NASA/TP—2010–216452

Refractory Metal Heat Pipe Life Test—Test Plan and Standard Operating Procedures

J.J. Martin and R.S. Reid
Marshall Space Flight Center, Marshall Space Flight Center, Alabama

December 2010

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LIST OF ACRONYMS AND SYMBOLS

Ar	argon
CDU	compact distillation unit
DE	destructive evaluation
Di	inside diameter
EBW	electron beam weld
H	hydrogen
HCI	hydrogen chloride
He	helium
HOV	hand-operated valve
H_2O_2	hydrogen peroxide
LANL	Los Alamos National Laboratory
Mo	molybdenum
MSFC	Marshall Space Flight Center
N	nitrogen
Na	sodium
NAA	neutron activation analysis
NaOH	sodium hydroxide
Nb	niobium
NDE	nondestructive evaluation
Ni	nitrogen

LIST OF ACRONYMS AND SYMBOLS (Continued)

PG	pressure gauge
PLC	programmable logic controller
Re	rhenium
RF	radio frequency
RGA	residual gas analyzer
SAFE	safe affordable fission engine
TFE	tetrafluoroethylene
TIG	tungsten inert gas
TP	Technical Publication
V	vanadium

NOMENCLATURE

A	area (m^2)
A_v	orifice area (m^2)
$C_{\underline{O}(Na)F}$	final concentration of oxygen in sodium (ppm)
$C_{\underline{O}(Na)I}$	initial concentration of oxygen in sodium (ppm)
C_p	coolant specific heat (J/kg-K)
$C_{\underline{O}(V)}$	concentration of oxygen dissolved in vanadium (wt%)
d_{Na}	diameter of sodium in container
d_V	diameter of vanadium wire
g	acceleration of gravity (m/s^2)
h	height (m); height sodium column (m)
L_c	condenser length
L_e	evaporator length
L_{Na}	length of sodium in container
L_V	length of vanadium wire in test volume
$M_{\underline{O}(Na)F}$	final mass fraction of oxygen dissolved in sodium
$M_{\underline{O}(Na)I}$	initial mass fraction of oxygen dissolved in sodium
$M_{\underline{O}(V)}$	mass fraction of oxygen dissolved in vanadium
M	mass (kg)
M_O	oxygen concentration (ppm)
$m_{\underline{O}(V)F}$	oxygen content of wire (mg)
$m_{\underline{O}(V)I}$	oxygen content of fusion blank (mg)
m_V	weight of wire (mg)
m	mass
$\dot{m}$	coolant mass flow rate (kg/s)
MW_{Na}	molecular weight of sodium (22.98997)

NOMENCLATURE (Continued)

MW_O	molecular weight of oxygen (15.9994)
MW_V	molecular weight of vanadium (50.9415)
$N_{\underline{O}(Na)}$	atom fraction of oxygen dissolved in sodium
$N_{\underline{O}(V)}$	atom fraction of oxygen dissolved in vanadium
O	distillate mass ratio $\left(\frac{\text{kgO}}{\text{kgNa}}\right)$
P_{sat}	sodium saturation pressure (MPa)
$\dot{Q}_{calorimeter}$	power absorbed by the calorimeter (W)
$\dot{Q}_{support}$	power absorbed by the support structure (W)
$\dot{Q}_{total}$	total power transferred by the condenser (W)
q	radiation heat transfer (W/cm^3)
q_{rad}	total power (W)
R	universal gas constant (J/mole-K)
r	radius; fill tube inner radius (m)
T	temperature (K)
t	time (s)
T_{inlet}	coolant inlet temperature (K)
T_{outlet}	coolant outlet temperature (K)
T_{sat}	sodium saturation temperature (K)
α(T)	normalized Arrhenius diffusion rate
ρ	density (kg/m^3)
ρ_{Na}	density of liquid sodium at 750 °C (7.727 g/cc)
ρ_V	density of vanadium (6.1 g/cc)
γ	ratio of specific heats
ρ_{sat}	sodium saturation density (kg/m^3)
σ	surface tension (N/m)
τ	time interval (s)

TECHNICAL PUBLICATION

REFRACTORY METAL HEAT PIPE LIFE TEST—TEST PLAN AND STANDARD OPERATING PROCEDURES

1. INTRODUCTION

The objective of this effort is to characterize the aging of molybdenum- (Mo-) rhenium (Re) alloy heat pipes by accelerating life-limiting factors through testing over a range of operating temperatures and mass fluences. To accomplish this goal, an experimental study has been proposed that will make use of two test series centered about a baseline design point (app. A). The first series, based on ASTM G68-80[1] and referred to as the G-series, investigates time-dependent corrosion effects. The second series, based on a Fisher central composite[2] design and referred to as the F-series, is intended to cross correlate temperature and mass fluence effects. The experimental hardware consists of sixteen ≈12-in-long Mo-44.5%Re heat pipes with annular crescent wick structures (fabricated from seven layers of Mo-5%Re 400 × 400 mesh cloth) and filled with sodium working fluid, as shown in figure 1. The heat pipes shall be evaluated in environmental test chambers to provide the required test conditions. Power will be applied to the heat pipes from a set of noncontact radio frequency (RF) inductive coils. Heat will be removed from the condensers with noncontact water-cooled laminar flow calorimeters through a static gas gap (test chamber is flooded with an inert low-pressure mixture of helium (He) and argon (Ar)). The temperature of each heat pipe is measured with a two-band optical pyrometer focused at the evaporator exit through a small gap between the RF inductor and calorimeter.

To govern planned operations during the fill, processing, and evaluation of the heat pipe units, a general test plan and set of standard operating procedures have been developed and are presented in this Technical Publication (TP). These plans and procedures are general outlines based on the current design and operations as understood at the time this TP was generated. During actual hardware buildup and checkout testing, these plans and procedures shall be updated and modified as required to incorporate the as-configured setup and lessons learned. The outlined plans and operations presented in this TP are based on experience gained during the buildup and testing of stainless steel/sodium heat pipe modules used in the safe, affordable fission engine (SAFE), 100-kW test program.[3,4]

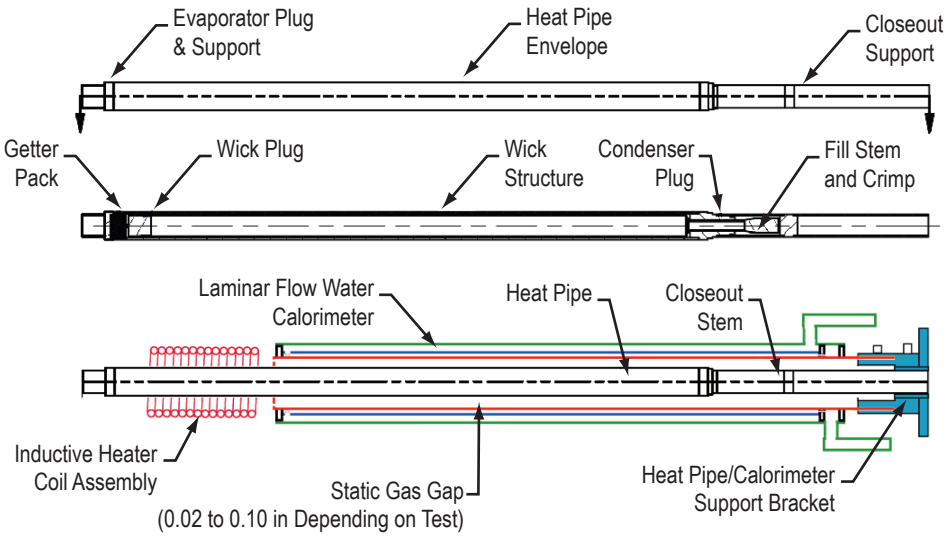

Figure 1. Heat pipe cross section.

2. GENERAL TEST PLAN

A general process flow, referred to as the general test plan, has been developed. This plan will serve as a guide to the sequence of events and standard operating procedures developed to support this project. Implementation of this plan will begin once completed heat pipe units are received from the selected fabrication vendor. All aspects of the heat pipe fabrication are regulated by the statement of work as specified in the final procurement contract. An overview of the general process is illustrated in figure 2.

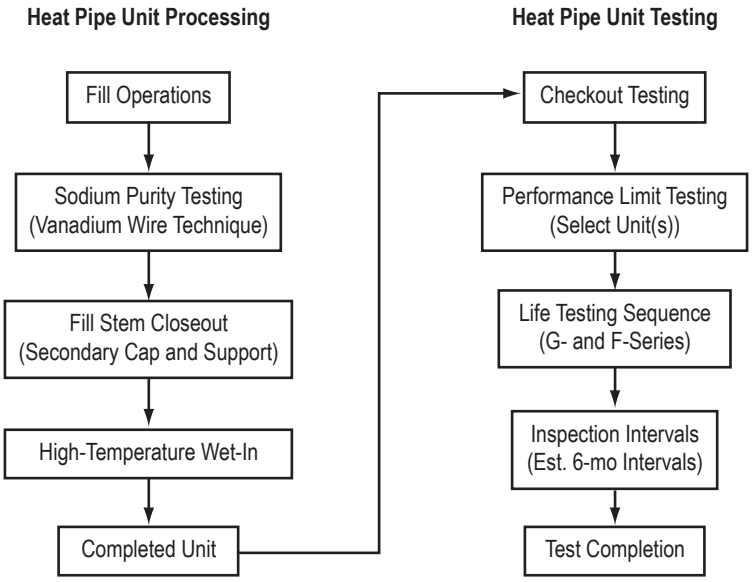

Figure 2. General operational sequence.

General standard operating procedures have been developed to cover the fill and processing segment of the test plan and will be discussed in section 3. The remainder of this section discusses the proposed test matrix, hardware checkout testing, performance testing to examine boiling limits, and long-term accelerated life testing, the primary emphasis of this effort.

2.1 Test Matrix Description

The project goal is to set up a series of tests that produce data that can (1) be compared to the historical database of previous heat pipe tests and (2) demonstrate a procedure to systematically assess heat pipe aging. The primary concern with heat pipe life is the long-term effects of corrosion and their impact on performance. To gather these data in a timely fashion, a means of accelerating the test evaluations is required. Because corrosion rate is proportional to temperature and the accumulation of nonmetallic elements in the evaporator, increasing temperature and mass fluence hastens aging. Elevated temperatures provide an increase in both the diffusion rate of impurities

from the structural elements and the rate at which these impurities attack the heat pipe evaporator material. A high mass fluence provides a mechanism that rapidly sweeps available impurities to the evaporator section, maintaining the highest impurity concentration at the hottest location. With this understanding, test conditions can be selected that establish and isolate corrosion trends as a function of temperature and mass fluence. The proposed test matrix, shown in table 1, lists a series of conditions that satisfy the accelerated test requirements. Sixteen tests have been baselined in the test matrix, using short heat pipes (active heat pipe length of ≈12 in) that have thick condenser/evaporator walls (0.625-in diameter with a 0.05-in wall thickness). This heat pipe design provides a condenser-to-evaporator-length ratio of approximately 3:1 and boiling limits that are invariant with condenser length; therefore, the shortened heat pipe units should perform similarly to the full-length versions.

Table 1. Proposed heat pipe life test matrix.

Test Identifier	Power Level (W)	Heat Pipe Temperature (K)	Gas Gap Mixture	Gas Gap Width (in)	Coolant Flow (gal/min)	Coolant Delta Temperature (K)	Number of Units
G-Series	3,000	1,273	He-32%Ar	0.025	0.61	18.8	7
F(-4)	5,000	1,273	He-6%Ar	0.025	0.77	24.6	1
F(-3)	1,000	1,273	He-32%Ar	0.103	0.11	35.2	1
F(-2)	3,000	1,373	He-32%Ar	0.031	0.56	21.1	1
F(-1)	3,000	1,173	He-32%Ar	0.021	0.65	17.6	1
F(0)	3,000	1,273	He-32%Ar	0.025	0.61	18.8	1
F(1)	2,000	1,223	He-32%Ar	0.037	0.54	13.9	1
F(2)	4,000	1,223	He-32%Ar	0.017	0.67	22.8	1
F(3)	2,000	1,323	He-32%Ar	0.046	0.50	15.2	1
F(4)	4,000	1,323	He-32%Ar	0.020	0.63	24.0	1

The proposed series of accelerated life heat pipe tests permits extrapolation of corrosion effects of a reference design from separate tests conducted over varying durations. The specified test matrix includes seven test units to address long-term corrosion rates (constant temperature and power with variable mass fluence) following guidelines specified in ASTM G68-80.[1] These seven tests are referred to as the G-series. The test matrix also includes nine tests that examine the cross-correlation trends between variable mass fluence and temperature on corrosion rate, using a Fisher central composite test design. These nine tests are referred to as the F-series. During this test program a combination of nondestructive and destructive examinations shall be performed to track the progression of any observed corrosion; the first destructive evaluation will be performed within 6 mo after the start of testing (the first G-series heat pipe unit). Possible identification of random manufacturing and processing defects should be somewhat easier since a series of heat pipes with overlapping operating condition will be evaluated.

The planned test matrix is aggressive in some cases, with the targeted operating temperature and power combinations producing radial heat fluxes up to 10 times that of a flight heat pipe design. For example, Figure 3 shows that test cases F(-4), F(-2), and F(4) are very near the predicted boiling limits for a 2-μm nucleation site radius. Heat pipe performance limits were assessed using the HTPIPE code.[5] This assessment indicated that the uncertain effects of aging and the onset of corrosion could cause a normally operating heat pipe unit to reach a boiling limit after many hours are accumulated because of a slow increase in the nucleation site radius. Strict attention to manufacturing details such as surface finish and fluid charging methods—e.g., distillation filling to minimize residual noncondensable gas in the heat pipe—should help mitigate boiling concerns.

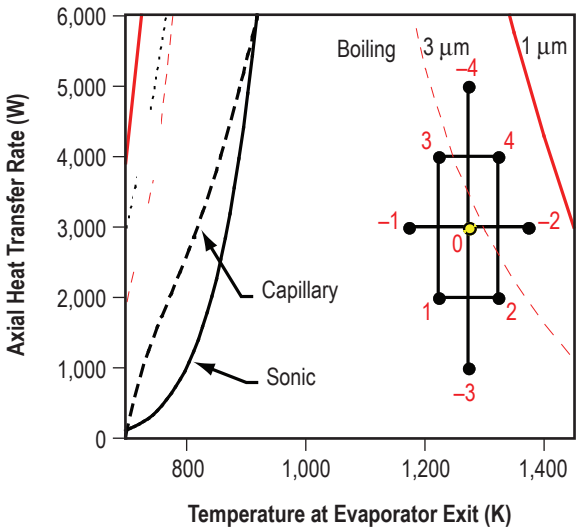

Figure 3. Proposed life test matrix compared to operating limits at two nucleation site radii.

A performance test on the first completed heat pipe is key to understanding the operating limits achievable with current fabrication/processing approaches. Specifics of the performance test will be discussed in section 2.3. Results of these tests will be used to determine if a possible reduction in operating conditions is necessary to provide long-term test capability while avoiding a boiling limit, which would prematurely remove a heat pipe unit from the test matrix. On the contrary, reduced power/temperature conditions would require an increase in test time to achieve an equivalent mass fluence.

In addition to the operating temperature and power goals, table 1 also specifies the primary test support system requirements to meet goals. These include the following:

- Static gas gap—a fixed parameter sized to the applicable calorimeter during fabrication (based on calculated performance). Once selected, only test chamber gas composition can be varied to adjust condenser/calorimeter coupling.

- Gas mixture—the test chamber gas mixture is the only way to make gross adjustments to the final thermal coupling; for the present calculations, this composition has been set to He-32%Ar by mass.

- Coolant flow rate—adjusted to maintain a sufficiently low calorimeter temperature to avoid a boiling condition in the coolant and also to provide a reasonable value for measurement (middle of flowmeter range).

- Coolant water temperature rise—balanced with the flow rate condition to provide an easily discernible temperature increase to simplify measurement.

For all test operations, the environmental chamber(s) shall be maintained at an absolute pressure in the 50- to 100-torr range, low enough to reduce convection yet high enough to minimize the concern of voltage breakdown. The planned hardware layout would place five heat pipe units per test chamber (heat pipes grouped by like requirements). This would necessitate three test chambers to handle all outlined test cases with the exception of case F(-4); this high-powered case requires a different gas mixture and will be tested in a separate test chamber setup. A discussion of the life test operations (sec. 2.4) addresses the test chamber arrangement and configuration in more detail.

2.2 Checkout Testing Operations

To improve the potential for overall success, a number of checkout evaluations shall be performed during the course of the project. These checkouts shall focus on the operations, procedures, and hardware layouts necessary to support both heat pipe fill/processing and heat pipe test chamber setup, verifying the integrity, performance, and repeatability of each system element necessary to achieve the overall project goal. Ancillary systems that shall be evaluated by checkout during build include, but are not limited to, the following:

- Fill, sampling, closeout, and wet-in of prototype heat pipe units.
- Feed-through/primary flange sealing and placement for environmental test chamber setup.
- Inert gas system supply and purification network used to regulate the environmental chamber atmosphere.
- Vacuum and bake-out system used to condition the test chamber and inert gas feed systems.
- Cooling water configuration to supply calorimeter system and other devices.
- Radio frequency heating system including inductive coils, interconnecting bus network, and power monitoring.
- Instrumentation configuration to verify data sampling and health monitoring.
- Control test methodology to verify performance and response to redline cuts, component failure, and power-out conditions.

As individual systems are successfully evaluated, they will be integrated to form a complete hardware test arrangement allowing full system performance to be evaluated; i.e., a full dress rehearsal. These fully integrated tests shall make use of one or more 321 stainless steel/sodium heat pipe prototype units, shown in figure 4. These heat pipes were fabricated to the same specification as the proposed Mo-44.5%Re heat pipe units and make use of a crescent annular wick composed of seven layers of 400×400 mesh 304 stainless steel wire cloth. These units are not equipped with an internal getter (to minimize cost), nor do they have the 0.5-in-long external evaporator end support, which can be added if necessary. These stainless steel heat pipes will perform similarly to the Mo-Re heat pipes; however, their high-temperature operating condition would be limited to below ≈1,150 K.

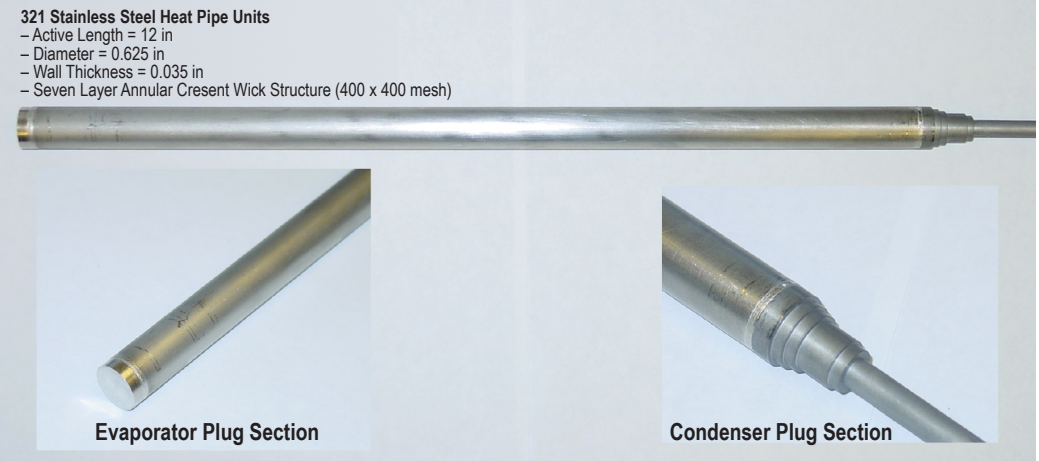

Figure 4. Stainless steel heat pipe prototypes.

Although high-temperature material strength limitations prevent the stainless steel units from being used to evaluate boiling limits, lower temperature operation will provide sufficient thermal performance data to confirm sizing of the heat pipe/calorimeter gas gap and RF system coupling. Any significant deviation from the original design analysis—flagged by these checkout tests—can be adjusted and calorimeters resized to meet power and temperature goals. These units shall serve as forerunners to the refractory metal test components, providing valuable insight into the processing and operational aspects and giving early experience to guide the final configuration without the worry of damaging expensive and long-lead refractory metal components. As progress is made in setting up the ancillary hardware systems, checkout evaluations shall be performed.

2.3 Performance Test Operations

The operating temperature and power in the currently planned test matrix push heat pipe performance, exceeding those of normal operation. In several cases, the test conditions push the heat pipes very close to the boiling limit. Figure 5 illustrates this limit with two average evaporator inner wall surface roughness factors—potential boiling nucleation sites. It is apparent from this data that the test conditions F(-4), F(4), and F(-2) fall in a range in which minor variations in the as-manufactured surface finish can significantly impact performance. In the procurement specifications, the required surface roughness is 1 μm for heat pipe internal surfaces. Given this sensitivity, the following factors must be considered in the successful execution of the planned test matrix:

- As mentioned previously, the final as-fabricated roughness on the inner surface of the heat pipe evaporator envelope could affect heat pipe performance. Honing of the inner surface should produce the required finish; however, other factors, such as the manner in which the material is formed; e.g., powder metallurgy or arc cast, may limit the achievable finish.

- The possible presence of residual trace argon purge gas introduced into the heat pipe during fill that is not fully evolved during vacuum outgassing. Argon, should it be carried along the capillary channel, can reduce boiling limits by activating potential nucleation sites in the evaporator. The use of vacuum distillation filling, which is more complex and time consuming, should significantly reduce residual argon.

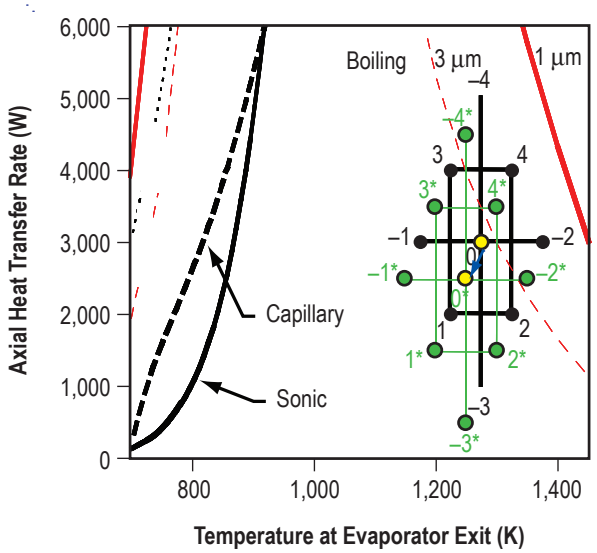

Figure 5. Proposed life test matrix with possible temperature and power adjusted sequence (denoted by '*').

- The unknown effects of aging could result in a gradual reduction of the boiling limit over time due to surface corrosion, resulting in increased local surface roughness with time. As a result, test conditions that lie closest to the boiling limit may exceed the limit over time, shutting down those heat pipe units.

Based on this sensitivity, it is critical that the first completed Mo-Re heat pipe be subjected to a performance test. This assessment will provide an indication as to the overall success, with respect to the calculated limit, of the as-fabricated hardware and sodium loading procedure. For this evaluation, a minimum of two power levels (in the 3,000- to 6,000-W range) should be examined to establish the limit curve. If the experimental boiling limit is numerically similar to the calculated value (for the nominal 1-µm-radius nucleation site), the current hardware and procedures should be sufficient to meet the test matrix conditions with acceptable margin. If a lower boiling limit is encountered, a reduction in overall test matrix temperatures or powers may be required to maintain a reasonable margin between normal operation and the limit, as illustrated by the slightly displaced '*' cases in figure 5. The outlined performance tests shed no light as to the effects aging may have on the boiling limit; therefore, sufficient margin should be incorporated to account for corrosion-induced degradation. Periodic nondestructive evaluations may be able to provide sufficient resolution to track the potential for boiling. The current plan will assess at least one, and possibly two, heat pipe units with a performance test to verify consistency.

2.3.1 Typical Hardware Configuration and Operation

The performance test will be conducted in the environmental test chamber designed for the 5,000-W accelerated life case (F(-4)). This chamber, illustrated in figure 6, has a diameter of 10 to 12 in with a length of ≈30 in. All seals used on the main flanges and feed-through will be metal knife-edge type seals to minimize the chance of air leakage into the system.

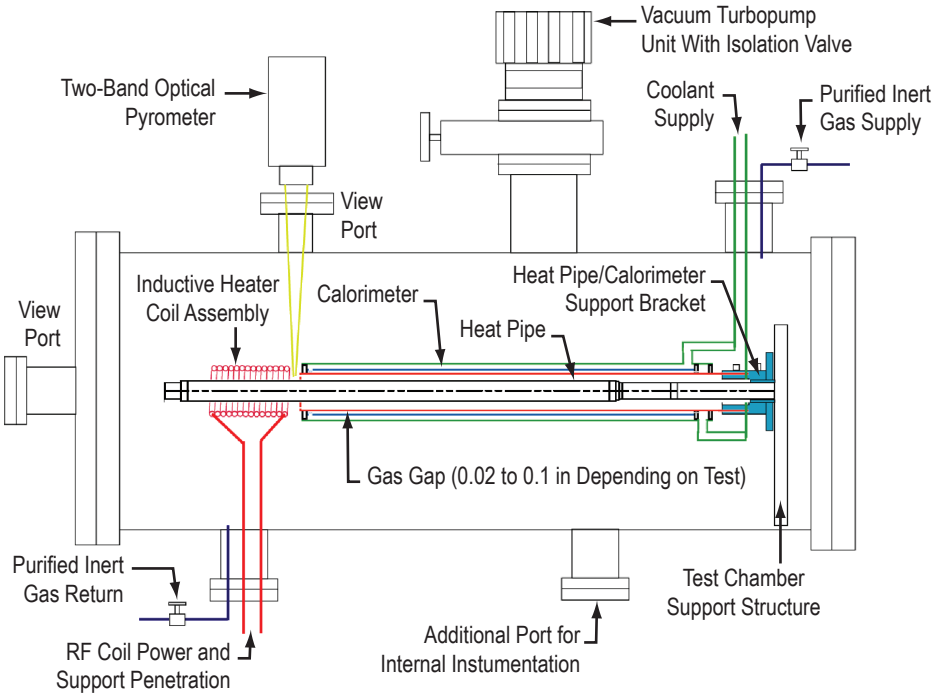

Figure 6. General layout for performance test setup.

The chamber is equipped with a vacuum pump and an inert purified gas system that provide a reduced pressure (50 to 100 torr) He-Ar mixture necessary to thermally couple the heat pipe to the calorimeter. The heat pipe and calorimeter are mounted to an attachment bracket that is secured to the test chamber support structure. The RF inductive coil is mounted to a feed-through that provides power. The coil does not touch the heat pipe unit. A view port allows a two-band optical pyrometer to monitor the evaporator exit temperature. An optical access port positioned at the heat pipe evaporator end allows for additional viewing instruments.

During a typical performance test, the heat pipe is expected to follow the sonic limit curve during startup to a point at which the condenser coupling to the calorimeter provides the limiting thermal resistance. At this point, the heat pipe will move off the sonic limit curve and track across the operating envelope to a target condition at the boiling limit. When the boiling limit is reached, there will be a drop in condenser power with a corresponding increase in evaporator outlet temperature, accompanied by bright spots or flashes from the heat pipe evaporator. Because of the tight wrapping of the RF inductive coil around the evaporator, visual inspection will be difficult and great care must be exercised as the boiling limit is reached. Optical observation, condenser power, and evaporator outlet temperature will be used to quantify the boiling limit during testing. Figure 7 illustrates a typical power versus temperature trace for a performance test using the test F(-4) hardware setup with two different test chamber gas species concentrations to vary the condenser/calorimeter coupling.

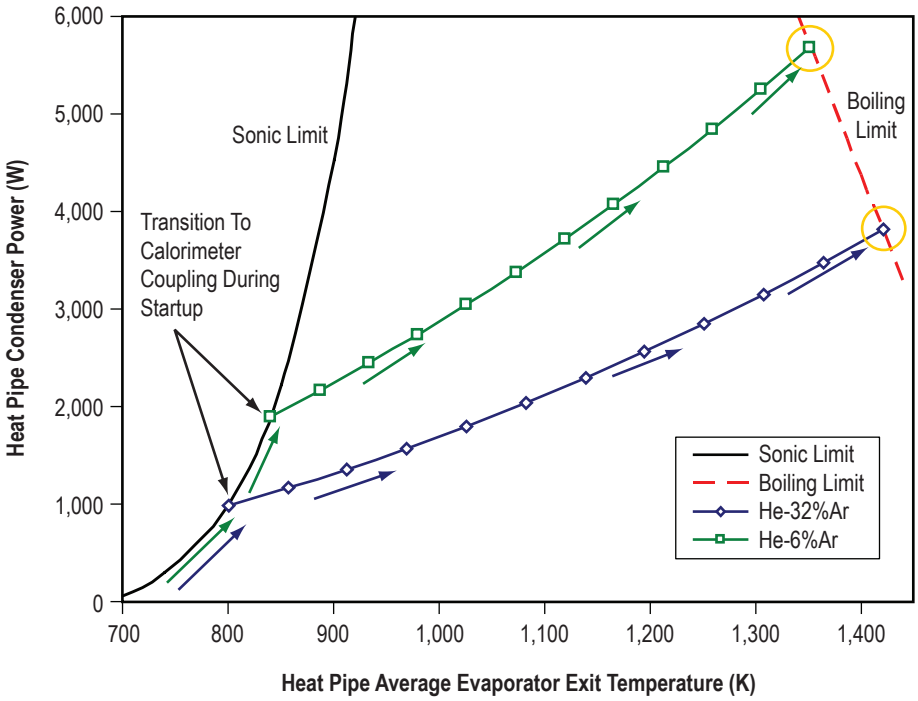

Figure 7. Nominal heat pipe response during performance test.

During testing, the heat pipe evaporator exit temperature will be monitored by the two-band optical pyrometer while calorimeter power is calculated in real time using the measured test parameters for flow and sensible temperature rise. The calorimeter power is given by:

$$\dot{Q}_{\text{calorimeter}} = \dot{m} C_p \left(T_{\text{outlet}} - T_{\text{inlet}}\right) \ , \tag{1}$$

where $\dot{m}$ is coolant mass flow rate, C_p is the coolant specific heat, and T is the temperature at the inlet and outlet of the calorimeter. To further refine this calculation, a detailed thermal model will need to be constructed based on the final heat pipe and support geometry. The total condenser power is given by:

$$\dot{Q}_{\text{total}} = \dot{Q}_{\text{calorimeter}} + \dot{Q}_{\text{support}} \ , \tag{2}$$

where $\dot{Q}_{\text{support}}$ reflects the transmitted condenser power absorbed by the support structure, by solid and gas conduction, that is not accounted for by the calorimeter. These values are expected to be low based on simplified assessments. The thermal losses to the environment from the evaporator surface are not a factor, because this heat energy is not transferred into the heat pipe working fluid.

2.3.2 General Performance Test Procedure

To execute a typical performance test (fig. 7), a general test procedure outlining the sequence of events has been developed. This outline covers items necessary to the successful operation of the baseline setup and addresses the following:

- Heat pipe and test chamber assembly preparation:
  – Basic cleaning and loading of the hardware.
  – Evacuation and leak checking of all components to verify seals.
  – Thermal bake-out of test chamber, gas system, and heat pipe units.
  – Charging of inert gas system and purging of the test chamber.

- Heat pipe operation:
  – Setup of cooling system, RF power system, and data/control system.
  – Ramping sequence to achieve desired test conditions.
  – Monitoring to determine limits and to keep system safe (redline cuts in place).

- System shutdown:
  – Sequence of cooling, power, and control systems for shutdown.
  – Preparing chamber environment for opening.

This general outline shall be expanded to include more specifics as hardware and test systems are detailed during the buildup and checkout phases of this program. In addition, a documentation file for each heat pipe unit, referred to as a 'traveler,' shall be carried throughout the process. Each traveler shall contain pertinent information regarding operations, test conditions, observations, and experimental findings for each unit subjected to a performance test. The general hardware and support systems used in this description are outlined in *NASA/TP—2010–216435*.[6]

2.3.2.1 Heat Pipe and Test Chamber Preparation. The following steps are used in preparation of the heat pipe and test chamber:

(1) Clean heat pipe unit using the approved procedure and handle only with powder-free, alcohol-cleaned, surgical gloves. This procedure includes removal of residue by immersing heat pipes in freon and ethanol in an ultrasonic cleaner.

(2) Wipe down test chamber with freon and alcohol to remove any potential grease, solvents, fingerprints, etc. Also perform complete visual inspection to verify that no foreign materials are present. Allow the test chamber to dry for a minimum of 1 hr.

(3) Install cleaned heat pipe unit in the test chamber heat pipe/calorimeter support bracket and secure.

(4) Close the test chamber and secure all flanges, using appropriate tightening sequence.

(5) Evacuate test chamber and inert gas feed system lines to 10^{-6} torr or lower and perform a helium leak check to a sensitivity of 10^{-10} std-cc/s He on all flanges, feed-throughs, and view ports. Tighten any leaky fittings using the appropriate tightening sequence; leaky view ports must be exchanged.

(6) Turn on the test chamber wall and inert gas feed system heater tapes and bring to a temperature of ≈200 °C to outgas all trapped water vapor and other volatiles. Hold at this heated condition for ≈24 hr or until pressure reaches the mid 10^{-8}-torr range; periodically monitor test chamber

vacuum pressure. Note: Test chamber pressure should increase during heating and then begin to fall after a steady temperature has been maintained for 1 to 2 hr. If pressure continues to rise or does not drop, there may be a leak caused by heating of the flanges. Perform a leak test while at temperature to locate the leak and tighten the offending flange.

(7) Initiate water flow in the calorimeter and set to minimize the uncertainty interval associated with measurement of $\dot{Q}_{\text{total}}$.

(8) Verify that all data systems are operational and the evaporator temperature measurement system (two-band optical pyrometer) is focused on the correct heat pipe location.

(9) Turn on the RF power system and bring the heat pipe temperature to ≈1,000 K as measured on the heat pipe evaporator over a period of 1 hr. Note: The test chamber pressure will increase as additional outgassing occurs.

(10) Maintain the heat pipe at a constant temperature of ≈1,000 K for ≈2 hr and monitor test chamber pressure; the pressure should begin to fall off after reaching a high value.

(11) Turn off the RF power system and all heaters, and monitor the test chamber vacuum level as the system cools; pressure should drop approximately one decade after the entire system is cooled to room temperature.

(12) Isolate the inert gas system from the vacuum chamber and charge the system with the required gas mixture ratio; typical gasses will be He with the desired percent Ar specified by weight. This is in preparation for multiple dilution cycles of the test chamber to sweep the environment.

(13) Isolate the test chamber from the vacuum system and then introduce the inert gas mixture purge into the test chamber to a pressure of ≈70 torr. Isolate the test chamber and let stand for 2 min.

(14) Isolate the inert gas system and open the vacuum system to remove the inert gas mixture from the test chamber. Allow the vacuum to reach the 10^{-6}-torr range.

(15) Perform two more inert gas dilution cycles, and then bring the test chamber pressure to ≈70 torr. Using the residual gas analyzer (RGA), take readings during the cycles.

(16) Engage the gas purifier and pump system; allow the system to circulate for 12 hr. Monitor the RGA during this process. The oxygen concentration should reach a level on the order of 0.3 parts per billion (ppb).

2.3.2.2 Heat Pipe Performance Test. The following steps are used to test heat pipe performance:

(1) Verify that the purified test chamber environment has been established to ≈0.3 ppb oxygen as read on the RGA.

(2) Initiate water flow in the calorimeter and set to a value consistent with the expected final power levels.

(3) Verify that all data systems are operational and that the evaporator temperature measurement system (two-band optical pyrometer) is focused on the correct heat pipe location. Set data rate to one sample per second.

(4) Turn on RF power system, bring heat pipe temperature to ≈950 K over a period of 1 hr, and allow it to stabilize. Note: The heat pipe temperature and power will place its operation to the right of the sonic limit in a heat transfer region controlled by the thermal coupling between the heat pipe condenser and calorimeter.

(5) Increase applied RF power slowly, increasing the temperature over ≈1 hr up to the boiling limit. This would correspond to a rate of approximately 7 to 10 °C/min.

(6) Engage test chamber wall cooling if required; do not allow the chamber surface temperature to exceed 150 °C.

(7) As the boiling limit is approached, provide visual surveillance of the heat pipe evaporator, as possible, due to RF coil obstructions, and monitor the data system outputs for heat pipe evaporator temperature and calorimeter power. Radio frequency power can be cut manually at any time if hot spots, flashes, or temperature/power excursions are observed. Note: Should an increase in evaporator temperature or loss of calorimeter power be measured beyond set redline conditions, the data/control system shall automatically cut RF power.

(8) Once the boiling limit has been reached and the RF power is interrupted, the system should be cooled to a temperature of ≈1,000 K and the RF power engaged and set to a power level sufficient to hold constant temperature. The heat pipe shall be held at this condition for several hours to make sure all surfaces are wetted. In extreme cases, a full length wet-in in a vacuum furnace may be required.

(9) After completion of the test, turn off the RF power and allow the system to cool to room temperature.

General Note: Throughout the operation, the RGA shall be monitored to verify that the inert test environment remains at ≈1 ppb oxygen or lower.

2.3.2.3 Heat Pipe Performance Test Shutdown. The following steps are used to shut down the heat pipe performance test:

(1) Isolate the test chamber inert gas system and shut down the calorimeter cooling loop.

(2) Using air to prevent asphyxiation issues, bring the test chamber to 1 atm so that it can be opened and the heat pipe removed.

(3) Close the chamber as soon as the heat pipe handling operation is completed and pull a vacuum on the test chamber; this will keep the chamber clean, simplifying the next test cycle.

2.4 Accelerated Life Time Test Operations

With completion of performance testing, the collected data shall be used to reassess the specific operating conditions in the overall test matrix, specifically, power and temperature for each of the F- and G-series conditions. The current planned test conditions are listed in table 1 and are illustrated in figure 8 in terms of evaporator exit temperature and mass fluence. Once the final test conditions are locked down, the 16 heat pipes will be divided into a number of test groups based on similarity of power level and inert gas composition. As shown in table 1, 15 of the tests require the same test environment (He-32%Ar), allowing them to share the same or similarly configured gas conditioning and processing system(s). The 16th heat pipe (condition F(-4)) requires a He-6%Ar mixture, due to the high heat flux, and will therefore require a completely separate test setup.

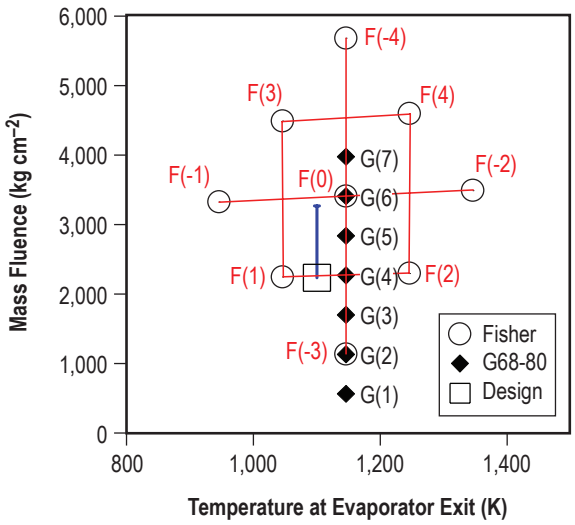

Figure 8. Life test matrix assuming a maximum 1-μm nucleation site radius for F- and G-series tests.

Subsequent to the test environment, the final grouping configuration is based on power level and the ease of setting up the RF power distribution. The design of the calorimeters is such that the gas conduction gap between the heat pipe and calorimeter is the limiting thermal resistance. The gas

gap was varied in the design to provide the desired operating temperature for a fixed heat transfer. This allows for a more compact grouping arrangement in the final test setup.

2.4.1 Typical Hardware Configuration and Operation

To manage the size of each setup, three environmental test chamber segments have been identified, with each segment containing five heat pipe units. These segments share a common vacuum, atmosphere control, and data/control systems. Radio frequency power would be provided to each test segment, driving the five heat pipe inductive coils in a series combination. The inductive coils contained in each test segment will be designed to provide the appropriate power level required by the test matrix, specific to the fabrication of the inductive coil assembly. This provides the flexibility of using one or more RF supplies, allowing test chamber segments to be hooked in series or operated independently as necessary. It may also be desirable to separate each of the test chamber segments for independent operation; e.g., independent operation may be desired to manage the possibility of heat pipe leakage due to corrosion or other breaching mechanisms. However, accomplishing this task would require duplication of many of the ancillary systems. The baseline layout for environmental test chamber segments to support the planned test matrix is described in sections 2.4.1.1 through 2.4.1.4.

2.4.1.1 Environmental Test Chamber Segment 1. The setup contains G-series heat pipe units in an He-32%Ar atmosphere. Each of the RF inductive coils is connected in series and designed to dissipate the same power.

Heat pipe units G1 to G5:

Operating temperature	1,273 K
Operating power	3,000 W

2.4.1.2 Environmental Test Chamber Segment 2. This setup contains the remaining G-series heat pipes and F-series heat pipe units with the power requirement. The atmosphere is He-32%Ar, and each of the five RF inductive coils is connected in series and designed to dissipate the same power.

Heat pipe units G6 and G7:

Operating temperature	1,273 K
Operating power	3,000 W

Heat pipe unit F(0):

Operating temperature	1,273 K
Operating power	3,000 W

Heat pipe unit F(-1):

Operating temperature	1,173 K
Operating power	3,000 W

Heat pipe unit F(-2):

Operating temperature	1,373 K
Operating power	3,000 W

2.4.1.3 Environmental Test Chamber Segment 3. The setup contains all F-series heat pipe units with varying power levels. The atmosphere is He-32%Ar. Each of the five RF inductive coils is connected in series and designed to provide a power ratio of 1, 2, and 4 to meet the following variable requirements:

Heat pipe unit F(-3)	
Operating temperature	1,273 K
Operating power	1,000 W
Heat pipe unit F(1)	
Operating temperature	1,223 K
Operating power	2,000 W
Heat pipe unit F(2)	
Operating temperature	1,223 K
Operating power	4,000 W
Heat pipe unit F(3)	
Operating temperature	1,323 K
Operating power	2,000 W
Heat pipe unit F(4)	
Operating temperature	1,323 K
Operating power	4,000 W

2.4.1.4 Environmental Test Chamber Segment 4. The setup contains a single F-series heat pipe unit that will be operated at the highest power level; the atmosphere will be He-6%Ar to improve heat transfer.

Heat pipe unit F(-4)	
Operating temperature	1,273 K
Operating power	5,000 W

The environmental chamber used to support performance testing operations described in the previous section shall be used as test segment 4 for the F(-4) accelerated life test. The performance test chamber will also serve as a forerunner to the buildup of the remaining test chamber segments so that any lessons learned can be incorporated. The other test chamber segments shall be designed in a similar fashion; however, these chambers will have a larger diameter and will have more and larger penetrations to accommodate the additional heat pipe units. Figure 9 illustrates the tentative layout of test chamber segments 1 to 3, configured for end-to-end attachment such that they may share common ancillary systems. Each test segment will have a diameter of ≈24 in and a length of 36 in. The large chamber flanges use a wire seal design; feed-throughs use a knife-edge type seal to minimize the potential for air leakage into the system. In addition, all fluid connections for cooling and RF power are either welded to the appropriate flange surfaces or brazed to an alumina-insulated feed-through to eliminate mechanical connections. The internal heat pipe and calorimeter arrangement is supported by a movable frame structure that is attached to the internal wall of the test chamber. The RF inductive coil arrangement is mounted on its own support structure, attached to the test chamber wall, and provides power to the heat pipes without making contact. The emphasis of the overall test chamber design and layout is to provide a configuration that minimizes the potential for air or water leakage to its interior in an effort to create a more trouble-free environment.

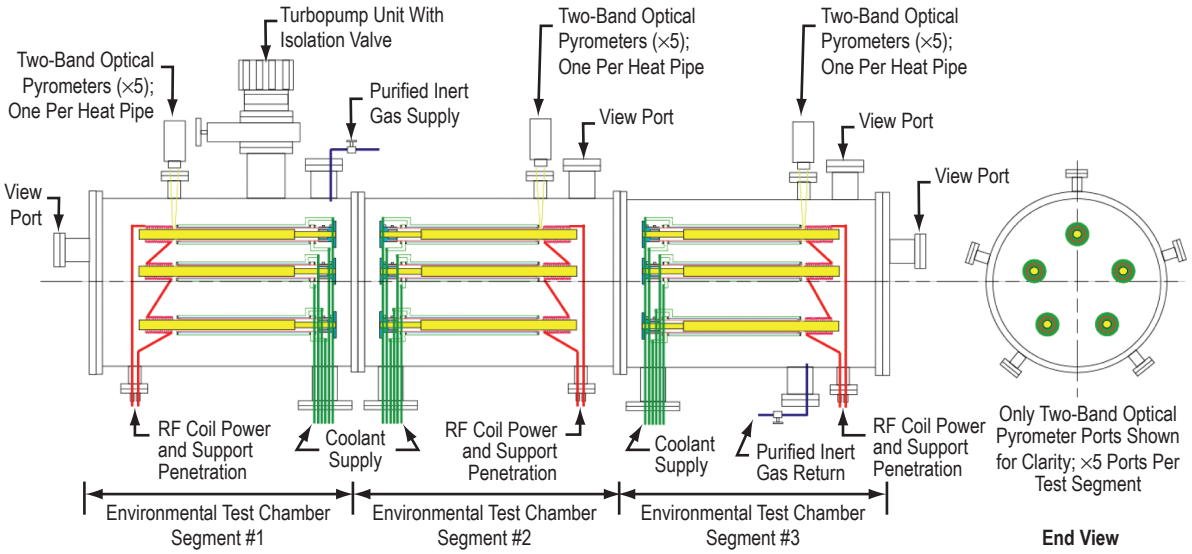

Figure 9. General layout for accelerated life test environmental test chamber setup.

Externally, the test chambers are equipped with ancillary systems to provide coolant water, RF power, and vacuum/inert purified gas. These chambers shall be operated at reduced pressure (50 to 100 torr) similar to that used on the performance test evaluation and will use a common mixture of helium and argon gas to achieve the desired thermal coupling. Additionally, sight glass access for two-band optical pyrometer temperature measurement instrumentation is provided. Each heat pipe will be equipped with its own pyrometer, positioned to provide an unobstructed view of the evaporator exit through the 6-mm gap between the RF inductive coil and calorimeter assemblies.

If the integrated test configuration discussed in figure 9 is used, all heat pipe units will need to be completed prior to starting the accelerated life testing cycle. Dividing each of the test chamber segments into a separate, independent setup allows for staging of the operation; however, this approach also introduces complexity. For independent segment operation, additional ancillary systems must be fabricated and verified and more variability may be introduced into the overall test environment, potentially impacting project goals. As a baseline, it is assumed that all three test chamber segments shall be operated as a single integrated unit. The expected startup transient and steady state conditions are shown for four cases (G-series and F(-4), F(-3), and F(-2)) in figure 10.

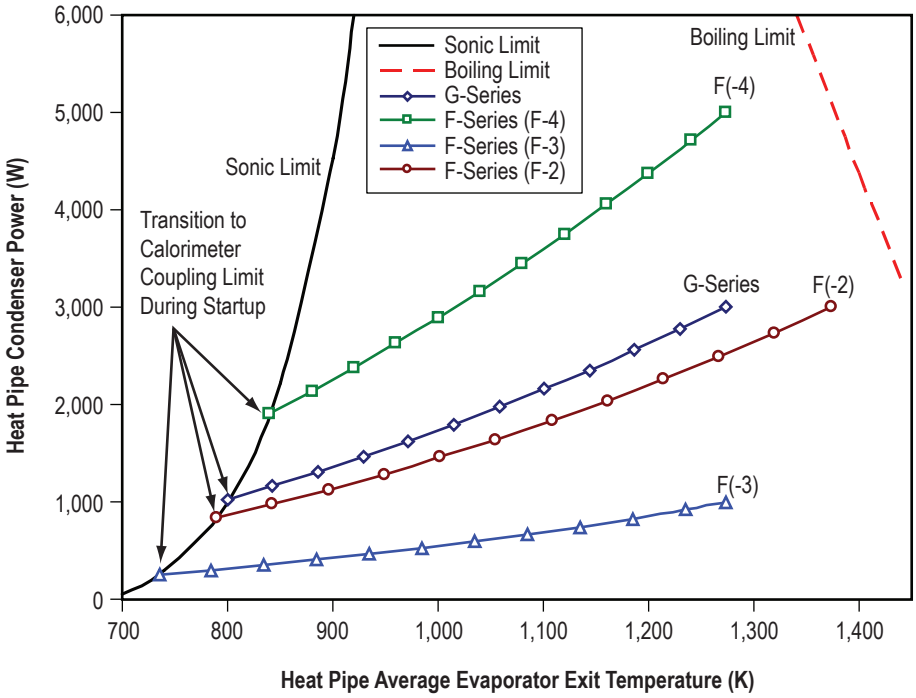

Figure 10. Typical heat pipe startup transient for accelerated life test operations.

As with the performance test, the heat pipe units are expected to follow the sonic limit curve during the initial portion of the startup transient, until thermal coupling between the calorimeter and the heat pipe condenser becomes the controlling heat transfer mechanism. Once this coupling limit is reached, performance will move off the sonic limit, transitioning across the operating envelope as power and temperature increase to the final steady state power and temperature conditions. A typical startup transient should be approximately 1 to 2 hr, with the steady state condition then maintained for periods up to 6 mo—the estimated inspection interval. The temperature and power balance will be assessed in the manner described in section 2.3.1, using equations (1) and (2) to evaluate axial power throughput with the same thermal modeling approach to account for losses. Should power outages or other facility interruptions result in an unexpected shutdown of the accelerated life test system, the cause of the shutdown shall be determined. The entire setup will be restarted if no damage has occurred to the heat pipe units and if all operational parameters, such as coolant flow and environmental conditions, are within required ranges.

2.4.2 General Accelerated Life Test Procedure

The following procedure identifies a series of steps required to set up and operate test hardware for accelerated life testing. The procedures assume a baseline setup in which test chamber segments 1–3 are coupled to form a single environmental test chamber. Chamber segment 4 will be operated independently because of its different gas composition requirement; however, the test procedure would be nearly identical. The general outlined approach captures the following basic sequences:

- Heat pipe and test chamber assembly preparation:
  – Basic cleaning and loading of the hardware.
  – Evacuation and leak checking of all components to verify seals.
  – Thermal bake-out of test chamber, gas system, and heat pipe units.
  – Charging of inert gas system and purging of the test chamber.

- Heat pipe operation:
  – Setup of cooling systems, RF power systems, and data/control systems.
  – Ramping sequence to achieve desired condition.
  – Monitoring to provide for 24-hr/day operation (redline cuts in place).

- System shutdown:
  – Sequence of cooling, power, and control system shutdown.
  – Preparing chamber environment for opening.

The following operations outline serves as a baseline approach and shall be expanded to include specifics as hardware and test system details are identified during the buildup and checkout phase of this program. In addition, a documentation file for each heat pipe unit, referred to as a 'traveler,' shall be carried throughout the process. Each traveler shall contain pertinent information regarding operations, test conditions, observations, and experimental findings for each unit.

2.4.2.1 Heat Pipe and Test Chamber Preparation. The following steps are used in preparation of the heat pipe and test chamber:

(1) All test chamber segments should be separated to allow access to both ends for connection of ancillary systems and loading of heat pipe units.

(2) Clean all heat pipe units with approved procedure and handle only with powder-free, alcohol-cleaned, surgical gloves.

(3) Wipe down all test chamber segments with freon and alcohol to remove potential grease, solvents, fingerprints, etc. Also perform complete visual inspection to verify no foreign materials are present. Let each chamber system dry for a minimum of 1 hr.

(4) Install five cleaned heat pipe units into each test chamber segment and secure in the heat pipe/calorimeter support bracket. Record the location of each heat pipe unit and its clocking within the support bracket.

(5) Move test chamber segments together, attach end plates, and secure all flanges, using the appropriate tightening sequence.

(6) Evacuate test chamber assembly and inert gas feed system to 10^{-6} torr or lower, and perform a helium leak check to a sensitivity of 10^{-10} std-cc/s He on all flanges, feed-throughs, and view ports. Tighten any leaky fittings using the appropriate tightening sequence; leaky view ports must be exchanged.

(7) Turn on the test chamber assembly wall and inert gas feed line heater tapes, and bring the entire system to a temperature of ≈200 °C to outgas all trapped water vapor and other volatiles. Hold at this heated condition for ≈24 hr or until pressure reaches the mid 10^{-8}-torr range; periodically monitor test chamber vacuum pressure. Throughout this and subsequent vacuum cycles, monitor chamber environment, using the RGA. Note: The test chamber pressure should increase during heating and then begin to fall after a steady temperature has been maintained for 4 to 6 hr. If pressure continues to rise or does not drop, there may be a leak caused by heating of the flanges. Perform a leak test while at temperature to locate the leak and tighten the offending flange.

(8) Prepare to outgas heat pipe units. Initiate water coolant flow in all heat pipe calorimeter loops. Set flow to ≈0.5 gal/min in each calorimeter loop.

(9) Verify that all data and control systems are operational. Verify that all heat pipe evaporator exit temperature measurement devices—two-band optical pyrometers—are focused on the correct heat pipe location.

(10) Turn on RF power systems and apply sufficient power to bring all heat pipes to a uniform temperature of ≈1,000 K over a period of 1 to 2 hr, as measured on the heat pipe evaporator exit. Note: The test chamber pressure will rise due to increased outgassing.

(11) Maintain all heat pipes at a constant temperature of ≈1,000 K for roughly 2 hr and monitor test chamber pressure. The pressure should begin to fall off shortly after the heat pipe temperatures stabilize.

(12) Turn off RF power system and all test chamber and inert gas system tape heaters, and monitor test chamber vacuum level as the system cools. It should drop approximately one decade once the entire system is cooled to room temperature.

(13) Isolate the inert gas system from the vacuum chamber and charge the system with the required gas mixture ratio. Typical gasses will be He, with the desired percent Ar specified by weight. This is in preparation for multiple dilution cycles of the test chamber to sweep the environment.

(14) Isolate the test chamber assembly from the vacuum system and then introduce the inert gas mixture into the test chamber; bring to a pressure of ≈70 torr. Isolate the test chamber and let stand for 2 min.

(15) Isolate the inert gas system and open the vacuum system to evacuate the inert gas mixture from the test chamber assembly. Allow the vacuum to reach the 10^{-6}-torr range. Use the RGA to monitor the test chamber during the dilution cycles.

(16) Perform two additional inert gas dilution cycles and then charge the test chamber assembly to a final pressure of ≈70 torr.

(17) Engage the inert gas system—the hot getter purifier unit and pump—and allow the system to circulate for 12 hr. Monitor the RGA during this process. The oxygen concentration should reach a level on the order of 0.3 ppb.

2.4.2.2 Heat Pipe Accelerated Life Testing. The following steps are used to conduct accelerated life testing:

(1) Verify that the purified test chamber assembly environment has been established at or below 0.3 ppb, using the hot getter purifier and recirculation system. Use the RGA unit to verify conditions of the test environment.

(2) Initiate coolant flow to each heat pipe unit calorimeter and set to a flow value consistent with that required to satisfy the calculated final steady state power condition.

(3) Check that all data and control systems are connected to uninterruptible backup power capable of providing at least 10 min of operation in the event of a facility power loss—sufficient time to transfer the system to a safe state. Verify that all data and control systems are operational and all heat pipe evaporator temperature measurement systems (two-band optical pyrometers) are focused on the correct heat pipe location. Set the data rate to one sample per 10 s.

(4) Engage the RF power system and increase power slowly over a period of 2 hr to the planned operating conditions. Note: As power is applied, the heat pipe temperature will follow the sonic limit during startup until the heat transfer is controlled by the limiting thermal resistance between the heat pipe condenser and calorimeter.

(5) Engage the test chamber wall cooling if required; do not allow the chamber surface temperature to exceed 150 °C.

(6) Once heat pipe temperatures have stabilized, minor adjustments to both the inert gas (He/Ar) mixture ratio and calorimeter cooling flow will likely be required to achieve test matrix conditions—specific power and temperature on each heat pipe unit. Note: Adjustments to the calorimeter cooling flow will provide only a very minor adjustment to the overall heat transfer; i.e., at the few percent level, since it regulates the calorimeter wall temperature. If larger adjustments are required, increasing the He concentration will improve thermal conduction, providing more power transfer at a given heat pipe temperature. In contrast, adding Ar will reduce thermal conduction, decreasing the extracted power capability at a given heat pipe temperature. A baseline condition of 32%Ar (design point) provides an effective thermal conductivity that is approximately half that of pure He, providing significant margin for adjustment if required.

(7) During normal operation, the data system shall monitor a number of test article and system parameters and compare these to redline cut values in order to 'safe' the system should it be required. Parameters that specifically address the heat pipe include the following:

- Heat pipe evaporator exit temperature will be monitored using the two-band optical pyrometers. An increase or decrease of more than 20 K about the nominal value indicates a potential problem and provides a warning. An increase or decrease of >40 K about the nominal value will result in immediate shutdown of RF power without operator intervention.

- Calorimeter power is monitored to provide an indication of the total power transferred to the calorimeter at a given evaporator exit temperature. A drop in calorimeter power by 5% to 10% from

the nominal value indicates a potential problem and provides a warning. An increase or decrease by >10% about the nominal value will result in immediate shutdown of RF power without operator intervention.

- Environmental test chamber pressure and gas concentrations are monitored to verify leak-tightness of the setup. A pressure change of 10% from the nominal operating value causes the system to alert the operator; shutdown will result if no intervention is made and if the pressure deviates by more than 20%. The RGA shall also be used to monitor gas composition within the environmental test chamber.

Facility parameters that affect the test operations include the following:

- The coolant system shall be monitored (flowmeter and pumps); a loss of flow requires the immediate shutdown of all RF power units to prevent damage to the heat pipes and calorimeters. Sufficient backup power is required for ≈5 min of operation to maintain a reduced circulation capacity to remove stored heat.

- The RF power system shall be monitored to track output conditions, reporting any power surges that could indicate that a voltage breakdown may be occurring. If a power spike is observed, the system will alert the operator; system shutdown will result if the deviation continues and there is no operator intervention.

- The gas recirculation system is monitored to verify that flow is maintained. If gas circulation halts, a warning will be provided so that the source of the interruption can be identified. Flow loss for short periods of time is not expected to cause problems; the RGA sampling will be used to determine appropriate cuts.

(8) Once every 48 hr, data files should be backed up to minimize the potential loss of data in the event of a computer crash.

(9) Expected test operation is 6 mo (4,380 hr) between inspection intervals. Once this test interval has been reached, the accelerated life testing will be stopped to remove the heat pipes specified for destructive or nondestructive evaluation.

2.4.2.3 Heat Pipe Accelerated Life Test Shutdown. The following steps are used to shut down heat pipe accelerated life testing:

(1) Isolate the test chamber from the inert gas system.

(2) Once the test interval is completed, the RF power is turned off and the system is allowed to cool to room temperature. If a slower cooling trend is required, the test chamber can be evacuated immediately after RF power is turned off, to reduce the thermal conductivity between the heat pipe and the calorimeter.

(3) Once components are near room temperature, the calorimeter and test chamber cooling systems can be shut down.

(4) Bring the test chamber to 1 atm pressure, which is 760 torr, using air to prevent asphyxiation issues, so that the chamber can be opened and the specified heat pipes removed. Test chamber segments will have to be separated to allow sufficient access. Position and clocking should be checked in the traveler log.

(5) Temporarily reconnect the environmental test chamber segments as soon as the heat pipe removal operation is completed, and establish a vacuum to preserve a clean condition. This will simplify the preparations for the next cycle.

2.5 Inspection Schedule

Planned heat pipe accelerated life testing shall be broken into intervals of ≈6 mo, as described in table 2. This will allow for a combination of nondestructive and destructive evaluations (NDE/DE) to be performed. The first planned inspection will be of the as-fabricated heat pipes prior to test, to provide a baseline. The current planned method for NDE will make use of a three-dimensional x-ray tomography method (fig. 11), using Hytec hardware. To accurately compare data collected from the heat pipe units throughout the 3 yr of expected testing, two or more fiducials shall be placed on each unit. This will allow for accurate alignment of x-ray images taken at each inspection interval, so that changes can be readily interpreted. Possible locations for these fiducials are the evaporator and fill stem plugs. Los Alamos National Laboratory (LANL) shall be responsible for all aspects of these inspections (types, processes, location, etc.) and the Marshall Space Flight Center (MSFC) shall coordinate fabrication and test operations to comply with the LANL requirements. Hytec inspection (fig. 12) has a native resolution of 0.004 in (0.01 cm). To achieve the maximum possible resolution with Mo-Re alloys, the voltage will be increased and exposure time extended to enable measurement of working fluid distributions and distortions on the order of <0.002 in (0.005 cm).

Table 2. Proposed heat pipe inspection intervals.

Interval	Type	Comments
Initial Condition	NDE: 3D X-Ray Tomography (Hytec)	As-built and filled condition
6 mo	NDE: 3D X-Ray Tomography (Hytec) DE: one G-series unit	Interim inspection
12 mo	NDE: 3D X-Ray Tomography (Hytec) DE: one G-series unit	Interim inspection
18 mo	NDE: 3D X-Ray Tomography (Hytec) DE: one G-series unit	Interim inspection
24 mo	NDE: 3D X-Ray Tomography (Hytec) DE: one G-series unit	Interim inspection
30 mo	NDE: 3D X-Ray Tomography (Hytec) DE: one G-series unit	Interim inspection
36 mo	NDE: 3D X-Ray Tomography (Hytec) DE: one G-series unit	Interim inspection and determination whether to continue or conduct DE on remaining heat pipes

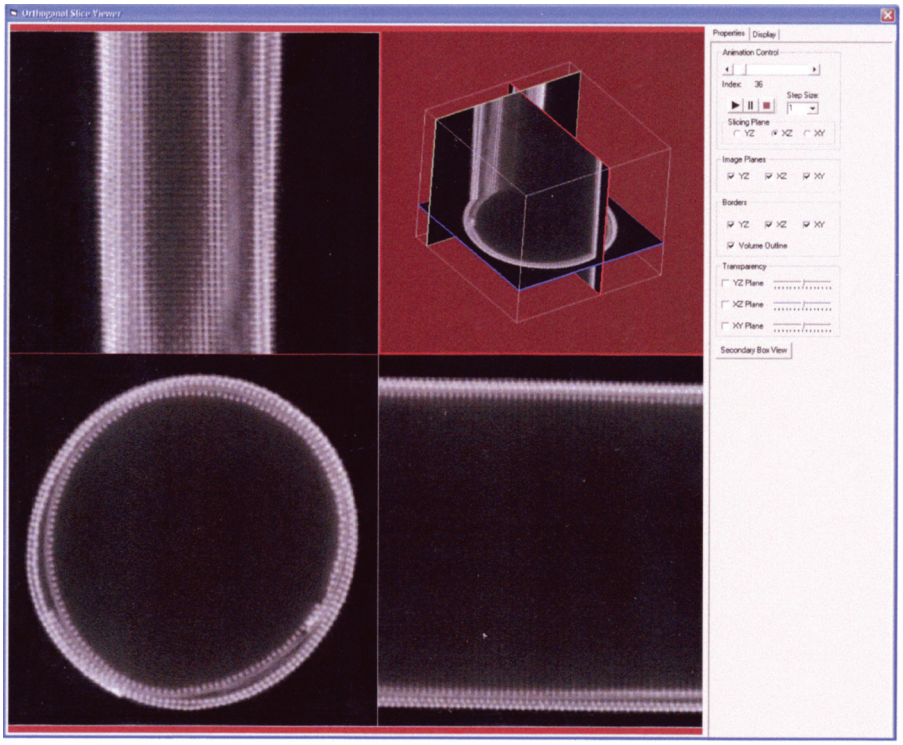

Figure 11. Hytec tomographic examination of SAFE-30 composite stainless steel heat pipe wick, orthogonal views of cross section. Diameter of object is ≈0.875 in.

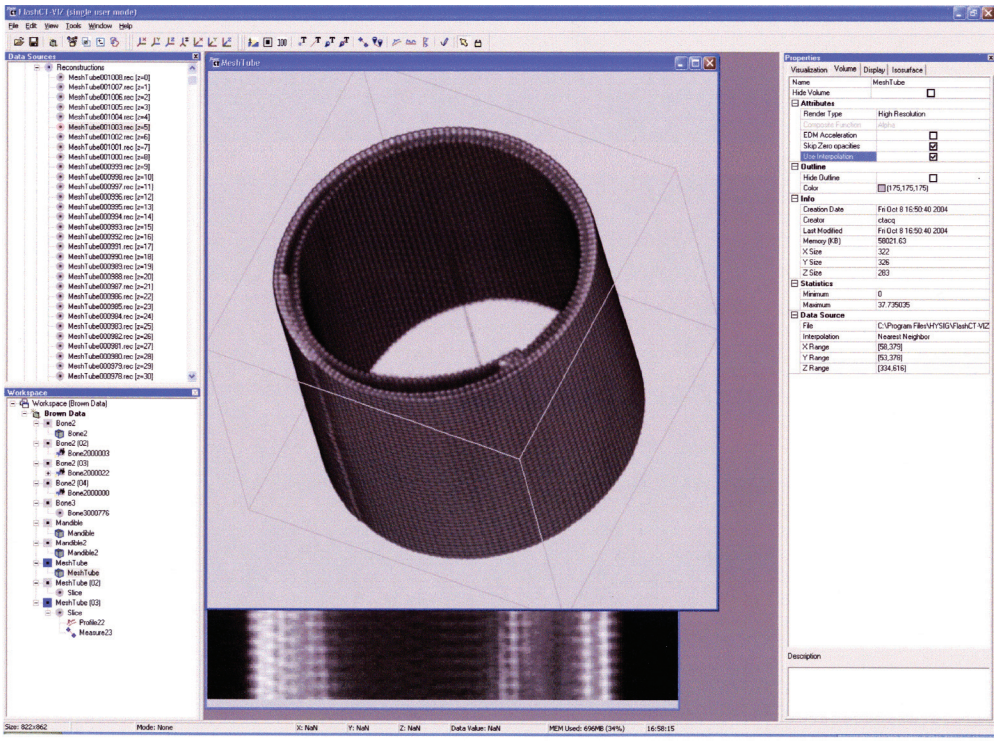

Figure 12. Hytec tomographic examination of SAFE-30 composite stainless steel heat pipe wick, perspective view of cross section. Resolution in this case approaches ≈0.004 in.

3. HEAT PIPE PROCESSING—STANDARD OPERATING PROCEDURES

To govern the tasks of preparing the heat pipe units for testing, a general set of operating procedures has been developed to establish and document the process. These procedures shall be updated and modified as required during the actual hardware operations, to incorporate changes, improvements, and lessons learned. Each heat pipe 'traveler' shall contain information regarding the process configuration and operational steps used to prepare the unit. Before the heat pipe units are received from the vendor(s), the fabrication, assembly, and documentation process will be governed by the specifications contained in the statement of work issued with the procurement. The hardware systems and processes described in this section are based on work supporting the buildup of the heat pipe modules integrated into the SAFE-100a system.

3.1 Alkali Metal Handling—Glove Box System

To perform the liquid metal fill and preparation of the heat pipe units, the existing MSFC fill machine shall be used. The fill machine consists of an inert gas glove box system equipped with a sodium supply. This equipment was used previously to fill a series of stainless steel/sodium heat pipe modules for the SAFE-100a program. The glove box, a Nexus system manufactured by Vacuum Atmosphere Corporation, Hawthorne, CA, is shown in figure 13. The glove box provides the inert Ar environment with gas purifiers to scrub oxygen and water vapor (Dri-Train), and hydrogen and nitrogen (Ni-Train). A source of high-purity Ar gas is maintained to replenish the glove box system; this Ar is supplied as boil-off from a liquid Ar dewar. The system is equipped with a number of antechambers that provide controlled access to the glove box workspace. It is also equipped with power and instrumentation feed-throughs, inert pressurant gas and vacuum lines, and allocations for a tungsten inert gas (TIG) welder. The glove box environment is continually controlled and monitored by an oxygen meter, a dewpoint meter, and a nitrogen/hydrogen analyzer to provide environmental data that include impurity concentration, in parts per million (ppm); typical impurity levels are <1 ppm.

Control and data acquisition related to the alkali metal distribution plumbing—power, temperature, and pressure—are provided by a National Instruments LabVIEW system. This configuration provides a method of tracking operations through real-time computer screen displays, which can be recorded by screen captures, and recorded data files. The general LabVIEW interface window is shown in figure 14. This software shall be modified as required during checkout of the heat pipe processing operations. Internal documentation outlining the fill machine plumbing/component setup and basic operation is in place. Before any alkali metal operations are performed, the glove box environment must be verified to be <5 ppm oxygen and 10 ppm water vapor. If these conditions are not present, sufficient time must be allowed for the Dri-Train to scrub the inert environment.

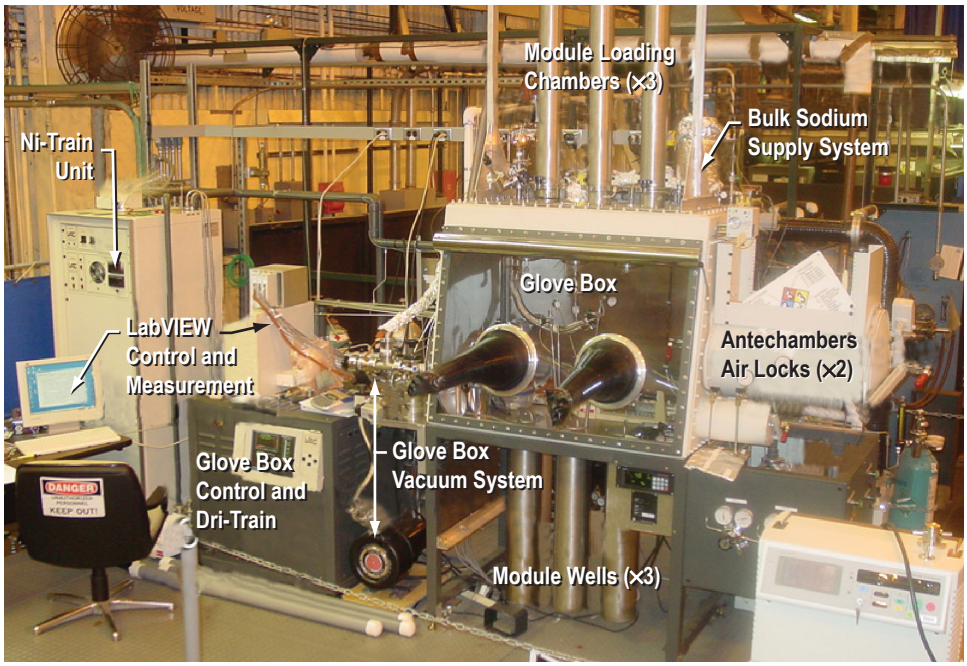

Figure 13. Alkali metal fill machine—glove box.

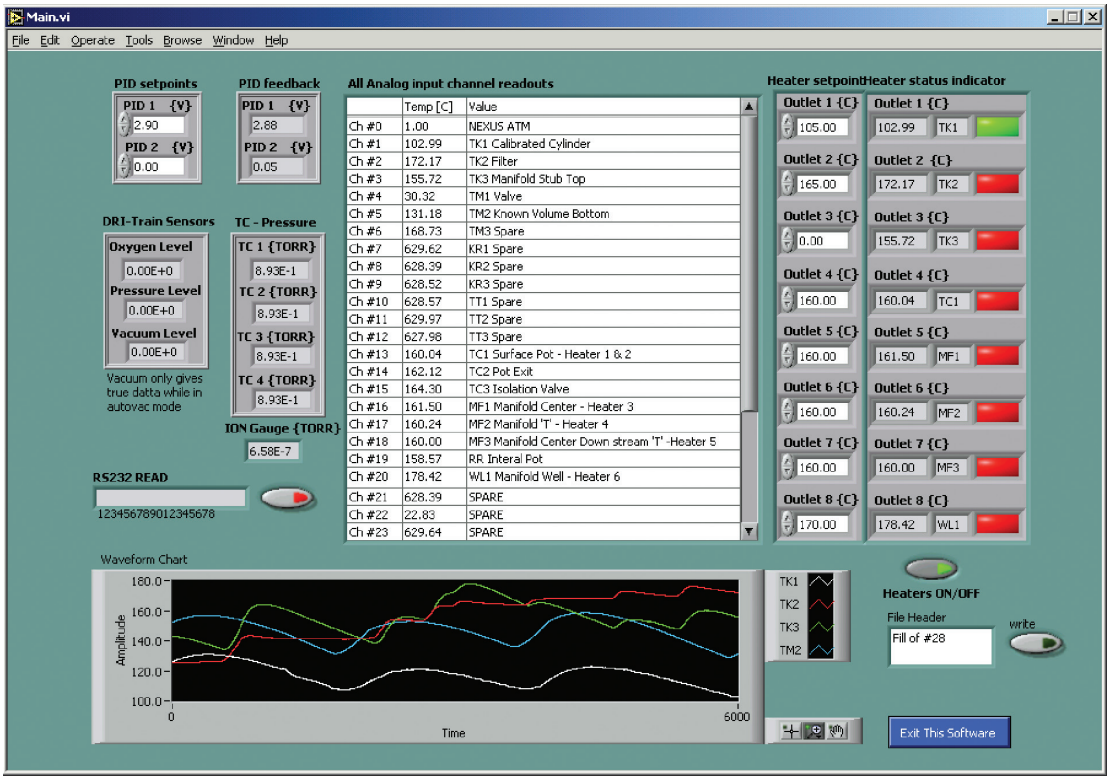

Figure 14. Example LabVIEW interface used for general data acquisition and control.

The sodium supply currently attached to the glove box alkali metal distribution system has been baselined for use in this project to fill the Mo-Re heat pipe units. The sodium stock, a 3-kg canister, was distillation purified at LANL. Initial stock was DuPont reactor grade sodium, trademarked as NiPure, having an initial oxygen concentration of ≈105 ppm as measured by neutron activation analysis (NAA) at the Neutron Activation Analysis Laboratory, Center for Chemical Characterization and Analysis, located at Texas A&M University. Distillation is expected to reduce the oxygen content to less than 10 ppm. After integration of the sodium canister with the fill machine at MSFC, a sodium sample was taken and submitted for NAA at the NAA Laboratory at Texas A&M University. Results from these tests indicate an average of 18.7 ppm oxygen in the dispensed sample; however, these results are below the advertised minimum determination limit of 50 ppm for the NAA method.

3.2 Heat Pipe Module Fill—Known Volume Method

The baseline method selected for filling heat pipes with a predetermined quantity of sodium (≈10 g) uses a metering technique commonly referred to as the 'known volume method.' This approach requires that a sample or 'charge' cylinder be fabricated with specific geometric dimensions to contain the required sodium mass. Implementation of this technique requires a two-step process: (1) Initial fill of the volume, followed by (2) transfer into the heat pipe. At each step of the process, cylinder weights are recorded to verify the actual quantity of sodium transferred. The current baseline charge cylinder has a diameter of 1 in and a length of 1.25 in; it shall be fabricated from stainless steel, with a material wall thickness of 0.035 in. Each end of the cylinder shall be equipped with 0.25-in-diameter tube stubs serving as connection points for transfer and vacuum operations. The canister welds shall be either TIG welds made in an inert atmosphere glove box or vacuum electron beam welds. Figure 15 shows the general layout of the proposed sample cylinder (fig. 15(a)) and a photo of a cylinder used for previous heat pipe work (fig. 15(b)). The stainless steel shall be cleaned per the procedure listed in appendix B. Connection to the heat pipe fill stem shall be made using compression type fittings (Swagelok) and stainless steel welded metal bellows valves, such as the NUPRO SS-4H. Dissimilar material contamination should not be a problem because the transfer temperatures are low (<200 °C). Each of the fill steps is described in section 3.2.1.

3.2.1 Known Volume Cylinder Fill

The initial step to filling a heat pipe unit is to meter out the specific quantity of sodium. During fill it is typically best to err on the high side, because additional sodium will merely increase the size of the heat pipe condenser liquid pool (located at the end of the heat pipe), which is not detrimental to operation. Undershooting the required sodium quantity may result in an insufficient liquid sodium pool at the end of the condenser and a loss of capillary continuity. This condition prevents the heat pipe from operating normally, because the pool is required to seal the end of the internal wick structure, providing a closed capillary flow path to the evaporator. The hardware setup used in past operations is shown schematically in figure 16(a) and photographically in figure 16(b); note that the heaters are not in place. The setup includes a feed system isolation valve, a filter to trap precipitates, the known volume, and a vacuum isolation valve.

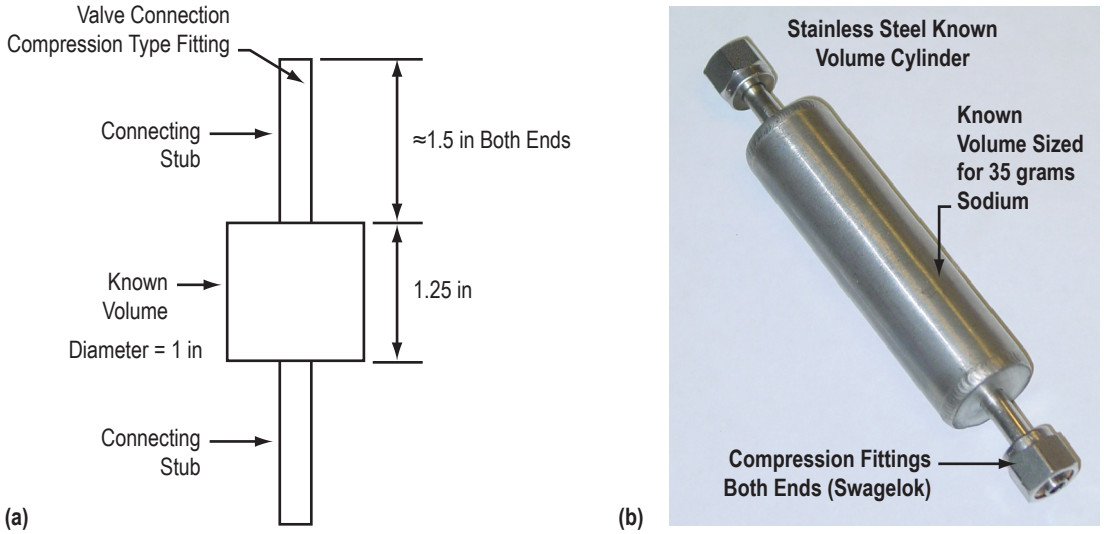

Figure 15. Proposed sample cylinder: (a) General known volume layout and (b) picture of a previously used known volume.

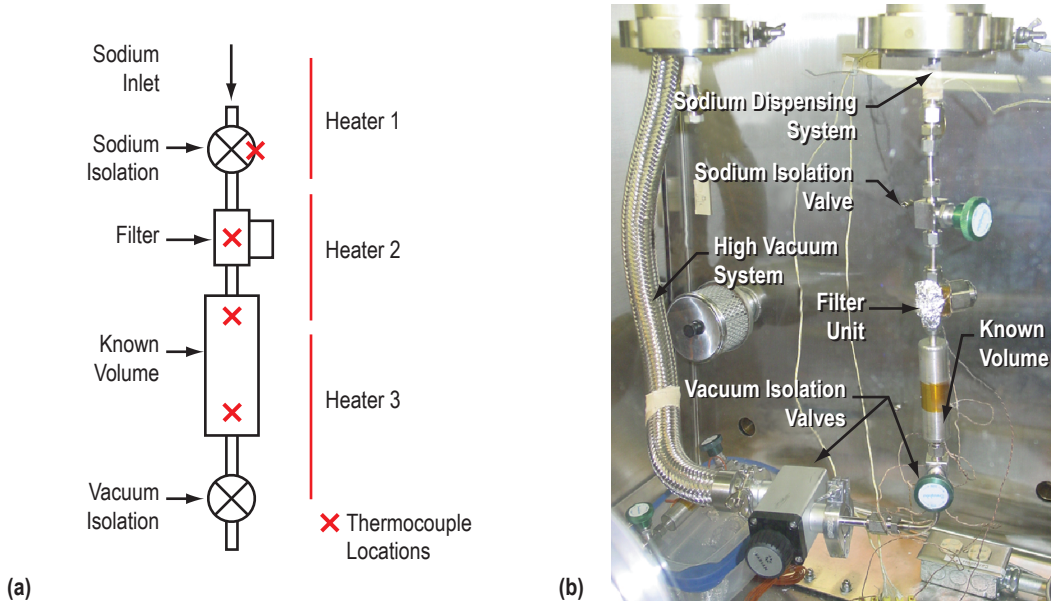

Figure 16. Hardware setup: (a) Known volume cylinder fill and (b) photographically.

The typical fill operation begins by heating all components of the sodium distribution system external to the glove box to a temperature in the range of 150 to 160 °C; these components include lines, valves, and bulk storage canister. The emphasis is to maintain a sodium temperature sufficient for transfer yet low enough to minimize oxygen solubility. Figure 17 graphs the solubility of oxygen as a function of temperature.[7] The empty known volume cylinder is weighed and connected to the sodium dispensing station (within the glove box). Before the internal components are heated, the glove box high-vacuum system is connected to the lower port of the known volume cylinder and is evacuated to a pressure in the low 10^{-5}-torr range. This is necessary to provide the differential driving pressure required for the pressurized bulk sodium to completely fill the known volume. Once a vacuum is established, the vacuum isolation valve is closed and heaters are placed over the sodium fill tubing, isolation valve, filter, and known volume cylinder. These individual heaters are adjusted to provide a temperature gradient with the sodium inlet at the highest temperature, ≈160 °C, and the known volume canister at the lowest temperature, ≈120 °C. The 14-μ filter used to trap particulates and precipitate is located between these two extremes; the filter temperature is monitored to determine approximate oxygen solubility.

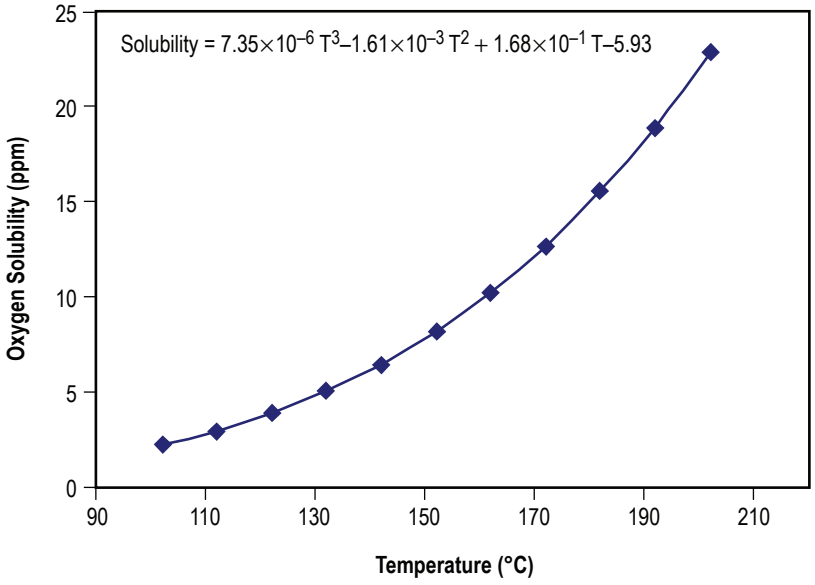

Figure 17. Oxygen solubility in Na as a function of temperature.

Establishing a temperature gradient across these components provides a straightforward mechanism for verifying the success of the fill operation by observation of each component's temperature response. All heater control and thermocouple measurements are processed and displayed using a customized LabVIEW interface. A graphical display is used to monitor the temperature trends for the isolation valve, filter, and known volume, providing the operator with an immediate visual indicator as to the success of the operation.

Once all temperatures have reached required levels, the bulk sodium reservoir is pressurized to 760 torr (with high purity Ar) and the sodium isolation valve inside the glove box is opened. The sodium immediately flows into the known volume cylinder, resulting in a rapid rise in the temperature of all wetted components. Another key indicator of a successful transfer is that both top and

bottom cylinder temperature measurements track together. Figure 18 illustrates these trends with sample data taken during fill of a known volume cylinder. To eliminate the potential for voids, the known volume and other components are tapped periodically while heat is maintained for at least one min before closing the sodium isolation valve. The heaters are then shut off and removed from the known volume, allowing it to cool rapidly. Another characteristic of a successful transfer is a sodium liquid-to-solid phase change plateau as it cools through the region at ≈100 °C; this is very evident in figure 18. Once the volume has cooled, it is removed, weighed, and then compared to its prefill weight, to verify that the required sodium mass has been transferred. The known volume cylinder is now ready for transfer to a heat pipe unit. Appendix C provides an initial standard operating procedure outlining the process steps to fill a known volume. During the actual filling procedures, these process steps would be completed and operation specifics would be recorded (temperatures, pressure, etc.) and placed in the heat pipe traveler. Screen captures are typically recorded at key intervals: Just prior to opening the sodium isolation valve, after transfer but just prior to turning off the heaters, and at the end of cooldown.

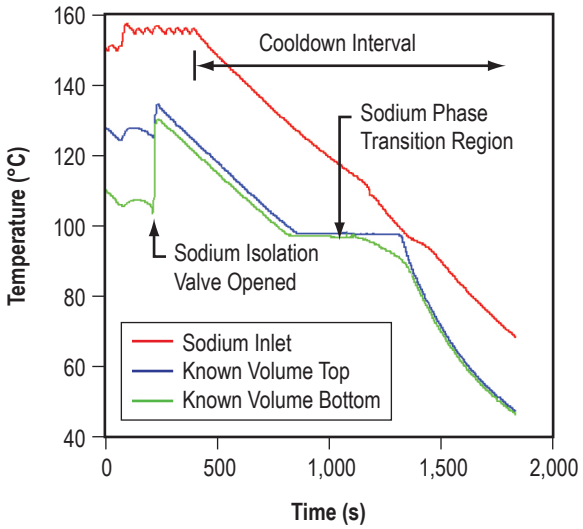

Figure 18. General temperature trends during known volume cylinder fill.

3.2.2 Known Volume Cylinder Transfer to Heat Pipe

The next step in the fill sequence is to transfer the sodium from the known volume cylinder into the heat pipe unit. To prepare the heat pipe to receive the sodium, its fill stem must be fitted with an isolation valve and then connected to the glove box high vacuum system. The unit should evacuate into the low 10^{-5}-torr range, as read on the vacuum system pressure gauge. Compression fittings should be tightened to eliminate any leaks. The vacuum condition is required to allow the sodium to flow freely into the heat pipe unit, using the glove box pressure as the driving force. With the heat pipe unit evacuated, the known volume cylinder is attached to the unit's isolation valve, as shown schematically in figure 19(a) and photographically in figure 19(b). A thermocouple is attached to the base of the heat pipe fill stem to monitor temperature variation during the transfer procedure, indicating that sodium has flowed into the unit. Heaters are attached to the known volume and heat pipe stem. Heaters are turned on with a set temperature of 160 to 180 °C for the known volume and 130 °C for the module stem; setting up this temperature

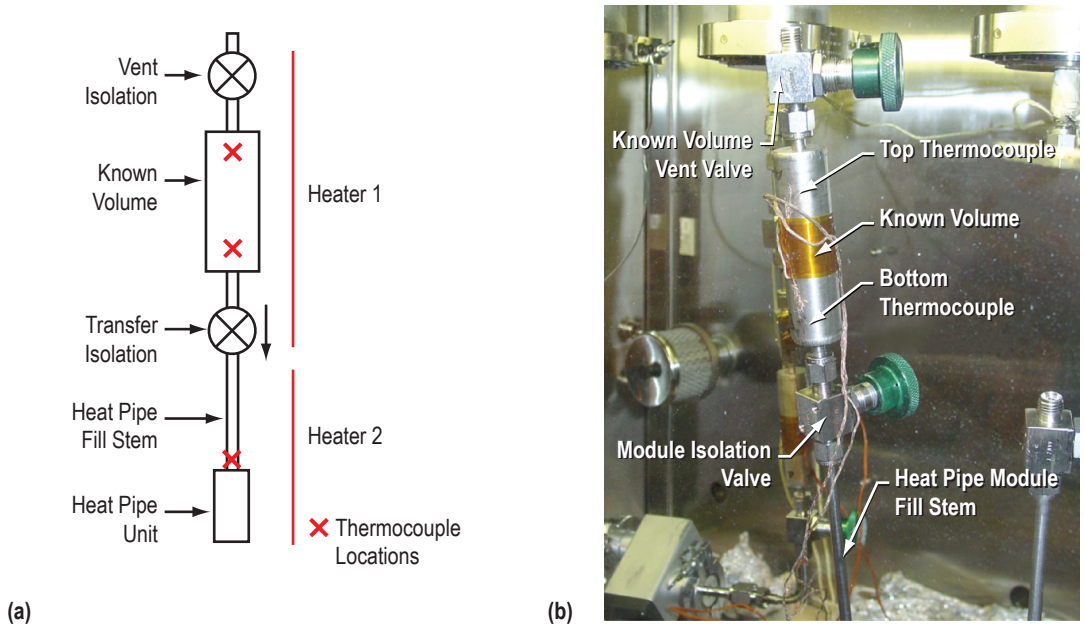

Figure 19. Volume cylinder setup: (a) Known volume cylinder transfer to heat pipe and (b) photographically.

difference provides a visual indicator of a successful transfer. It is important to configure the heaters such that the top heater covers the known volume and the isolation valve, ensuring that the larger components are thoroughly heat soaked. This minimizes the chance of solidification and introduces a hot sodium flow into the heat pipe fill stem.

Once temperatures have reached target values, the heat pipe isolation and vent valves are opened, allowing sodium to flow rapidly into the module. Figure 20 illustrates the rapid increase in temperature along the stem as sodium passes into the module. The module and known volume canister are tapped to ensure transfer, and heating is maintained for several minutes. Typically, the known volume canister temperature will increase (with its top and bottom temperatures drifting apart) after the sodium is transferred; this results from a lower thermal mass available to absorb the heater power, producing a tendency to overshoot. The heaters are then shut off, isolation and vent valves closed, and the heaters and insulation removed, resulting in a rapid temperature drop. A clear indication that all sodium has been transferred and that both the known volume and heat pipe fill stem are clear is the absence of a liquid-to-solid phase transition plateau during the cooldown. The known volume cylinder is removed and weighed to verify the amount of sodium transferred. The heat pipe unit is fitted with a new isolation valve to eliminate the chance of residual sodium in the valve seat, which would interfere with valve sealing.

The heat pipe unit is connected to the glove box high-vacuum system and evacuated into the low 10^{-6}-torr range; fittings are tightened as necessary to eliminate leakage. Appendix D provides a standard operating procedure outlining the process steps to transfer the sodium from a known volume into a heat pipe unit.

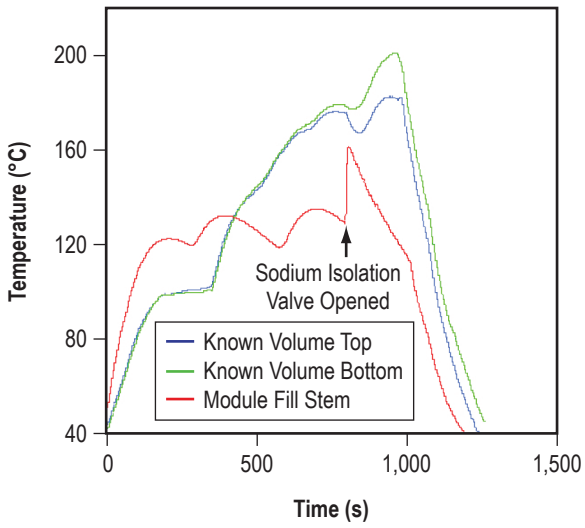

Figure 20. General temperature trends during transfer from known volume to heat pipe.

During actual implementation, this procedure would be completed with operational specific data, such as temperatures and pressure, recorded and placed in the heat pipe traveler. Computer screen captures are typically recorded at key intervals: Just prior to opening the heat pipe isolation valve, after transfer but just prior to turning off the heaters, and at the end of cooldown.

3.2.3 Heat Pipe Unit Vacuum Conditioning

During sodium transfer into the heat pipe unit, purified Ar is introduced into the heat pipe, serving as the driving force to move the sodium. As a result, Ar can potentially be trapped within the sodium as it cools, creating trapped pockets. If this Ar remains, it can interfere with the operation of a heat pipe unit, especially when the unit is operated near boiling limits. The Ar can be transported along the capillary flow channel to the evaporator, activating potential nucleation sites where boiling can be initiated. Removing the residual Ar requires a low-level bake-out to thoroughly outgas the interior volume of the heat pipe. To accomplish this task, a small tube-type heater arrangement is required inside the glove box. The heater shell must be fabricated from a suitable material, such as titanium or stainless steel, for compatibility. The heat pipe is placed within the tube heater, and its isolation valve is connected to the glove box high-vacuum system and evacuated into the low 10^{-6}-torr range. This setup is illustrated in figure 21.

To maintain a vacuum condition within each heat pipe unit during and after vacuum conditioning, each unit's vacuum isolation valve fittings must be leak checked, using a helium leak detector (such as a Varian model 979 leak detector) that is connected to the glove box high-vacuum roughing system. The leak detector is allowed to self calibrate and is then brought online. The baseline leak rate for the detector is typically 0.2×10^{-11} std cc/s He when isolated from the system. Depending on the amount of He already saturating the glove box system from previous operations, the leak detector baseline will typically increase into the 10^{-9} or low 10^{-8} std cc/s range when its sense port is opened to the glove box vacuum system. Because the glove box is a closed

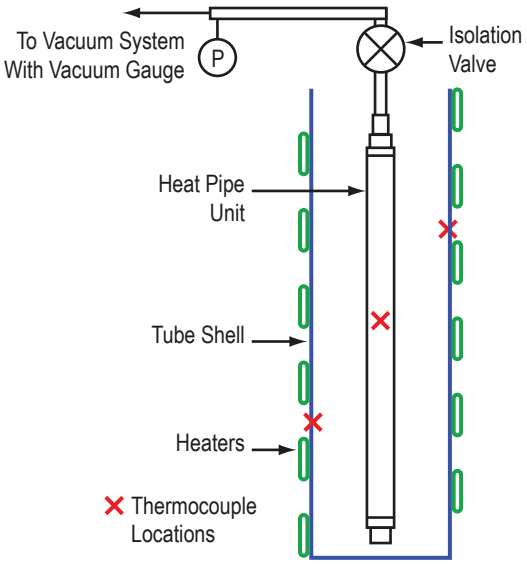

Figure 21. Setup for heat pipe unit vacuum conditioning.

environment and traps the helium that easily diffuses through the vacuum system O-ring seals, it is very difficult to maintain a leak rate baseline $<10^{-9}$ std cc/s range. Additionally, the changing out of heat pipe units (connecting/disconnecting) introduces significant helium into the vacuum system, which can quickly saturate the leak detector, requiring considerable time to clear because of the length of the lines (low vacuum conductance). Typically, the first leak check of the day will have the lowest baseline; this baseline climbs with each subsequent unit that is evaluated. However, the leak detector has a built-in zeroing function, which resets the sampling range to its lowest level (0.2×10^{-11} std cc/s) by subtracting out the current background baseline. Helium can then be sprayed around the fittings under investigation, and any increase in leak rate produced by locally higher helium concentration introduced by a 'real' leak is readily picked up and displayed. All fittings are systematically tested for integrity and tightened as necessary, maintaining the zeroed helium leak rate at $<10^{-10}$ std cc/s. The system is allowed to stabilize for ≈15 min and a final helium leak check is performed on all heat pipe unit isolation valve fittings. As an additional checkout, the unit's isolation valve is closed for several minutes to lock up the heat pipe volume. Noting the vacuum system pressure, the unit's isolation valve is reopened and any change in the vacuum level is recorded and investigated. If the valve fittings are leak tight, no increase in pressure is expected. Appendix E provides a standard operating procedure outlining the leak testing process; a completed version of this procedure would be placed in the heat pipe traveler.

To initiate the vacuum conditioning process, the heat pipe vacuum isolation valve is opened to the glove box high vacuum system, and the tube heater surrounding the heat pipe is turned on. The heat pipe temperature is increased to 200 to 250 °C over a period of ≈1 hr and held. During this period, the pressure initially rises as the sodium melts, and trapped argon is released. The pressure subsequently begins to fall after all trapped Ar has been released. The heat pipe unit should be periodically tapped gently during the heating period to assist in freeing the trapped Ar. Any sudden Ar releases will be visible as sudden jumps in the vacuum level, as indicated on the glove box display. Figure 22 shows a plot of a typical vacuum conditioning cycle performed on a heat pipe module.

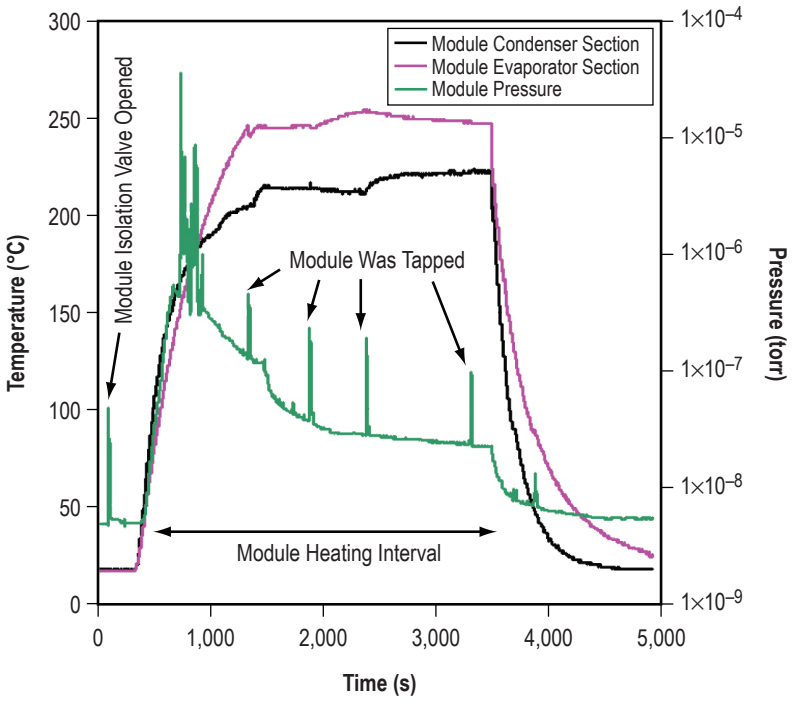

Figure 22. Typical vacuum pressure response during vacuum conditioning.

Once the pressure has stabilized and no further pressure spikes are present, typically on the order of 1 hr, the heaters can be turned off and the system continually evacuated until all hardware has cooled to room temperature. The vacuum isolation valve can now be closed. The heat pipe unit is now ready for sodium purity sampling or final closeout. Appendix F contains a standard operating procedure outlining the vacuum conditioning steps discussed. During actual vacuum conditioning, a completed version of this procedure would be placed in the heat pipe traveler.

3.3 Heat Pipe Module Fill—Distillation With Vanadium Wire Method

An alternate to the fill procedure described in the previous section is to distill sodium into the heat pipes. An advantage of distillation is that the transfer of low-pressure sodium vapor[8] is performed in vacuum, avoiding the introduction of purified glove box Ar into the heat pipe during charging. An additional benefit of distillation is that the sodium can be purified to <1 ppm oxygen. Distillation coupled with hot trapping provided by the vanadium (V) wire technique ensures that closed heat pipes contain known low levels of nonmetallic impurities (to a level potentially «1ppm). Distillation requires that sodium be metered into a known volume cylinder, using hardware and procedures identical to those described in section 3.2. The general approach involves connecting a filled known volume to a distillation pot (located in the glove box) that is hooked to a heat pipe. Figure 23 illustrates a general layout for the distillation hardware. The known volume, along with connective tubing, is covered with heaters so that the vapor pressure of sodium can be increased. Turbo pumps are used to evacuate the heat pipe and transfer tubing to the 10^{-6} torr range. The connective tubing is baked at 400 °C before the transfer is initiated; both the heat pipe and sodium isolation valves should be closed during bake-out. A typical distillation temperature set

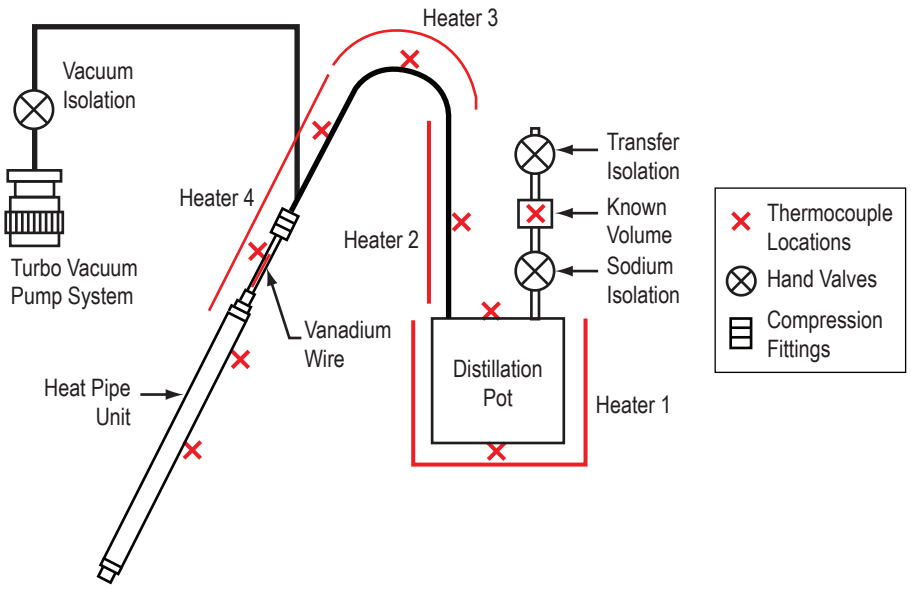

Figure 23. General layout for distillation heat pipe fill system.

point, heater 1, is in the 350 to 450 °C range, adjusted to minimize transfer time and to maximize purity. The vapor pressure of sodium is given by equation (3) and is illustrated in figure 24:

$$\ln P_{sat} = 11.2916 \pm 0.5077 - (12532.694 \pm 87.141)/T_{sat} - (0.3869 \pm 0.0600) \ln T_{sat} \quad , \qquad (3)$$

where P_{sat} is the saturation pressure of sodium (in MPa) at the saturation temperature T_{sat} (in K). Heaters 2 and 3 are set to a lower range of approximately 250 to 300 °C, and heater 4 is set to <150 °C; most of the heat pipe unit itself is unheated.

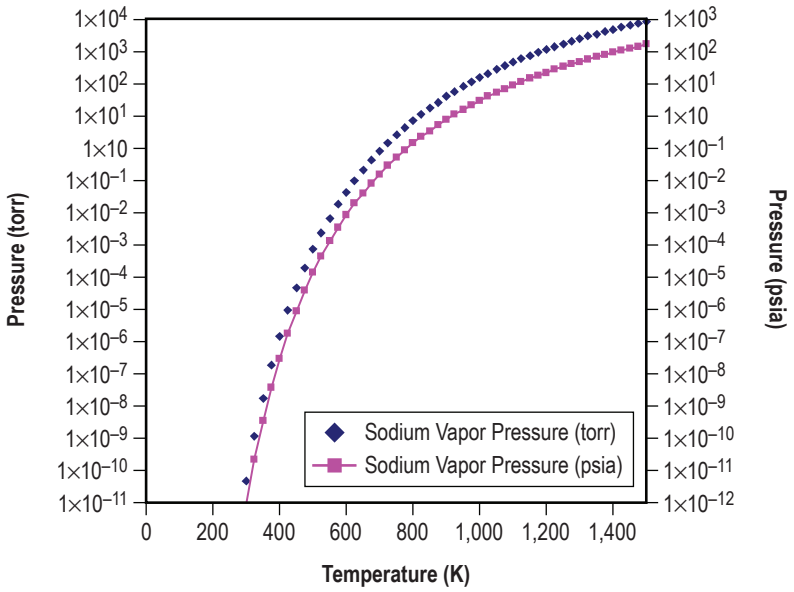

Figure 24. Sodium vapor pressure as a function of temperature.

3.3.1 General Vacuum Distillation/Vanadium Wire Procedure Summary

All parts that are exposed to sodium during charging are degreased and vacuum fired before use. The heat pipe is charged with sodium using the hardware shown schematically in figures 23, 25, and 26. Inside an inert gas glove box, the manufacturer's closure hardware is removed from the heat pipe body. An Mo-Re wire—referred to as a bow—that contains a vanadium wire is inserted into the fill stem bore, and the end of the bow is tack welded or mechanically attached to the fill stem's inside diameter. The heat pipe fill stem is connected to the compact distillation unit either with a compression fitting or with a TIG weld. A known volume is attached with compression fittings to a sodium storage container. The known volume is then evacuated. The general fill of the known volume with sodium from the storage container is depicted in figure 25. The sodium inside the storage container is melted with resistance heaters, and the known volume is heated above the sodium melting point. The Ar above the sodium-free surface in the storage container is pressurized, forcing sodium from the bottom of the storage container into the known volume. This process can be accomplished using the procedure described in section 3.2.1.

Once filled, the known volume and sodium storage container are separated under inert gas and sealed with compression fittings. The known volume is then attached to the distillation pot. This distillation configuration is shown in figure 26. The temperature of the known volume container is then increased to the sodium melting point, and the sodium is pushed into the distillation pot with Ar gas. A positive Ar pressure is maintained inside the distillation pot as the known volume container is removed, and the pot is sealed with compression fittings. The distillation pot is leak tested to verify the integrity of all compression joints. The internal volume of the heat pipe and distillation hardware is attached to a vacuum pumping system as a means of degassing the system before distillation. This same pumping system is used to remove off-gasses from the sodium during the fill process. Distillation of sodium into the heat pipe consists of the following steps:

(1) Movement of sodium from a storage container to a loading pot of a known volume.

(2) Transfer of sodium from the known volume to the distillation pot.

(3) Vaporization of sodium in the distillation pot and condensation of the sodium in a line that allows the distillate to gravity-feed into the heat pipe.

(4) Formation of a sodium freeze plug in the heat pipe fill stem (dependent on closeout technique).

(5) Isolation of the fill stem from the distillation assembly.

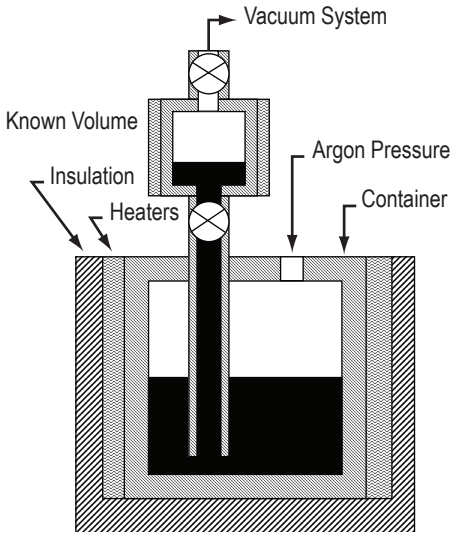

Figure 25. Fill of known volume container with Na from storage container.

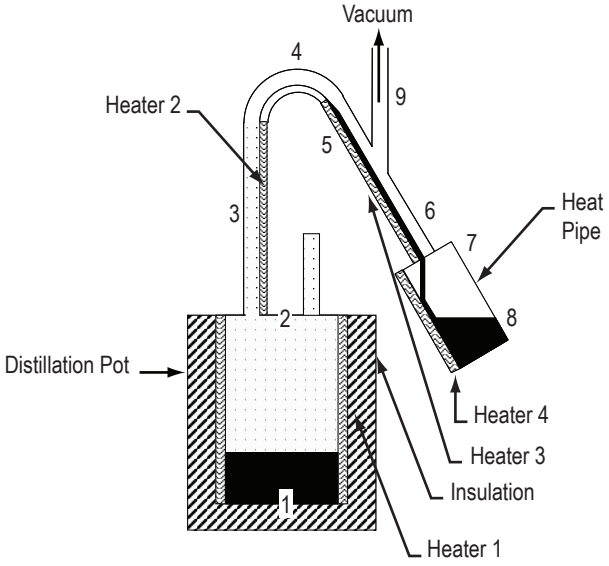

Figure 26. Sodium distillation apparatus used to fill life-test heat pipes. The inside diameter of the transfer line is ≈0.5 cm.

The distillation pot is evacuated to 10^{-6} torr and heated to 400 °C with heater tapes. Sodium vapor moves at sonic velocity up the transfer line and condenses on the far side of the bend. The condensed sodium then flows into the heat pipe by gravity. Figure 27 illustrates the general temperature trends that might be expected during a typical distillation fill. The numbers associated with each temperature curve reference the thermocouple locations shown in figure 26. The heat pipe and compact distillation unit (CDU) assembly are brought to the distillation temperature distribution

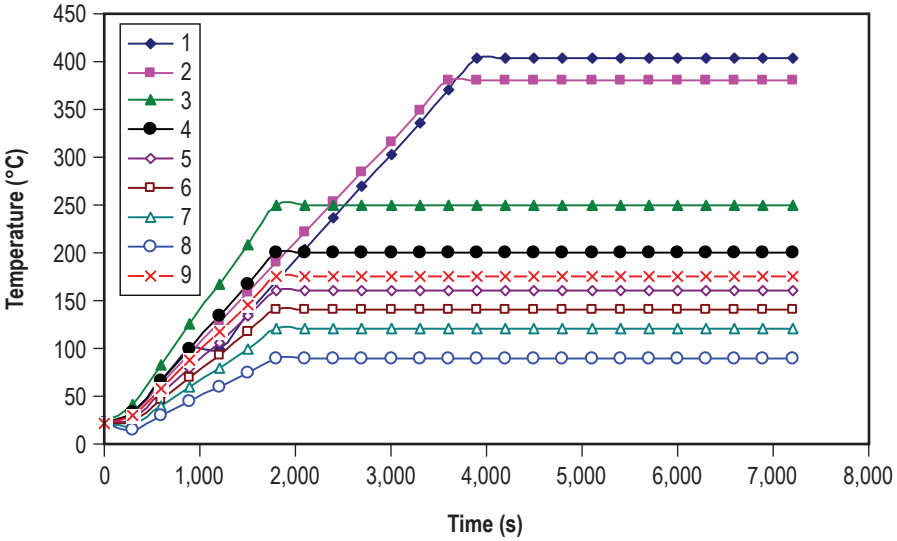

Figure 27. Temperature versus time during the vacuum distillation of Na into life-test heat. The total Na transferred during this fill operation is ≈10 g.

as indicated in figure 27. Sodium distillation into the heat pipe requires ≈1 hr at a distillation pot temperature of ≈400 °C as shown in figure 28. The density of saturated sodium vapor is given by the following equation:

$$\rho_{sat} = \exp\left[-86.671 + 0.30672T - 4.9716\times10^{-4}T^2 + 4.3234\times10^{-7}T^3 - 1.925\times10^{-10}T^4 + 3.4434\times10^{-14}T^5\right] \quad (4)$$

During distillation, the sonic velocity of the vapor flowing through the transfer line limits the sodium mass transfer rate between the distillation pot and the heat pipe. The mass transfer rate of sodium vapor through an orifice with area A_v, at temperature T, is approximately as follows:

$$\delta m \sim \rho_{sat}\left[\frac{\gamma RT}{2(\gamma+1)}\right]^{0.5} A_v \delta t \quad . \quad (5)$$

Filling continues for a precalibrated time interval. At no point should the distillation pot dry out, as this would expose and heat volatile impurities in the pot. Dry out would be indicated by the increase in the temperature versus time slope read at thermocouple location No. 1. The heat pipe fill stem is then pinched below the connecting compression fitting and sealed with a TIG pass, forming a seal that isolates the fill stem. The pinch seals the heat pipe interior until a more permanent cover tube can be welded over the pinch before vanadium wire equilibration. The heat pipe is then inverted, and sodium is moved to the condenser end by warming the heat pipe unit. The heat pipe temperature is then increased to the equilibration temperature of ≈750 °C for ≈4 hr. The heat pipe is allowed to cool, and the fill stem is severed to remove the vanadium wire sample; the heat pipe is then closed out by a final electron beam weld. These processes will be covered in more detail

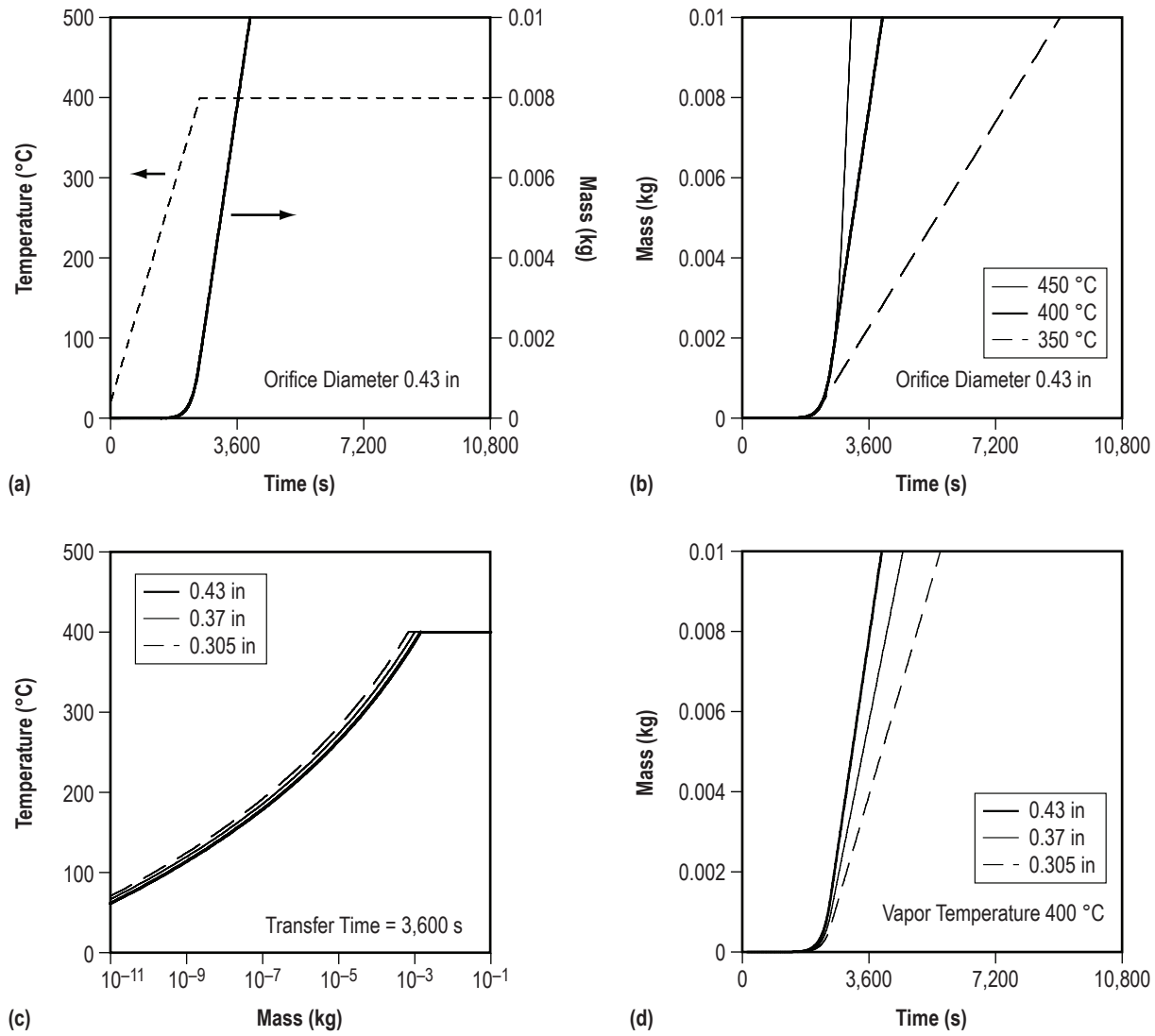

Figure 28. Distillation operations at various vapor temperatures and orifice diameters.

in the following sections. Note: The use of a larger fill tube would reduce sodium hold-up and would potentially accommodate sodium circulation for the vanadium wire during equilibration. However, the current hardware design basis makes use of a 0.25-in-diameter fill line.

Performing the integration in equation (6) with the measured temperature data yields an oxygen concentration in the distillate in parts per million:

$$m_o = A\int_\tau \rho_{\text{sat}}\left(T(t)\right)\left[\gamma RT(t)\right]^{1/2}\left[O\right]\left(T(t)\right)dt \quad . \tag{6}$$

Wall temperature data at the choke point can be used to estimate the purity of the condensate, given a dissociation pressure of compounds such as sodium monoxide that remain in the distillation pan. This estimated concentration value has been shown to be in good agreement with the neutron activation measurements.

3.4 Heat Pipe Module Sodium Purity Testing—Vanadium Wire Equilibration

As part of this program, a technique to sample the purity of sodium within the heat pipe units will be developed. The current technique of choice involves bringing a section of vanadium wire into equilibrium with the oxygen contained within the dispensed sodium. This technique, referred to as the vanadium wire equilibration method, is described in ASTM 997-83[9] and in reference 10. A procedure adapted from the ASTM standard to test for oxygen in vanadium wire samples extracted from a heat pipe has been published, *NASA/TM—2005–213902*.[11] Appendix G provides a segment of this TM detailing the procedure for testing the vanadium wire once it has been extracted from the heat pipe unit. The viability of this approach must be confirmed before use during heat pipe fill. Work is underway to apply this technique using the sodium supply currently attached to the MSFC fill machine. If, after evaluation, the vanadium wire equilibration technique appears viable, it will be incorporated into the heat pipe fill sequence. The most challenging part of this approach is to integrate sampling into the heat pipe fill and closeout procedure so that the following conditions are met:

- The entire procedure must be kept as simple as possible (few steps).
- The procedure should have few (if any) changes to the existing heat pipe design.
- The procedure must leave the heat pipe in a known state after closeout.

A possible sequence of steps that could accomplish this task is outlined in figure 29. The stainless steel prototype heat pipes can be used to practice these procedures.

3.4.1 Vanadium Wire Equilibration Operations Outline

The following operations are used to achieve vanadium wire equilibration:

(1) A vanadium wire segment (on the order of 6 cm long) must first be attached to the internal wall of the heat pipe fill stem. This wire should be positioned ≈2.5 cm above the end of the condenser plug.

(2) Sodium is introduced into the heat pipe using either of the transfer methods described previously; i.e., direct liquid transfer—section 3.2 or distillation transfer—section 3.3.

(3) Once transfer and vacuum processing are completed, in the case of direct liquid transfer, the end of the fill stem is sealed by pinching and TIG or electron beam welding. Note: An additional protective cap may be required to cover the pinch weld, providing a second level of containment.

(4) The heat pipe is brought to a low wet-in temperature (900 to 1,050 K range) for up to 48 hr to thoroughly saturate its interior with sodium. This allows for impurities within the heat pipe interior to be brought into solution and distributed throughout the sodium. This operation must be performed in a vacuum oven to prevent contamination of the heat pipe surface.

(5) The sodium is settled, condenser side down, such that the heat pipe fill stem is filled with sodium.

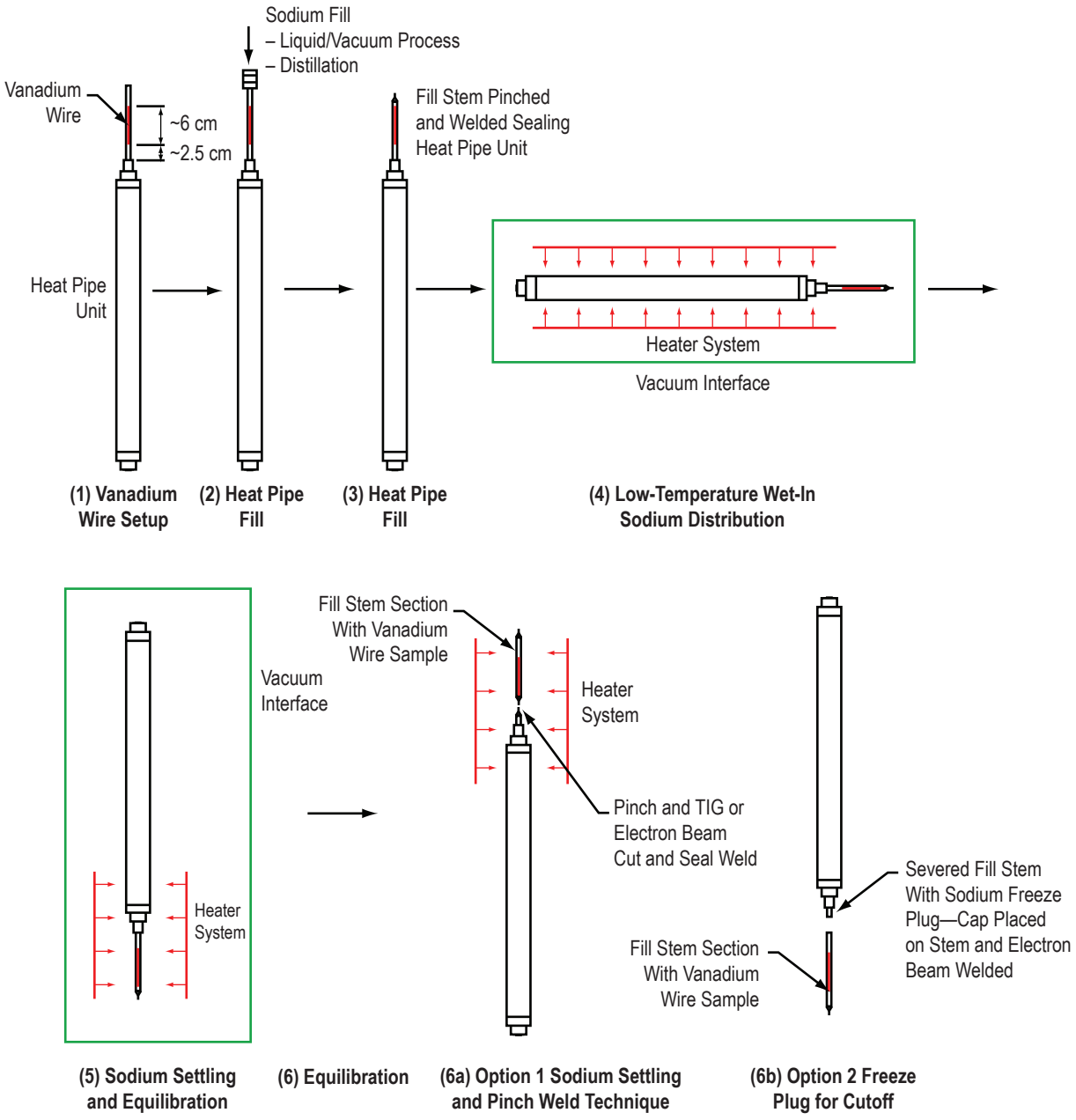

Figure 29. Possible vanadium wire purity sampling heat pipe process sequence.

(6) The heat pipe fill stem region is brought to the equilibration temperature (900 to 1,050 K range) and held for the required time interval (3 to 4 hr). The vanadium wire purifies the sodium locally; however, oxygen will diffuse through the bulk settled sodium into the fill stem volume, where it will be absorbed by the vanadium wire.

(7) To remove the vanadium wire sample from the heat pipe unit, two possible approaches are outlined below:

(a) The first approach is to drain the sodium from the fill stem back into the heat pipe, allowing the fill stem to be pinched and seal welded. The pinch cannot be performed with sodium in the stem. To accomplish this task, rotate the heat pipe with the evaporator side down to allow the sodium to flow out of the condenser and fill stem region. A small amount of sodium will remain due to surface tension forces. This can be evaluated by balancing the pressures generated by the sodium mass and surface tension within the fill stem, as given by:

$$\rho g h = \frac{\sigma(2\pi r)}{\pi r^2} \tag{7}$$

and

$$h = \frac{2\sigma}{\rho g r} \ , \tag{8}$$

where ρ is the density of sodium (kg/m^3), g is the acceleration of gravity (m/s^2), h is the height of the sodium column (m), σ is the sodium surface tension (N/m), and r is the tube internal radius (m). Sodium surface tension (N/m) and density (kg/m^3)[12] are given by the expressions:

$$\sigma = 206.7 \times 10^{-3} - 1 \times 10^{-4} (\text{T-273.15}) \tag{9}$$

and

$$\rho = 1011.8 - 0.22054(T) - 1.9226 \times 10^{-5}(T^2) + 5.637 \times 10^{-9}(T^3) \ , \tag{10}$$

where T is in kelvin (range of 370.98 to 1,300 for σ and 370.98 to 1,644.24 for ρ). Estimates of the sodium column height and mass remaining in the fill stem are shown in figure 30 as a function of settling temperature. As indicated, very little mass remains (≈3% of the total heat pipe inventory).

Once the sodium is settled, the fill stem could be pinched and a fusion weld performed using either a TIG or electron beam welder to seal the fill stem, as illustrated in figure 31. This process will be discussed in more detail in section 3.5.1.

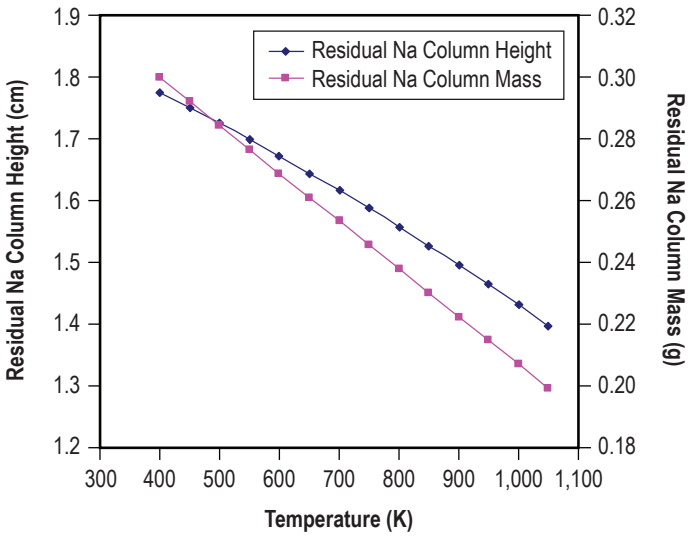

Figure 30. Estimated residual Na column in heat pipe fill stem after settling.

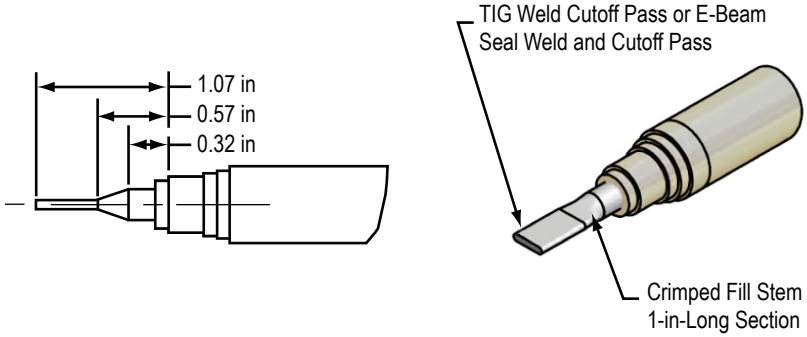

Figure 31. Pinch and seal weld option for heat pipe fill stem closeout.

The pinch and seal weld approach prevents the heat pipe internal volume and sodium from being exposed to the external environment (glove box or electron beam welder). However, there is risk associated with the pinch and weld technique because the presence of sodium in the welding zone presents a possibility for voids or formation of low-melting-point intermetallic compounds in the weld. Additionally, there is a pressure differential across the pinch which tends to open the weld, making it susceptible to creep failure during long-term, high-temperature operation. A secondary (evacuated) enclosure is placed over the fill stem, providing containment; however, potential leakage past the pinch weld after long-term, high-temperature operation must still be considered as a possibility. Note: This leakage is not expected to impact performance, since the secondary enclosure is evacuated (no inert gas will be introduced) and the potential loss of sodium into the secondary containment volume is small.

(b) A second approach to extracting the vanadium wire sample requires fewer overall steps but results in more sodium being removed from the heat pipe (requiring overfill to compensate).

At the conclusion of the equilibration step, the sodium is allowed to remain in the heat pipe fill stem. The sodium within the fill stem forms a freeze plug, protecting the heat pipe internal volume. To remove the segment of fill stem containing the vanadium wire, a clean cutting tool (type and material composition to be determined) would be used to cut the fill stem ≈0.5 cm from the condenser plug. The end of the fill stem is then fitted with a cap that is welded to the condenser plug, as shown in figure 32. This process will be discussed in more detail in section 3.5.2.

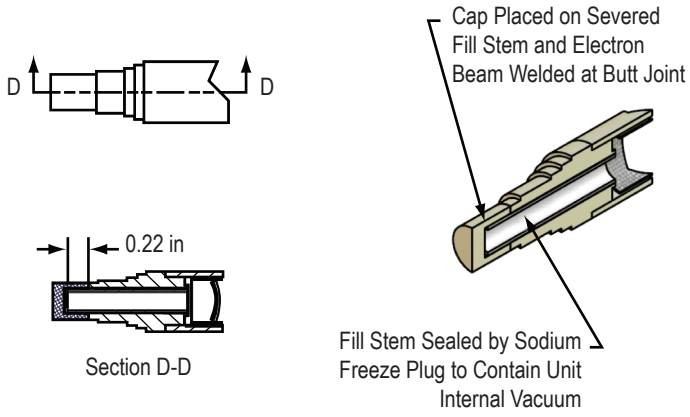

Figure 32. Cutoff and plug seal weld option for heat pipe fill stem closeout.

With this approach, the heat pipe internal sodium (fill stem freeze plug) is exposed to the glove box environment when it is severed and will begin to pick up contaminants. There is an additional risk of further contamination when the heat pipe is transported from the glove box to the electron beam welder for attachment of the cap to the condenser plug. With this technique, there should be minimal risk of introducing sodium in the weld area, so high-temperature operation should present no issues. Secondary protection to the cap closeout is provided by an outer evacuated enclosure welded to the heat pipe condenser plug. Note: Exposure time to the glove box and electron beam welder environment should be minimized once the fill stem is severed. Inert gas carriers will need to be built to transport the heat pipe from the glove box to the welder; the welder environment will also require inert gas fill. Checkout tests will be necessary to verify operations and to make recommendations regarding potential cap design variations (which include the secondary enclosure).

(8) Once the vanadium wire section of the fill stem has been removed, it can be processed and evaluated for oxygen content as described in appendix G.

(9) The heat pipe unit with sealed fill stem is now ready for final closeout and wet-in operations.

To execute these operations, additional support hardware will be required for glove box operations. Items will include Mo-Re-compatible point heaters for low-temperature operations (up to 650 K); a small vacuum furnace capable of providing equilibration temperatures (up to 1,050 K); chill blocks, as necessary; and associated instrumentation and fluid connections to accomplish the tasks outlined above.

3.5 Heat Pipe Unit Final Closeout and Wet-In

After completion of the sodium fill and sampling operations, the heat pipe fill stem is sealed, using one of the two possible methods (or a variation thereof) that were mentioned in section 3.4. The following discussion provides additional details on the implementation of the two closeout techniques. Attachment of a secondary evacuated containment/structural support tube assembly and the final wet-in process to uniformly distribute and wet internal surfaces with the sodium working fluid are also outlined. For all operations, a series of checkout evaluations will be implemented using a combination of stainless steel prototype heat pipes and Mo-Re material samples to verify each step of the process.

3.5.1 Heat Pipe Fill Stem Pinch Closeout

The first method of sealing a heat pipe fill stem is referred to as the 'pinch' method. Illustrated in figure 31, this approach requires that a portion of the stem tubing be flattened, similar to the practice that is used after filling. The fill stem is first crimped and then fusion cut/welded, followed by additional seal weld passes as necessary. To implement this approach, the sodium must first be settled to the evaporator end of the heat pipe to clear the lower end of the fill stem. This allows the stem to be tightly compressed, which is necessary for a successful weld. Some residual sodium, <1-in length, remains at the top of the fill stem, held in place by surface tension; however, it should not interfere with the crimping operation on the lower portion of the stem.

With the fill stem clear, an ≈1-in (2 to 2.5 cm) length of the fill stem is compressed (centered 2.5 in from the end of the condenser plug), using a hydraulic pressing device incorporated into the glove box. The press dies are made of hardened steel with molybdenum foil attached to the die faces to minimize material contamination. The pinch process flattens the fill stem tube with approximately 0.002 to 0.005 in of plastic deformation to minimize material springback; a minimal material gap is required to achieve a successful leak-tight fusion weld that seals as it cuts. The use of a fusion weld/cut pass prevents introduction of the external atmosphere into the heat pipe, because the molten material blocks the potential entry path; the tight pinch provides a very small path that prevents entrainment of molten steel back into the heat pipe unit. Figure 33 shows two weld techniques that can be used to make this cut. The first uses a TIG torch within the glove box to make a high-current pass across the midsection of the crimp, simultaneously cutting and fusing the material and allowing the outer portion of the fill stem to fall away. The TIG torch can then be used at a lower current setting with a filler material to trace back over the fused crimp end, adding material to build up the weld thickness to approximately match that of the tube wall thickness, typically 0.030 in.

The second welding approach makes use of an electron beam welder with a two-pass process to seal the crimp. The first pass is a seal weld, at the heat pipe end of the crimp, with the beam welder energy adjusted to provide a full penetration weld. For the second pass, ≈0.2 in from the seal weld, the beam energy is set sufficiently high to completely cut the material, fusing the end of the crimp and allowing the free end of the fill stem to fall away. A general outline for this approach is provided in appendix H.

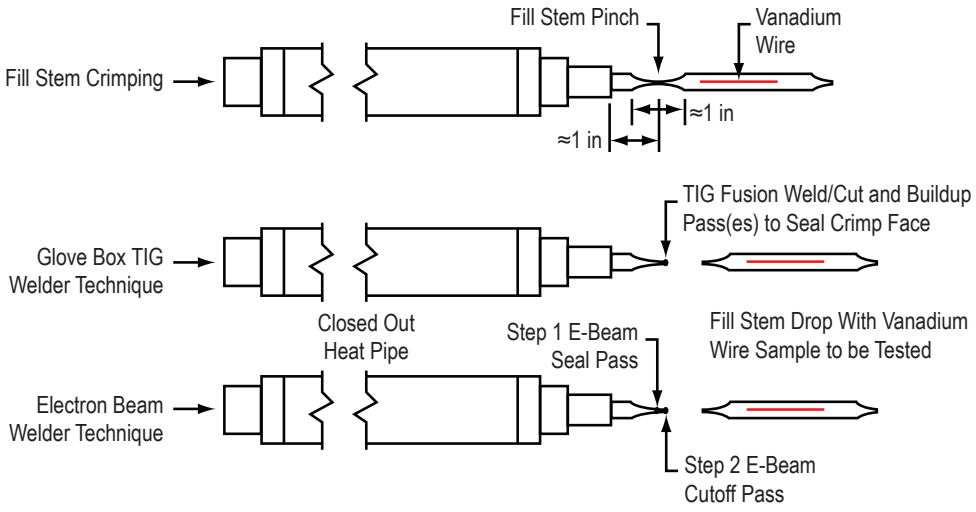

Figure 33. General fill stem fusion weld/cutoff techniques for the pinch method.

The free portion of the fill stem contains the vanadium wire segment. This sample can be opened and analyzed to verify sodium purity based on the test procedure described in appendix G. The use of the 'pinch' closeout approach provides for a closeout technique that does not expose the heat pipe internal sodium to an external environment. However, the presence of sodium in the weld zone introduces the possibility of voids and the formation of low-melting-point, intermetallic compounds, placing a risk factor on the long-term performance of the pinch weld as a primary seal. This is solved by the use of a secondary, evacuated, protective containment tube placed over the closeout fill stem; this outer tube also serves as a structural support member.

3.5.2 Heat Pipe Fill Stem Cap Closeout

The second fill stem closeout approach is referred to as the 'cap' method, because it makes use of a cap arrangement to close out the end of the fill stem. A cross-sectional view of this technique was shown in figure 32. In this approach, the sodium remains in the fill stem, since it is required as a freeze plug, preventing inert Ar from the glove box from entering the heat pipe. At a distance of ≈ 0.2 in from the condenser plug, a cleaned cutting tool is used to sever the outer length of the fill stem from the heat pipe unit. The removed portion of the fill stem contains the vanadium wire segment, which will be tested using the procedure in appendix G to determine sodium purity.

To seal the fill stem stub, a snug-fitting Mo-Re cap is slid over the fill stem and pressed tightly against the face of the heat pipe condenser plug. The cap is sealed to the condenser plug by use of a circumferential, full-penetration, electron beam weld. Since this weld must be made in vacuum, the heat pipe has to be transported from the safety of the inert glove box to the electron beam welder. To perform this transfer, the heat pipe must be placed in an inert gas carrier and then bagged while in the electron beam welder, where it is positioned in the alignment assembly. This is the riskiest portion of the operation, and a great deal will depend on the speed and precision of the transport and setup operations. Additionally, it is very important that once the heat pipe is set up in the electron beam welder, all inert Ar is evacuated from the cap interior so that it will not be introduced into the heat

pipe volume, once the freeze plug melts during actual heat pipe operation. To assist in eliminating this gas, it may be necessary to provide a very small groove along the inner surfaces of the cap that is sufficient for venting but is not large enough to interfere with the welding process. Once the electron beam welder is evacuated to the 10^{-6}-torr range, a step that will take ≈3 hr to ensure venting, a full-penetration weld will be made around the circumference to seal the heat pipe. Figure 34 illustrates this process. Checkout tests will be required to verify that no sodium melt enters the weld zone as a result of heating from the welding process.

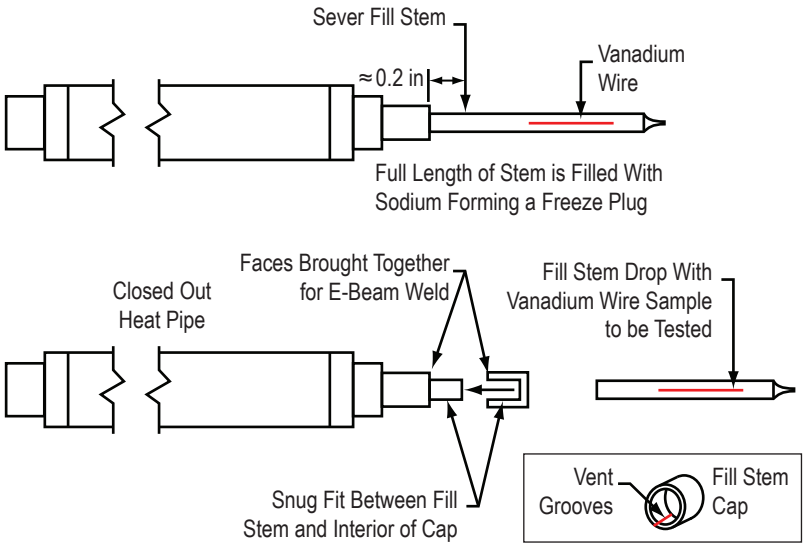

Figure 34. General fill stem cutoff and weld techniques for the cap method.

Although the cap approach has fewer steps and a final closeout weld that should not have sodium contamination, as compared to the pinch technique, it has a significant potential for the introduction of contamination at the sodium freeze plug interface. This could potentially void all the effort put into purifying and sampling and could introduce unknowns regarding the final state of the heat pipe working fluid. Variations to this approach need to be investigated to alleviate the potentially troublesome portions of the described operations. Appendix I includes a sample procedure for implementing this closeout approach.

3.5.3 Heat Pipe Final Closeout and Support

To provide secondary containment for the heat pipe fill stem seal and to offer a means of supporting the heat pipe in the test fixture, a series of outer tubes is attached to the heat pipe condenser plug. The secondary containment is provided by an evacuated tube section, with a diameter of 0.5 in, a wall thickness of ≈0.030 in, and a length of 1.5 in; the support tube is ≈3.5 in long, with the same diameter and wall thickness. An intermediate transition plug connects the two tube sections. Figure 35 illustrates the general layout of the final closeout for the case, using the fill stem 'cap' method. The outlined closeout approach should be compatible with the current identified fill stem closeouts or reasonable variations on those closeouts.

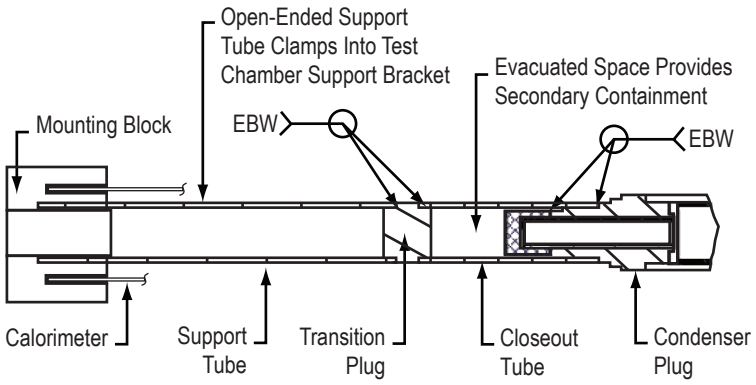

Figure 35. General fill stem final closeout with secondary containment and support tube.

Alignment of the containment and support tubes is critical to the assembly, because these members will be used to center the heat pipe in the calorimeter unit. To assist in the assembly, very tight slip-to-interference fits with ample shouldering will be employed on both the heat pipe condenser plug and the transition plug; this should significantly minimize wobbling. If necessary, an alignment fixture can be fabricated to hold straightness during assembly. Full-penetration electron beam welding will be used for all three closeout welds to minimize heat-induced warping and to provide an evacuated condition within the volume of the closeout tube. Checkout evaluations of these techniques will be performed during assembly of the stainless steel heat pipe prototypes to verify that the assembled straightness is sufficient to allow integration with the tight-tolerance calorimeter units.

Another consideration in the design is the volume of the evacuated secondary containment space; this region is large enough to hold 1.5 to 2 g of sodium, depending on temperature, should it be completely filled in the event of a fill stem seal failure. This failure/loss must be considered during assessment of the heat pipe sodium fill requirement. However, this condition must also be weighed with the probability or the type of weld failure, which is dependent on the fill stem closeout technique selected. In most cases the failure would be a small opening that propagates through interconnecting porosity, possibly caused by the presence of sodium in the weld; the geometric constraints of the flow path would limit the amount of sodium transferred. The length of the closeout tube could be adjusted to reduce the overall volume available to leakage, and the support tube could be extended to maintain the same overall support geometry. Sample procedures outlining the general assembly steps for the final closeout are listed in appendix J.

3.5.4 Heat Pipe Final Wet-In

The final step in processing heat pipe units is to perform a high-temperature, wet-in to wet all internal surfaces, including the wick structure, with the sodium working fluid. Fully distributing the sodium throughout the heat pipe and completely filling the capillary channel improves startup characteristics and is especially important for operation at high heat flux, approaching the boiling limit. The wet-in will be performed in a clean vacuum furnace operating in the 10^{-6} torr range at a steady-state temperature of 50 to 100 °C above maximum operating temperature for a duration of 24 to 48 hr.

Before the heat pipe units are loaded into the vacuum furnace, their exterior surface must be cleaned to remove potential surface contamination using PF® solvent and alcohol inside an ultrasonic bath. Once loaded into the vacuum furnace, the chamber must be evacuated and baked out at a minimum of 200 °C to assist in removing water vapor and other surface contaminants that could affect the heat pipe unit. The wet-in startup heating transient typically requires 2 hr to reach the desired temperature. Shutdown employs an ambient furnace cooling cycle, which requires several hours. Appendix K provides a general outline of the wet-in process.

4. SUMMARY

A general test plan and evaluation sequence has been identified. The proposed procedures will apply to the accelerated life testing of the 16 Mo-Re alloy heat pipe units outlined in this project. These tests will examine a range of operating conditions, including variation of temperature and mass fluence, to investigate the long-term effects of heat pipe aging. Standard operating procedures have been developed for all hardware operations that address this goal; general outlines have been provided for heat pipe fill, closeout, and test. The documented information is intended to serve as an initial step, identifying the currently envisioned options and potential impacts. These processes will be updated as hardware is produced and checkout testing, using stainless steel precursor heat pipes, is performed, producing new information on the viability of each process. Intervals for both nondestructive and destructive evaluation of the heat pipe units are incorporated into the testing sequence. One possible NDE technique includes a three-dimensional x-ray tomography system that may provide sufficient resolution to detect the onset of corrosion.

A variety of options were outlined and discussed for heat pipe preparation. Two methods were identified for the fill process: direct liquid transfer and distillation. The distillation method produces a higher purity sodium fill and also limits residual glove box argon remaining in the final heat pipe assembly. The planned performance test will be instrumental in determining if liquid transfer is sufficient or if distillation is required. Performance test results will also indicate if the proposed high heating rates required in the baseline test matrix can be reliably achieved; adjustments in processing techniques and variation of the test matrix may be required. To monitor sodium purity, a workable method of incorporating a vanadium sampling wire into the heat pipe fill stem has been identified. The only potential concern regarding the outlined approach is the current diameter of the baseline heat pipe fill stem; this diameter may restrict circulation of the hot sodium. Increasing the fill stem size would impact many of the current heat pipe activities, affecting cost and extending schedule; alternatives will be investigated. The final heat pipe closeout is a critical operation; two candidate approaches have been identified, each with pros/cons related to the number of operational steps and potential for contamination. Hands-on checkout operations with sample hardware will be required to determine the final closeout variation to be used on the 16 Mo-Re heat pipe units. The final operational procedures developed during this project could be used as a starting point for the detailed development and testing process required of a flight prototype heat pipe program.

APPENDIX A—REFERENCE DESIGN AND TEST MATRIX CONDITIONS

The baseline heat pipe design and the conditions for accelerated life tests in neutral gravity are provided in table 3. Life tests and reference designs for the F-series, cross-correlating temperature and mass fluence effects, are provided in table 4. Life tests and reference designs for the G-series, investigating time-dependent corrosion effects, are provided in table 5.

Table 3. Life test heat pipe design and conditions—neutral gravity.

Scale	Test Heat Pipe Values	Revised Reference Design Values
Wick shape	Annular gap	Annular gap
Evaporator length	$L_e = 0.075$ m	$L_e = 0.75$ m
Adiabatic length	$L_a = \approx 0$ m	$L_a = \approx 0.50$ m
Condenser length	$L_c = 0.25$ m	$L_c = 0.75$ m
Container inside radius	$R = 0.705$ cm	$R = 0.552$ cm
Channel dimension	$A = \approx 0.056$ cm	$A = \approx 0.05$ cm
Wick pore radius	$r = 35$ μm nominal	$r = 15$ μm nominal
Nucleation site radius	$n = \approx 1$ μm	$n = \approx 1$ μm
Solid thermal conductivity	$k = 60$ W·m^{-1}·K^{-1}	$k = 60$ W·m^{-1}·K^{-1}
Working fluid	$f =$ Na	$f =$ Na
Temperature	$T = \{1{,}173$ to $1{,}373$ K$\}$	$T = \{1{,}173$ to $1{,}373$ K$\}$
Design heat pipe power	$Q = 3$ kW$_t$	$Q = 8$ kW$_t$

Table 4. Life tests: Fisher series (F-series) with reference design.

Heat Pipe	Wall/Wick Material/Fluid	L_c/L_e	Di (cm)	T (K)	α (T)	q (W)	q_{rad} (W/cm^2)	G (kg/cm^2-s)	t (hr)	M" (kg/cm^2)
Design	Mo-Re/Na	2	1.41	1,250	1.00	5,752	22	0.059	105,120	2,233
F(-4)	Mo-Re/Na	3	1.41	1,273	1.30	5,000	226	0.599	26,280	5,669
F(-3)	Mo-Re/Na	3	1.41	1,273	1.30	1,000	45	0.120	26,280	1,134
F(-2)	Mo-Re/Na	3	1.41	1,373	3.74	3,000	136	0.368	26,280	3,483
F(-1)	Mo-Re/Na	3	1.41	1,173	0.38	3,000	136	0.351	26,280	3,321
F(0)	Mo-Re/Na	3	1.41	1,273	1.30	3,000	136	0.360	26,280	3,401
F(1)	Mo-Re/Na	3	1.41	1,223	0.72	2,000	90	0.237	26,280	2,241
F(2)	Mo-Re/Na	3	1.41	1,223	0.72	4,000	181	0.474	26,280	4,481
F(3)	Mo-Re/Na	3	1.41	1,323	2.25	2,000	90	0.243	26,280	2,295
F(4)	Mo-Re/Na	3	1.41	1,323	2.25	4,000	181	0.485	26,280	4,589

Table 5. Life tests: G68-80 series (G-series) with reference design.

Heat Pipe	Wall/Wick Material/Fluid	L_c/L_e	Di (cm)	T (K)	α (T)	q (W)	q_{rad} (W/cm^2)	G (kg/cm^2-s)	t (hr)	M" (kg/cm^2)
Design	Mo-Re/Na	2	1.41	1,250	1.0	5,752	22	0.059	105,120	2,233
G-1	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	4,380	567
G-2	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	8,760	1,134
G-3	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	13,140	1,701
G-4	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	17,520	2,267
G-5	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	21,900	2,834
G-6	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	26,280	3,401
G-7	Mo-Re/Na	3	1.41	1,273	1.3	3,000	136	0.360	30,660	3,968

APPENDIX B—GENERAL MATERIAL CLEANING PROCEDURES

B.1 Stainless Steel Cleaning Procedure

This procedure applies to austenitic stainless steel in the as-milled condition. It may be used for screen and wire cloth for heat pipes, tubing, plate, and other forms of material that, although machined in part, contain surfaces in the as-milled condition. The term 'wash' constitutes full immersion in fluid.

(1) Wash part in PF solvent in an ultrasonic cleaner for at least 5 min to remove all signs of grease.

(2) Wash part in ultrasonic cleaner containing a caustic solution consisting of 11 parts (by volume) deionized water, 1 part sodium hydroxide, and 1 part hydrogen peroxide for up to 5 min.

(3) Wash part in hot deionized water for at least 5 min.

(4) Repeat steps (2) and (3) three times.

(5) Wash part in hot deionized water in ultrasonic cleaner for at least 5 min.

(6) Wash part in ethanol in ultrasonic cleaner for at least 5 min.

(7) Proceed to vacuum bake-out; establish a pressure of 10^{-6} torr and a temperature of 50 °C over the maximum operating temperature.

B.2 Molybdenum-Rhenium Alloy Cleaning Procedure

This procedure applies to all Mo and Mo-Re alloys. It may be used for screen and wire cloth for heat pipes, tubing, plate, and other forms of material that, although machined in part, contain surfaces in the as-milled condition. The term 'wash' constitutes full immersion in fluid. Alloys with low percentages of Re will suffer from recrystallization and lose ductility.

(1) Wash piece in PF solvent until all signs of grease have been removed.

(2) Soak piece for 1 to 2 min in 1 part by volume HCl and 1 part by volume deionized water to remove residual iron surface impurities.

(3) Soak piece for 5 min in caustic cleaning solution consisting of 11 parts by volume deionized water, 1 part by volume NaOH, and 1 part by volume H_2O_2. Remove piece from caustic bath. Replenish or replace solution as required.

(4) Wash part in hot deionized water for at least 5 min.

(5) Repeat steps (3) and (4) three times.

(6) Rinse piece for 5 min in ethanol inside an ultrasonic cleaner.

(7) Vacuum- or hydrogen-fire the part at a temperature 50 °C over the maximum operating temperature.

B.3 Molybdenum-Rhenium Alloy Cleaning Procedure for General Handling

This procedure is to be used to clean up all exterior surfaces of a heat pipe that is ready for testing to remove contaminants that might have been picked up from inappropriate handling, such as not using gloves.

APPENDIX C—GENERAL KNOWN VOLUME FILL PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____

Known Volume Cylinder Identifier:	_____	Initial Mass Known Volume:	____g
Date Known Volume Fill:	_____	Final Mass Known Volume:	____g
		Sodium in Known Volume:	____g

C.1 General Notes

The following general notes apply:

(1) During periods when sodium metal and parts wetted by sodium metal are exposed to the glove box atmosphere, the oxygen and water vapor levels must be maintained below 5 ppm (–75 dewpoint for water vapor).

(2) A copy of this record shall be completed and placed in the heat pipe traveler.

(3) The sodium known volume cylinders must be chemically cleaned and vacuum baked to 10^{-5} torr and at least 10 °C above the maximum transfer temperature. After baking, internal—to be wetted—surfaces shall be exposed only to the glove box atmosphere.

(4) Each known volume cylinder must be marked to allow identification when it is later used to transfer sodium to a heat pipe unit. The only exception is when the known volume is to be immediately transferred to a waiting heat pipe.

(5) A known volume cylinder may be used multiple times during the heat pipe filling process. The typical limiting factor is failure of the compression seals to hold a vacuum.

(6) When a known volume cylinder that has been used previously is to be stored, it should be connected to the fill station, evacuated, and valved off to maintain an internal vacuum.

(7) Heat the sodium distribution system to the minimum temperature required to achieve a successful transfer, so as to minimize oxygen solubility.

C.2 Procedures for Known Volume Fill

The following procedures are used to fill the known volume. Refer to figure 36.

(1) Heat the sodium reservoir to a temperature in the 150 to 200 °C range.

(2) Record glove box: oxygen, ____ppm, and water vapor, ____dewpoint.

(3) Record tare weight of the known volume cylinder, _____ g.

(4) Remove the compression plug sealing the sodium fill system filter, F2.

(5) Seat the known volume cylinder into the filter port. The Nupro isolation valve should be on the bottom of the known volume cylinder.

(6) Attach a high vacuum line to the Nupro isolation valve at the bottom of the known volume cylinder.

(7) Open the Nupro isolation valve and hand-operated valve (HOV) H08 connecting the known volume to the high vacuum system.

(8) Evacuate the known volume cylinder, gauge PG H04, into the low 10^{-5}-torr range: _____torr.

(9) Place heater tape on internal sodium lines, valve, filter, and known volume cylinder.

(10) Wrap insulating aluminum foil over lines, valve, filter, and known volume as required.

(11) Heat sodium lines external to the glove box to a temperature in the 150 to 200 °C range.

(12) Open HOV H03a.

(13) Open HOV H03.

(14) Open HOV H05.

(15) Bring programmable logic controller (PLC) L02 to 2.9 Vdc (14 psia).

(16) Close valves (HOV H08 and Nupro isolation) at bottom of known volume to prevent sodium from entering the vacuum system.

(17) Heat sodium lines inside glove box to a temperature in the 150 to 200 °C range.

(18) Heat the sodium filter (F2) to a temperature in the 120 to 170 °C range.

(19) Heat the known volume cylinder to a temperature in the 100 to 150 °C range.

(20) Record all temperatures on traveler sheet, figure 36.

(21) Record a screen capture of data.

(22) Open HOV S02.

(23) Tap on known volume cylinder during transfer to ensure even sodium distribution.

(24) Watch for increase in temperature on TK1 and TM2, indicating sodium has transferred.

(25) Wait 1 min.

(26) Close HOV S02.

(27) Record a screen capture of data.

(28) Shut off heaters inside the glove box and allow components to cool to 30 °C (remove insulation and heaters as necessary).

(29) Set PLC L02 to 0 psia and close HOV H05.

(30) Record box atmosphere: ___ppm O_2, ___ °C dewpoint.

(31) Record a screen capture of data once system has cooled.

(32) Remove known volume cylinder from sodium filter.

(33) Replace sodium filter compression plug seal.

(34) Turn off heaters external to glove box if no additional fills are to be performed.

(35) Weigh newly filled known volume cylinder: _____g

C.3 General Notes or Significant Observations

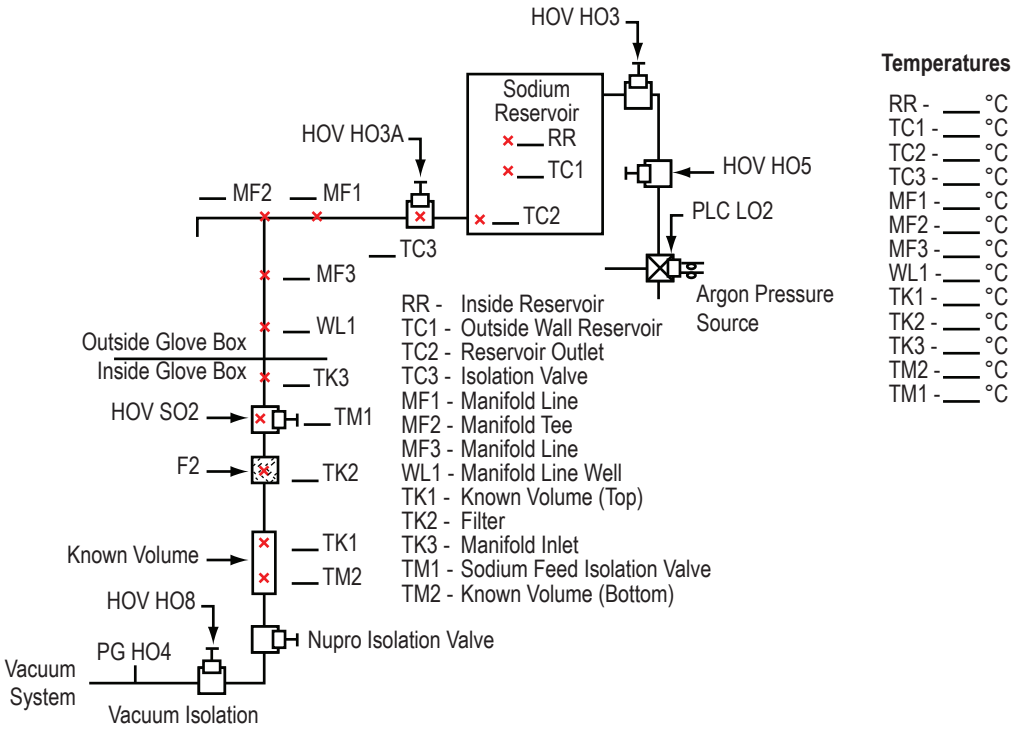

Figure 36. Known volume cylinder fill schematic.

APPENDIX D—GENERAL KNOWN VOLUME TO HEAT PIPE SODIUM TRANSFER PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____

Known Volume Cylinder Identifier:	_____	Initial Mass Known Volume:	____g
Date Heat Pipe Fill:	_____	Final Mass Known Volume:	____g
Heat Pipe Identifier:	_____	Sodium in Transferred-to Heat Pipe:	____g

D.1 General Notes

The following general notes apply:

(1) During periods when sodium metal and parts wetted by sodium metal are exposed to the glove box atmosphere, the oxygen and water vapor levels must be maintained below 5 ppm (–75 dewpoint for water vapor).

(2) A copy of this record shall be completed and placed in the heat pipe traveler.

(3) New valves attached to the heat pipe after filling should be chemically cleaned and vacuum baked to 250 °C.

(4) Load heat pipe units into glove box gallows per glove box standard operating procedure.

D.2 Procedures for Sodium Transfer to Heat Pipe Unit

The following procedures are used to transfer sodium from the known volume to the heat pipe unit:

(1) Weigh known volume tare: ___ g.

(2) Record box atmosphere oxygen, ___ ppm, and water vapor, ___ dewpoint.

(3) Attach the sodium transfer isolation Nupro valve to heat pipe fill stem.

(4) Connect the glove box high vacuum system to the heat pipe isolation valve.

(5) Open HOV H08 and the heat pipe isolation valve and evacuate the heat pipe into the mid 10^{-5}-torr range: _____ torr.

(6) Close HOV H08 and the heat pipe isolation valve and disconnect from the vacuum system.

(7) Attach known volume cylinder to heat pipe isolation valve at fill stem.

(8) Place heater tape on volume cylinder, fill stem, and upper portion of heat pipe unit.

(9) Wrap insulating aluminum foil over the known volume cylinder, fill stem, and upper portion of the heat pipe as needed.

(10) Bring the known volume to a temperature in the 150 to 200 °C range: value: ____°C.

(11) Bring the heat pipe heater to a minimum temperature of 120 °C: value: ____ °C.

(12) Record temperatures on figure 37.

(13) Record a screen capture of data.

(14) Open the isolation valve connecting the heat pipe and known volume cylinder.

(15) Open known volume cylinder vent valve if present.

(16) Tap on assembly to allow sodium to flow into heat pipe; wait 1 min.

(17) Watch for an increase in the temperature on TK2 to verify sodium has transferred.

(18) Record a screen capture of data.

(19) Turn off heaters and allow assemblies to cool to room temperature. Remove insulation as necessary to assist in cooling.

(20) Record a screen capture of data once temperatures have cooled to <50 °C.

(21) Remove the known volume cylinder and valves from heat pipe unit.

(22) Weigh the known volume cylinder: _____ g.

(23) Place new, clean, vacuum-baked isolation valve onto the heat pipe fill stem.

(24) Attach the glove box high vacuum system to the heat pipe isolation valve.

(25) Evacuate heat pipe for 15 min and tighten fittings as necessary to achieve a pressure in the mid to low 10^{-6}-torr range; record value: _____ torr.

(26) Close the heat pipe isolation valve and vacuum system valve (HOV H08) and disconnect the heat pipe unit.

D.3 General Notes or Significant Observations

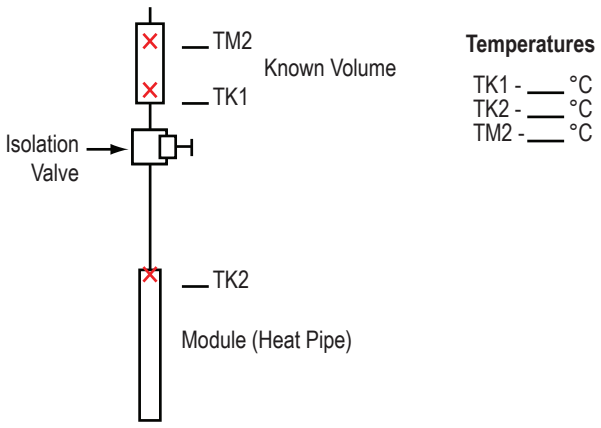

Figure 37. Schematic for Na transfer from known volume cylinder to heat pipe unit.

APPENDIX E—GENERAL HEAT PIPE LEAK CHECKING PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____
Heat Pipe Identifier: _____
Operation Date: _____

E.1 General Notes

The following general notes apply.

(1) Verify that the heat pipe isolation valve is a new, clean valve and has been chemically cleaned and vacuum baked to 250 °C.

(2) A copy of this record shall be completed and placed in the heat pipe traveler.

(3) During leak checks, He accumulates inside the glove box, resulting in an increase in the leak detector baseline by: (a) diffusion through the O-ring seals on the vacuum system, and (b) infiltration into vacuum lines when leaks occur and when heat pipe units are changed out.

(4) Typically it is very difficult to maintain a leak rate in the 10^{-9} std atm cc/s range and nearly impossible to achieve the 10^{-0} std atm cc/s range, since the glove box is a closed environment; see figure 38. However, the Varian leak detector, model 979, has a very useful zeroing feature that, when engaged, resets the sampling range to the 10^{-10} std atm cc/s (subtracting the background baseline). Helium can then be sprayed around the fittings under investigation, and any increase in leak rate—helium concentration—is readily sensed.

(5) If processes can be adjusted such that the heat pipe activities can be performed completely in the glove box or the heat pipe internal volume can be sealed by welding as soon as possible while pulling vacuum, a better internal vacuum condition can be achieved, because all mechanical fittings and valves typically have some leak rate.

E.2 Procedures for Heat Pipe Leak Check

The following procedures are used to check the heat pipes for leaking:

(1) Connect the heat pipe isolation valve to the glove box high-vacuum system.

(2) Open HOV H08 and monitor the vacuum pressure on the ion gauge (PG H04); leave the isolation valve closed.

(3) Connect the leak detector to the glove box high-vacuum system turbo roughing line.

(4) Bring the leak detector online and, when ready, rough down connecting lines.

(5) Open the leak detector isolation valve to the glove box vacuum system.

(6) Close the glove box high-vacuum system roughing isolation valve. All flow is now directed into the leak detector.

(7) Using a portable He bottle inside the glove box, spray He around the isolation valve fitting vacuum system side; tighten fittings as necessary to eliminate large leaks; monitor the ion gauge vacuum level, which should be in the mid to low 10^{-6}-torr range: _____ torr.

(8) Record the leak detector baseline leak rate; it should be in the 10^{-9} std atm-cc/s range (depending on the number of prior leak checks performed that day). Record the leak rate value: _____std atm-cc/s. Allow several minutes to stabilize.

(9) Enable the leak detector zeroing function if required.

(10) Open the heat pipe isolation valve and record the highest pressure: _____ torr.

(11) Spray He on heat pipe isolation valve fittings, monitoring the leak rate, and tighten fittings as needed; allow several minutes to stabilize; record value: _____ std atm-cc/s.

(12) Evacuate heat pipe for at least 15 min record vacuum pressure: _____ torr.

(13) Final check—close the heat pipe isolation valve for 2 to 5 min; record value: _____ torr.

(14) Open heat pipe isolation valve and record pressure spike: _____ torr. A large increase indicates a leak, while a small increase is most likely caused by outgassing.

(15) Close the heat pipe isolation valve.

(16) The heat pipe is leak tight and ready for the next stage of operation.

E.3 General Notes or Significant Observations

The general glove box with leak detector setup is shown in figure 38.

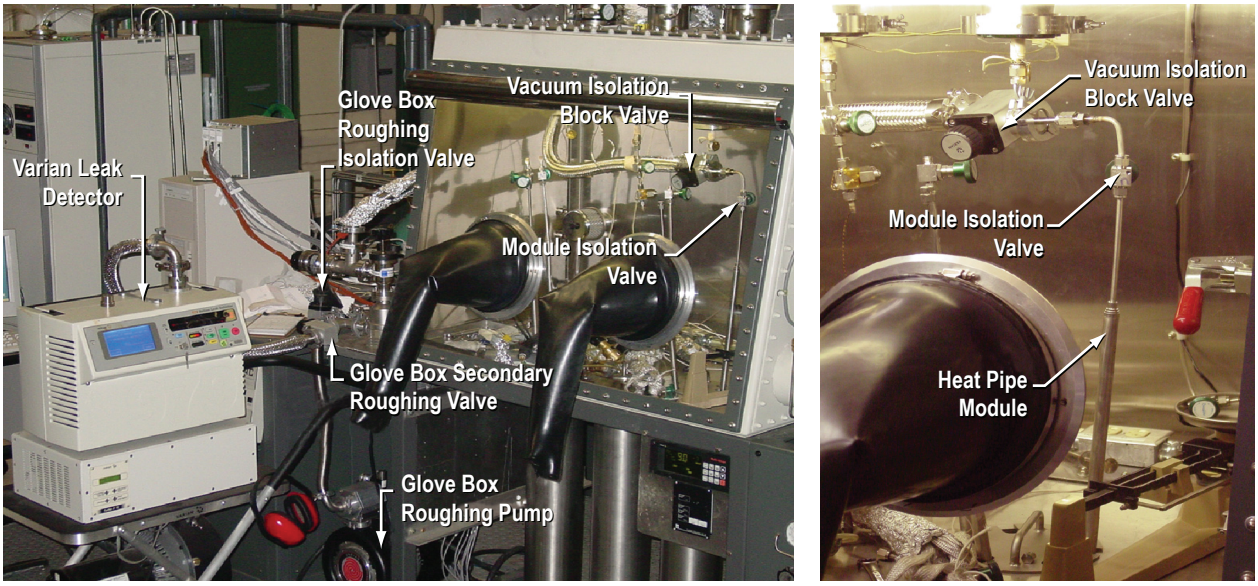

Figure 38. General glove box with leak detector setup.

APPENDIX F—GENERAL HEAT PIPE VACUUM CONDITIONING PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____
Heat Pipe Identifier: _____
Operation Date: _____

F.1 General Notes

The following general notes apply:

(1) The glove box high-vacuum system shall be used to evacuate the heat pipe for this operation; see figure 39.

(2) A copy of this record shall be completed and placed in the heat pipe traveler.

(3) A helium leak detector shall be connected to the system so that all fittings can be checked.

(4) A low-temperature tube heater setup inside the glove box is needed that is capable of providing a temperature range of 200 to 250 °C.

(5) This operation should release any residual Ar trapped within the heat pipe unit.

F.2 Procedures for Heat Pipe Vacuum Conditioning

The following procedures are used to vacuum condition the heat pipe module:

(1) Connect the heat pipe to the glove box high-vacuum pump system.

(2) Use the vacuum system to evacuate up to the heat pipe isolation valve. Bake the vacuum system as necessary, for 1 to 4 hr at 150 to 200 °C, to obtain vacuum in the mid to low 10^{-8}-torr range. Repair any fitting leaks.

(3) Record the baseline pressure of the vacuum system: _____ torr.

(4) Open the heat pipe isolation valve and record the maximum pressure spike: _____ torr.

(5) Note how long to return to baseline: _____ seconds to _____ torr.

(6) Attach thermocouples to the heat pipe and place it in the tube heater setup.

(7) Bring the heat pipe to ≈200 °C and monitor the pressure. Record the highest value: _____ torr.

(8) Maintain heating for ≈2 hr or until the pressure falls back into the low 10^{-7}- or 10^{-8}-torr range. Periodically tap on the heat pipe to help release trapped gasses.

(9) Record temperatures on figure 39.

(10) Turn off heaters and record the module pressure at room temperature: _____ torr.

(11) Record a screen capture of data.

(12) Evacuate for an additional 30 min and record pressure: _____ torr.

(13) Close the heat pipe isolation valve and detach from the glove box high-vacuum system.

F.3 General Notes or Significant Observations

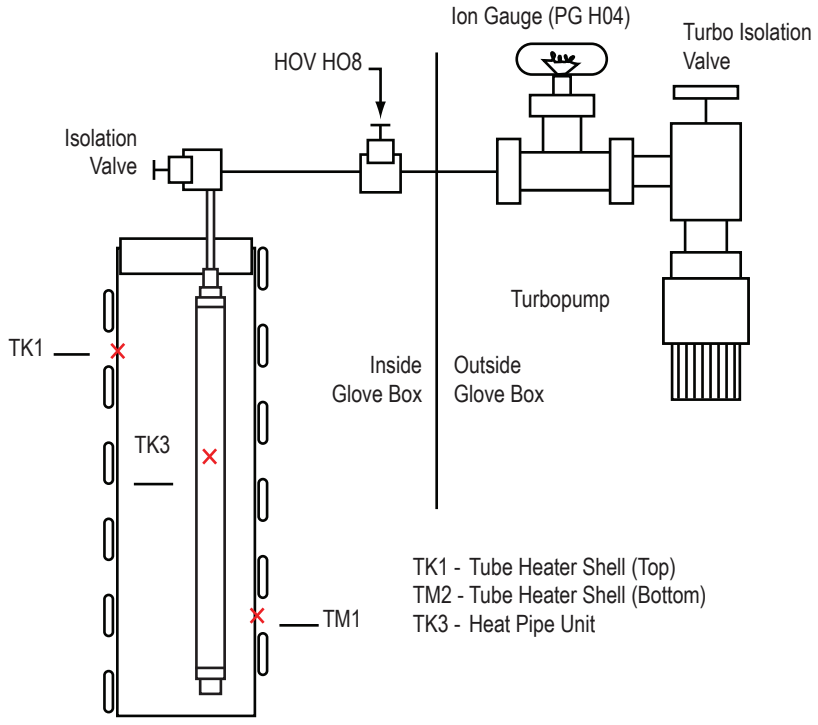

Figure 39. Hardware setup for vacuum conditioning a heat pipe unit in the glove box.

APPENDIX G—GENERAL HEAT PIPE PURITY TESTING PROCEDURE—VANADIUM WIRE EQUILIBRATION

Note: The following information is extracted with some modifications from appendix B of a NASA Technical Memorandum titled, "Method for Determination of Less Than 5 ppm Oxygen in Sodium Samples."[11]

STANDARD TECHNIQUE FOR ANALYSIS OF NUCLEAR-GRADE SODIUM HEAT PIPES BY THE EQUILIBRATION METHOD USING VANADIUM WIRES

This procedure is based on ASTM C997-83,[9] sections 65 to 74.

1. Scope

1.1. This method is applicable for determining oxygen in sodium using the Wire and Foil Equilibration Sampling Procedure. This procedure requires 3 to 4 hr, excluding equilibration time.

1.2. This method appears applicable in the range of 10 to 1,000 μg of oxygen in vanadium (0.1 to 15 μg/g of oxygen in sodium with the amount of vanadium wire usually available). The range may be extended down to 0.003 μg of oxygen in sodium, if vanadium wires of 0.1 g are available.

2. Summary of Method

2.1. A vanadium wire is immersed in sodium at 750 °C (1,382 °F) for a time sufficient to establish equilibrium with respect to oxygen. Subsequent measurement of the oxygen concentration in the wire is related to the concentration of active oxygen in sodium at that temperature by means of the distribution coefficient.

3. Interferences

3.1. Temperature-induced equilibrium shifts, involving oxygen and other impurities, can theoretically affect the oxygen concentration determined by this procedure, if the equilibration occurs at a temperature other than the system temperature. Extensive experience indicates that this is not a problem in measuring the oxygen in a 300 to 650 °C (572 to 1,202 °F) system.

4. Apparatus

4.1. Specimen-Equilibration Device Options—Figure 40 is a schematic drawing of the holder for use with sodium heat pipes and small sodium specimens. Figure 41 is a schematic drawing showing steps in the equilibration process for sodium heat pipes and small sodium containers (A—fill, B—equilibration, C—sever, and D—heat pipe closeout).

4.2. Electropolishing Apparatus—Figure 42 shows a typical electropolishing apparatus. The electrolysis cell consists of a 250-mL, tall-form beaker with a cylindrical cathode, >1,000 mm^2, near the bottom. Platinum and tantalum are suitable cathode materials. The lead from this electrode is insulated with shrink-fit TFE-fluorocarbon or polyethylene. Anode contact is made through spring-loaded forceps with platinum tips. The electrolysis cell rests on a magnetic stirrer. Direct current is supplied from batteries or a rectifier capable of providing up to 4 A at 4 to 25 V.

4.3. Oxygen-Determination Apparatus—Capable of determining 0.1% to 1.5% oxygen in vanadium metal by an inert gas or vacuum-fusion technique. A LECO RO-16 (currently marketed equivalent TC500) O Determinator has been used successfully.

4.4. Magnetic Stirrer—TFE-fluorocarbon-coated stirring bars.

4.5. Forceps—Self-locking type.

5. Reagents and Materials

5.1. Acetone, technical grade.

5.2. Electropolishing Solution—Cautiously add 200 mL of concentrated sulfuric acid to 800 mL of chilled methanol while stirring. Store in a glass bottle. Discard after use.

5.3. Ethanol, technical grade.

5.4. Lintless Tissue, Cel-Fibe Wipes No. 1745, or equivalent.

5.5. Nickel Flux, LECO part 763-065 or equivalent.

5.6. Oxygen Standards, ≈100 and 300 μg/g oxygen in steel. LECO Oxygen Standards. Stock Numbers 501-645 and 501-646 have been found satisfactory.

5.7. Vanadium Wire High Purity, annealed, 0.25-mm (0.010-in) or 0.50-mm (0.020-in) diameter with a tolerance of 0.005 mm (0.0002 in). Typical impurity concentrations are: <300 μg/g total metallic impurities—titanium + zirconium + hafnium shall be ≈20 μg/g; and ≈300 μg/g total of oxygen, nitrogen, hydrogen, and carbon—none of which shall be > 150 μg/g. The wire surface shall be smooth and free of scale, showing only line drawing marks. This surface must be free of galling and pitting marks. Ductility and surface condition of the wire must permit bending the wire 180° about its own diameter without surface cracking. The ductility of the wire must be sufficient to withstand, without fracture, six bends about its own diameter. A general description of the bend test is found in sections 14 and S22 of ASTM A370.[13] Vanadium wire of sufficient purity has been obtained from the Materials Research Corp., Orangeburg, NY 10962. Other potential V-wire sources are Goodfellow and All-Chemie.

6. Precautions

6.1. Observe the usual precautions for handling sodium, acids, and flammable liquids. Avoid electrical sparks when electropolishing to prevent ignition of the polishing solution.

7. Calibration of Vacuum-Fusion Analyzer

7.1. Check the instrument in accordance with the instruction manual and the precautions in section 6.1. Determine a crucible blank, and standardize the instrument with one high (≈300 μg of oxygen) and one low (≈100 μg of oxygen) standard.

8. Procedure

8.1. Wire Preparation and Equilibration:

8.1.1. Cut the vanadium wire into lengths suitable for the intended holder, and coil or straighten as required; see figure 40. Equal lengths of wire should be prepared for the heat pipe and test volume.

8.1.2. Degrease the wire with acetone. Handle the degreased wire with forceps or clean cotton gloves.

8.1.3. Place wires in wire holders located on the heat pipe/test volume end caps assembly. If a holder like that in figure 40 is used, fix the wires in place by bending their ends around the holder. Nickel is the preferred material for the wire holder and the test volume. Typically, 50 to 75 mm of 0.25-mm-diameter wire or 20 to 30 mm of 0.50-mm-diameter wire is exposed in an equilibration. Figure 41 depicts the subsequent sequence of steps.

8.1.4. Weld the holder to the heat pipe and the test volume bodies in an inert atmosphere.

8.1.5. Inside an inert atmosphere, introduce sodium into the heat pipe and test volume by transfer through the fill stem (step A), with a quantity of sodium sufficient to occupy the fill stem, test volume, and connecting holder tube, and cover the V wire contained within the heat pipe. The heat pipe orientation during the fill operation is typically with the condenser end above the evaporator.

8.1.6. Choose an equilibration time from figure 43 from the estimated concentration of oxygen in the sodium; if no reliable concentration estimate is available, assume 0.01 μg/g. The equilibration time for 0.25-mm-diameter wires must be in the 4- to 30-hr range. The equilibration time for 0.50-mm-diameter wires must be in the 16- to 120-hr range.

8.1.7. Place the heat pipe assembly condenser side down in a vacuum furnace that can be tilted 180° while at 750 °C (1,382 °F). Orient the heat pipe assembly as shown in figure 41, step B. Bring the furnace to 10^{-5} torr or better. Turn on thefurnace heaters; once the sodium melting temperature, ≈100 °C (≈212 °F), is reached, tap the assembly as needed to move sodium to the condenser end of the heat pipe assembly. Bring the heat pipe assembly to 750 ± 2 °C (1,382 ± 4 °F) for the chosen time.
8.2. Post-Equilibration Treatment:
8.2.1. Procedure for Nonradioactive Systems:
8.2.1.1. After the chosen equilibration period has elapsed, tilt the vacuum furnace at 750 °C to bring the heat pipe condenser above the evaporator. Tap the furnace to assist the sodium flow from the condenser end to the evaporator end. Shut off furnace heaters.
8.2.1.2. Cool the heat pipe assembly to a convenient temperature, not less than 110 °C (230 °F).
8.2.1.3. Tilt the vacuum furnace to return the heat pipe condenser below the evaporator; this will move the sodium back to the condenser end. Tap the assembly as needed. Cool the heat pipe to room temperature.
8.2.1.4. Sever the test volume from the heat pipe inside an Ar-purged dry box; see figure 41, step C.
8.2.1.5. The wire and a sodium sample are extracted from the test volume for analysis.
8.2.1.6. A cap is attached to the heat pipe, using an electron beam welder connected in an inert atmosphere; see figure 41, step D.
8.2.1.7. Dissolve the sodium adhering to the vanadium wires in about 1,000 mL of technical-grade ethanol. The large volume of ethanol prevents excessive wire heating.
8.2.1.8. Rinse holder and wires with water and allow the wires to dry.
Note: For the rest of the procedure, handle the wires with forceps.
8.2.1.9. Remove the wires from the holder. Use only straight portions of the wire for analysis. Make cuts, as necessary, at least 3 mm from each bend.
8.2.1.10. Separate the wires for archival storage from those for immediate analysis.
8.2.1.11. Store the archival wires in a properly identified, capped vial.
8.2.1.12. Fill the electrolytic cell with electropolishing solution. Grasp the wire with the forceps and adjust the anode position so that the forcep tips just contact the liquid and the wire is centered in the cell. If the wire is too long, cut or bend it into a "J" shape. With the stirrer at a low speed, start the electrolytic current. Adjust the voltage to provide a current of 5 to 10 mA/mm for 0.25-mm wire or 10 to 20 mA/mm for 0.50-mm wire. Polish each end of the wire for 30 s to reduce the diameter 0.03 to 0.05 mm. Rinse the wires in water and then methanol. Use only forceps to handle the clean wires.
8.2.1.13. Determine the oxygen content of the wire by a standard inert-gas-fusion or vacuum-fusion technique; i.e., by Method ASTM E146 or, if a vacuum-fusion analyzer is used, by the procedure described in section 8.3.
8.3. Determination of Oxygen by Vacuum Fusion Analyzer:
8.3.1. Cut 0.25 in (6.4 mm) off each end of the wire.
8.3.2. Cut the rest of the wire into lengths just under 3/8 in (9.5 mm) and place them into clean glass vials; about 10 pieces are obtained per wire.
8.3.3. Select and weigh a wire, based on the estimated oxygen concentration that will contain 100 to 300 µg of oxygen.
8.3.4. Put a nickel flux spiral into a new graphite crucible and insert the crucible into the lower electrode (without the nickel flux, the wires do not always completely fuse).
8.3.5. Using forceps, transfer the weighed group of wire sections to the empty wire loader. Using a flashlight, ascertain that all wires are at the bottom of the loader. Occasionally, a wire will not fall to the bottom and may hang up in the loader.

8.3.6. Slide the wire holder to the left after ascertaining that the furnace assembly is open. The furnace assembly must be open to prevent nitrogen pressure from blowing wires out of the holder.
8.3.7. Close the furnace assembly and proceed according to the instruction manual.
Note: Successful operation requires that both a purge and a measure pressure be approximately 12 psig (83 kPa) and that they be equal within 0.1 psig (0.7 kPa).
Note: Effective operation requires the maintenance of a fixed nitrogen purge rate of 0.8 to 2.0 L/min. To prevent blockage of the purge gas-exit orifice by particulates, the LECO RO-16 instrument is equipped with a paper filter in the line. This filter may become plugged and will require removal and replacement. The LECO instruction manual covers this maintenance step.
8.3.8. Record the readout.
8.3.9. Open the furnace assembly to relieve the nitrogen pressure when the determination is complete. Using a flashlight and a mirror, check up into the cavity to ascertain that no wires are hung up. If a wire section has hung up, remove and weigh it, and correct the wire weight.
8.3.10. Analyze a standard that will correspond to the level of oxygen in the wires after approximately every six determinations.

9. Calculation

9.1. Calculate the oxygen concentration in the vanadium wire.

$$C_{\underline{O}(V)} = \left(m_{O(V)F} - m_{O(V)I}\right) \cdot 10^2 \Big/ m_V \quad , \tag{11}$$

where:

$m_{\underline{O}(V)F}$ = oxygen content of wire, mg,
$m_{\underline{O}(V)I}$ = oxygen content of fusion blank, mg, and
m_V = weight of wire, mg.

9.1.1. Determine the oxygen concentration in sodium, in micrograms per gram, corresponding to the weight percent oxygen in the equilibrated vanadium wire by reference to figure 44. Since the V wire further purifies the sodium during equilibration, correction will be necessary to establish the true O concentration in the sodium before and after equilibration.
9.1.2. Figure 44 was prepared to establish the true O concentration in the sodium before equilibration, applicable to the equilibrium oxygen distribution between vanadium and sodium at 750 °C (1,382 °F):

$$M_{\underline{O}(V)} = C_{\underline{O}(V)} \cdot 10^{-2} \tag{12}$$

$$N_{\underline{O}(V)} = \frac{M_{\underline{O}(V)} / MW_O}{\left(1 - M_{\underline{O}(V)}\right) / MW_V + M_{\underline{O}(V)} / MW_O} \tag{13}$$

$$N_{\underline{O}(Na)} = N_{\underline{O}(V)} \exp\left[28.22 - 39.42\left(1 - N_{\underline{O}(V)}\right)^2\right] \tag{14}$$

$$M_{\underline{O}(Na)F} = \frac{N_{\underline{O}(Na)} MW_O / MW_{Na}}{1 + N_{\underline{O}(Na)}\left(MW_O / MW_{Na} - 1\right)} \tag{15}$$

$$M_{\underline{O}(Na)I} = M_{\underline{O}(Na)F} + M_{\underline{O}(V)}\left(\frac{\rho_V}{\rho_{Na}}\right)\left(\frac{d_V}{d_{Na}}\right)^2\left(\frac{L_V}{L_{Na}}\right) \quad (16)$$

$$C_{\underline{O}(Na)F} = M_{\underline{O}(Na)F} \cdot 10^6 \quad (17)$$

$$C_{\underline{O}(Na)I} = M_{\underline{O}(Na)I} \cdot 10^6 \quad (18)$$

where:

$C_{\underline{O}(V)}$ = concentration of oxygen dissolved in vanadium (wt%)
$M_{\underline{O}(V)}$ = mass fraction of oxygen dissolved in vanadium
MW_O = molecular weight of oxygen (15.9994)
MW_V = molecular weight of vanadium (50.9415)
$N_{\underline{O}(V)}$ = atom fraction of oxygen dissolved in vanadium
$N_{\underline{O}(Na)}$ = atom fraction of oxygen dissolved in sodium
MW_{Na} = molecular weight of sodium (22.98997)
$M_{\underline{O}(Na)F}$ = final mass fraction of oxygen dissolved in sodium
$M_{\underline{O}(Na)I}$ = initial mass fraction of oxygen dissolved in sodium
ρ_V = density of vanadium (6.1 g/cc)
ρ_{Na} = density of liquid sodium at 750 °C (7.727 g/cc)
d_V = diameter of vanadium wire (0.01 in)
d_{Na} = diameter of sodium in container (0.555 in)
L_V = length of vanadium wire in test volume
L_{Na} = length of sodium in container
$C_{\underline{O}(Na)F}$ = final concentration of oxygen in sodium (ppm)
$C_{\underline{O}(Na)I}$ = initial concentration of oxygen in sodium (ppm).

10. Precision and Accuracy

10.1. Precision—For the concentration range of 0.5 to 5 µg/g of oxygen in sodium, the relative standard deviation is expected to be within 10%.[8] For results in that concentration range, one laboratory reported relative standard deviations ranging from 1 to 7% for 10 sets of triplicate determinations made over a period of several months.

10.2. Accuracy—No standards are available for accuracy assessment. The oxygen analyzer is calibrated to eliminate bias in the measurement of oxygen contained in the vanadium wire.

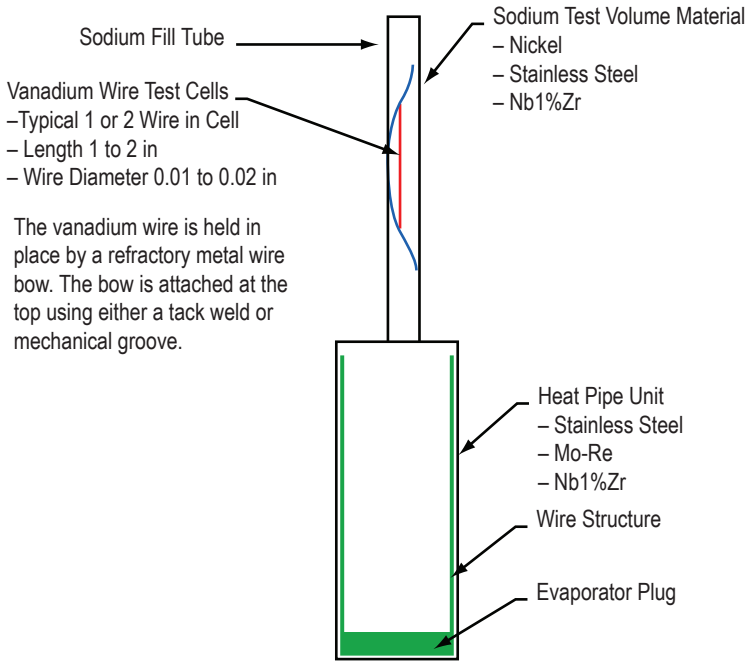

Figure 40. Schematic of an equilibration holder for use with heat pipe assemblies.

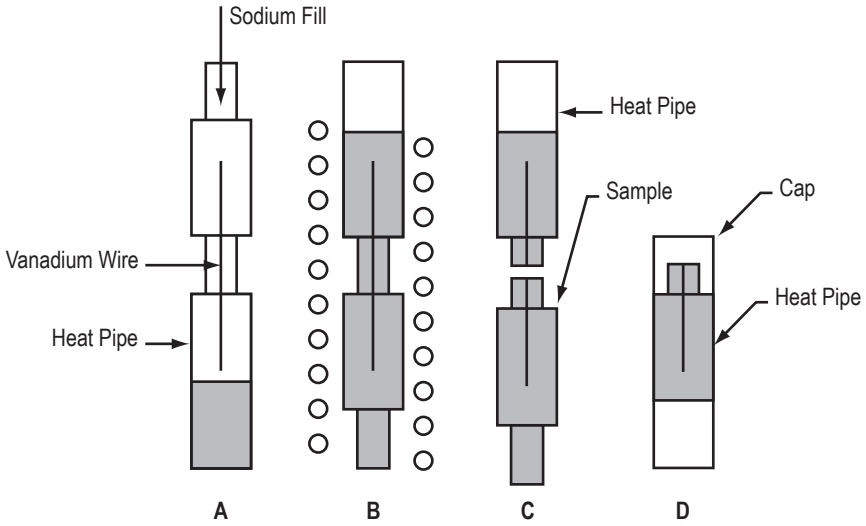

Figure 41. Method to measure oxygen concentration in Na-filled heat pipes.

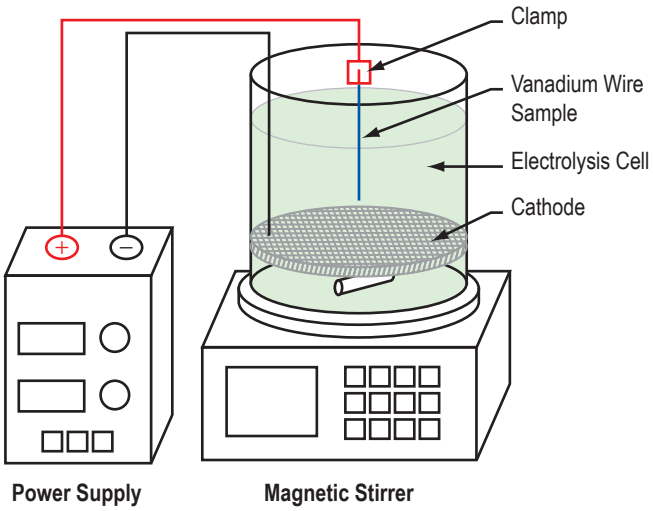

Figure 42. Typical electropolishing apparatus.

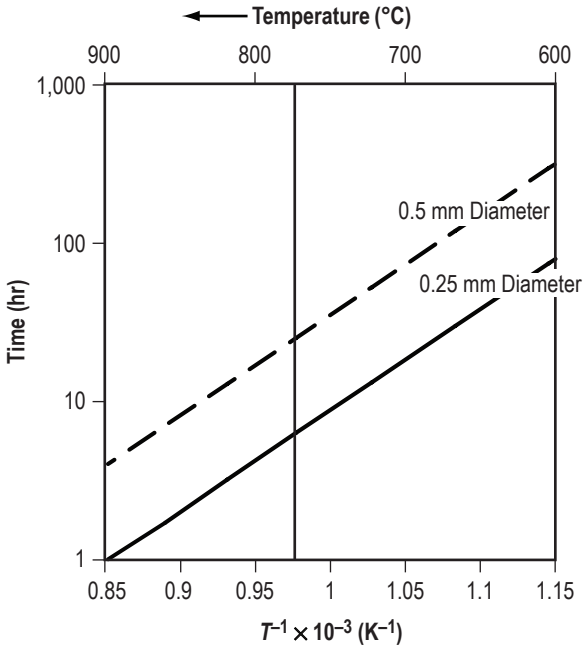

Figure 43. Estimated time required for equilibrium oxygen concentrations in static-sodium-immersed vanadium wires.

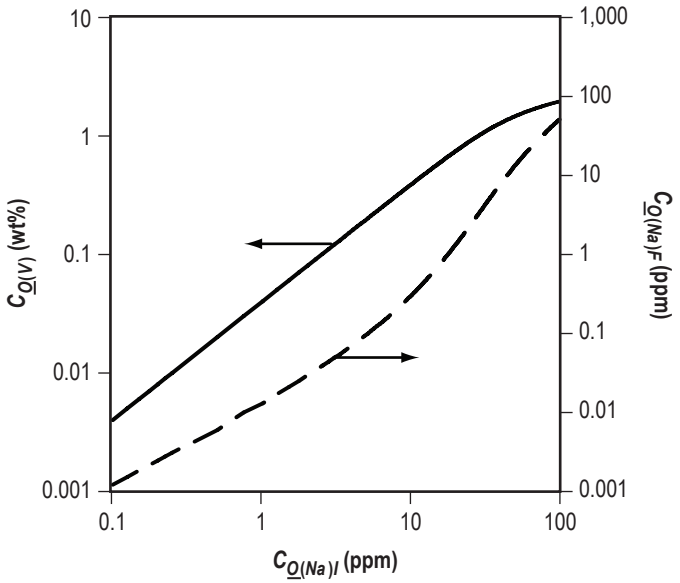

Figure 44. Corresponding equilibrium oxygen concentrations, vanadium versus Na at 750 °C (1,382 °F).

APPENDIX H—GENERAL HEAT PIPE FILL STEM PINCH METHOD CLOSEOUT PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____

Heat Pipe Identifier: _____

Operation Date: _____

H.1 General Notes

The following general notes apply:

(1) Verify that the fill machine glove box TIG welder is operational.

(2) Verify that the glove box hydraulic press is functional, with the correct steel die, and the Mo foil protection is in place.

(3) Check that the typical TIG setting for performing the fusion weld/cut off is _____ A.

(4) Check that the typical TIG setting for performing a seal weld over the fused end is _____ A.

(5) Verify that the heat pipe is well grounded to prevent TIG welder high RF output from interfering with normal glove box operations.

(6) Visual evidence of a yellow flame during welding operations indicates the presence of sodium in the weld zone.

(7) The cut-off portion of the fill stem contains the vanadium wire sample, to be analyzed to assess dispensed sodium purity. This portion must be kept and submitted for testing.

(8) A copy of this record shall be completed and placed in the heat pipe traveler.

H.2 Procedure for Fill Stem Pinch Method Closeout (TIG version)

The following pinch method procedure is used to close out the fill stem by means of TIG welding:

(1) Sodium must be settled out of the heat pipe fill stem, so place the heat pipe inside the tube shell heater, with the condenser end down.

(2) Place at least one thermocouple on the heat pipe fill stem.

(3) Turn on heaters and bring heat pipe temperature to 200 °C.

(4) Once the fill stem is at temperature, tap the fill stem tube to encourage sodium to flow out of the fill stem. Approximately 1 in of sodium will remain at the top of the fill stem, but this should not impact planned operations.

(5) While at temperature, tap periodically during a >5-min period.

(6) Turn off heaters and allow the heat pipe to cool.

(7) Record box atmosphere oxygen, ___ ppm, and water vapor, ___ dewpoint.

(8) If an isolation valve was used to seal the heat pipe fill stem, hook it up to the glove box high-vacuum system; otherwise, jump to step (11).

(9) Evacuate up to the heat pipe isolation valve by opening HOV H08 and watching HOV P04.

(10) When vacuum pressure is in the 10^{-7}-torr range, open heat pipe isolation valve and record pressure spike, _____ torr and the recovery time, _____ min.

(11) Position heat pipe with fill stem inside hydraulic ram dies; use either a ¾- or 1-in die assembly). Place center of the die ≈1 in from the end of the condenser plug.

(12) Apply pressure and flatten the fill stem tube; the die stops engage, yielding ≈0.002 to 0.005 in of plastic deformation.

(13) Release the press and use a dial caliper to verify the width of the pinched tube section at several locations; record: _____ in.

(14) Repeat pressing to achieve the goal by resetting the press die stops.

(15) Position the heat pipe for the most desirable fill stem angle for TIG welding.

(16) Fusion weld across the center of the flattened pinched portion of the fill stem, allowing the penetration to cut free the valve end of the stem and seal the heat pipe end.

(17) Note the TIG flame color _____ ; if the flame has a yellow tint, it is due to residual sodium in the fill stem.

(18) Inspect the fill stem fusion weld and follow up across the fused end with a subsequent seal weld to ensure good penetration and closure. Use the appropriate filler rod for Mo-Re alloy and make sure that a clean TIG flame is achieved—no sodium. The objective is to build up a sufficient nugget to at least match the material wall thickness.

(19) Record the box atmosphere oxygen, ___ ppm, and water vapor, ___ dewpoint.

(20) Let the heat pipe cool; it is now ready for the next operation.

(21) Remove the cut-off fill stem that contains the vanadium wire sample and submit it for purity processing.

H.3 General Notes or Significant Observations

APPENDIX I—GENERAL HEAT PIPE FILL STEM CAP METHOD CLOSEOUT PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____
Heat Pipe Identifier: _____
Operation Date: _____

I.1 General Notes

The following general notes apply:

(1) Check typical electron beam welder setting for performing the fusion weld: _____ energy, _____focus, _____current.

(2) Verify that bags are present both inside the glove box and outside the glove box to hold inert argon so that the heat pipe can be transported safely from the glove box to the electron beam welder.

(3) Verify that the alignment rig inside the electron beam welder is set to hold the heat pipe with the fill stem cap at the right height for welding. This is required to speed up the process of setting up the heat pipe in the welder, minimizing exposure of the heat pipe outside the glove box.

(4) During welding, the visual evidence of a yellow flame indicates the presence of sodium in the weld zone.

(5) The cut-off portion of the fill stem contains the vanadium wire sample (to be analyzed to assess dispensed sodium purity); this portion must be kept and submitted for testing.

(6) A copy of this record shall be completed and placed in the heat pipe traveler.

I.2 Procedure for Fill Stem Cap Method Closeout

The following cap method procedure is used to close out the fill stem by means of electron beam welding:

(1) Verify that sodium has been settled into the fill stem, forming a freeze plug, before proceeding.

(2) Record box atmosphere oxygen, ___ ppm, and water vapor, ___ dewpoint.

(3) Using a cleaned cutting tool, sever the heat pipe fill stem 0.2 in from the end of the condenser plug.

(4) Clean sodium away from the cut end of the fill stem.

(5) Place the fill stem cap over the severed fill stem and tap into place (it should be a snug fit). The cap should be flush with the end of the condenser plug when fully engaged.

(6) Place the heat pipe into a double bag that contains the glove box atmosphere.

(7) Transport the heat pipe from the glove box (out the air lock) to the electron beam welder.

(8) Keep the bag attached to the fill stem end and hook the evaporator end into the alignment rotation stage of the electron beam welder; verify that the fill stem cap is positioned at the correct height for welding.

(9) Remove the protective bags from the fill stem end.

(10) Rapidly close the electron beam welder housing and evacuate. Pump into the 10^{-6}-torr range and allow at least ____ hr to ensure the cap is sufficiently vented.

(11) Using the previously tested setting, perform a full penetration weld around the circumference of the heat pipe fill stem cap.

(12) Note if there is a change in color of the weld spot:_____. If a yellow tint is observed, it is caused by residual sodium in the fill stem.

(13) Let heat pipe cool under vacuum and then remove from the electron beam welder.

(14) Return heat pipe to the glove box.

(15) Remove the cut off fill stem that contains the vanadium wire sample and submit it for purity processing.

I.3 General Notes or Significant Observations

APPENDIX J—GENERAL HEAT PIPE FINAL CLOSEOUT AND SUPPORTS PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____
Heat Pipe Identifier: _____
Operation Date: _____

J.1 General Notes

The following general notes apply:

(1) Check the typical electron beam welder setting for performing the fusion weld: _____ energy, _____focus, _____current.

(2) Fit check all closeout tube sections and plug components to verify a snug fit, allowing for good alignment.

(3) Clean the heat pipe condenser plug end and closeout components, to eliminate high oxygen concerns in the weld zone.

(4) A copy of this record shall be completed and placed in the heat pipe traveler.

J.2 Procedure for Fill Stem Final Closeout

The following procedure is used for final closeout of the fill stem:

(1) Perform fit check of the closeout tube, transition plug, and support tube.

(2) Clean each component as necessary to achieve a successful weld.

(3) Tap the closeout tube in place over the condenser plug; this should be a snug fit.

(4) Check straightness with the appropriate fixture or tooling.

(5) Place the heat pipe in an electron beam welder and hook the evaporator end into the alignment rotation stage of the electron beam welder; verify that the closeout tube weld location is positioned at the correct height for welding.

(6) Close the electron beam welder housing and evacuate. Pump into the 10^{-6}-torr range and allow at least ____ hr.

(7) Using the previously tested setting, perform a full penetration weld around the circumference of the closeout tube, attaching it to the condenser plug.

(8) Let the heat pipe cool under vacuum and then remove it from the electron beam welder.

(9) Tap the transition plug in place into the closeout tube; this should be a snug fit.

(10) Check straightness with the appropriate fixture or tooling.

(11) Place the heat pipe in the electron beam welder and hook the evaporator end into the alignment rotation stage of the electron beam welder. Verify that the transition plug weld location is positioned at the correct height for welding.

(12) Close the electron beam welder housing and evacuate. Pump into the 10^{-6}-torr range and allow at least ____ hr to provide sufficient time for the volume inside the closeout tube to vent.

(13) Using the previously tested setting, perform a full penetration weld around the circumference of the transition plug, attaching it to the closeout tube.

(14) Let the heat pipe cool under vacuum and then remove it from the electron beam welder.

(15) Tap the support tube in place into the transition tube; this should be a snug fit.

(16) Check straightness with the appropriate fixture or tooling.

(17) Place the heat pipe in an electron beam welder and hook the evaporator end into the alignment rotation stage of the electron beam welder. Verify that the support tube weld location is positioned at the correct height for welding.

(18) Close the electron beam welder housing and evacuate. Pump into the 10^{-6}-torr range and allow at least ____ hr.

(19) Using the previously tested setting, perform a full penetration weld around the circumference of the support tube, attaching it to the transition plug.

(20) Let the heat pipe cool under vacuum and then remove it from the electron beam welder.

(21) Return the heat pipe to the glove box.

J.3 General Notes or Significant Observations

APPENDIX K—GENERAL HEAT PIPE WET-IN PROCEDURE

Note: The following outlined procedure is adapted from a procedure developed for processing stainless steel/sodium heat pipe modules using the MSFC alkali metal handling glove box. These procedures shall be modified as required during checkout evaluation for the current project.

Operators: _____
Heat Pipe Identifier: ____
Operation Date: _____

K.1 General Notes

The following general notes apply:

(1) The vacuum furnace should be very clean—cleaned by means of turbo pumps—and should be baked out at 200 to 250 °C during evacuation to remove all water vapor and other condensable materials.

(2) Heat pipe units shall typically be wet-in one to three at a time.

(3) Prior to wet-in, all external surfaces shall be cleaned by use of an approved procedure to remove any contamination that would prove detrimental to the heat pipe at high temperature. During all handling, the operator shall wear approved gloves to prevent additional contamination.

(4) The furnace should have sufficient equipment to record temperature and pressure during the operation.

(5) A copy of this record shall be completed and placed in the heat pipe traveler.

K.2 Procedure for Module Wet-In

The following procedure is used for module wet-in:

(1) Clean the surface of all heat pipes prior to placement in the vacuum furnace.

(2) Place heat pipes into the furnace and support at the ends using Mo-based material structures.

(3) Begin recording time, temperature, and pressure history for the vacuum furnace.

(4) Evacuate the system into the 10^{-6}-torr range or lower.

(5) Turn on the surface heaters and bake out the furnace chamber at approximately 200 to 250 °C. Allow the pressure to fall back into the 10^{-6}-torr range before proceeding.

(6) Set the furnace to ramp the temperature up to a temperature 50 to 100 °C over maxiumum operating temperature over a period of 2 hr.

(7) Hold the temperature on the module for ____ hr (24 to 48 hr range); record the average vacuum pressure, _____ torr, while the furnace is at temperature.

(8) After the hold period has expired, the furnace shall be shut off and allowed to cool naturally to room temperature; this will take 3 hr or more.

(9) Make note of any abnormalities during the wet-in process; i.e., pressure/temperature spikes, power loss, etc.

(10) Remove the heat pipe units from the furnace and inspect for damage.

(11) Record the final weight and place the units in transport containers.

K.3 General Notes or Significant Observations

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14. ABSTRACT

Refractory metal heat pipes developed during this project shall be subjected to various operating conditions to evaluate life-limiting corrosion factors. To accomplish this objective, various parameters shall be investigated, including the effect of temperature and mass fluence on long-term corrosion rate. The test series will begin with a performance test of one module to evaluate its performance and to establish the temperature and power settings for the remaining modules. The performance test will be followed by round-the-clock testing of 16 heat pipes. All heat pipes shall be nondestructively inspected at 6-month intervals. At longer intervals, specific modules will be destructively evaluated. Both the nondestructive and destructive evaluations shall be coordinated with Los Alamos National Laboratory. During the processing, setup, and testing of the heat pipes, standard operating procedures shall be developed. Initial procedures are listed here and, as hardware is developed, will be updated, incorporating findings and lessons learned.

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alkali metals, sodium, heat pipes, refractory metals, molybdenum, rhenium, radiofrequency heating, fission power system, life testing

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