or material transport in the reflux-cleaned vessel. The evaporator wick in both heat pipes, however, collapsed during operation. This collapse was attributed to the surface tension in the liquid pulling downward on the wick surface.

Efforts to mitigate the corrosion effects are still under development. It may be possible to accelerate the reduction of the native oxide layers with a higher temperature (greater than 900°C) vacuum bake out and refluxing. A series of capsule tests is now underway to determine a cost-effective system for processing liquid metal heat pipes.

POSTSCRIPT

The collapse of the felt wick remains an issue. Prior to operation, the wick was approximately 5-mm thick, and, after the tests, the evaporator wick was less than 1-mm thick on each of the two heat pipes. This collapse was probably caused by the surface tension at the liquid/vapor interface. Both heat pipes failed when the systems were restarted from a routine maintenance shutdown. The collapsed wick was unable to supply the flow of sodium necessary to cool the heat pipe absorber surface. In future tests, a support screen will be sintered on top of the felt to prevent the collapse of the wick.

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