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LOW COST HIGH PERFORMANCE GENERATOR TECHNOLOGY PROGRAM

LCHPG Heat Pipe Topical
Appendices
July 1975

PREPARED FOR NUCLEAR RESEARCH AND APPLICATIONS DIVISION, ENERGY RESEARCH AND DEVELOPMENT
ADMINISTRATION UNDER ERDA CONTRACT E(49-15) -3075 (FORMERLY SNSO-3)

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APPENDIX A

Technical Summary Report for Development, Fabrication and Testing
of Heat Pipes for High Performance Generator, Dynatherm Corporation,
DTM-73-9, August 31, 1973.

DTM-73-9

TECHNICAL SUMMARY REPORT

for

DEVELOPMENT, FABRICATION AND
TESTING OF HEAT PIPES FOR
HIGH PERFORMANCE GENERATOR

August 31, 1973

Prepared under Purchase Order B-64931

for

Teledyne Isotopes, Inc.
Energy Systems Division

by

Dynatherm Corporation
Cockeysville, Maryland

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1. INTRODUCTION

This report describes the work performed by Dynatherm Corporation for Teledyne Isotopes during a program entitled "Heat Pipe Fabrication, Associated Technical Support and Reporting." The program was initiated on November 29, 1972; the main objectives were accomplished with the delivery of the heat pipes for the HPG. Life testing of selected heat pipe specimens is continuing to and beyond the present date.

The program consisted of the following tasks, each of which will be described in more detail in subsequent sections of this report:

Task I. Heat Pipe Development of Process Definition

Under this task the HPG oriented technology of Cu-H₂O which was developed under a previous program* was further refined. The necessary fabrication processes were developed, and fabrication and test procedures were generated.

Task II. Prototype Heat Pipes for Fin Segment Test

Eight (8) prototype heat pipes which were fabricated in accordance to the specifications and procedures developed under Task I were delivered to Isotopes. These heat pipes were then installed by Isotopes into a mock-up of one HPG fin and further tested on a subassembly level.

Task III. HPG Heat Pipe Fabrication and Testing

Fifty-six (56) heat pipes of the same specifications were delivered under this task. Each heat pipe was fabricated and tested under a rigid Q/A

*LES 8/9 Integration Study, Teledyne Isotopes Contract with Dynatherm Corporation under Purchase Order Number B-64592

program and in accordance with the previously developed procedures. A complete data package was delivered with every heat pipe.

Task IV. Controlled Heat Pipe Life Test

This task was an extension of a previous effort under the LES 8/9 Integration Study. Two (2) sample heat pipes were attached to a representative section of an HPG fin, performance mapped, and then placed on life test. Because of some technical difficulties in the early phases of the program, the two life test samples are of slightly different design and were placed on test at different times. The performance to date, however, of both samples is nearly identical. Also, as part of this task, four (4) additional heat pipes are being life tested. Although not covered by the present purchase order, these life tests are being continued since they provide valuable information about the long-term performance of such heat pipes at various operating temperatures.

Task V. Heat Pipe Film Coefficient Determination

Under this task a specimen configured to duplicate the basic heat pipe parameters was to be designed, fabricated, and tested to obtain film coefficient test data. Because more applicable engineering data on the evaporator and condenser heat transfer became available from the testing of the prototype heat pipes, the experimental portion of this task was deleted and the test program of Tasks 2 and 3 expanded in order to generate these data.

2. DEVELOPMENT AND PROCESS DEFINITION (TASK I)

The heat pipe specifications were defined by Isotopes Document HPGA 2180500 Rev. C and Isotopes Interface Drawing HPGA 2180000 Rev. C. The heat pipes must be capable of transporting 60 watts minimum, applied over approximately two (2) inches near one end and removed from the remainder of the heat pipe. This performance requirement applies over the temperature range from 150 to 350⁰F. When operated with a heat input of 60 watts, the overall temperature difference must not exceed 24⁰F which corresponds to a heat pipe resistance of 0.4⁰F/watt.

During the LES 8/9 Integration Study, a basic Cu-H₂O heat pipe design was developed which met all performance specifications for the HPG heat pipes. This design is shown in Figures 1 and 2. It consists of a 1/4-inch diameter tube bent to an "L" shape for optimum interfacing with both the generator housing and the radiator fins. The heat pipe wall thickness is .022 inch. This allows for circumferential grooving of the tube wall with standard tooling. A total of eighty (80) grooves per inch, each approximately .005 inch deep, are used. These grooves provide the necessary flow paths from the main wick to the heat transfer surface on the circumference of the tube. To take maximum advantage of the high film coefficients, the main wick is located on the side opposite the heat input/output areas of the heat pipe. These areas are interfaced directly with the generator housing and fins.

The main wick consists of a 100-30-100 mesh screen composite slab held against the wall with a .020-inch diameter beryllium-copper spring. The spring serves to maintain the wick away from the heat input/output areas and to retain the wick in position in dynamic environments. In order to provide proper surface wetting with water, both the internal surface of the tube and the main wick are chemically treated with a surface

FIGURE 1
LES 8-9 HEAT PIPE DESIGN

Detail 'A'
Fill Tube Detail

Detail 'B'
End Cap Detail

FIGURE 2
FILL TUBE AND END CAP DETAIL

coating which has been successfully demonstrated in prior applications.

Breadboard models based on the above design were fabricated and tested -- both as individual components and after soldering the heat pipe to a representative section of the HPG fin. Test results obtained with the heat pipe/fin assembly were reported in an earlier report.* The measured overall thermal resistance ranged from 0.2 to 0.25^oF/watt at a nominal heat input of 60 watts and for evaporator elevations of up to 1 inch. This result compares favorably with the specified maximum resistance of 0.4^oF/watt. The maximum heat transport capability was found to be approximately 90 watts in the adverse -1 inch orientation and in excess of 120 watts in the simulated "zero-g" condition. Thus, the heat pipe design met the performance specifications and provided a comfortable performance margin.

The early phases of this program consisted of an extensive effort to develop the processes and techniques required for fabricating and testing production lots of these heat pipes. Although the design met the performance specifications, difficulties were encountered in reproducing the results. The following major problem areas were identified:

- During an initial bake-out cycle at 350^oF, substantial quantities of noncondensing gas were generated. This was expected and was the purpose for conducting the cycle. But it proved to be difficult to remove this gas without losing too much working fluid. Only 1.6 grams of water are used in this design, and the performance is sensitive to underfill and overfill.

*Dynatherm Report DTM-73-4, "Addendum to Final Report for HPG/LES 8-9 Integration Study," March 1973

- The Ebonol surface coating, which is used to enhance wetting, was destroyed near the ends of the heat pipe due to excessive heating during welding of the end cap and fill tube. This was particularly detrimental at the evaporator end where good wetting is required to achieve good performance.
- The test fixture, which was developed for rapid acceptance testing of many heat pipes, introduced a thermal impedance in the measured data approximately equal to the impedance of the heat pipe. The problem was attributed to the location of the thermocouples which were attached to the fixture rather than the heat pipe in order to permit rapid exchangeability of pipes during testing of production lots.

The above difficulties led to a complete reevaluation of the heat pipe design from a point of reproducible fabrication and ease of processing and testing. The revised design is shown in Figures 3 and 4. The wick structure was changed from a composite* slab consisting of 100-30-100 mesh screen to a homogeneous* slab fabricated only from 30 mesh screen. Although the homogeneous slab does not have the same ultimate performance capability as a composite slab, its performance is adequate for this application, much more reproducible, and less dependent on a perfect surface oxide layer for good wetting. The end cap at the evaporator was modified to include a small diameter tubular extension (see Figure 4). This permitted the welding of the end cap to the heat pipe tube before applying the surface treatment. The final weld closure of the small extension

* For a discussion on homogeneous and composite wicks, see "Heat Pipe Design Handbook" prepared by Dynatherm Corporation for NASA-MSC, August 1972

FIGURE 3
HPG HEAT PIPE DESIGN

FIGURE 4
HPG FILL TUBE AND END CAP DETAIL

occurs far enough away from the end of the wick that the surface coating remains intact.

A complete reevaluation of the bake-out and charging procedure was performed. It was recognized that the outgassing temperature should be increased to at least 400^oF in order to be commensurate with the soldering temperature of the heat pipes into the HPG fin. Obviously, this temperature increase would result in even more gas generation during this bake-out cycle. Thus, a charging, bake-out, and pinch-off procedure had to be devised which would allow for bleeding of the generated gas without excessive loss of working fluid. After considerable development effort, a pinch tube design along with the proper procedures evolved which accomplished the above objectives. In Figure 4, the final design of the fill tube is shown which consists of a long and small diameter tube which can be used for several pinches and does not trap any liquid. The exact procedures for charging, baking, and pinch-off are listed elsewhere in this report. Basically, the heat pipe which is equipped with the long fill tube is charged under atmospheric conditions. It is charged with an amount of fluid, which is slightly in excess of the desired charge, using a hypodermic needle and a syringe. It is then heated to the boiling point of the fluid until steam emerges from the fill tube. At this point, it is pinched and welded at the "long" end of the fill tube. Subsequently, the heat pipe is subjected to the 400^oF bake-out cycle. After releasing the gas, a second pinch-off is performed using basically the same technique as during the first pinch-off. This method led to virtually gas-free heat pipes in which the final fluid charge could be controlled to within ± 0.05 gram.

With regard to the test fixture, extensive modifications were also made. This consisted of spring-loading thermocouples directly against the heat pipe as opposed to attaching them to the fixture. This modification still permitted rapid exchange of pipes during the test phase without biasing the results by the temperature of the fixture. The

test stand is shown schematically in Figure 5. A total of 8 thermocouples -- 3 along the evaporator, 1 in the adiabatic section, and 4 along the condenser -- are used. This distribution of TC's permitted a separate measurement of evaporator and condenser impedances. Heat is applied and removed from only one side of the heat pipe which corresponds to the environment of the HPG. Thus, the revised test fixture permitted reasonably accurate measurements of the heat pipe's performance under test conditions which simulated the installation into the HPG.

Part of the development phase was also a weld qualification program. The original design was based on type 110 copper for the end cap and fill tube hardware. Although the closures were helium leak-tight as tested up to 600 psia, some porosity was noted in microphotographs. Consequently, the basic material was changed to type 102 oxygen-free copper. The resulting welds were considerably less porous and were judged acceptable for this application. Following this qualification effort, a detailed welding procedure was written and released.

Finally, a qualification and acceptance test procedure was generated, reviewed, and released. The major steps of the acceptance test procedure are listed in Table I; those of the qualification test procedure are listed in Table II. The qualification testing was conducted on two heat pipes which were also first subjected to acceptance testing. All delivered heat pipes (prototypes and HPG hardware) underwent the acceptance testing.

Several breadboard heat pipes of the revised design were processed and tested according to the finalized procedures. After sufficient confidence had been obtained that an acceptable design and reproducible process had been developed, the fabrication of the eight (8) deliverable prototypes was initiated.

Step	Procedure	Acceptance Criteria
1. Helium Leak Test	Evacuate and spray with Helium	Leak rate less than 10^{-8} STD cc/sec
2. Proof Pressure Test	Pressurize with Argon	No burst
3. Leak Test of Final Closure Weld	Select one heat pipe from each lot, open and subject closure weld to Steps 1 and 2	Leak rate less than 10^{-8} STD cc/sec, No burst
4. Noncondens-ible Gas Test	Install in test fixture with condenser elevated. Record temperature gradients at $\sim 150^{\circ}\text{F}$	None
5. Functional Performance Test	Test in fixture at 150 and 350°F with evaporator elevated by 1 inch and with 60 watts input	Overall conductance at 150 and 350°F greater than 2.5 Watts/ $^{\circ}\text{F}$ with 60 watts input

TABLE I
HIGHLIGHTS OF ACCEPTANCE TEST PROCEDURE

Step	Procedure	Acceptance Criteria
1. Thermal Cycling	Heat in oven to 500°F for 30 minutes. Perform noncondensable gas test.	None
2. Thermal Performance Mapping	Attach bent heat pipes to housings/fin section. Test to burn-out at 150 and 350°F and at evaporator elevations of 0, 1, and 2 inches.	Overall conductance at 150 and 350°F and at 0 and 1 inch elevation greater than 2.5 Watts/°F.
3. Recovery Test	Operate with 30 watts at 1/8 inch evaporator elevation. Burn-out by raising evaporator to 3 inches for 3 minutes. Return to 1/8 inch elevation at 150 and 350°F.	None
4. Life Testing	Operate continuously with 50 watts input at 315°F and with condenser elevated by 1 inch. Record data every 2 weeks. Record data with evaporator elevated by 1 inch every month.	10,000 hour life without catastrophic failure and with conductance remaining greater than 2.5 Watts/°F.

Note: Heat pipes used for qualification testing must first pass all acceptance tests.

TABLE II
HIGHLIGHTS OF QUALIFICATION TEST PROCEDURE

FIGURE 5

PROTOTYPE MODEL ASSEMBLY AND TEST SETUP

3. PROTOTYPE HEAT PIPES FOR HPG FIN SEGMENT (TASK II)

The contract called for delivery of 8 prototype heat pipes to be fabricated and tested in accordance with the procedures generated under Task I. These heat pipes were intended for installation into a mock-up of a single HPG fin and housing section. A lot of 13 heat pipes was fabricated. All pipes met or exceeded all performance specifications. Eight (8) pipes were selected from the group and delivered to Teledyne Isotopes. The pertinent performance parameters of the delivered pipes are given in Table III. The last column in that table lists the average values and the standard deviation of the measured parameters.

Serial No.	Overall Resistance 150°F (°F/Watt)	Overall Resistance 300°F (°F/Watt)	Noncondensable Gas Test (°F)	Fluid Charge (grams)	Shipping Weight (grams)
01	.213	.180	2.0	1.595	34.592
02	.232	.227	2.3	1.579	33.677
03	.198	.212	1.4	1.610	34.168
05	.183	.183	2.3	1.606	34.342
06	.213	.234	2.4	1.608	34.097
07	.188	.211	2.3	1.610	35.508
10	.194	.187	2.0	1.621	34.441
13	.214	.158	2.0	1.613	34.613
Average	.204	.199	2.1	1.605	34.430
Deviation	.016	.026	0.3	0.184	0.531

TABLE III
DATA SUMMARY FOR LOT 1A
DELIVERED PROTOTYPE HEAT PIPES

4. HPG HEAT PIPES FABRICATION AND TESTING (TASK III)

In order to meet the required delivery of 56 heat pipes for the HPG generator (48 to be installed plus 8 spares) a total of 80 heat pipes were processed in three lots. The relatively large number of spares was necessitated by a problem occurring during the bending operation. Some of the heat pipe tubes had not been sufficiently annealed and did not permit bending into the final shape. Thus, most of the excess heat pipes were not spares, in the true sense, but replacements for pipes with the wrong temper. Of the 73 heat pipes processed, only 8 failed to meet the performance criteria. The pertinent parameters of the delivered heat pipes are given in Table IV. Again, the last column lists the average values and the standard deviation of the measured parameters.

Five (5) heat pipes -- two from each of the first two production lots and one from the last production lot -- were destructively tested in order to qualify the final closure weld. All 5 pipes met the acceptance criteria.

Serial No.	Overall Resistance 150°F (°F/Watt)	Overall Resistance 300°F (°F/Watt)	Noncondensable Gas Test (°F)	Fluid Charge (grams)	Shipping Weight (grams)
01	.297	.202	2.0	1.585	34.244
02	.270	.240	2.3	1.596	35.260
03	.205	.187	2.3	1.587	33.962
04	.263	.215	2.3	1.621	35.333
05	.221	.157	1.3	1.572	34.026
06	.207	.225	1.7	1.575	36.180
08	.273	.256	1.7	1.574	35.657
11	.297	.214	2.0	1.627	34.871
12	.360	.214	2.3	1.589	33.874
14	.305	.240	1.7	1.597	34.783
16	.218	.212	2.0	1.580	35.374
17	.247	.203	6.3	1.604	35.489
18	.268	.209	1.4	1.586	34.116
19	.298	.275	2.0	1.597	35.836
22	.190	.231	1.3	1.596	34.116
24	.168	.207	1.3	1.589	35.749
25	.234	.202	1.7	1.572	35.076
27	.177	.175	1.3	1.608	34.469
28	.180	.182	2.4	1.612	34.737
30	.283	.245	1.7	1.604	33.955
33	.193	.193	1.7	1.611	34.953
34	.305	.188	9.5	1.612	33.553
35	.198	.178	2.0	1.556	34.265
Average	.246	.211	2.4	1.593	34.777
Deviation	.052	.028	1.8	0.018	0.741

TABLE IV
DATA SUMMARY FOR LOT 2A
DELIVERED HEAT PIPES

Serial No.	Overall Resistance 150°F (°F/Watt)	Overall Resistance 300°F (°F/Watt)	Noncondensable Gas Test (°F)	Fluid Charge (grams)	Shipping Weight (grams)
01	.277	.185	11.6	1.538	33.674
02	.330	.233	17.0	1.607	34.355
03	.247	.195	4.7	1.605	34.281
04	.264	.253	4.0	1.604	34.589
05	.363	.304	12.4	1.607	34.384
06	.277	.266	2.0	1.603	35.529
07	.251	.225	2.0	1.606	36.061
08	.170	.193	1.7	1.587	35.404
12	.247	.220	1.0	1.605	35.714
13	.213	.182	10.3	1.503	35.163
14	.258	.228	2.0	2.593	35.593
16	.200	.206	3.0	1.608	35.068
18	.167	.191	1.7	1.586	37.112
20	.231	.229	3.3	1.610	36.196
21	.228	.267	6.0	1.606	36.549
22	.197	.208	0.0	1.596	36.336
24	.204	.170	1.3	1.592	36.591
Average	.243	.221	4.9	1.592	35.447
Deviation	.052	.059	4.7	0.029	0.964

TABLE IV

DATA SUMMARY FOR LOT 3A
DELIVERED HEAT PIPES

Serial No.	Overall Resistance 150°F (°F/Watt)	Overall Resistance 300°F (°F/Watt)	Noncondensable Gas Test (°F)	Fluid Charge (grams)	Shipping Weight (grams)
01	.188	.165	1.3	1.605	32.988
02	.217	.173	7.0	1.565	34.186
03	.178	.151	1.7	1.589	33.657
04	.185	.142	2.3	1.601	32.838
05	.187	.153	2.7	1.603	33.364
06	.165	.160	1.7	1.595	33.351
07	.170	.131	3.0	1.589	33.977
08	.217	.155	11.8	1.559	33.728
11	.212	.165	7.3	1.608	32.279
12	.208	.157	2.7	1.602	32.712
13	.202	.137	2.3	1.600	34.694
14	.193	.177	1.3	1.543	33.435
15	.183	.140	5.0	1.604	32.902
16	.183	.150	2.4	1.603	33.711
17	.185	.138	0.6	1.602	33.952
18	.195	.140	4.0	1.608	35.843
Average	.192	.152	3.6	1.592	33.601
Deviation	.016	.014	2.9	0.019	0.859

TABLE IV
DATA SUMMARY FOR LOT 4A
DELIVERED HEAT PIPES

5. CONTROLLED HEAT PIPE LIFE TESTING (TASK IV)

The effort under this task consisted not only of life testing but included also the qualification test program. The latter was conducted under this task because it required that the heat pipes be soldered into a simulated fin/housing section. Two such assemblies were fabricated and tested. The design of both test assemblies is shown in Figure 5 and the instrumentation in Figure 6. The first one was assembled and tested with a heat pipe of the original LES 8/9 design (see Section 2). The results of the performance mapping on this first assembly were reported in DTM 73-4.* The second assembly was equipped with a heat pipe from the prototype lot, and it was completely qualification tested in accordance with the finalized test procedure.

The results of the performance mapping of this second assembly are shown in Figures 7 and 8. In these figures, the overall thermal resistance is plotted against input power for three evaporator elevations of 0, 1, and 2 inches. It is seen that, at 0 and 1 inch elevation and at the design heat load of 60 watts, the resistance is less than $0.25^{\circ}\text{F/watt}$ which compares favorably to the specification requirement of 0.4°F/watt . The results of the recovery tests are shown in Figure 9. The purpose of the recovery tests was to evaluate the performance of the heat pipe after a burn-out has been induced, such as might occur in a dynamic environment. The tests involved operating the pipe at various input power levels, inducing a forced burn-out by elevating the evaporator by 3 inches with the power applied, and then monitoring the recovery. The data prior to the forced burn-out compare closely with those obtained during performance mapping (see Figures 7 and 8). At the 150°F level, the recovery after forced burn-out appears to be not complete at inputs exceeding 60 watts. At the 350°F level, complete recovery is indicated

*Dynatherm Report DTM-73-4, "Addendum to Final Report for HPG/LES 8-9 Integration Study," March 1973

FIGURE 6
HEAT PIPE PERFORMANCE TEST SETUP

FIGURE 7
OVERALL THERMAL RESISTANCE OF HPG HEAT PIPE FIN SN1A12 at 150°F

FIGURE 8
OVERALL THERMAL RESISTANCE OF HPG HEAT PIPE FIN SN1A12 AT 350°F

FIGURE 9
THERMAL RECOVERY PERFORMANCE OF HEAT PIPE SN1A12

up to 90 watts.

After performance mapping and recovery testing the second assembly was placed on life test. At that time, the first assembly had already accumulated 3600 hours. However, during that time period, only temperature gradients along the pipe were measured while it was oriented in a slight reflux mode. Since the second unit was placed on life test, both units are being operated and data recorded in accordance with the final test procedure. These data are shown in Figures 10 and 11. After 1700 and 5300 hours respectively, both units are still well within specifications and no trend of degradation is detectable.

In order to obtain more information about the long-term stability of Cu-H₂O heat pipes at temperatures which are representative of RTG radiators, four additional heat pipes were placed on life test. This effort was not included in the Statement of Work of this program. It was done to advance the state-of-the-art of Cu-H₂O heat pipe technology and to increase the confidence level in the chosen design. The four heat pipes are of the HPG design but in the straight configuration. They are being operated in the reflux mode; i. e., a resistance heater was wrapped around the evaporator end and heat is rejected by natural convection to air while operating vertically with the evaporator end at the bottom. The operating temperatures are 300, 350, 380, and 400°F, respectively. Initially, one of the pipes was run at 450°F; but, because of the rapid gas generation at that temperature, this particular test was terminated after 168 hours and the operating temperature of this pipe readjusted to 380°F. To date, these refluxers have accumulated 4658 hours and the testing is being continued.

A new technique was developed for determining the amount of noncondensable gas generated during life testing. Periodically, the life test heat pipes are turned off; i. e., allowed to cool to room temperature. After complete thermal equilibrium at room

FIGURE 10
HPG LIFE TEST OF HEAT PIPE FIN SN1A12

FIGURE 11
HPG LIFE TEST OF HEAT PIPE FIN SN4

temperature has been achieved heat is again applied. During the transient heat-up cycle, temperatures are being recorded from the thermocouples shown in Figure 12. The differential thermocouple near the condenser end registers the passage of the vapor-gas interface between its two junctions. Whenever gas is present it will be compressed at the end of the condenser; the location of the interface being only a function of the vapor pressure and of the amount of gas. During heat-up, which is accompanied by rising vapor pressure, this interface moves gradually toward the condenser end. When it passes between the junctions of the differential thermocouple its output reaches a peak. The additional thermocouple in the adiabatic section of the heat pipe registers the vapor temperature at the time the differential thermocouple records its peak output. Typical transient temperatures of such a heat-up cycle are shown in Figure 13. Since the location of the differential thermocouple remains fixed, the vapor temperature associated with the peak of the differential output is a measure of the amount of noncondensable gas present. This amount can then be determined with the following assumptions:

- At the time the peak occurs, the interface between vapor and gas is located halfway between the junctions of the differential thermocouple.
- The inactive end of the condenser is at room temperature.
- The interface is sharp; i. e. , only gas is present beyond the interface.
- The gas obeys the ideal gas law.
- The pressure of the gas is equal to the vapor pressure of the working fluid as determined by the vapor temperature.

FIGURE 12
TEST SETUP AND INSTRUMENTATION OF

FIGURE 13
TYPICAL RECORDED VOLTAGE PROFILE DURING GAS TEST

With the above assumptions, the amount of gas which is present during a particular cycle can be calculated from the following equation which is a form of the ideal gas law:

$$m = \frac{p_v(T) A_c L}{R T_o} \quad (1)$$

In this equation, $p_v(T)$ is the vapor pressure of the working fluid at the temperature at which the peak occurs. A_c is the cross-sectional area of the vapor space in the condenser, L is the distance between the center of the differential thermocouple and the end of the heat pipe, R is the gas constant of the noncondensable gas (always hydrogen), and T_o denotes room temperature.

The advantage of this new technique is twofold. Firstly, it yields a quantitative value for the amount of noncondensing gas without having to make a judgement as to the location of the interface which is always required when steady-state measurements, with fixed location thermocouples, are performed. Secondly, the inactive portion of the condenser may be safely assumed to be at room temperature, since data are taken during a transient heat-up, while the inactive section of the condenser is not yet heated. With the conventional steady-state technique, the effective temperature of the noncondensing gas (and the associated partial pressure of the working fluid) has to be determined numerically and is subject to considerable error.

The differential transient technique has been applied to the four Cu-H₂O refluxers operating at various temperatures. The results are shown in Figures 14 and 15. In Figure 14, the absolute amount of gas is plotted as a function of operating time. In Figure 15, the corresponding calculated inactive condenser lengths at each of the four operating temperatures are shown. The data indicate that at 300°F no gas is being generated.

FIGURE 14
HEAT PIPE GAS GENERATION TEST

FIGURE 15
INACTIVE CONDENSER LENGTH AT OPERATING TEMPERATURE AFTER t HOURS

The refluxer with the nominal test temperature of 350°F did not generate any gas during the first 2500 hours of operation. But it was found that the actual test temperature had only been 325°F during that period. When the temperature was readjusted to 350°F, gas generation started with some recent sign of leveling off. The other two heat pipes, which are being tested at 380 and 400°F respectively, generated much gas during an initial period. But within the last 2000 hours they appeared to have reached an equilibrium state.

It must be emphasized that these results apply only to a copper-water system in which all surfaces are oxidized with Ebonol to achieve good wetting. With this in mind, several conclusions can be drawn.

- No gas is generated at operating temperatures below approximately 325°F.
- At higher temperatures, gas is generated but at a rate which is diminishing with time.

Much additional work needs to be done to resolve questions, such as: (1) what is the optimal surface treatment; (2) what is the precise threshold temperature for gas generation; (3) does the gas generation really level off; and (4) would gas generation resume if the gas were removed after an initial burn-in period. Some of the work required to resolve these questions is recommended as a follow-on effort in Section 8 of this report.

6. HEAT PIPE FILM COEFFICIENT DETERMINATION (TASK V)

The determination of the effective heat transfer (film) coefficients at evaporator and condenser is necessary for the thermal design of any heat pipe application. Experimental data for several wick designs are available. However, the effective heat transfer coefficients depend strongly on the wick design, wick and wall material, working fluid, heat pipe geometry, and interfacing with the heat source and heat sink. Thus, extrapolation of existing data from other heat pipes to the HPG application would, at best, be marginal. For this reason, a test was planned in which the coefficients for this particular design were to be measured. This test was to be designed and conducted in such a manner that "true" evaporator film coefficients would have been measured. A "true" film coefficient in this context is defined as the heat transfer coefficient associated with the evaporation or condensation process in the heat pipe. Mathematically, it is expressed as follows:

$$Q = h A \Delta T \quad (2)$$

Q is the total heat throughput, h the film coefficient, A the evaporator or condenser surface area measured on the inside of the heat pipe, and ΔT the difference between the inside wall temperature and the saturation vapor temperature of the fluid.

Although the true film coefficient is of considerable theoretical interest, its use in design calculations is often difficult. Frequently, the actual evaporator area is not precisely known and temperature drops through the heat pipe wall and interfaces have to be included. This situation is particularly true for the HPG design where the wick is located eccentrically and heat is applied over one-half of the circumference only. A more meaningful parameter is the effective heat transfer coefficient which is defined as in Equation 2 but where A is based on the external evaporator area and where ΔT is the

difference between the heat pipe's outside wall temperature and the vapor temperature.

The effective heat transfer coefficients can best be measured on an actual heat pipe of the reference design and with heat applied and removed in a manner which is representative for an HPG application. Thus, the actual test data from the prototype heat pipes have been analyzed and the results are shown in Table V. The effective heat transfer coefficients given in that table are based on total outside evaporator and condenser area and are representative for an installation in which heat is applied to and removed from one-half of the circumference. These data can be used for design calculations for similar applications and designs.

		Evaporator	Condenser
Effective Area (Ft ²)		.011	.048
Average Measured Resistance (°F/Watt)	150°F	.149	.056
	350°F	.156	.043
Effective Heat Transfer Coefficient (BTU/Hr-Ft ² -°F)	150°F	2080	1269
	350°F	1987	1652

TABLE V
EFFECTIVE EVAPORATOR AND CONDENSER
HEAT TRANSFER COEFFICIENTS

7. LISTING OF DRAWINGS AND PROCEDURES

The following drawings and procedures were generated under this program.

Copies of these documents are retained in Dynatherm's Engineering File.

- Procedures

001-1000	Ebonol-C Treatment for Copper
074-1010	Welding Procedure
074-1011	Charging and Pinch-Off Procedure
074-1012	Tinning Procedure
074-1013	Acceptance and Qualification Test Specification
DTM-M-100 (Rev. B)	Quality Assurance and Inspection Procedure

- Drawings

074-1000	Heat Pipe Assembly
074-1001	Heat Pipe Tubing
074-1002	Fill Tube
074-1003	End Cap
074-1004	Wick
074-1005	Spring, Retainer
074-1007	Straight Pipe Assembly
074-1015	Bending Fixture

8. RECOMMENDED FOLLOW-ON EFFORT

During this program, a heat pipe configuration was developed which meets all basic performance objectives. In addition, processes and procedures were developed for the fabrication of prototype hardware. Application of this hardware to the high reliability requirements of spaceflight systems will require rigid qualification of the fabrication, processing, and testing procedures, and also the establishment of consistent performance characteristics of the design.

During the fabrication and testing of the prototypes, some performance inconsistencies were noted. These were primarily attributed to variations in the surface wetting treatment which occurred because of flow passage restriction during chemical treatment. In addition, temperature cycling during the soldering operation is suspected to detrimentally affect ultimate performance. Process modification and control will be required to develop consistency in internal surface treatment, and this is expected to guarantee optimum and repeatable performance. In addition, it is necessary to establish the upper threshold temperature which is experienced during the temperature cycle and beyond which irreparable damage results.

Since the existing surface treatment appears to be marginal for both the short-term soldering cycle and the long-term operating temperature of the High Performance Generator, alternate surface treatment should be considered. Promising approaches are high temperature steam and/or concentrated oxygen processes developed by RCA -- a technology recently purchased by Dynatherm. The objectives of applying these processes to the HPG heat pipes would be to develop a higher soldering temperature capability and improved long-term operating stability.

Parallel to the development of improved surface treatments, the evaluation of the currently used black-oxide treatment should be continued. Two heat pipe/fin segments and four refluxers were placed on life test during this program. Life testing and data evaluation should be continued in order to establish a confidence basis for this design. Also, the results obtained with the refluxers are very amenable to interpretation by a kinetic corrosion model. Correlation with the model would permit accurate prediction of gas generation as a function of time and operating temperature. A numerical analysis could then be conducted to predict temperature profile and length of the inactive zone of the heat pipe at various operating temperatures.

In addition to development of a qualified internal surface wetting treatment, establishment of qualified fabrication, processing, and testing procedures is required. The basic procedures have been developed during the prototype fabrication phase. A review of these procedures, definition of all critical steps, and the development of appropriate Quality Assurance provisions will be required for application to the production of space-flight qualified hardware. This includes, but is not limited to, the development of a test fixture and procedure for thermal performance testing of the heat pipe in the bent configuration, certification requirements for the welding operation, criteria for sampling and testing of the weld joint designs, criteria for sampling and testing of the final pinch-off, non-destructive testing of the heat pipe leak-tightness and strength, material controls, and fabrication and processing control.

APPENDIX B

1. Ebonol-C Treatment for Copper
2. Welding Procedure for Heat Pipes for
Teledyne Isotopes 150 Watt SNAP 19 HPG
3. Charging and Pinch-off Procedure for Heat
Pipes for Teledyne Isotopes 150 Watt SNAP 19
HPG
4. Tinning Procedure for Heat Pipes for Teledyne
Isotopes 150 Watt SNAP 19 HPG
5. Acceptance and Qualification Test Procedure
Heat Pipes for 150 Watt SNAP 19 HPG

PAGE	DESCRIPTION	DATE	APPROV

CONTRACT NO.		<i>dynaltherm</i>		COCKEYSVILLE, MARYLAND	
		CORPORATION		21030	
PREPARED BY <i>T. Zarnoch</i>		DATE <i>11/11/71</i>		TITLE EBONOL-C TREATMENT FOR COPPER	
REVIEWED/APPROVED <i>St. J. P. P.</i>		DATE <i>11/12/71</i>			
APPROVED <i>J. J. P.</i>		DATE <i>11/12/71</i>			
APPROVED <i>C. J. P.</i>		DATE <i>11/19/71</i>			
APPROVED		DATE			
		SIZE <i>A</i>	CODE IDENT NO. <i>34881</i>	DRAWING NO. 001-1000	
		SCALE:		SHEET OF	

1.0 SCOPE

This procedure defines the Ebonol-C treatment for copper to improve the wetting capability of the wick for heat pipe use.

2.0 APPLICABLE DOCUMENTS

Federal:	O-M-232	Methyl Alcohol (Methanol)
	O-N-350	Nitric Acid, Technical
Military:	Mil-T-27602A	Trichloroethylene, Oxygen Propellant Compatible
Commercial:	Ebonol C Data Sheet Enthone Inc. West Haven, Conn.	or
	Mil-F-495C	Finish, Chemical, Black, for Copper Alloys

3.0 WARNINGS & CAUTIONS

- 3.1 Use in a ventilated area. Avoid prolonged or repeated breathing of trichloroethylene, nitric acid, and methanol vapors.
- 3.2 Avoid prolonged contact with skin. Wear rubber gloves while handling trichloroethylene and nitric acid.
- 3.3 Trichloroethylene and methanol should be kept away from flames and other ignition sources when used where vapors may be concentrated.

4.0 PROCEDURE

4.1 Degreasing

- 4.1.1 Degrease parts in trichloroethylene at 120-130°F for 15 minutes.

4.1.2 Rinse thoroughly with methanol.

4.1.3 Dry parts.

4.2 Oxidizing

4.2.1 Dip parts into a 50% (by volume) solution of nitric acid, for 15 seconds, at room temperature and agitate.

4.2.2 Rinse thoroughly with water. Agitate parts during rinse. HCl

4.2.3 Dip parts into a solution (37 grams/200 cc) of Ebonol-C, for 5 minutes, at 215-220°F.

4.2.4 Rinse thoroughly with running tap water (cold) for 2 minutes.

4.2.5 Rinse thoroughly with running tap water (hot) for 2 minutes.

4.2.6 Rinse thoroughly with distilled water and drain for 20 minutes.

LTR	PAGE	DESCRIPTION	DATE	APPROVED

CONTRACT NO.

P.O. No. B-64931

dynaltherm

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TITLE

WELDING PROCEDURE FOR HEAT PIPES

FOR

TELEDYNE ISOTOPES 150 WATT SNAP 19 HPG

SIZE

A

CODE IDENT NO.

34881

DRAWING NO.

074-1010

SCALE:

SHEET 1 OF 5

1.0 PURPOSE

The purpose of this procedure is to delineate the steps to be taken to prepare and weld the copper parts of the heat pipes for the Teledyne Isotopes 150 Watt SNAP 19 HPG.

2.0 APPLICABLE DOCUMENTS

074-1001	Tube, Heat Pipe
074-1002	Fill Tube
074-1003	End Cap
074-1007	Straight Heat Pipe Assembly
074-1011	Charging and Pinch-Off Procedure

3.0 EQUIPMENT AND MATERIALS

Airco 200 AMP AC-DC Welding Transformer
Airco 200 AMP H35B Welding Torch
Argon Gas Supply for Shielding and Back-Up
DTM Model - Welding Stand
O-N-350 - Nitric Acid, Technical
TT-I-735 - Isopropyl

4.0 HEAT PIPE WELDING

The Teledyne Isotopes heat pipe consists of the 074-1007-029 tube body and wick assembly, the 074-1002 fill tube, and the 074-1003 end cap. These parts are to be welded into the 074-1007-009 straight heat pipe assembly. After this assembly is ebanoled, the end cap is to be closure welded, then after leak and pressure testing and charging, the first pinch-off seal weld is to be made. Finally the final pinch-off closure weld is to be made following the 12 hour bake.

4.1 Assembly Welding

4.1.1 Procedure

- 4.1.1.1 Dip rinse 074-1007-029 assembly, 074-1002 fill tube and 074-1003 end cap in 50% solution of nitric acid at room temperature.
- 4.1.1.2 Rinse thoroughly with demineralized or distilled water.
- 4.1.1.3 Rinse thoroughly with Isopropyl alcohol.
- 4.1.1.4 Dry in oven at $225 \pm 25^{\circ}$ for one hour.
- 4.1.1.5 Set Airco Welding Transformer to the 5-50 amp welding current range.
- 4.1.1.6 Place the 074-1003 end cap in the end of the 074-1007-029 tube body assembly per 074-1007 marked with the heat pipe serial number and hold parts together on the welding stand.
- 4.1.1.7 Attach argon gas tube to other end of 074-1007-029 assembly and adjust for good back-up gas flow through parts to be welded.
- 4.1.1.8 Adjust shielding gas (argon) flow through the welding torch.
- 4.1.1.9 Detach back-up gas tube from 074-1007-029 assembly and strike welding arc at area to be welded. (NOTE: Do not use welding rod or wire.)
- 4.1.1.10 Adjust welding current with foot control to obtain proper metal flow while rotating parts to obtain completed weld.
- 4.1.1.11 Allow welded parts to cool to room temperature.

4.1.1.12 Place 074-1002 fill tube in the other end of 074-1007-029 assembly per 074-1007 and hold parts together on welding stand.

4.1.1.13 Repeat steps 4.1.1.7 through 4.1.1.12.

4.2 End Cap Closure Weld

4.2.1 Procedure

- 4.2.1.1 Hold 074-1007-009 heat pipe with the end cap end down and abrasive clean the end cap tip. Wipe end cap tip clean before turning it up for welding.
- 4.2.1.2 Place end cap end of heat pipe in a heat sink.
- 4.2.1.3 Set Welding Transformer to the 5-50 amp welding current range and adjust shielding gas (argon) flow from welding torch.
- 4.2.1.4 Strike arc and make closure weld. (Do not use welding rod or wire.)
- 4.2.1.5 Allow to cool at room temperature.

4.3 Fill Tube Closure Weld

4.3.1 Procedure

- 4.3.1.1 Prior to pinch-off per 074-1011, set welding transformer to the 10-160 amp welding current range and adjust the shielding gas (argon) flow from welding torch.
- 4.3.1.2 Immediately following pinch-off per 074-1011, strike arc and make closure weld. (NOTE: Do not use welding rod or wire.)

4.3.1.3 Allow to cool to room temperature.

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CONTRACT NO.

P.O. No. B-64931

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TITLE

CHARGING AND PINCH-OFF PROCEDURE FOR

HEAT PIPES FOR TELEDYNE ISOTOPES

.150 WATT SNAP 19 HPQ

SIZE

CODE IDENT NO.

DRAWING NO.

A

34881

074-1011

SCALE:

SHEET 1 OF 4

1.0 PURPOSE

The purpose of this procedure is to delineate the steps to be taken to charge and pinch-off the Teledyne Isotopes heat pipes.

2.0 APPLICABLE DOCUMENTS

074-1007	Straight Heat Pipe Assembly
074-1010	Welding Procedure

3.0 EQUIPMENT AND MATERIALS

One Mililiter-capacity Medical Type Syringe

Pinch-Off Tool - DTM Model

Distilled Water

Variable Transformer

4.0 HEAT PIPE CHARGING

The Teledyne Isotopes heat pipes are to be charged with the type and amount of distilled water specified on 074-1007.

4.1 Procedure

- 4.1.1 Fill syringe with the required amount of distilled water per 074-1007.
- 4.1.2 Hold heat pipe vertical with fill tube end up.
- 4.1.3 Insert syringe in fill tube and charge heat pipe.
- 4.1.4 Place charged heat pipes in container that will keep them vertical (fill tube end up) until the fill tube has been pinched-off and closure weld made.

5.0 HEAT PIPE PINCH-OFF

5.1 Initial Pinch-Off

5.1.1 Procedure

- 5.1.1.1 Position heat pipe in pinch-off tool to provide the position of the pinch-off as required by 074-1007.
- 5.1.1.2 Snip off tip of fill tube protruding above pinch-off tool in such a manner as to produce a "pin-hole" opening.
- 5.1.1.3 Heat lower two to three inches of the heat pipe evaporator section with the electrical heater controlled by the variable transformer.
- 5.1.1.4 Place a small glass plate over the "pin-hole" opening and watch for condensing water vapor.
- 5.1.1.5 Pinch-off fill tube when water starts to condense on the glass and make closure weld per 074-1010.

5.2 Final Pinch-Off

5.2.1 Procedure

- 5.2.1.1 Hold heat pipes under cold water (or place them in ice water) with the evaporator end down.
- 5.2.1.2 After heat pipes are cold, heat the fill tube end to approximately 212⁰ F with a torch to drive out any water collected there.
- 5.2.1.3 Keep pipes vertical and place them in ice water to keep them ready for pinch-off.
- 5.2.1.4 Place heat pipe to be pinched-off in pinch-off tool to locate pinch-off as required per 074-1007 but oriented so that the

pinch-off will be across the heat pipe when looking at the wick location scribe line.

5.2.1.5 Heat evaporator end as in Step 5.1.1.3 until the gas to water vapor interface raises to within one inch of the fill tube end of the heat pipe and then immediately snip off the end of the fill tube as in Step 5.1.1.2.

5.2.1.6 Repeat Steps 5.1.1.4 and 5.1.1.5.

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CONTRACT NO.

P.O.No. B-64931

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TITLE

TINNING PROCEDURE FOR HEAT PIPES FOR

TELEDYNE ISOTOPES 150 WATT SNAP 19 HPG

SIZE

A

CODE IDENT NO.

34881

DRAWING NO.

074-1012

SCALE:

SHEET 1 OF 3

1.0 PURPOSE

The purpose of this procedure is to delineate the steps to be taken to tin the heat pipes for the Teledyne Isotopes 150 Watt SNAP 19 HPG.

2.0 APPLICABLE DOCUMENT

074-1000 Heat Pipe for 150 Watt SNAP 19 HPG

MIL-S-6872 Type III General Spec for Soldering Process

3.0 EQUIPMENT AND MATERIALS

MIL-T-81533 Trichloroethane

O-N-350 Nitric Acid, Technical

TT-I-735 Isopropyl

Tin Flux (stay clean)

QQ-S-571d - Alpha Pure Tin, "Vaculoy" grade

Dynatherm Equipment Number 1177 - Tin Pot and Heater

Stainless Steel Wool

4.0 TINNING HEAT PIPES

The Teledyne Isotopes heat pipes are to be tinned per 074-1000 prior to delivery. This is to facilitate the soldering of these heat pipes to the 150 Watt SNAP 19 HPG cooling fins.

4.1 Procedure

4.1.1 Degrease heat pipe in trichloroethane.

4.1.2 Rinse heat pipes in 50% solution of Nitric Acid at room temperature.

- 4.1.3 Rinse thoroughly with tap water.
- 4.1.4 Rinse thoroughly with Isopropyl alcohol and dry.
- 4.1.5 Heat tin (Vaculoy grade tin solder bar, Alpha Metals, or equivalent) in clean stainless steel solder pot to 500-550°F.
- 4.1.6 Dip condenser end of heat pipe to be tinned into liquid in-organic acid flux, (Alpha Metals 2001 Stay-Clean or equivalent), and wipe off the excess and then dip the pipe into the Molten tin. (NOTE: Do not allow tin to get on the pipe which has not been fluxed).
- 4.1.7 Hold pipe in the molten tin until the tin menisci around the pipe changes from concave to convex and then remove the heat pipe from the tin at a rate of $1/2 \pm 1/4$ inch per second until the tip is reached. Hold the tip in the molten tin for 5-10 seconds to allow any excess tin to drain off.
- 4.1.8 Remove heat pipe from the tin pot and allow it to cool for 40 ± 10 seconds, then rinse thoroughly in tepid water and wipe clean with a lint-free cloth or paper towel.
- 4.1.9 Repeat steps 4.1.7 through 4.1.9 for the evaporator end of the heat pipe.

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CONTRACT NO.

P.O. Number B-64931

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TITLE

ACCEPTANCE AND QUALIFICATION TEST PROCEDURE

HEAT PIPES FOR 150 WATT SNAP 19 HPG

SIZE

A

CODE IDENT NO.

34881

DRAWING NO.

074-1013

SCALE:

SHEET OF

1.0 PURPOSE

The purpose of this procedure is to define the tests and the steps to be taken to satisfy the acceptance and qualification requirements for the Teledyne Isotope Heat Pipes for 150 Watt SNAP 19 HPG as delineated in Specification HPGA2180500.

2.0 APPLICABLE DOCUMENTS

HPGA2180500	Specification: Heat Pipes for 150 Watt SNAP 19 HPG
074-1000	Top Drawing: Heat Pipe for 150 Watt SNAP 19 HPG
074-1007	Drawing: Straight Heat Pipe Assembly (for 150 Watt SNAP 19 HPG)
063-1001	Drawing: Demonstration Section Assembly (Simulated RTG Housing and Heat Pipe Fin)

3.0 ACCEPTANCE TESTS (Perform on all Heat Pipes)

The objective of these tests is to verify that each Teledyne Isotope Heat Pipe meets the acceptance requirements of Specification HPGA2180500 and are therefore acceptable for operational use on the 150 Watt HPG.

3.1 Equipment and Materials

- 3.1.1 Veeco Helium Leak Detector - Model MS 90-AB, Maximum detection sensitivity 2×10^{-10} std. cc/sec.
- 3.1.2 Helium supply for Leak Testing
- 3.1.3 Pressure Regulator and Gauge - Dynatherm Equipment No. D-1063
- 3.1.4 Two (2) Variable Power Supplies - Variac or equivalent
- 3.1.5 Wattmeter - Weston Model 310 or equivalent
- 3.1.6 Thermocouple Switch
- 3.1.7 Icebath - Thermocouple cold junction
- 3.1.8 Digital Voltmeter

3.2 Helium Leak Test

Each Teledyne Isotope Heat Pipe must be helium leak checked prior to charging with working fluid to verify that the leak rate is less than 10^{-8} std. cc/sec.

3.2.1 Procedure

3.2.1.1 Connect heat pipe to helium leak detector.

3.2.1.2 With helium leak detector set at a leak detection sensitivity of at least 10^{-9} std. cc/sec., spray helium over heat pipe and in particular over areas of welds.

3.2.2 Acceptance Criteria

Accept heat pipes which have a helium leak rate of 10^{-8} std. cc/sec. or less as a result of the above (3.2.1) tests.

3.3 Proof Pressure Test

Each Teledyne Isotope heat pipe must be proof pressure tested at a pressure of 1000 psi at room temperature to verify its capability to withstand operating pressure.

3.3.1 Procedure

3.3.1.1 Connect heat pipe with swagelock fitting to argon, or dry nitrogen bottle using appropriate regulator and gauge.

3.3.1.2 Place heat pipe in an area where operator is protected in case of rupture during test.

3.3.1.3 Raise pressure to 1000 ± 50 psia and hold for 3 ± 1 minutes.

3.3.1.4 Release pressure and remove heat pipe from test stand.

3.3.2 Acceptance Criteria

Accept those heat pipes which do not burst and show no visual evidence of deformation.

3.4 Final Closure Leak Tightness Verification

Heat pipes which have been selected under 074-1006, Section 3.2, for destructive testing are to be used to qualify the integrity of the final closure weld of the deliverable heat pipes.

3.4.1 Procedure

- 3.4.1.1 Remove approximately 1/4 inch of the evaporator end of the heat pipe.
- 3.4.1.2 Remove working fluid and dry.
- 3.4.1.3 Helium leak test opened heat pipe in accordance with Section 3.2 of this procedure.
- 3.4.1.4 Proof pressure test opened heat pipe in accordance with Section 3.3 of this procedure.
- 3.4.1.5 Repeat Step 3.4.1.3

3.5 Thermal Performance Test

Each Teledyne Isotope heat pipe must be tested to verify that it is reasonably free of noncondensing gas and that it has a conductance of at least 2.5 watts/^oF at average vapor temperatures of 150^oF and 350^oF with a heat input of at least 60 watts with the evaporator elevated one inch above the condenser.

To facilitate the accomplishment of these tests, all of these heat pipes will be tested prior to the final bending operation. Only a random sample of two heat pipes will be tested after bending in accordance with Section 4.0 of this procedure.

3.5.1 Test Fixture Calibration

- 3.5.1.1 Place a two (2) inch long section of 1/4 inch diameter copper tubing in the heater section of the test fixture (Figure 3.1 .
- 3.5.1.2 Hook up one regulated power source to the wattmeter and the heat pipe evaporator heating element in test fixture as shown in Figure 3.2.
- 3.5.1.3 Hook up the thermocouple leads to the thermocouple switch, the icebath cold junction and the digital voltmeter as shown in Figure 3.2.
- 3.5.1.4 Apply at least two different heat inputs to the heater and read temperatures after equilibrium has been attained.
- 3.5.1.5 Plot heat input versus average heater temperature and use this graph to determine heat losses at each temperature setting during heat pipe testing.

3.5.2 Non-Condensable Gas Test

- 3.5.2.1 Install the 074-1007 straight heat pipes in the Figure 3.1 Performance Test fixture with wick opposite the simulated housing and fin.
- 3.5.2.2 Elevate the condenser end of the heat pipe by 0.5 ± 0.02 inch above the evaporator end.
- 3.5.2.3 Apply sufficient power to heater to attain an equilibrium vapor temperature of 150 ± 25 °F.
- 3.5.2.4 Record temperatures and other indicated information on Data Sheet

Figure 3.1
Heat Pipe Performance Test Fixture

Figure 3.2
Heat Pipe Performance Test Set-up

3.5.3 Functional Performance Test

Verify the capability of the heat pipe to transport 60 watts with the evaporator elevated one (1) inch above the outboard end of the condenser by accomplishing the following steps:

- 3.5.3.1 With the heat pipe installed in the test fixture as in 3.5.2.1, elevate the evaporator end by 1.0 ± 0.125 inch above condenser.
- 3.5.3.2 Apply 60 watts to the evaporator heating element and regulate the cooling fan to obtain a stabilized reading of $150^{\circ}\text{F} \pm 10^{\circ}\text{F}$ in the adiabatic section of the heat pipe.
- 3.5.3.3 Record the readings of all thermocouples and other information on Data Sheet. Convert the milivolt readings to degrees fahrenheit. Calculate the overall heat pipe resistance based on average evaporator temperature (TC#1, 2 and 3) and average condenser temperature (TC #5, 6, 7 and 8).
- 3.5.3.4 Repeat steps 3.5.3.2 and 3.5.3.3 above, for a stabilized adiabatic section temperature of $350^{\circ}\text{F} \pm 10^{\circ}\text{F}$ at 60 watts.

3.5.4 Acceptance Criteria

Accept those heat pipes which have an overall temperature difference of 24°F or less (corresponding to a conductance of $2.5\text{W}/^{\circ}\text{F}$) between the average evaporator temperature and the average condenser temperature with 60 watt input at average vapor temperatures of 150°F and 350°F .

4.0 QUALIFICATION TESTING

Two (2) heat pipes, designated as prototypes, shall be subjected to the following sequence of qualification testing in addition to the acceptance tests specified in Section 3.

4.1 Thermal Cycling

4.1.1 Procedure

- 4.1.1.1 Following the acceptance testing, place the two heat pipes into an oven.
- 4.1.1.2 Attach a thermocouple to one of the heat pipes and provide a suitable read-out device.
- 4.1.1.3 Raise the oven temperature to 500 ± 20 °F and hold for 30 ± 5 minutes.
- 4.1.1.4 Allow oven to cool to room temperature.

4.2 Thermal Performance Mapping

4.2.1 Procedure

- 4.2.1.1 Bend heat pipes to final configuration in accordance to 074-1000.
- 4.2.1.2 Tin heat pipes in accordance with procedure 074-1012.
- 4.2.1.3 Solder heat pipes to simulated housing/fin section per 063-1001. Instrument the simulated housing/fin section and the heat pipes as shown in Figure 3.1.
- 4.2.1.4 Orient the housing/fin section such that the heat pipe is horizontal with respect to gravity.
- 4.2.1.5 Apply 30 watts of heater power and maintain average temperature at 150 ± 10 °F. Record temperatures and other pertinent information on Data Sheet. Thermal insulation or fan

cooling may be used in order to maintain the specified average temperature of the heat pipe.

- 4.2.1.6 Increase heater power in steps of 10 watts while maintaining the average heat pipe temperature at $150 \pm 10^{\circ}\text{F}$.
Record information on Data Sheet. Continue until the measured heat pipe conductance is lower than 2.5 watts/ $^{\circ}\text{F}$.
- 4.2.1.7 Repeat steps 4.2.1.5 and 4.2.1.6 for an average heat pipe temperature of $350 \pm 10^{\circ}\text{F}$, and at evaporator elevations of 1.0 ± 0.125 and 2.0 ± 0.125 inches.
- 4.2.1.8 The heat pipes shall be considered acceptable if their conductance, as measured by the average evaporator and condenser temperature when installed into the simulated housing/fin section, exceeds 2.5 watts/ $^{\circ}\text{F}$ with a gross heat input of 60 watts. This criterion applies to both the 150 and 350°F temperature level and to evaporator elevations of 0 and 1.0 inches, respectively.

4.3 Recovery Testing

4.3.1 Procedure

The purpose of these tests is to determine the capability of the heat pipes to recover to an operating condition following an induced failure.

- 4.3.1.1 With the heat pipe installed into the simulated housing/fin section, orient it such that the evaporator is elevated at least $1/8$ but not more than $1/4$ inch above the condenser.

- 4.3.1.2 Apply 30 watts of power to the evaporator heater and maintain average heat pipe temperature at $150 \pm 10^{\circ}\text{F}$ by using either insulation on the fin or fan cooling. Record temperatures and other pertinent information on Data Sheet.
- 4.3.1.3 With the heater power applied, raise evaporator by $3 \pm 1/4$ inch above condenser for a period of 3 ± 0.1 minutes.
- 4.3.1.4 Lower evaporator to the orientation specified in Section 4.3.1.1 and record data 10 minutes after lowering evaporator. If the heat pipe's conductance has returned to a value of at least $2.5 \text{ watts}/^{\circ}\text{F}$ at that time, allow it to achieve complete equilibrium and record data again. If the heat pipe has not recovered after 10 minutes to a conductance of at least $2.5 \text{ watts}/^{\circ}\text{F}$, proceed to Step 4.3.1.6 or 4.3.1.7.
- 4.3.1.5 Increase heater power in steps of 10 watts and repeat Steps 4.3.1.2 through 4.3.1.4.
- 4.3.1.6 Repeat Steps 4.3.1.2 through 4.3.1.5 for an average heat pipe temperature of $350 \pm 10^{\circ}\text{F}$.
- 4.3.1.7 Terminate test, record maximum heater power at which heat pipe recovered at both the 150 and 350°F temperature level.

4.4 Life Testing

Following completion of the thermal mapping and the recovery tests, the two heat pipes shall be placed on a life test.

4.4.1 Procedure

- 4.4.1.1 Orient the simulated housing/fin section such that the evaporator of the heat pipe is elevated $1.0 \pm 1/4$ inch below the condenser.
- 4.4.1.2 Apply $50 \begin{smallmatrix} + 10 \\ - 0 \end{smallmatrix}$ watts to the heater and adjust insulation or fan such that the average heat pipe temperature is 315 ± 10 °F.
- 4.4.1.3 Obtain reading of pertinent data and record on Data Sheet.
- 4.4.1.4 Maintain at this condition and record pertinent data at least every two (2) weeks.
- 4.4.1.5 Once every 2 weeks, re-orient heat pipe such that the evaporator is elevated $1.0 \pm 1/8$ inch above the condenser. With $50 \begin{smallmatrix} + 10 \\ - 0 \end{smallmatrix}$ watts applied to the heater, record data.
- 4.4.1.6 Terminate life test if any one of the following conditions applies:
- Rupture of the heat pipe envelope.
 - Failure to meet the 2.5 watt/°F conductance criterion during Step 4.4.1.4 and 4.4.1.5.
 - 10,000 hours have been accumulated.

THERMAL TESTING DATA SHEET

FOR

HEAT PIPES FOR TELEDYNE ISOTOPES 150 WATT SNAP 19 HPG

Date										
Time										
Serial Number										
Test Temperature ($^{\circ}\text{F}$)										
Elevation (inches)										
Net Power Input (watts)										
Thermocouples	mv	$^{\circ}\text{F}$	mv	$^{\circ}\text{F}$	mv	$^{\circ}\text{F}$	mv	$^{\circ}\text{F}$	mv	$^{\circ}\text{F}$
No. 1										
No. 2										
No. 3										
No. 4										
No. 5										
No. 6										
No. 7										
No. 8										
No. 9										
Average Evaporator Temperature ($^{\circ}\text{F}$)										
Adiabatic Temperature ($^{\circ}\text{F}$)										
Average Condense Temperature ($^{\circ}\text{F}$)										
Thermal Resistance, Evaporator ($^{\circ}\text{F}/\text{watt}$)										
Thermal Resistance, Condenser ($^{\circ}\text{F}/\text{watt}$)										
Total Thermal Resistance ($^{\circ}\text{F}/\text{watt}$)										

APPENDIX C

Specification: HPGA 2100310, Heat Pipe Bonding Procedure,
Isotopes, Nuclear Systems Division, June 4, 1973.

SECURITY UNCL. CONF. SECRET
REVIEW DI RD GP: 1234

~~CLASSIFIER~~

REVISIONS: SEE SHEET _____

REV LEVEL

DWG

THIS
SHEET

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This drawing releases no parts - for reference only.

EFFECTIVE ON	CALC WT	DASH NUMBER	NEXT ASSEMBLY	USED ON	NEXT ASSY	FINAL ASSY	TEST
APPLICATION					QTY REQD		

CONTRACT NO.

DRAWN BY *W. J. Bayne* DATE *6/4/73*

CHECKER *A. Hall* *6/4/73*

STRESS ENGR.

WT. ENGR.

MATL. ENGR.

RELIABILITY

GR. ENGR.

PROJECT

ENGR.

ISOTOPES

NUCLEAR SYSTEMS DIVISION

A TELEDYNE COMPANY

Baltimore Maryland

HEAT PIPE BONDING PROCEDURE

SIZE

A

CODE IDENT NO.

30856

HPGA2100310

SCALE

SHEET 1 OF 46

REVISIONS

SYM	PAGE	DESCRIPTION	DATE	APPROVED

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CHG.

SCALE

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HEAT PIPE BONDING PROCEDURE

1.0 SCOPE

1.1 Application: This procedure establishes the requirements to be met by Teledyne Isotopes Energy Systems Division and subcontractors for the solder attachment of copper-water (forty-eight) heat pipes to the magnesium-thorium alloy housing subassembly, HM21A-T5 housing body and HK31A-H24 fins, of the HPG-JPL generator.

1.2 Engineering Information

1.2.1 This procedure shall be applicable for preparation of details and the fabricated assembly to the extent specified herein.

1.3 Equivalent Government Specifications: There is no equivalent government specification.

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2.0

APPLICABLE DOCUMENTS

2.1

The following specifications and/or documents of the issue in effect on the date of issue of this specification are applicable to the extent referenced herein.

HPGA2100010	Housing Assembly
HPGA2140000	Heat Pipe Bonding Fixture
HPGA2180000	Heat Pipe Interface Drawing
HPGA2140002	Fin Tinning (Heat Pipe) Tool
HPGA2140019	Heat Pipe Soldering Chamber
HPGA2140020	Heating Fixture Heat Pipe Installation
HPGA2140022	T/V Chamber Test Suspension Bracket Assembly
PS 0300033	Protection and Cleaning of Magnesium and Magnesium Alloys
PS 0300030	Ultrasonic Cleaning
PS 0700001	Handling of Dangerous & Toxic Materials
PS 0300002	Grit Blasting of Magnesium-Thorium Alloy Details and Assemblies

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3.0 REQUIREMENTS

3.1 Facilities

3.1.1 Plasma Spray Booth

3.2 Equipment

3.2.1 Metco Type 3MB Plasma spray gun and associated power supply.

3.2.2 Rolling mill - to accommodate the rolling of the solder shim stock.

3.2.3 Precision shear - 12" minimum length.

3.2.4 Solder preform female form die and rubber pad.

3.2.5 Chart recorder - L&N Speedomax; 6 point Type K; 0-1200°F span;
chart speed 0.5-1"/min.

3.2.6 Digital type K thermometer
Digitek
#590KF

3.2.7 Thermocouple switch
48 position

3.2.8 Surface pyrometer
Alnor Instruments
#4200A

3.2.9 Variable power transformer, 0-240 volt AC, 5KW.

3.2.10 Variable power transformer, 0-140 volt AC, 20 amp.

3.2.11 Nine (9) variable power transformers, 0-140 volt AC, 1 amp.

3.2.12 Fin tinning (heat pipe) tool, HPGA2140002.

3.2.13 Heating fixture heat pipe installation, HPGA1240020.

3.2.14 Heat pipe bonding fixture, HPGA2140000.

3.2.15 T/V chamber test suspension bracket assembly, HPGA2140022.

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3.2.16 Heat pipe soldering chamber, HPGA2140019.

3.2.17 Dake hydraulic press, 50,000 psi, Model 44226.

3.3 Materials

3.3.1 Silicone sponge rubber sheet, 3/8" thick. Spec.: AMS3196A.

3.3.2* RTV21 silicon moulding compound and Thermolite 12 catalyst - General Electric Company. Spec.: Commercial.

3.3.3 Masking Tape
Spec.: stockroom

3.3.4 Polyethylene bags
Spec.: stockroom

3.3.5* Argon
Spec.: evaporated liquid on MIL-A-18455

3.3.6 Brass sheet, 16 to 24 gauge (as required - plasma spray mask)
Spec.: Commerical.

3.3.7* Ni-4.5% Al powder
Spec.: Metco #450 powder

3.3.8* Copper powder
Spec.: Metco #55 powder

3.3.9 Cotton swabs
Spec.: stockroom

3.3.10* Flux, Kester's #1544
Spec.: Commercial

3.3.11* Puretin solid wire solder, 1/32" and 1/16" diameter (Kester's or Alpha Metals).
Spec.: Commercial

3.3.12* Bar solder, pure tin
Spec.: Vaculoy Grade (Alpha Metals)

*Hazardous Materials; refer to PS 0700001 for handling instructions.

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- 3.3.13 Bristle brush
Spec.: Commercial
- 3.3.14* Kester's AP-20 Cleaner
Spec.: Commercial
- 3.3.15* Alcohol, Methanol or Denatured Ethanol
Spec.: 95% minimum
- 3.3.16* Trichlorethylene
Spec.: Commercial
- 3.3.17* Freon TF
Spec.: Commercial
- 3.3.18 Disposable wipes, e.g., Scott 05313 or equivalent.
Spec.: e.g. Kimberly Clark #3425, Type 900L
- 3.3.19 Gloves, Nylon or cotton
Spec.: stockroom
- 3.3.20 Water, demineralized
Spec.: $\geq 50 \text{ K}\Omega$ resistivity
- 3.3.21 Type K Thermocouple wire, #30 and #36ga, fiberglass insulation.
Spec.: Commercial
- 3.3.22 Type K thermocouple connector plugs male and female (48 each).
Spec.: Commercial
- 3.3.23 46 mesh Alumina, Al_2O_3 , abrasive grit.
- 3.3.24 Hi-D PTFE Adhesive tape 503-3, 1" wide x 18 ft. roll, Cadillac Plastics.

3.4 Product

A soldered assembly of copper-water heat pipes (48) attached to the magnesium-thorium alloy housing assembly.

*Hazardous Materials; refer to PS 0700001 for handling instructions.

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3.5 Fabrication Control

3.5.1 Special Skills: Operators shall be sufficiently skilled in the operation of plasma spray equipment and chemical cleaning baths. The plasma spray operator must have prior experience in spraying similar sample and prototype hardware. For preparation of solder preforms, assembly of tooling and detail parts and soldering operations, the operator shall be sufficiently experienced to perform the operations as specified herein.

3.5.2 Safety: Some materials called out in this process can be hazardous. Refer to PS 0700001 for precautions and handling instructions.

3.5.3 Operators shall wear tinted welding face shield and safety muffs, respectively in an adequately ventilated spray booth when performing the flame spraying operations.

3.5.4 Safety goggles, and protective clothing (i.e., long rubber gloves, rubber apron) shall be worn during chemical cleaning operations.

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4.0

QUALITY ASSURANCE

4.1

Quality shall ensure conformance to the requirements of this process specification.

SIZE

CODE IDENT NO.

A

30856

HPGA2100310

REV

SCALE

SHEET 11

5.0 HEAT PIPE IDENTIFICATION

The identity of each of the forty-eight heat pipes must be maintained throughout the assembly process and in the completed housing assembly. The following identification system shall be employed.

5.1 Assign identification numbers to the fins and heat pipe grooves as follows:

5.1.a View the housing along its X-axis from the bottom end of the housing. Start with the fin nearest the Z-axis (identified on boss per on drawing HPGA2100010) and progressing in a clockwise direction, assign identification numbers, one thru six, to each of the fins.

5.1.b For each fin, assign an identification letter to each mating fin/case heat pipe groove. Starting from the top end of the housing (c.f. HPGA2100010) assign identification letters, A thru H, progressively to each of the mating fin/case grooves. Thus, each mating fin/case groove is identified by a digit (fin location) and a letter (position on the indicated fin), as illustrated in Figure 1.

5.2 Assign each of the forty-eight heat pipes, identified by the vendor assigned serial numbers, to each of the fin/case grooves. Tabulate fin/case groove identification versus heat pipe serial number.

5.2.1 For build of HPG S/N 02 use heat pipe location assignments as listed in Appendix A, Table II.

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FIGURE 1. HOUSING ORIENTATION AND CASE FIN GROOVE AND HEAT PIPE IDENTIFICATION SYSTEM.

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		SCALE			SHEET 13

6.0

SILICONE RUBBER COMPLIANT JOINT CLOSURES

Solder retention in the wide gap heat pipe case*/fin joint is effected by four compliant silicone rubber joint closures:

- a. The silicon rubber faced (3/8" thick silicone sponge rubber sheet per AMS3196A with an adhesively bonded facing of 1/16" thick 30 Durometer, A-scale, silicone rubber sheet) hold down shoe or pressure foot, HPGA 2140000-001 (forty-eight pieces).
- b. The six cast RTV silicone rubber case/grooved fin fillet strips each of which seals the transition sections between the case (evaporator) and fin condenser) sections of the eight heat pipes on each of the six fins.
- c. Hollow round slip-over plug which fits over the tubular seal at the evaporator end of the heat pipe and which is made from 3/8" thick silicone sponge rubber sheet (AMS3196A), forty-eight pieces.
- d. Hollow round slip-over plug which fits over the tubular closure at the condenser end of the heat pipe and which is made from 3/8" thick silicone sponge rubber sheet (AMS 3196A), forty-eight pieces.

6.1 Cast silicone rubber case/grooved fin fillet strips.

6.1.1 Orient the as-machined housing such that the case axes is horizontal, the grooved side of Fin No. 1 is facing upward and the fin makes an approximate +45° angle with the horizontal plane. Adjust the fin angle to yield the cast fillet strip cross-sectional profile as shown in Figure 2.

6.1.2 Plug mounting lug holes on ends of case located by Fin No. 1.

6.1.3 Place the serialized heat pipes in their assigned positions in Fin No. 1 grooves. Hand form each pipe as required for proper fit.

CAUTION: Heat pipes are easily deformed and shall be handled with extreme care.

6.1.4 Using the "fillet" or juncture of the No. 1 fin as a pattern, cast a replica of the fillet with heat pipes in place using RTV21 moulding compound with Thermolite 12 catalyst.

*NOTE: For clarification of terminology used in this text : Case - cylindrical body of the housing with/or without fins. Housing - case with fins attached.

		SIZE	CODE IDENT NO.		
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REV					
SCALE			SHEET 11

NOTE: $\pm .03$ TOLERANCE ON 2 PLACE DIMENSIONS

FIGURE 2. TYPICAL CROSS-SECTION CAST RTV-21 SILICONE RUBBER CASE/GROOVED FIN FILLET STRIP

REV	SIZE	CODE IDENT NO.	
	A	30856	HPGA2100310
	SCALE		SHEET 15

6.1.5 Cure in-place (temperature and time to be determined).

6.1.6 Identify each cast fin fillet strip by marking with the fin number on the strip end corresponding to the top end of the housing.

Slit the cast fillet strip per Engineering instructions to permit removal of the heat pipes. Strip the cast fillet strip from the mold and remove the heat pipes.

CAUTION: Heat pipes are fragile, handle with extreme care. Do not distort or otherwise damage the heat pipes.

6.1.7 Repeat steps 6.1.1 through 6.1.6 for each of the remaining fins, numbers 2 thru 6.

6.1.8 Post-cure the six cast silicone rubber fin fillet strips in air for one hour at 500°F.

6.2 Make forty-eight (48) evaporator end slip-over plugs, Figure 3A, from 3/8" thick silicone sponge rubber sheet (AMS3196A).

6.3 Make forty-eight (48) condenser end slip over plugs, Figure 3B, from 3/8" thick silicone sponge rubber sheet.

		SIZE	CODE IDENT NO.	
		A	30856	HPGA2100310
	REV			
SCALE				SHEET 16

NOTE: $\pm .03$ TOLERANCE ON 2 PLACE DIMENSIONS

FIGURE 3. FOAM SILICONE RUBBER SLIP-OVER PLUGS

	REV	SIZE	CODE IDENT NO.		
		A	30856	HPGA2100310	
		SCALE			SHEET 17

7.0

PREPARATION OF MAGNESIUM-THORIUM ALLOY HOUSING

Preparation of the magnesium-thorium alloy housing for heat pipe soldering entails: (1) grit blasting the groove surfaces in the case saddles and fins (c.f. Figure 1) and chemically cleaning the housing assembly, (2) plasma spraying the grooves with a Ni-4.5% Al (Metco 450) bondcoat and a copper (Metco 55) overlay, (3) tinning the plasma sprayed solder substrates at approximately 500°F using pure tin solder and an activated rosin liquid flux (Kester's #1544), and (4) cleaning the tinned housing assembly. The process steps for preparation of the "tinned" housing detail are described below.

- 1 NOTE: Prior to proceeding to step 7.1, the machined housing shall be employed for thermal testing and qualification of: (1) the Fin Tinning Tool (HPGA21400002) and Heat Pipe Installation Heating Fixture (HPGA21400020) used in tinning the housing heat pipe grooves, and (2) the Heat Pipe Soldering Chamber (HPGA21400019) and Heat Pipe Installation Heating Fixture (HPGA21400020) used in the heat pipe soldering operation.

Also the machined housing shall be used as a pattern for casting the six RTV silicone rubber case/grooved fin fillet strips as described in Section 5.1.

In addition to the housing, six magnesium-thorium alloy grooved fin samples, Figure 4, shall be processed per steps 7.1 thru 7.1.5.

- 7.1 Prepare the heat pipe groove areas on the magnesium-thorium alloy housing and grooved fin test samples for plasma spraying by cleaning and grit blasting per PS 0300033 Method 2 with the limitations and exceptions specified in 7.1.1 thru 7.1.5.

- 7.1.1 Mask applicable areas and grit blast (PS 0300002) the heat pipe fin grooves (48) and case saddle grooves (48), in the housing and groove in grooved-fin, test sample (6), as indicated in Figure 5, using 46 grit aluminum oxide.

- 7.1.2 All steps of PS 0300033 Method 2, except grit blasting, are applied to the whole grooved fin test samples and the external surfaces of the housing.

NOTE: Mask the I.D. surface of the case and the I.D. surfaces of the electrical connector bosses.

- 7.1.3 The time for bright pickling (c.f. PS 0300033 Method 2, step 9 of Figure 2 from specification) shall be 1 1/2 to 2 minutes for a bath temperature of 60-80°F.

		SIZE	CODE IDENT NO.		
		A	30056	HPGA2100310	
REV					
SCALE				SHEET	18

SEE HPGA2100011 FOR SECTION DIMENSIONS

NOTE: $\pm .03$ TOLERANCE ON 2 PLACE DIMENSIONS

FIGURE 4. GROOVED FIN TEST SPECIMEN

	REV	SIZE	CODE IDENT NO.		
		A	30356	HPGA2100310	
		SCALE			SHEET 19

BLAST & SPRAY TO EXTEND FROM FIN TIP TO WELD JOINT.

BLAST & SPRAY TO EXTEND FROM END TO END OF MACHINED SADDLE

FIGURE 5. HEAT PIPE GROOVE AREAS DELINEATED FOR GRIT BLASTING AND PLASMA SPRAYING

	REV	SIZE	CODE IDENT NO.		
		A	30356	HPGA2100310	
		SCALE			SHEET

- 7.1.4 Following bright pickling, immediately rinse the housing (and grooved fin test samples) using tap water spray systems.
- 7.1.5 If the housing and grooved fin samples cannot be plasma sprayed within four (4) hours after cleaning, package the parts in double, argon-filled, hermetically sealed, polyethylene bags and store in ETG shipping container.
- 7.2 Plasma spray heat pipe grooves (housing) & groove in grooved fin test samples.
- 7.2.1 Preparation of cleaned and grit blasted (heat pipe grooves only) housing for plasma spraying.
- 7.2.1.1 Handle all cleaned detail parts with clean, white, cotton gloves.
- 7.2.1.2 Mechanically attach one grooved fin test specimen to each fin. The grooved fin test specimen shall be mechanically attached to the end of the fin(s) (1/16" gap), such that there is good alignment (i. e. continuity) between the grooves, as shown in Figure 6. The test specimen should be matched with one of the central four heat pipe grooves on each fin.
- 7.2.1.3 Mask, using a brass template or equivalent, as required to restrict the plasma sprayed coating to the groove areas as shown in Figure 5.
- 7.2.2 Plasma Spray Procedure
- 7.2.2.1 Place the masked part (housing) with attached test pieces in the plasma spray booth.
- 7.2.2.2 Plasma spraying shall be accomplished with Metco Type 3MB plasma torch and the following operational parameters:

<u>Powder (Metco No.)</u>	<u>Argon Flow CFH</u>	<u>Hydrogen Flow CFH</u>	<u>Current Amps</u>	<u>Nozzle</u>	<u>Powder Part No.</u>	<u>Powder Feed RPM</u>
#55-Cu	150	5	500	GH	#1	20
#450- Ni 4.5% Al	100	15	500	GH	#2	20

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	A	30856	HPGA2100310
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FIGURE 6. TYPICAL MECHANICAL ATTACHMENT OF GROOVED FIN TEST SPECIMEN TO HOUSING FIN

	REV	SIZE	CODE IDENT NO.		
		A	30356	HPGA2100310	
		SCALE		SHEET	22

7.2.2.2 (Continued)

Torch work piece distance -- 6" to 8"

Spray direction(s) -- In a plane which is normal to the groove axis and +45° and -45° from the normal to the fin or case surface.

Pass sequence* -- The Ni-4.5% Al (Metco #450) bondcoat shall be manually applied in two passes in the +45° and two passes in the -45° direction. Immediately after sequential application of the Ni-4.5% Al bondcoat to eight heat pipe grooves (i. e., single fin/case segment) plus the grooved fin test sample, the copper topcoat shall be manually applied in three passes in the +45° direction plus three passes in the -45° direction.

7.2.2.3 Progressively flame spray each of the remaining five sets of heat pipe grooves plus grooved fin test sample (i. e. single fin/case segments) per the procedure of 7.2.2.2.

7.2.2.4 Remove masking from plasma sprayed housing.

7.2.2.5 Remove the grooved fin test samples, one from each fin. For each test sample identify the edge abutting against the fin, and the fin and groove location (c.f. 5.1). Cut metallographic samples (transverse to the groove axis and 1/4" and 3/4" from the abutting surface). Mount and polish samples. Measure coating thicknesses and provide data to Engineering for review. DO NOT PROCEED TO TINNING OPERATION WITHOUT PRIOR ENGINEERING APPROVAL.

7.2.2.6 If the plasma sprayed housing cannot be "tinned" (7.3) within four (4) hours after plasma spraying, package the part in a double argon-filled, hermetically sealed, polyethylene bag, or store in the ETG shipping container.

7.3 Tinning of plasma sprayed heat pipe grooves with pure tin solder.

7.3.1 Insert the core heater (Heating Fixture Heat Pipe Installation HPGA2140020) into the core of the housing and bolt to lugs on the top end of the housing.

7.3.2 Attach the Fin Tinning Tool (HPGA2140002) to the rib side of fin No. 1 using thermally insulated clamps.

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- 7.3.3 Electrically connect the Core Heater (HPGA2140020) and the Fin Tinning Tool (HPGA2140002) to control powerstats as determined in preliminary thermal tests (c.f. $\triangleleft 1$ in 7.0).

NOTE: The two heaters must be capable of heating the single fin (attached to Fin Tinning Tool) and mating case segment to 460 to 510°F. The temperature of the remaining five fins and mating case segments must be less than 350°F. Temperatures should be measured by a surface contact pyrometer (Alnor).

- 7.3.4 Tinning of the flame sprayed heat pipe fin and mating case grooves shall be accomplished one fin and mating case segment at a time starting with fin No. 1 (i.e. 1A thru 1H).
- 7.3.4.1 Orient the housing such that fin No. 1 is in a horizontal position, grooved side up.
- 7.3.4.2 Apply with cotton swabs Kester's #1544 activated rosin liquid flux to all flame sprayed surfaces of the heat pipe grooves (fin and case), 1A thru 1H.
- 7.3.4.3 Heat fin No. 1 and mating case segment at 15° to 30°F/minute and stabilize at 460 to 510°F. Check temperatures with an Alnor surface contact pyrometer.
- 7.3.4.4 Tin the plasma sprayed heat pipe grooves, 1A thru 1H, using pure tin solder either 1/16" or 1/32" diameter solid solder wire (Kester's or Alpha Metals). Use additional 1544 rosin flux and auxiliary heating from an electrically heated (500°F nominal tip temperature; 200 watt, or less) soldering iron as required to attain uniform tinning. Wiping with a clean, lint free cloth is permitted.
- 7.3.4.5 Total time in which the plasma sprayed copper substrate is exposed to the molten tin solder should be fifteen (15) minutes maximum, preferably less than ten (10) minutes. As soon as tinning of heat pipe grooves 1A thru 1H is completed (not to exceed fifteen minutes) turn off power and cool the housing to ambient temperature.
- 7.3.4.6 Remove the Fin Tinning Tool (HPGA2140002) from fin No. 1 and attach it to the rib side of fin No. 2. Re-orient the core heater (HPGA2140020) if required from results of preliminary thermal tests (c.f. $\triangleleft 1$). Orient the housing such that fin No. 2 is in a horizontal position, grooved side up.

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SCALE		SHEET 24	

7 3.4.7 Tin the remainder of the plasma sprayed heat pipe grooves per paragraphs 7.3.4.2 thru 7.3.4.6, proceeding sequentially thru fins Nos. 2,3,4,5, and 6.

7.3.5 Cleaning the pre-tinned housing.

7.3.5.1 Remove the core (HPGA2140020) and fin (HPGA2140002) heating tools.

7.3.5.2 Remove all residual flux by hand scrubbing with Kester's AP-20 rosin cleaner using a clean bristle brush and, if desired, Scotch-brite pads.

7.3.5.3 Clean with a vigorous hot water rinse.

7.3.5.4 Rinse with alcohol.

7.3.5.5 Blow dry with an inert gas.

7.3.5.6 Visually inspect for residual flux (1 to 10X magnification). If noted, repeat steps 7.3.5.2 thru 7.3.5.6.

7.3.5.7 If the pre-tinned housing is not to be used in the final solder operation (8.0) for a period greater than four hours, package the part in a double, argon filled, hermetically sealed, polyethylene bag, and store in the ETG shipping container.

7.4 Process flow chart

The process flow chart for preparation of the "tinned" magnesium-thorium alloy housing detail is outlined in Figure 7. This flow chart summarizes operations 7.0 thru 7.3.5.7.

REV	SIZE	CODE IDENT NO.	
	A	30856	HPGA2100310
SCALE		SHEET 25	

-
- ```
graph TD; 1[1. Clean welded and machined housing and six grooved fin test samples and grit blast heat pipe grooves, per PS 0300033 Method 2, except as noted(*).] --> 2[2. Vapor degrease - PS 0300009.]; 2 --> 3[3. Mask and grit blast heat pipe groove areas per PS 0300002 Use 46* grit alumina.]; 3 --> 4[4. Mask housing I. D. surface and I. D. surface of receptacle bosses and initiate chemical cleaning within 60 min. after grit blasting.]; 4 --> 5[5. Within 60 minutes]; 5 --> 6[6. Pickle Sodium Dichromate (Chloride Removal) PS 0300033.]; 6 --> 7[7. Tap water rinse.]; 7 --> 8[8. Alkaline Clean PS 0300033.]; 8 --> 9[9. Tap water rinse.]; 9 --> 10[10. Bright pickle PS 0300033 60°-80°F; 1 1/2 to 2* minutes.]; 10 --> 11[11. Tap water spray rinse.]; 11 --> 12[12. Demineralized water rinse.]; 12 --> 13[13. Dry]; 13 --> 14[14. Within 30 minutes]; 14 --> 15[15. Attach one grooved fin test sample to end of each fin with grooves aligned.]; 15 --> 16[16. Mask heat pipe groove areas.]; 16 --> 17[17. Plasma spray heat pipe groove areas Ni-4.5% Al (Metco #450) undercoat copper (Metco #55) topcoat.];
```
1. Clean welded and machined housing and six grooved fin test samples and grit blast heat pipe grooves, per PS 0300033 Method 2, except as noted(\*).
  2. Vapor degrease - PS 0300009.
  3. Mask and grit blast heat pipe groove areas per PS 0300002 Use 46\* grit alumina.
  4. Mask housing I. D. surface and I. D. surface of receptacle bosses and initiate chemical cleaning within 60 min. after grit blasting.
  5. Within 60 minutes
  6. Pickle Sodium Dichromate (Chloride Removal) PS 0300033.
  7. Tap water rinse.
  8. Alkaline Clean PS 0300033.
  9. Tap water rinse.
  10. Bright pickle PS 0300033 60°-80°F; 1 1/2 to 2\* minutes.
  11. Tap water spray rinse.
  12. Demineralized water rinse.
  13. Dry
  14. Within 30 minutes
  15. Attach one grooved fin test sample to end of each fin with grooves aligned.
  16. Mask heat pipe groove areas.
  17. Plasma spray heat pipe groove areas Ni-4.5% Al (Metco #450) undercoat copper (Metco #55) topcoat.

FIGURE 7. FLOW CHART PREPARATION OF "TINNED" HOUSING DETAIL

|  |     |       |                |             |          |
|--|-----|-------|----------------|-------------|----------|
|  | REV | SIZE  | CODE IDENT NO. |             |          |
|  |     | A     | 30856          | HPGA2100310 |          |
|  |     | SCALE |                |             | SHEET 26 |



18. Remove and metallographically examine six grooved fin test samples; proceed or re-work topcoat pending Engineering decision.
- ↓
19. Housing assembly with plasma sprayed heat pipe groove areas.
- ↓
20. Install core heater (HPGA 2140020) in center of housing.
- ↓
21. Attach fin tinning tool to Fin #1.
- ↓
22. Heat Fin #1 and mating grooved section of case to 450°-500°F using Kester 1544 flux on flame sprayed substrate.
- ↓
23. Tin the flame sprayed substrate on heat pipe grooves on Fin #1 and mating section of case using auxiliary soldering iron, pure tin solder and Kester's #1544 flux.
- ↓
24. Cool to ambient; move tinning tool to Fin #2 and repeat last two steps. Repeat on Fins 3 thru 6.
- ↓
25. Clean, using Kester's AP-20 Rosin remover; wash hot water, alcohol rinse, blow dry.

FIGURE 7. CONTINUED

|  |     | SIZE     | CODE IDENT NO. |             |          |
|--|-----|----------|----------------|-------------|----------|
|  |     | <b>A</b> | <b>30856</b>   | HPGA2100310 |          |
|  | REV |          |                |             |          |
|  |     | SCALE    |                |             | SHEET 27 |

8.0 PREPARATION OF HEAT PIPES FOR HEAT PIPE SOLDERING

8.1 De-grease forty-eight (48) pre-tinned heat pipes (HPGA2180000 and specification HPGA2180500).

NOTE: HEAT PIPES ARE EASILY DEFORMED AND SHALL BE HANDLED WITH EXTREME CARE AT ALL TIMES.

8.1.1 Wash with trichlorethylene.

8.1.2 Rinse with alcohol.

8.1.3 Blow dry with an inert gas.

8.1.4 Package each heat pipe in an argon filled, hermetically sealed, polyethylene bag.

|  |     |       |                |             |
|--|-----|-------|----------------|-------------|
|  |     | SIZE  | CODE IDENT NO. |             |
|  |     | A     | 30356          | HPGA2100310 |
|  | REV |       |                |             |
|  |     | SCALE |                | SHEET 28    |

9.0

PREPARATION OF TIN SOLDER PREFORMS

9.1

Roll 0.015"/0.016" thick tin solder shim stock.

9.1.1

Use Vaculoy grade (Alpha Metals) tin solder bar.

9.1.2

Cut the tin solder bar to convenient lengths commensurate with finish strip requirements and capabilities of the ultrasonic cleaning tank and rolling mill.

9.1.3

Ultrasonically clean the tin solder bar mullets in Freon TF (3.3.17) or trichlorethylene (3.3.16) per PS 0300030.

9.1.4

Saturate a wipe with alcohol (3.3.15). Wipe all rolls and the run out tables of the rolling mill.

9.1.5

Roll the tin solder bar to 1"  $\begin{smallmatrix} +1/8" \\ -1/16" \end{smallmatrix}$  width by 0.015"/0.016" thick strip; sixty four plus eight spare pieces,  $10\frac{1}{2} \pm 1/16$ " in lengths, are required. Roll at ambient temperature without a lubricant.

9.1.6

Ultrasonically clean the strips in Freon TF or trichlorethylene per PS 0300030.

9.1.7

Rinse with alcohol.

9.1.8

Blow dry with an inert gas.

9.1.9

If the time span before proceeding to operation 9.3 is greater than four (4) hours, package in an argon filled, hermetically sealed polyethylene bag.

9.2

Prepare 1/32" diameter solid wire tin solder preforms. (Kester's or Alpha Metals.)

9.2.1

Cut seventy  $10\frac{1}{2} \pm 1/8$ " lengths of 1/32" diameter solid wire tin solder.

9.2.2

Hand straighten.

9.2.3

Cut to following lengths:

No. PiecesLengths

56

 $2.25 \begin{smallmatrix} +1/32" \\ -0 \end{smallmatrix}$ 

56

 $9.60 \begin{smallmatrix} +1/32" \\ -0 \end{smallmatrix}$ 

SIZE

CODE IDENT NO.

A

30856

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REV

SCALE

SHEET 29

- 9.2.4 Ultrasonically clean the wires in Freon TF or trichlorethylene per PS 0300030.
- 9.2.5 Rinse with alcohol.
- 9.2.6 Blow dry with an inert gas.
- 9.2.7 If the time span before proceeding to step 9.4 is greater than four (4) hours, package in an argon filled, hermetically sealed polyethylene bag.
- 9.3 Form the 0.015/0.016" thick solder strips (9.1.9) to the desired preform shapes, Figure 8.

- 9.3.1 Cut the solder shim stock to the desired lengths.

| <u>No. Pieces</u> | <u>Length</u>        |
|-------------------|----------------------|
| 56                | 2.25" $+1/32"$<br>-0 |
| 56                | 9.60" $+1/32"$<br>-0 |

- 9.3.2 Form the strips to straight contour pieces (contour as shown in Figure 8) using a female die, rubber pad and 25 ton Dake hydraulic press.
- 9.3.3 Shear the contoured solder preforms to widths as noted in Figure 8 A and B.
- 9.3.4 Hand form the 2 1/4" long contoured blanks to conform to Figure 8A using as a tool a 0.250" diameter bar bent to the desired radius.
- 9.3.5 Ultrasonically clean the formed solder shims in Freon TF or trichlorethylene per PS 0300030.
- 9.3.5.1 Rinse with alcohol.
- 9.3.5.2 Blow dry with an inert gas.
- 9.3.5.3 Visually inspect. All pieces should be free of laps, seams, cracks, pits, surface inclusions or foreign particles, or other similar defects.

|  |       |                |             |
|--|-------|----------------|-------------|
|  | SIZE  | CODE IDENT NO. |             |
|  | A     | 30856          | HPGA2100310 |
|  | REV   |                |             |
|  | SCALE |                | SHEET 30    |

REV

SCALE

A

SIZE

30856

CODE IDENT NO.

HPGA210-210

SHEET 31

NOTE:  $\pm.010$  TOLERANCE ON 3 PLACE DIMENSIONS  
 $\pm.03$  TOLERANCE ON 2 PLACE DIMENSIONS } UNLESS OTHERWISE NOTED

FIGURE 8. SOLDER SHIMS

- 9.3.9 If the time span before proceeding to step 9.4 is greater than four (4) hours, package in argon filled, hermetically sealed polyethylene bags.
- 9.4 In order to simplify placement of the solder preforms, attach the wire preforms (9.2 - 9.2.7) to the formed shim stock solder preforms (9.3 - 9.3.9) by tack soldering in the position shown in Figure 9.
- 9.4.1 The 2 1/4" length wires and formed (curved) preforms are joined to form the evaporator section preforms - 56 pieces. Tack solder near the ends.
- 9.4.2 The 9.6" length wires and formed preforms are joined to form the condenser section preforms - 56 pieces. Tack solder at the center of the length and near each end.
- 9.4.3 Tack solder using Kester's #1544 rosin flux, a 50 watt electrically heated solder iron with 450°F tip temperature, and a minimal quantity of 67 Sn-33Pb solid wire solder applied to the tip of the iron.
- 9.4.4 Ultrasonically clear the preforms in Kester's AP-20 (3.3.14) rosin cleaner.
- 9.4.5 Wash with tap water.
- 9.4.6 Ultrasonically clean the preforms using Freon TF or trichlorethylene.
- 9.4.7 Rinse with alcohol.
- 9.4.8 Blow dry with an inert gas.
- 9.4.9 If the time span before proceeding to step 10 is greater than four (4) hours, package in argon filled, hermetically sealed polyethylene bags.

|     |       |                |             |
|-----|-------|----------------|-------------|
| REV | SIZE  | CODE IDENT NO. | HPGA2100310 |
|     | A     | 30856          |             |
|     | SCALE | SHEET 32       |             |

FIGURE 9. SOLDER PREFORM (FORMED SHIM PLUS WIRE)

|       |      |                |             |    |
|-------|------|----------------|-------------|----|
| REV   | SIZE | CODE IDENT NO. | HPGA2100310 |    |
|       | A    | 30856          |             |    |
| SCALE |      | SHEET          |             | 33 |

## 10.0 PRE-SOLDERING TOOLING AND PIECE PART ASSEMBLY AND INSTRUMENTATION

The functions of the soldering tool includes: (1) holding the solder preforms and heat pipes in proper position, (2) applying adequate pressure, via the Belleville spring actuated silicone rubber faced compliant hold down shoe, to attain positive seating of the heat pipes in the plasma sprayed and pre-tinned case/fin grooves on melting of the solder preforms, and (3) retaining the molten tin solder in the wide gap joint by physical entrapment by the compliant silicone rubber faced hold down shoe and auxiliary silicone rubber joint closures, as described in Section 6.0.

### 10.1 Thermocouple Installation

10.1.1 Thermocouple leads are to be held in place by teflon tape/silicone adhesive with the thermocouple bead being mechanically held in place by the compliant hold down shoe. Hence, thermocouple installation must precede installation of the hold down shoes.

10.1.2 Use Type K (chromel-alumel) #36 gage (alternate #30 gage) fiberglass insulated thermocouple wire.

10.1.3 Thermocouples installed adjacent to the case (evaporator) section of the heat pipe shall be located at the mid-length of the evaporator section on the edge of the lip of the saddle section (c.f. Figure 10). Thermocouples installed adjacent to the fin (condenser) section of the heat pipe shall be located on the grooved side of the fin, two inches  $\pm 1/4$ " from the fin tip and immediately adjacent to the pre-tinned area. For heat pipe location designated A, locate thermocouples on the bottom side of the heat pipe groove. For heat pipe locations designated B thru H locate thermocouples on the top side of the heat pipe groove.

10.1.4 Thermocouples shall be installed in the positions listed in Table IA and identified as indicated in Table IB.

NOTE: Thermocouples must be installed concurrently with the heat pipes, c.f. Section 10.2.2.9.

10.1.5 Attach all thermocouples to the 48 point T/C selector switch with readout on the Digitek. Check all thermocouples for continuity and proper identification. Six thermocouple locations, as designated in Table IB are attached to a recorder.

10.2 Assembly of heat pipe bonding tool (HPGA2140000) with heat pipes and solder preforms.

10.2.1 Preliminary assembly of heat pipe bonding tool (HPGA2140000).

|     |       |                |             |
|-----|-------|----------------|-------------|
| REV | SIZE  | CODE IDENT NO. |             |
|     | A     | 30856          | HPGA2100310 |
|     | SCALE |                | SHEET 3-1   |

FIGURE 10. THERMOCOUPLE INSTALLATION

|     |       |                |             |          |
|-----|-------|----------------|-------------|----------|
| REV | SIZE  | CODE IDENT NO. |             |          |
|     | A     | 30856          | HPGA2100310 |          |
|     | SCALE |                |             | SHEET 35 |

TABLE 1A

THERMOCOUPLE LOCATIONS

| <u>Heat Pipe</u> | <u>1</u> | <u>2</u> | <u>3</u> | <u>4</u> | <u>5</u> | 6 | <u>6</u> |
|------------------|----------|----------|----------|----------|----------|---|----------|
| A                | I        | I        | I        | I        | I        |   | I        |
| B                | II       | III      | II       | III      | II       |   | III      |
| C                |          | II       |          | III      |          |   | II       |
| D                | III      |          | III      |          | III      |   |          |
| E                |          | II       |          | II       |          |   | II       |
| F                | II       |          | III      |          | II       |   |          |
| G                | III      | II       | III      | II       | III      |   | II       |
| H                | I        | I        | I        | I        | I        |   | I        |

I Case (Evaporator) and Fin (Condenser).  
 II Case (Evaporator) only.  
 III Fin (Condenser) only.

SIZE  
**A**  
 SCALE

CODE IDENT NO.  
**30856**

HPGA2100310

REV

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TABLE 1B - THERMOCOUPLE LOCATIONS AND IDENTIFICATION NUMBERS

| Heat<br>Pipe | Location * | Thermocouple Identification Number<br>Fin Number |          |          |          |          |          |
|--------------|------------|--------------------------------------------------|----------|----------|----------|----------|----------|
|              |            | <u>1</u>                                         | <u>2</u> | <u>3</u> | <u>4</u> | <u>5</u> | <u>6</u> |
| A            | C          | 1                                                | 3        | 5        | 7**      | 9        | 11       |
|              | F          | 2**                                              | 4        | 6        | 8        | 10       | 12       |
| B            | C          | 13                                               | -        | 15       | -        | 17       | -        |
|              | F          | -                                                | 14       | -        | 16       | -        | 18       |
| C            | C          | -                                                | 19       | -        | -        | -        | 21       |
|              | F          | -                                                | -        | -        | 20       | -        | -        |
| D            | C          | -                                                | -        | -        | -        | -        | -        |
|              | F          | 22                                               | -        | 23**     | -        | 24       | -        |
| E            | C          | -                                                | 25       | -        | 26**     | -        | 27       |
|              | F          | -                                                | -        | -        | -        | -        | -        |
| F            | C          | 28                                               | -        | -        | -        | 30       | -        |
|              | F          | -                                                | -        | 29       | -        | -        | -        |
| G            | C          | -                                                | 32       | -        | 34       | -        | 36       |
|              | F          | 31                                               | -        | 33       | -        | 35       | -        |
| H            | C          | 37**                                             | 38       | 41       | 43       | 45       | 47       |
|              | F          | 38                                               | 40       | 42       | 44**     | 46       | 48       |

\* Case - C  
Fin - F

\*\* Connect these locations to both Digitek (via selector switch) and L&N  
Speedomax (continuous reading).

REV

SCALE

A

SIZE

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CODE IDENT NO.

HPGA2100310

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- 10.2.1.1 Attach the Heat Pipe Installation Heating Fixture (HPGA2140020-009), end plate (HPGA2100000-011) and T/V Chamber Test Suspension Bracket Assembly (HPGA2140022-009); use 1/4" spacer on short leg of bracket) in the cited sequence to the TOP end of the machined housing with plasma sprayed and tinned heat pipe grooves (7.3.5.7).
- 10.2.1.2 Attach the end plate (HPGA2140000-011) to the bottom end of the housing (HPGA2100010).
- 10.2.1.3 Assemble six welded assemblies, load backup beams (HPGA2140000-019) and the load backup feet (HPGA2140000-005), eight per assembly. Retract all load backup feet via adjusting bolt -007.
- 10.2.1.3.1 Insert each load backup beam assembly between the end plates and bolt in position. Check vertical alignment of the load backup feet with the fin ribs; adjust as needed by shimming the ends of the load backup beam assembly at the end plates.
- 10.2.1.3.2 Using adjusting screw -007, position each load backup foot so it just "bottoms out" on the fin rib. CHECK THAT ALL "LOAD FEET" ARE BOTTOMED OUT; DO NOT APPLY PRESSURE; Check that the -007 screw is approximately perpendicular to the fin rib edge, vertically.
- 10.2.1.4 Assemble the six sets of Pressure Feet (-039), eight per set. Use Ten Belleville springs per stack; adjust load nut to spring stack free height.
- 10.2.1.4.1 Retract all pressure feet to the maximum extent using adjusting screw, -017, and having negligible load on the Belleville springs.
- 10.2.1.4.2 Keep tie rods and spacers between the pressure feet loose.
- 10.2.1.4.3 Retract adjusting screw, -1001, to permit maximum travel of welded assembly -029. (Pressure pad on condenser or fin section of the heat pipe.)
- 10.2.2 Placement of heat pipes, solder shims and seals, and final bonding tool assembly and adjustment.
- 10.2.2.1 Orient the housing with bonding tool attached (10.2.1.3.2) such that the housing cylindrical axis is horizontal and Fin #1 is horizontal with its grooved side up.
- 10.2.2.2 Assign a set of solder preforms (9.4.9) to each of the heat pipe locations, 1A thru 1H. Locate in the case and fin groove with the "wire side of pre-form" positioned toward the TOP END OF HOUSING.

|     |       |                |             |
|-----|-------|----------------|-------------|
| REV | SIZE  | CODE IDENT NO. |             |
|     | A     | 30356          | HPGA2100310 |
|     | SCALE |                | SHEET 38    |

10.2.2.3 Hand form each solder preform as required to conform to its assigned heat pipe groove, 1A thru 1H.

10.2.2.4 With the hand formed solder preforms in place, check the fit-up of the heat pipes previously assigned to these locations, 1A thru 1H (5.2). Re-work by hand forming as required.

10.2.2.5 Place each heat pipe into an individual polyethylene bag together with its mating set of solder preform. Identify each bag by its assigned heat pipe number, a total of eight bags.

**CAUTION: DO NOT DEFORM IN HANDLING.**

10.2.2.6 Clean each heat pipe and mating set of solder preforms immediately before final assembly and soldering. **MAINTAIN IDENTITY.**

10.2.2.6.1 Ultrasonically clean using Freon TF or trichlorethylene.

10.2.2.6.2 Rinse with alcohol.

10.2.2.6.3 Blow dry with an inert gas.

10.2.2.6.4 Package in polyethylene bag. Identify bag with corresponding heat pipe location number.

10.2.2.7 Position the silicone rubber case/grooved fin fillet strip #1 in the mating root fillet of Fin #1.

10.2.2.8 Prepare and insert into each heat pipe groove the designated heat pipe and mating set of solder preforms, proceeding sequentially from position 1A thru 1H.

10.2.2.8.1 Apply a thin coat of Kester's #1544 flux to the tinned case and fin grooves.

10.2.2.8.2 Apply a thin coat of Kester's #1544 flux to all surfaces of the two mating solder preforms using clean cotton swabs. Insert the fluxed preform into the corresponding case groove and fin groove.

10.2.2.8.3 Apply a thin coat of Kester's #1544 flux to the heat pipe (joint mating surfaces only).

|     |       |                |             |
|-----|-------|----------------|-------------|
| REV | SIZE  | CODE IDENT NO. |             |
|     | A     | 30056          | HPGA2100310 |
|     | SCALE |                | SHEET 39    |

- 10.2.2.8.4 Fit the evaporator end and condenser end slip over silicone sponge rubber plugs (6.2. and 6.3) over the respective closures at the ends of the heat pipe.
- 10.2.2.8.5 Insert the fluxed heat pipe with end plugs into the proper position in the silicone rubber fin fillet strip and "bottom" on the fluxed solder preform. Check visually for proper fit-up. Rework, including re-cleaning and re-fluxing, as required.
- 10.2.2.8.6 Install the balance of the heat pipes on Fin #1, Nos. 1B thru 1H by repeating operations 10.2.2.8.1 thru 10.2.2.8.5.
- 10.2.2.9 Install the required thermocouples, as listed in Table I (Section 10.1.4) by the procedure specified in Section 10.1 thru 10.1.4.
- 10.2.2.10 Install and adjust pressure foot assembly (10.2.1.4).
- 10.2.2.10.1 Carefully lay the pressure foot assembly on top of the pre-placed heat pipes such that each heat pipe is approximately in the center of its silicone rubber hold down pad.
- 10.2.2.10.2 Apply sufficient hand pressure to the loading beam, -019, to align the beam with the endplate mounting holes. Insert shims at the end(s) of the "beam" as required to maintain lateral alignment of the beam and pressure feet. Install and tighten the beam end mounting bolts.
- 10.2.2.10.3 Adjust the angular position of the fin segment of the pressure foot, using adjusting screw, -1001, so as to obtain uniform deformation of this segment of the silicone rubber pressure pad.
- 10.2.2.10.4 Adjust the hold down pressure on each heat pipe as follows: (1) check that load nut on spring stack is adjusted to stack free height, zero pre-load, (2) advance load screw, -017, until rubber pressure pad contacts heat pipe evaporator section (Case), (3) re-check angular adjustment fin segment screw, -1001, for uniform pad contact, and (4) apply load by advancing load screw 0.40". THESE OPERATIONS MUST BE OBSERVED AND APPROVED BY ENGINEERING.
- 10.2.2.10.5 Check thermocouples for proper position of the bead, proper identification, and electrical continuity. Harness thermocouple leads as required to maintain integrity during subsequent handling of the housing.
- 10.2.2.11 Sequentially install solder preforms, heat pipes and thermocouples on Fin Nos. 2 thru 6 by repetition of operations 10.2.2.1 thru 10.2.2.10.5 for each fin.

|       |  |      |                |             |    |
|-------|--|------|----------------|-------------|----|
|       |  | SIZE | CODE IDENT NO. |             |    |
|       |  | A    | 30056          | HPGA2100310 |    |
| REV   |  |      |                |             |    |
| SCALE |  |      |                | SHEET       | 40 |

11.0 SOLDERING OPERATION

11.1 Install housing with attached fixturing and instrumentation (from 10.2.2.11) into the heat pipe soldering chamber, HPGA2140019.

11.1.1 Insert support bar through the T/V chamber test suspension bracket assembly (mounted on top of the housing) and engage the locator pin.

11.1.2 Carefully lift the assembly by the support bar (protect electrical and thermocouple leads during handling) and slowly lower into the chamber until the support bar is positioned on the brackets attached to the sides of the chamber.

11.1.3 Connect the core heater (i. e. heat pipe installation heating fixture) to its electrical control circuit. Check for proper heater resistance.

11.1.4 Connect all the thermocouples to the external multi-point switch maintaining proper identification of each couple and make a final check on the operation of each.

11.2 Solder thermal cycle

11.2.1 The time-power (electrical) profile for both the chamber and core heater as established in prior "Dummy Runs" shall be employed. Minor changes may be made with the concurrence of Engineering.

11.2.2 Heat to 400°F ±10°F at approximately 20-30°F/minute. Hold at 400°F to stabilize temperatures, 15 minutes maximum.

11.2.3 Heat to a nominal temperature of 480°F at approximately 30°F/minute. Continuously scan temperatures at all TC locations. All thermocouple locations shall indicate a minimum temperature of 460°F for a minimum time of one minute. The maximum permissible temperature at any location is 510°F. The total time at temperatures greater than 450°F (any location) shall be recorded, and if consistent with the above minimum and maximum temperature shall be held to ten minutes or less. After the above cited conditions are satisfied, turn off power and cool in the chamber.

11.2.4 After the maximum temperature has dropped to 350°F, remove the chamber cover and cool to room temperature.

|       |      |                |             |
|-------|------|----------------|-------------|
|       | SIZE | CODE IDENT NO. |             |
|       | A    | 30356          | HPGA2100310 |
| REV   |      |                |             |
| SCALE |      |                | SHEET 41    |

11.3 Post-soldering procedure

11.3.1 Disconnect the core heater electrical leads (ALL POWER OFF).

11.3.2 Disconnect all thermocouples.

11.3.3 Lift the housing and attached tooling from the chamber and set in vertical position.

11.3.4 Remove support bar.

11.3.5 Release the seating force from each heat pipe until the load foot is loose.

11.3.6 Retract, approximately 1/16 - 1/32", each pressure foot (support on fin ribs).

11.3.7 Remove all longitudinal square tube support beams (and attached load feet and pressure feet) from the tool end plates by unbolting both ends.

11.3.8 Remove the T/V chamber test suspension bracket, core heater and end plates.

11.3.9 Remove all thermocouples.

|  |     |                  |                                |             |    |
|--|-----|------------------|--------------------------------|-------------|----|
|  | REV | SIZE<br><b>A</b> | CODE IDENT NO.<br><b>30056</b> | HPGA2100310 |    |
|  |     | SCALE            |                                | SHEET       | 42 |



12.0 CLEANING, INSPECTION AND FINISHING

12.1 **Cleaning**

12.1.1 Remove all residual flux by scrubbing with Kester's AP-20 flux remover. Use clean bristle brush and pads are required.

12.1.2 Wash with warm water.

12.1.3 Rinse with alcohol.

12.1.4 Blow dry with inert gas.

12.2 **Inspection**

12.2.1 Visually inspect. Identify areas where excess solder should be removed and surface fillet voids which require "filling."

12.3 **Finishing**

12.3.1 Hand grind to remove excess solder as indicated in inspection and approved by Engineering.

12.3.2 Use ECCOBOND 58C (Emmerson & Cuming, Inc.) conductive cement to fill voids as designated by Engineering. Alternate techniques may be employed only with specific Engineering approval.

12.3.2.1 Use inert gas to blow any foreign particles from the void. Use spatula or other suitably shaped tool to hand fill the void with Eccobond 58-C cement (thinning of cement as directed by Manufacturing is permitted). Drive off volatiles and "semi-cure" by heating at 170°F ±10° for eight hours minimum.

|  |     |       |                |             |
|--|-----|-------|----------------|-------------|
|  | REV | SIZE  | CODE IDENT NO. |             |
|  |     | A     | 30356          | HPGA2167310 |
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13.0 DATA REQUIREMENTS

13.1 Tabulate heat pipe serial number versus fin/case groove identification number.

13.2 Temperature/time plots for the solder thermal cycle shall be continuously recorded, for the specific thermocouple locations defined in Section 10.1, using a L&N speedomax, six point, 0-1200°F strip chart recorder. Identify time scale, and location identity of each indicated thermocouple (on the chart).

13.3 During the solder thermal cycle record the total time at temperature above 450°F (maximum time for any thermocouple location).

|     |  |       |                |             |          |
|-----|--|-------|----------------|-------------|----------|
|     |  | SIZE  | CODE IDENT NO. |             |          |
|     |  | A     | 30056          | HPGA2100310 |          |
| REV |  |       |                |             |          |
|     |  | SCALE |                |             | SHLET 11 |

APPENDIX A

Heat pipe location assignments for build of HPG S/N 02 are summarized in Table A-1.

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TABLE A-1. HEAT PIPE LOCATION ASSIGNMENTS - 150 WATT HPG

| <u>Fin</u> | <u>Groove</u> | <u>Heat Pipe</u> | <u>Fin</u> | <u>Groove</u> | <u>Heat Pipe</u> | <u>Fin</u> | <u>Groove</u> | <u>Heat Pipe</u> |
|------------|---------------|------------------|------------|---------------|------------------|------------|---------------|------------------|
| 1          | A             | 2A01             | 3          | A             | 2A33             | 5          | A             | 3A22             |
|            | B             | 2A03             |            | B             | 2A34             |            | B             | 3A24             |
|            | C             | 2A04             |            | C             | 2A35             |            | C             | 4A01             |
|            | D             | 2A05             |            | D             | 3A01             |            | D             | 4A02             |
|            | E             | 2A06             |            | E             | 3A02             |            | E             | 4A03             |
|            | F             | 2A08             |            | F             | 3A03             |            | F             | 4A04             |
|            | G             | 2A11             |            | G             | 3A04             |            | G             | 4A05             |
|            | H             | 2A16             |            | H             | 3A05             |            | H             | 4A06             |
| 2          | A             | 2A17             | 4          | A             | 3A06             | 6          | A             | 4A07             |
|            | B             | 2A18             |            | B             | 3A08             |            | B             | 4A08             |
|            | C             | 2A19             |            | C             | 3A12             |            | C             | 4A11             |
|            | D             | 2A22             |            | D             | 3A13             |            | D             | 4A12             |
|            | E             | 2A24             |            | E             | 3A14             |            | E             | 4A13             |
|            | F             | 2A27             |            | F             | 3A16             |            | F             | 4A14             |
|            | G             | 2A28             |            | G             | 3A18             |            | G             | 4A16             |
|            | H             | 2A30             |            | H             | 3A21             |            | H             | 4A18             |

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APPENDIX D

MCL Report: Subject, Scanning Electron Microscopy (SEM)  
Electron-Excited, Energy-Dispersive X-ray Analysis (E/EDAX),  
and X-ray Diffraction (XRD) Examination of a Failed 1/4" ID  
Ebonol C Coated Copper Pipe Described as a Heat Exchanger  
Element, Job No. 7412042, December 30, 1974.

**MCL**

**X-Ray Diffraction—Scanning & Transmission Electron Microscopy**

DECEMBER 30, 1974

JOB NO. 7412042

SUBJECT      SCANNING ELECTRON MICROSCOPY (SEM),  
ELECTRON-EXCITED, ENERGY-DISPERSIVE  
X-RAY ANALYSIS (E/EDAX), AND X-RAY  
DIFFRACTION (XRD) EXAMINATION OF A  
FAILED 1 1/4" ID EBONOL C COATED COPPER  
PIPE DESCRIBED AS A HEAT EXCHANGER  
ELEMENT

EXPERIMENTAL PROCEDURE AND RESULTS

SEM AND E/EDAX - AN OPENED SECTION OF THE PIPE, A PIECE OF THE CU-BE SPRING, AND SOME OF THE BLACK RESIDUE WERE MOUNTED ON A HIGH PURITY GRAPHITE WAFER, COATED WITH APPROXIMATELY 200Å OF CARBON, AND EXAMINED IN THE SEM USING A 30 KV ACCELERATING VOLTAGE AND A TILT ANGLE OF 35-45°. (THE CARBON COATING PROVIDES AN "INVISIBLE" ELECTRICALLY CONDUCTING SURFACE WHICH IS NECESSARY FOR EFFICIENT SEM EXAMINATION, AND DOES NOT INTERFERE WITH THE SEM OR E/EDAX RESULTS.) THE SAMPLE WAS EXAMINED USING BOTH SECONDARY AND BACK-SCATTERED ELECTRON EMISSION.

ALL ELEMENTS WITH ATOMIC NUMBERS GREATER THAN EIGHT (OXYGEN) ARE DETECTED SIMULTANEOUSLY WITH E/EDAX; ELEMENTS WITH LOWER ATOMIC NUMBERS ARE NOT DETECTED. THIS ENABLES THE DETECTION OF ELEMENTS NOT NECESSARILY EXPECTED CONCURRENTLY WITH THE ELEMENTS OF PRINCIPAL INTEREST.

BOTH THE PIPE AND THE SPRING HAD A CHARACTERISTIC AMORPHOUS CORROSION PRODUCT SURFACE. THE BLACK RESIDUE ALSO HAD AN AMORPHOUS APPEARANCE CHARACTERISTIC OF A CORROSION PRODUCT (SEE ENCLOSED MICROGRAPHS).

E/EDAX ANALYSIS INDICATED THE FOLLOWING:

|          | <u>COATING ON<br/>SPRING</u> | <u>COATING ON<br/>PIPE</u> | <u>BLACK<br/>RESIDUE</u> |
|----------|------------------------------|----------------------------|--------------------------|
| COPPER   | MAJOR                        | MAJOR                      | MAJOR                    |
| COBALT   | TRACE                        | VERY MINOR                 | ND                       |
| IRON     | TRACE                        | VERY MINOR                 | VERY MINOR               |
| TITANIUM | TRACE                        | ND                         | TRACE                    |
| CHLORINE | ND                           | TRACE                      | ND                       |
| SULFUR   | TRACE                        | VERY MINOR                 | TRACE                    |
| SILICON  | TRACE                        | VERY MINOR                 | TRACE                    |

SEE ENCLOSED E/EDAX SPECTRA.

XRD (DIFFRACTOMETRY) - A PORTION OF THE BLACK RESIDUE WAS LOADED INTO A SMALL GLASS TRAY FOR X-RAY DIFFRACTOMETRY ANALYSIS. THE SAMPLES WERE THEN EXPOSED TO COPPER RADIATION OVER THE BRAGG REFLECTION ANGLE OF  $7^{\circ}$  TO  $68^{\circ}$  TWO THETA USING A CURVED (FOCUSING) GRAPHITE CRYSTAL DIFFRACTED BEAM MONOCHROMATOR. THE PEAKS ON THE PATTERN WERE RELATIVELY SHARP INDICATING THAT THE SAMPLE WAS WELL CRYSTALLIZED. THE BRAGG REFLECTIONS OF THE CRYSTALLINE FRACTION OF THE SAMPLE WERE COMPATIBLE WITH THE STRUCTURES AND THE VERY APPROXIMATE CONCENTRATIONS OF:

|                                   |           |
|-----------------------------------|-----------|
| Cu <sub>2</sub> O (CUPROUS OXIDE) | ~70 VOL % |
| CuO (CUPRIC OXIDE)                | ~20 VOL % |
| Cu (COPPER)                       | ~10 VOL % |

IN ADDITION, THE BACKGROUND SHAPE RESULTING FROM INCOHERENT X-RAY SCATTERING INDICATED A VERY MINOR NON-CRYSTALLINE FRACTION.

THE VOLUME PERCENTAGES OF THE CRYSTALLINE FRACTION SHOWN ABOVE WERE CALCULATED FROM THE RATIOS OF THE PRINCIPAL BRAGG REFLECTION PEAK HEIGHTS AND ARE CONSIDERED TO BE ACCURATE (95% CONFIDENCE LEVEL) WITHIN  $\pm 35$  VOLUME PERCENT OF THE AMOUNT PRESENT.

#### COMMENTS

- THE FAILURE APPEARS TO BE OF A CORROSION TYPE. THIS OBSERVATION IS SUPPORTED BY THE RELATIVELY HIGH CONCENTRATION OF  $\text{Cu}_2\text{O}$  (COPPER RICH OXIDE) IN THE BLACK RESIDUE.
- SULFUR IS THE MOST SUSPECT ELEMENT WHICH MAY BE PLAYING A ROLE IN THE CORROSION MECHANISM. IT SHOULD BE NOTED THAT THE SULFUR LEVEL IS SIGNIFICANTLY HIGHER IN THE PIPE COATING THAN IT IS IN THE BLACK RESIDUE. THE SOURCE OF THE SULFUR MAY BE IMPROPER CLEANING DURING FABRICATION.
- VERY LOW LEVELS OF CHLORINE WERE ALSO OBSERVED IN THE CORROSION PRODUCT. BECAUSE OF THE VERY LOW LEVEL, THE CHLORINE IS ASSUMED TO PLAY A MUCH LESS SIGNIFICANT ROLE IN THE CORROSION MECHANISM.

#### SUGGESTIONS

- WE SUGGEST THAT THE CHEMISTRY OF THE CORROSION CAN BE ELUCIDATED BY ANALYZING THE WATER FROM A UNIT BY X/EDAX AND THE RESIDUE OF THE WATER AFTER EVAPORATING TO DRYNESS BY E/EDAX.
- WE ALSO SUGGEST THAT A NEW UNUSED UNIT BE EXAMINED BY SEM AND E/EDAX.



Black Residue

Expanded  
Vertical  
Scale

Black Residue

No. 8999  
1000X

No. 9000  
2000X

E/DDAX of Spring

Matrix

Cu Si S Fe Co Cu

Oxide  
Coating

Cu Si S Ti Fe Cu

Spring

No. 9004

350X

No. 9005

1000X

Pipe

Pipe

No. 9003  
1000X

Cu Si S Cl

Pipe M

No. 9001  
20X

No. 9002  
40X

APPENDIX E

Specification: HPGA2180500, Heat Pipes for 150 Watt  
SNAP 19 HPG, Isotopes, Nuclear Systems Division,  
November 17, 1972



SECURITY UNCL. CONF. SECRET  
REVIEW DI RD GP. 1234

CLASSIFIER

REVISIONS: SEE SHEET 243

REV LEVEL

DWG

THIS  
SHEET

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A

HPGA2180500

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S/N 02

HPGA2180000

HPG

EFFECTIVE ON

CALC  
WT

DASH  
NUMBER

NEXT ASSEMBLY

USED ON

NEXT  
ASSY

FINAL  
ASSY

TEST

APPLICATION

QTY REQ'D

CONTRACT NO.

DRAWN BY *M. Kreitz* DATE 11-17-72

CHECKER *M. Hall* 11-17-72

STRESS ENGR.

WT. ENGR.

MATL. ENGR.

RELIABILITY

GR. ENGR. *M. Hall* 11-17-72

PROJECT  
ENGR.

ISOTOPES

NUCLEAR SYSTEMS DIVISION

A TELEDYNE COMPANY

Baltimore Maryland

SPECIFICATION: HEAT PIPES FOR 150 WATT  
SNAP 19 HPG

SIZE

A

CODE IDENT NO.

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HPGA2180500

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# REVISIONS

| SYM | PAGE | DESCRIPTION                                                                                                                  | DATE     | APPROVED |
|-----|------|------------------------------------------------------------------------------------------------------------------------------|----------|----------|
| A   | 8    | Para. 3.4.1.1. Added ..... and the evaporator elevation shall be 1 inch over the length of the pipe.                         | 11/17/72 | QAF      |
|     | 11   | Rewrote para. 4.4.2.2.                                                                                                       |          |          |
|     | 12   | Modified para. 4.4.2.3.<br>Redefined life test duration in 4.4.2.4.                                                          |          |          |
|     | 1    | ADDED EFFECTIVITY, N.A., USED ON. & REV. SMT. NO.                                                                            |          |          |
| B   | 9    | Para. 3.4.1.4 - Replaced TBD with 1.0.<br>Para. 3.4.2.1 - Replaced TBD with .... As defined in Appendix A.                   | 12/1.72  | QAF      |
|     | 11   | Para. 4.4.1.4 - Rewrote second sentence as follows: With .... 1.0 inch above condenser, 60 watts minimum shall be ....350°F. |          |          |
|     | 14   | Added Appendix A.                                                                                                            |          |          |
| C   | 7    | Para. 3.2.14 added " the amount of <u>fluid</u> ocharge                                                                      | 3/2/73   | QAF      |
|     | 7    | Para. 3.2.2 modified with regard to internal gas quantities                                                                  |          |          |
|     | 7    | Para. 3.2.3 added $12 \pm 1$ & $400 \pm 10^{\circ}\text{F}$ for TBD and replaced "operated in a reflux mode" - with "baked". |          |          |
|     | 8    | Para. 3.3.1.3 added $34.4 \pm 1.6$ gms. for TBD                                                                              |          |          |
|     | 8    | Para 3.4.1.1 modified                                                                                                        |          |          |
|     | 9    | Para. 3.4.2.1 added " the heat pipe .....as specified in Appendix A                                                          |          |          |
|     | 9    | Para 3.4.2.2 deleted "10 <sup>-11</sup> torr" and replaced with "High vacuum"                                                |          |          |
|     | 11   | Para 4.4.1.2 added "..... at room temperature ....."                                                                         |          |          |
|     | 11   | Para. 4.4.1.3 added "samples for this ..... between buyer and seller"                                                        |          |          |
|     | 12   | Added Para. 4.4.3                                                                                                            |          |          |
|     | 12   | Para. 4.4.2.4 - deleted " .....in a heat pipe mode .... "                                                                    |          |          |

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SCALE

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| 7     | C       |       |     |       |     |
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| 12    | A, C    |       |     |       |     |
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HPGA2180500

C

**SCALE**

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**A**

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**SPECIFICATION: HEAT PIPES FOR 150 WATT SNAP 19 HPG**

**1.0      SCOPE**

This specification establishes the requirements for design, performance and testing of the heat pipes which augment the heat rejection fins of the 150 watt SNAP 19 HPG.

**2.0      APPLICABLE DOCUMENTS**

The documents listed below, with amendments or revisions indicated, form a part of this specification.

|             |                                 |
|-------------|---------------------------------|
| WW-T-799    | Tube, Copper Seamless           |
| QQ-C-502    | Copper Rods and Shapes          |
| RR-W-360    | Industrial Wire Fabric          |
| QQ-W-343D   | Electrical Wire                 |
| QQ-C-530    | Beryllium Copper Alloy          |
| MIL-C-45662 | Calibration System Requirements |

**Applicable Drawings**

|              |                                  |
|--------------|----------------------------------|
| HPGA 2180000 | Isotopes Interface Drawing       |
| 0063-1001    | Dynatherm Fin and Block Assembly |

**3.0      REQUIREMENTS**

**3.1      Item Definition**

The heat pipe is a nearly isothermal device consisting of a closed chamber or tube, a working fluid and a wick. Heat is transported by evaporation of the fluid in the heat source region, vapor flow from the source region to the sink region, and condensation

|     |          |                |             |
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of the vapor at the heat sink region followed by return of the liquid through the wick to the heat source region. This specification defines heat pipes which are to be installed in the heat rejection subsystem of a 150 watt SNAP 19 HPG. The purpose of the heat pipes is to increase the effectiveness of the heat rejection fins of the generator. The heat pipes are defined by this specification and Isotopes Interface Drawing HPGA2180000.

### 3.2 Materials, Parts and Processes

#### 3.2.1 Materials and Parts

Standard parts and components shall be used wherever they are suitable for the purpose.

##### 3.2.1.1 Heat Pipe Material

The heat pipe tube material shall be alloy 122 copper per WW-T-799. End caps and fill tube shall be alloy 102 copper per QQ-C-502.

##### 3.2.1.2 Wicking Material

The wicking material shall be square weave copper wire cloth per RR-W-360. The wire material shall be electrolytic copper (99.9% pure) per QQ-W-343D.

##### 3.2.1.3 Retainer Spring

The wick retainer spring shall be made from Beryllium Copper Alloy 25 (172) per QQ-C-530.

##### ⊙ 3.2.1.4 Working Fluid

The working fluid shall be distilled water with the following minimum purity:

|                                          |                          |
|------------------------------------------|--------------------------|
| Total solids                             | < 4 ppm                  |
| Maximum conductivity                     | < $8 \times 10^6$ mho/cm |
| Combined dissolved<br>Cu, Na, Mg, Ca, Fe | < 1 ppm                  |

|       |          |                |             |
|-------|----------|----------------|-------------|
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The amount of fluid charge shall be specified on the vendor drawings after completion of development tests.

© 3.2.2 Surface Treatment

All interior surfaces of the heat pipe shall be treated such that an essentially zero wetting angle is achieved between the working fluid and the metal surfaces. This surface condition shall be sufficiently stable in a water vapor atmosphere up to 500°F so that all heat pipe performance requirements of this specification shall be met or exceeded after soldering the heat pipes to the generator housing and fins.

© 3.2.3 Heat Pipe Outgassing After Charging

After charging the heat pipe assembly with the working fluid, it shall be baked for at least  $12 \pm 1$  hours at  $400 \pm 10^\circ\text{F}$ . If any gas has generated during that period, it shall be bled off prior to final closure.

3.2.4 Welding After Heat Pipe Charging - Seal Off

Absolute closure of the heat pipe (following charging) shall be achieved by welding the pinch-off region of fill tube in addition to the cold weld achieved during pinch-off.

3.3 Design

The heat pipe shall be designed to meet or exceed the requirements of this specification in all operating, non-operating, and storage conditions, including the space environment of zero g.

3.3.1 Physical Description

The heat pipes shall be fabricated to meet the requirements shown in Isotopes drawing HPGA2180000 and this specification.

|          |           |                         |             |
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### 3.3.1.1 Installation

The heat pipes are to be installed by Teledyne Isotopes into the HPG generator and will be required to withstand at least one thermal cycle between room temperature and 500°F and meet the performance requirements of this specification after that cycle.

### 3.3.1.2 Interchangeability

The heat pipes shall be identified by tagging with the applicable drawing number and a serial number. Heat pipes having the same drawing number shall be interchangeable with regard to form, fit and function.

### ③ 3.3.1.3 Weight

The total weight of the heat pipe shall be  $34.4 \pm 1.6$  grams

### 3.3.2 Construction

The heat pipes shall be designed to withstand the internal pressures of the working fluid at all operating, non-operating and processing temperatures without loss of seal integrity.

### 3.3.2.1 Pressure Safety Factor

The heat pipe shall be designed to withstand an internal pressure of 1360 psig at 500°F.

### 3.4 Performance

### 3.4.1 Functional Performance

### ① 3.4.1.1 Heat Transport

③ The heat pipe shall be capable of transporting 60 watts minimum, applied over a maximum of two (2) inches near one end and removed from the remainder of the heat pipe. Heat input and removal shall be through aluminum saddles covering no more than one half of the pipe circumference and the evaporator elevation shall be 1 inch over the length of the pipe. During all demonstration and acceptance testing the heat pipe shall be oriented with regard to heat input and removal in a manner identical to that which will exist during operation.

|          |           |                         |             |
|----------|-----------|-------------------------|-------------|
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#### 3.4.1.2 Temperature Range

The heat pipe shall meet the performance specified in 3.4.1.1 over the temperature range from 150 to 350°F.

#### 3.4.1.3 Heat Pipe Conductance

When operated with a heat input of 60 watts, the overall temperature difference shall not exceed 24°F. The overall temperature difference is defined as the difference between average evaporator and average condenser temperatures measured on the outside of the heat pipe. The above requirement corresponds to a heat pipe conductance of 2.5 watts/°F.

#### Ⓑ 3.4.1.4 Test Environment Performance

The heat pipe shall perform within the specified limits in a one "g" environment with the evaporator end of the pipe raised at least 1.0 inch above the condenser end.

#### 3.4.2 Environment

The heat pipe shall be capable of meeting the performance requirements after exposure to the following environments:

- Ⓒ 3.4.2.1 Dynamic Environment: The heat pipe shall be capable of meeting performance requirements in section 3.4.1 except it shall be at 0.25 in. evaporator elevation during and immediately after exposure to dynamic environments as specified in Appendix A

#### Ⓒ 3.4.2.2 Pressure

External pressure on the heat pipe will range from atmospheric during ground activities to high vacuum in orbit.

#### 3.4.2.3 Temperature

The heat pipe shall be capable of withstanding and operating within the conditions and temperature ranges specified in paragraphs 3.3.1.1, and 3.4.1.2.

|          |       |                |             |
|----------|-------|----------------|-------------|
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#### 4.0 QUALITY ASSURANCE PROVISIONS

##### 4.1 General

Quality Assurance inspections and tests shall be performed on the heat pipes in accordance with MIL-Q-9858. Test equipment used to inspect and/or provide quantitative data shall be calibrated in accordance with MIL-C-45662.

##### 4.2 Material Certification

Purchased raw materials shall be accompanied with chemical and/or physical test results.

##### 4.3 Inspection

Sufficient inspections shall be performed to ensure that all parts and assemblies conform to all applicable drawings and this specification with respect to all details including workmanship, finish, construction and identification. Teledyne Isotopes Quality Control Representatives shall have access to witness and review operations and procedures performed by Dynatherm Corporation related to fabrication of heat pipes covered by this specification.

##### 4.4 Tests

All tests shall be conducted in accordance with Teledyne Isotopes approved Test Procedures.

###### 4.4.1 Acceptance Testing

Each deliverable heat pipe shall be subjected to the following acceptance tests:

###### 4.4.1.1 Helium Leak Test Prior to Pinch-Off

After assembly but prior to charging with the working fluid, the heat pipe shall be helium leak tested with a leak detection sensitivity of at least  $10^{-9}$  std cc/sec.

Acceptance limit shall be a total leak rate of  $10^{-8}$  std cc/sec or less.

|       |      |                |             |
|-------|------|----------------|-------------|
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③ 4.4.1.2 Proof Pressure Testing

The heat pipe shall be proof pressure tested to 1000 psig at room temperature prior to charging.

③ 4.4.1.3 Leak Test of Final Closure Weld

The leak tightness of the final closure weld shall be verified through the leak testing to a sensitivity of at least  $10^{-9}$  std cc/sec. Acceptance limit shall be a total leak rate of  $10^{-8}$  std cc/sec or less. Samples for this test shall be destructively tested and will be randomly selected from the stock fabricated to meet delivery requirements. Number of samples shall be mutually agreed upon between buyer and seller.

③ 4.4.1.4 Thermal Performance Testing

The thermal performance of the heat pipes:

shall be measured in the following manner. With the heat pipe vaporator elevated 1.0 inch above the condenser, 60 watts minimum shall be transported with the condenser section temperature at 150°F and 350°F.

4.4.2 Qualification Testing

Two (2) Prototype Units, identical to deliverable units shall be subjected to the following qualification tests in addition to the acceptance tests specified in 4.4.1.

4.4.2.1 Thermal Cycling

After pinch-off, the prototype heat pipes shall be subjected to a thermal soak at 500°F for at least 30 minutes.

① 4.4.2.2 Thermal Performance Mapping

The prototype heat pipes shall each be bonded in a simulated section of the RTG housing and fin in accordance with DTM drawing 063-1001.

Temperature gradients will be measured along the fin section assembly at power input increments of 10 watts starting at 30 watts until burnout is achieved. Measurements will be obtained for operating temperatures of 150°F and 350°F and at elevations of 0, 1 and 2 inches. At the horizontal orientation, maximum power at which the heat pipe can recover from burnout will be determined at both temperatures.

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|          | SIZE  | CODE IDENT NO. |             |
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(A) 4.4.2.3 Performance Testing with Simulated Housing/Fin Section

Sufficient performance testing shall be done on the bonded assembly to verify an overall conductance of at least 2.5 watts/°F as measured between the simulated heater block and the simulated fin at locations immediately adjacent to the heat pipe.

(A) 4.4.2.4 Life Testing

Following completion of 4.4.2.3, both prototype units shall be operated with a heat transport of  $50 \pm 10_0$  watts at  $315 \pm 10^\circ\text{F}$  to evaluate life performance of the units. Failure shall be defined as either a rupture of the heat pipe envelope, or failing to transport 50 watts, or a continuing decrease of the overall conductance at such a rate that an extrapolation to 5 years would render the heat pipe useless for the HPG application at that time. At the end of 10,000 hours the life test shall be terminated.

(C) 4.4.3 Radiographic Inspection

Each deliverable heat pipe shall be accompanied by a full length radiograph sufficient to define the location and orientation of the internal components (wick and helical spring) of the heat pipe.

5.0 PREPARATION FOR DELIVERY

5.1 Handling

The vendor shall protect materials during all phases of fabrication, processing and storage to prevent handling damage and foreign material contamination.

5.2 Storage

Materials to be stored shall be protected against deterioration and damage. Procedures shall be generated and utilized to ensure the positive identification of materials.

|          |       |                |             |    |
|----------|-------|----------------|-------------|----|
|          | SIZE  | CODE IDENT NO. |             |    |
|          | A     | 30856          | HPGA2180500 |    |
| REV<br>C | SCALE |                | SHEET       | 12 |

5.3 Marking

All markings on shipping containers shall be clearly legible and may be applied by stencil, rubber stamp or lacquer over coated gummed label.

5.4 Packaging

Packaging procedures and instructions shall be utilized and provide for protection to materials while at the vendor's plant, during transportation to destination, and upon arrival at destination.

5.5 Packing

The vendor shall provide for cushioning, blocking, bracing or bolting, as applicable to prevent undesired free movement within containers and physical damage due to transmission of shock and vibration.

|     |       |                |              |
|-----|-------|----------------|--------------|
|     | SIZE  | CODE IDENT NO. |              |
|     | A     | 30856          | HPGA 2180500 |
| REV | SCALE |                | SHEET 13     |

## APPENDIX A

### I. SINUSOIDAL VIBRATION LEVEL

- A. Acceptance Level: Sinewave vibration in each of three (3) orthogonal axes (Xy, Y, Z) as defined by the following parameters:

| <u>Vibration Level</u>    | <u>Frequency (Hz)</u>                      |
|---------------------------|--------------------------------------------|
| 0.4 inch double amplitude | 5-10                                       |
| $\pm 2.0$ g's (0-peak)    | 10-2000                                    |
|                           | } constant sweep<br>rate from<br>0-2000 Hz |

### II. RANDOM VIBRATION LEVELS

- A. Acceptance Level: Random vibration in each of three (3) orthogonal axes (X, Y, Z) as defined by the following parameters:

| <u>Power Spectral Density</u> | <u>Frequency (Hz)</u> |
|-------------------------------|-----------------------|
| Increasing @ 3 db/octave      | 10-900                |
| $0.075 \text{ g}^2/\text{Hz}$ | 900-1400              |
| Decreasing @ 15 db/octave     | 1400-2000             |

Test duration: 60 seconds per axis

Overall level: 9.54 g's (RMS)

- B. Qualification Level: Random vibration in each of three (3) orthogonal axes (X, Y, Z) as defined by the following parameters:

| <u>Power Spectral Density</u> | <u>Frequency (Hz)</u> |
|-------------------------------|-----------------------|
| Increasing @ 3 db/octave      | 10-900                |
| $0.1875 \text{ g's/Hz}$       | 900-1400              |
| Decreasing @ 15 db/octave     | 1400-2000             |

Test duration: 3 minutes per axis

Overall level: 15.1 g's (RMS)

SIZE

CODE IDENT NO.

A

30856

HPGA2180500

REV

B

SCALE

SHEET 14

### III. ACCELERATION LEVEL

- A. Acceptance Level: The test specimen shall be tested successively in each direction along each of its three (3) mutually perpendicular axes. An acceleration of 10 g's longitudinal and 6.25 g's lateral applied simultaneously shall be attained, stabilized, and maintained for a period of not less than three (3) minutes or more than four (4) minutes.

### IV. SHOCK LEVEL

- A. Acceptance Level: The test specimen shall be subjected to three 775 g shocks, or the equivalent shock spectrum shown in Figure 1, in each direction along each of its orthogonal axes for a total of 18 shocks. Time to peak of each shock shall be 0.2 milliseconds,  $\pm 0.1$  milliseconds, half cycle sine.
- B. Qualification Level: The test specimen shall be subjected to three 15 g shocks in each direction along each of its three mutually perpendicular axes for a total of 18 shocks. Time to peak of each shock shall be 11 milliseconds  $\pm 10\%$ , time of decay shall be equal to or less than 2 milliseconds (half sine wave). The equivalent shock spectrum, see Figure 2, may be substituted for the half sine wave. Repeat the acceptance levels above for second part of test.

### V. ACOUSTIC NOISE LEVEL

- A. Qualification Level: The test specimen shall be subjected to the following sound pressure levels:

|          |       |                |             |
|----------|-------|----------------|-------------|
|          | SIZE  | CODE IDENT NO. |             |
|          | A     | 30856          | HPGA2180500 |
| REV<br>B | SCALE | SHEET 15       |             |

1/3 Octave Band  
Center Frequency  
(Hz)

40  
50  
63  
80  
100  
125  
160  
200  
250  
315  
400  
500  
630  
800  
1000  
1250  
1600  
2000  
2500  
3150  
4000  
5000  
6300  
8000

1/3 Octave Band  
Sound Pressure Level  
(db)\*

124  
125.5  
127  
129  
130.5  
131.5  
132.5  
133.5  
134  
134.5  
134.5  
134  
133.5  
133  
132  
131  
129.5  
128.5  
126.5  
125  
123  
121.5  
120  
118

\*db (Re: 0.0002 dynes/cm<sup>2</sup>)

Test Duration: 2 minutes

Overall Level: 145 db

SIZE

A

CODE IDENT NO.

30856

HPGA 2180500

REV

B

SCALE

SHEET 16



FIGURE #1  
SHOCK RESPONSE SPECTRUM  
40 g HALF SINE - 18 MS  
(Q = 10)

TES-SNSO-3-30

APPENDIX F

Investigation of Surface Treatments for Copper Heat Pipes,  
Battelle Columbus Laboratories, July 7, 1975.

REPORT

on

INVESTIGATION OF SURFACE TREATMENTS FOR  
COPPER HEAT PIPES

to

TELEDYNE ENERGY SYSTEMS

by

John E. Slater and Walter K. Boyd

July 7, 1975

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Columbus Laboratories  
505 King Avenue  
Columbus, Ohio 43201

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REPORT  
on  
INVESTIGATION OF SURFACE TREATMENTS FOR  
COPPER HEAT PIPES

by  
John E. Slater and Walter K. Boyd

July 7, 1975

INTRODUCTION AND BACKGROUND

Teledyne Isotopes Inc. uses heat pipes to transfer heat from thermoelectric generators to cooling fins. The heat pipes are constructed from CD Alloy No. 122 copper tubing. They are approximately 20 inches long, in the shape of an elongated 'L', with the short (near) end attached to the heat source and the far end to the fin. The piping diameter used has varied, but attention is now being paid to the use of 1/4 inch tubing, with a wall thickness of 0.021 inch.

The heat pipes contain triply distilled, deionized water, initially degassed; the water evaporates close to the heat source, condenses at the far end, and returns to the source by capillary action through spiral grooves cut into the inside wall of the tubing. The return of the water is also aided by an internal copper mesh 'wick' which extends the length of the pipe and half way around the circumference. It is held in place by a beryllium copper spring.

It is essential that the water should 'wet' the inner surface, for the capillary return action to be effective, and thus a proprietary surface treatment is given to the inner surface. This treatment goes under the trade name "Ebonal C"; a solution of oxidizing agent and strong base is applied to the copper, and a layer of oxide, supposedly  $\text{CuO}$ , is laid down. This layer apparently has excellent wetting characteristics.

The heat pipes operate essentially isothermally in use, at temperatures up to 400 F. Teledyne has tested pipes at various temperatures

for times running into thousands of hours. Although no failures have yet been observed at low (< 300 F) or high (400 F) temperatures, pipes operated in the temperature region of 300 - 325 F have failed after times of approximately 8000 hours. As far as can be ascertained from the preliminary failure analysis undertaken for Teledyne, failure occurred as a result of pitting, mainly close to the bend in the tube.

As a result of this failure, Teledyne initiated a research program at Battelle's Columbus Laboratories to provide information on the following:

- the cause of the pitting failure
- future avoidance of similar failures
- the long-term stability of the inner surface layer on the pipe
- the nature of any possible electrochemical reactions between heat pipe components under service conditions.

A two phase program was conceived as the approach of choice to provide this information. Phase I was to concentrate on work which more clearly defined the specific nature of the problem, and the theoretical stabilities and reactivities of the heat tube components and surfaces; Phase II would explore experimentally these theoretical predictions, and would pursue the optimum method of surface treatment. Only Phase I was described in detail, since Phase II would be markedly dependent on the results from Phase I.

Tasks in Phase I are:

#### Task 1. Literature Search

The objective of this search, aided by the computerized data bank at the Copper Data Center at Battelle, is to determine the current knowledge of high-temperature aqueous corrosion of copper. This search will be valuable in gathering together any previously published work which may have a bearing on the current problem.



## Task 2. Destructive Analysis of Heat Pipes

This task is designed to destructively examine both life-tested and untested heat pipes to investigate the nature of the corrosion, the stability of the film, and the action of any contaminants. Examinations are to include:

- Analysis of gas contained in pipes - to gain information on degassing efficiency and possible cathodic reactions
- Analysis of water, for pH,  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ , and  $\text{Cu}^{2+}$ , to give information on the rinsing efficiency after surface treatment, and on the nature of the corrosion reaction.
- Examination of inner surface of pipe (by optical and SEM methods), to determine nature and extent of corrosion and/or coating degradation.
- Surface analysis of coating (by EDAX/SEM) to determine concentration of any contaminants, having regard to the three prior investigations in this Task.

## Task 3. Computation of Elevated Temperature Pourbaix Diagrams for Copper

These diagrams are to be used to predict the thermodynamic stability and corrosion tendencies of copper in water at the temperatures of importance for the heat pipes, and particularly to give information on the stability of  $\text{CuO}$  and  $\text{Cu}_2\text{O}$ . Changes in such diagrams as a result of  $\text{Cl}^-$  or  $\text{SO}_4^{2-}$  ions are extremely important. The diagrams also allow predictions of possible cell activity between copper oxides laid down on the copper surface, and the substrate itself. In short, they allow further interpolation of results from Task 2.

Because of funding and time restrictions, only Task 1 and the first two parts of Task 2 have been initiated. This report presents the results of the investigation thus far.

## RESULTS AND DISCUSSION

### Task 1. Literature Survey: Corrosion of Cu, Cu<sub>2</sub>O, and CuO in High Temperature Water

The particular application of copper as the construction material for heat pipes involves the prolonged contact of Cu with relatively pure water at temperatures up to 400 F. The lay-down of a copper oxide coating on the inner surface of the pipe by the presently-used process brings further variables into the system: the presence of the oxide itself, and the presence of reactants not well rinsed after the oxide treatment. Since the treatment solution is strongly basic, the effect of higher pH's on corrosion must be considered. It appears that  $\text{Cl}^-$  ion may be a product of the reaction, and therefore its effect on corrosion must also be considered.

This literature survey was undertaken to acquire current knowledge on the behavior of copper in aqueous solutions under the above conditions. It was conducted using the resources of the Copper Data Center at Battelle's Columbus Laboratories, the partial literature search by Teledyne Energy Systems, and a further search by the Corrosion and Electrochemical Technology Section. Because of the short time available for this study, certain material was not available in time for the present survey. Additional material will be added to the survey in the final report.

#### Theoretical Studies

##### Thermodynamic Aspects

Any survey of the corrosion behavior of copper in aqueous solutions must consider the thermodynamic aspects of the situation. Figure 1 shows the Pourbaix diagram<sup>(1)</sup> for the copper/water system at 25 C, considering Cu, Cu<sub>2</sub>O and CuO to be the stable phases. The diagram indicates that copper is a relatively noble metal, and should not be corroded by noncomplexing solutions free from oxidizing agents. Corrosion would be expected

---

(1) References are listed on page 22

FIGURE 1. POTENTIAL pH EQUILIBRIUM DIAGRAM FOR THE SYSTEM COPPER WATER AT 25 C

in low- or high-pH environments containing oxidizing agents. Neutral or slightly alkaline solutions would be expected to passivate the copper surface.

The presence of oxygen in acid solution would be expected to lead to the corrosion of copper, since the potential of the  $\text{Cu}/\text{Cu}^{2+}$  couple is well below that for oxygen reduction. Oxygen-induced corrosion in neutral solutions should be less severe, again because of the passive regions.

The Pourbaix diagram gives thermodynamic domains of stability for the compounds in aqueous solutions at 25 C with no complexing ions present. The diagram gives no information on the shift in domains with increasing temperature or with other ions in solution. Methods are available for extending the predictions of the diagram to high temperatures. Thus Brook<sup>(2)</sup> has calculated the potential-pH diagram for Cu at temperatures of 120 and 150 C. This diagram is shown in Figure 2. The salient features are

1. The stability domain of  $\text{Cu}_2\text{O}$  diminishes rapidly with increasing temperature.
2. The stability domain of  $\text{CuO}$  shifts to lower pH's with increasing temperatures (consistent with the shift in the neutral point of water).
3. The stability of the  $\text{CuO}_2^{=}$  anion is markedly increased at lower pH values, at increased temperatures.

Pourbaix<sup>(3)</sup> has calculated the potential-pH diagram for the system  $\text{Cu}-\text{Cl}^--\text{H}_2\text{O}$  for solutions containing 355ppm ( $10^{-2}\text{M}$ )  $\text{Cl}^-$ . Several new species enter into the system, including  $\text{CuCl}$  and  $3\text{Cu}(\text{OH})_2 \cdot \text{CuCl}_2 \gamma$  (Figure 3). Pourbaix also includes a diagram of the system  $\text{Cu}-\text{Cl}-\text{SO}_3-\text{H}_2\text{O}$  to explain the pitting of copper in Brussels tap water. Ives and Rawson<sup>(4)</sup> have computed various equilibria for Cu in the  $\text{Cu}-\text{CO}_2-\text{O}_2-\text{H}_2\text{O}$  system, showing the vulnerability of Cu to attack in such solutions, the critical effects of oxygen and the less than adequate protective nature of the surface films which form.

The literature search has been unable thus far to find any high-temperature thermodynamic calculations or diagrams which include a treatment of  $\text{Cl}^-$  effects.

FIGURE 2. POTENTIAL-pH DIAGRAM FOR THE Cu-H<sub>2</sub>O SYSTEM  
Concentration of soluble species  $10^{-6}$  M.

FIGURE 3. POTENTIAL-pH EQUILIBRIUM DIAGRAM FOR THE 3-COMPONENT SYSTEM, Cu-Cl-H<sub>2</sub>O, FOR SOLUTIONS CONTAINING 355 ppm Cl<sup>-</sup> (10<sup>-2</sup> g ion/liter)

### Mechanisms of Corrosion and Pit Formation

As far as a theoretical mechanism for copper corrosion in systems is concerned, the basic thermodynamic tenets have been applied to a model by Ives and Rawson<sup>(5)</sup>. They hypothesize that copper, immersed in an oxygenated aqueous medium, is covered with an adherent film of cuprous oxide. However, as the film grows, the outer layer becomes non-adherent, and the outer structure is porous and never protective. Thus a true "passive film" does not develop. These authors envisage that this duplex film behaves as a "double cell" with the following reactions occurring (see Figure 4).

At Cu/adherent film interface:

At adherent film/porous film interface (redox):

At porous film/solution interface:

In this model, film dissolution proceeds in step with film growth until the film becomes very thick, when poor conductivity favors film-dissolution.

Many workers have put forward theories for the pitting which can occur during the use of copper pipes and tubes in water service. These theories must all explain the structure of a pit on copper, as described initially by May<sup>(6)</sup>. This structure is shown in Figure 5. May explained this structure on the basis of a large cathode/small anode, differential oxygen cell theory, with the precipitation of CuCl within the pit. This theory has been amended by Campbell<sup>(7)</sup> who noted the importance of carbon

FIGURE 4. HYPOTHETICAL DOUBLE CELL INVOLVED  
IN COPPER CORROSION

FIGURE 5. STRUCTURE OF A CORROSION PIT  
IN COPPER AS OBSERVED BY MAY

surface films in promoting pitting. Workers at CEBELCOR in Belgium have conducted research on this subject for some years, and their conclusions are well summarized by Pourbaix<sup>(3)</sup>. Lucey, in two recent articles<sup>(8,9)</sup>, has summarized succinctly current thinking on the pitting process. The correlation between reactions and pit structure, in the so-called "Membrane Cell", is shown in Figure 6. It is interesting to compare this with the function of the compact layer of  $\text{Cu}_2\text{O}$  in Ives and Banden's work<sup>(5)</sup>. For the membrane cell to operate, the following factors are essential:

- (1) There must be a membrane
- (2) The  $\text{Cu}^+$  concentration inside the membrane must be greater than outside the membrane, so the anodic reaction occurs on the inside surface and the cathodic reaction on the outer
- (3) Reducible ions must reach the outside surface at a fast enough rate to maintain the flow of current between the faces.

It is not clear at this time whether the presence of carbonate species in solution is absolutely necessary to this mechanism of pit formation.

### Experimental Investigation

Because of the extensive use of copper piping in the transport of potable and hot water, there is a large body of information on the influence of alloy, water quality, temperature, and fluid velocity on the corrosion of copper in these environments. Only a limited portion of this literature is germane to the present subject. Copper and, more commonly, copper-base alloys are also used for feedwater heaters in power plants. There are several parameters which are of interest here. These are:

- Effect of temperature
- Effect of anion level of solution (e.g.  $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$  content).
- Effect of oxygen of solution.

It has not been possible to separate out all these variables in this survey. Thus, the treatment will be chronological, rather than analytical.

Obrecht<sup>(10)</sup> studied the corrosion of copper tubing of unspecified purity in flowing soft water for times to 500 days, at different temperatures. Cuprous oxide was formed on the lower-temperature tubes, whereas cupric oxide

FIGURE 6. DIAGRAMMATIC REPRESENTATION OF THE ARRANGEMENT OF CORROSION PRODUCTS AND OF THE REACTIONS INVOLVED IN PITTING CORROSION OF COPPER

was predominant at 200 F. Oxidation to  $\text{CuO}$  appeared to be enhanced by higher temperatures. This worker also investigated the effect of solution additions on corrosion in the copper-water system at 140 F. He found that oxygen is essential for corrosion to occur, as predicted thermodynamically, and when the oxygen is consumed, corrosion stops. In an open system, with access of oxygen, the rate law for corrosion is roughly parabolic; however, copper ion in solution was at a low value, indicating that film thickening was the predominant reaction (presumably  $\text{Cu}_2\text{O}$ ). Oxygen is still necessary for corrosion to occur in water containing  $\text{CO}_2/\text{HCO}_3^-$ , but the  $\text{CO}_3^{2-}/\text{HCO}_3^-$  equilibrium did not affect corrosion rates. However, the presence of 10 ppm  $\text{Cl}^-$  or 40 ppm  $\text{SO}_4^{2-}$  in the water gave an increased corrosion rate.

North and Pryor<sup>(11)</sup> studied the corrosion of DHP Cu (CDA 122) in boiling 3 percent NaCl, as part of an investigation to determine the role of corrosion product structure on the corrosion of Cu-Ni alloys. Corrosion was affected in a Soxhlet apparatus, and the solution (initially aerated) was replaced every 7 days. The corrosion rate appeared almost linear through 60 days, with a value of  $5.67 \text{ mg/dm}^2/\text{day}$  ( $\sim 1.2 \text{ mpy}$ ). The corrosion product was  $\text{Cu}_2\text{O}$ , with a thickness of 2800 Å after 30 days.

In an early paper, Boden<sup>(12)</sup> had shown that Cu can suffer severe corrosion under boiling heat transfer conditions, as compared with zero heat transfer conditions. In a subsequent paper<sup>(13)</sup> he attempted to resolve this anomalous increase in terms of thermogalvanic and/or local corrosion effects. Thermogalvanic effects were determined in aerated and deaerated 1 percent and 3 percent NaCl solutions as a function of flow rate and temperature differential, using a cathode (cold) area to anode (hot) area of 150. The greatest effects were noted at a temperature of 140 C, in aerated solutions, where corrosion rates of  $>90 \text{ mg/cm}^2/\text{day}$  were experienced. This corrosion rate was 1/2 to 1/10 attributable to thermogalvanic corrosion, the remainder being probably due to an oxygen differential cell.

Somewhat earlier, Berry<sup>(14)</sup> had presented data on the thermogalvanic corrosion of copper in copper sulfate solutions of different concentrations. The experiments were performed with a disadvantageously large anode: cathode ratio of a fixed temperature difference of 150 F, but corrosion rates of

86 mpy were seen in 0.940 M  $\text{CuSO}_4$ , falling to 2 mpy in 0.0002 M  $\text{CuSO}_4$ .

There appeared to be no attempt to deaerate the solution, however, so again differential oxygen concentration cells could be in operation.

Mattson and Frederickson<sup>(15)</sup> conducted a comprehensive investigation to determine parameters affecting pit formation in hard-drawn copper tubes containing hot water. Their experimental studies were conducted at 75 C in an electrolytic cell containing a copper anode and cathode, across which a voltage of 0.5 V was applied to simulate the pitting condition. The solution variables were:

- pH - adjusted to 6, 7, 8, and 9 by varying the  $\text{O}_2/\text{CO}_2$  ratio
- $\text{SO}_4^{2-}$  - from 0.1 to 2.0 mE/l
- $\text{Cl}^-$  - 1, 2, 4, mE/l (36, 71, 142 ppm).

These workers found that the corrosion current was a function of pH, but that in  $\text{Cl}^-$ -containing solutions, the corrosion rates decreased with increasing pH, whereas the opposite effect was noted for sulfate-containing solutions.

In a very comprehensive series of investigations, Thomas and Tiller<sup>(16, 17)</sup> have studied the effect of anion concentration, pH and temperature on the formation and breakdown of surface films on OFHC copper in air-saturated solutions. Most of their experimentation involved the determination of anodic polarization curves, together with the cathodic reduction and hence identification of the surface films formed during polarization.

In  $\text{Cl}^-$ -containing solutions at 25 C, the current remained steady at potentials up to 0.19  $V_H$  in 0.01 M solutions (~350 ppm), when a rapid increase occurred. Increasing the  $\text{Cl}^-$ -concentration gave a less well defined constant current region with larger currents, and a lower "break-down" potential. Cathodic reduction showed cuprous chloride to be present in films on surfaces polarized in all  $\text{Cl}^-$ -containing solutions, with  $\text{CuO}$  present only at the 0.01 M concentration. In discussion of their results, the authors admit that the initially fairly protective film formed an anodic polarization, will lose its protective nature if allowed to stand in the solution. They therefore hypothesize that pitting is associated with local variations in a fairly high rate of general corrosion.

Polarization curves run at 25, 60, and 90 C in 0.01 M NaCl after 1 hour and 24 hour immersion indicated that the surface films which formed were nonprotective, with a relatively linear increase in current with applied potential. After polarization to  $0.3 V_H$ , the specimens were covered with a thick dark blue-grey film, with localized red-brown areas. Pits were present. To confirm this behavior, specimens were immersed in the same solution at the same temperatures for 7 days under freely corroding conditions. After cleaning, the specimens were examined for pitting. Pitting tendency increased with increasing temperature. The authors suggest that the greater the corrosion current passing through the film, the greater the tendency for pit formation to occur.

Ives and Rawson<sup>(18)</sup> found that chloride ion additions at 100 ppm level to water containing varying concentrations of  $O_2$  and  $CO_2$  did not always promote corrosion of copper. Thus, for low  $CO_2$  concentrations and a constant  $p_{O_2} = 0.30$  atm, the presence of chloride retarded both film growth and dissolution. Chloride was found in the film after these experiments, in the magnitude of 0.7 to 1.5 percent. The  $Cu_2O$  films formed in chloride solutions were also much less adherent; they were punctured by small holes, and the authors conjectured that the chloride ion caused a change from a general corrosion mechanism to a more localized attack.

At present, the most meaningful paper relating to corrosion of Cu in high temperature water is that by Byalobzhetskii and Anurova<sup>(19)</sup>. These authors investigated the effect of temperatures up to 250 C on the corrosion of Cu in distilled water, and studied the gas evolved and  $O_2$  adsorbed during the corrosion process. At 250 C in a Pyrex capsule, pure copper consumed  $177 \text{ cm}^3/\text{m}^2/\text{hr}$  of  $O_2$  and liberated  $0.9 \text{ cm}^3/\text{m}^2/\text{hr}$  of  $H_2$ . For a copper autoclave, with an air atmosphere, the results for a 5 hr exposure were:

| <u>Temp C</u> | <u><math>H_2</math> Liberated<br/>(<math>\text{cm}^3/\text{m}^2/\text{hr}</math>)</u> | <u><math>O_2</math> Consumed</u> |
|---------------|---------------------------------------------------------------------------------------|----------------------------------|
| 100           | None                                                                                  | 18.0                             |
| 150           | 1.2                                                                                   | Consumed                         |
| 200           | 1.7                                                                                   | Consumed                         |
| 250           | 21.9                                                                                  | Consumed                         |

and for an oxygen atmosphere:

| <u>Temp C</u> | <u>H<sub>2</sub> Liberated</u><br><u>(cm<sup>3</sup>/m<sup>2</sup>/hr)</u> | <u>O<sub>2</sub> Consumed</u> | <u>Calculated</u><br><u>Corrosion Rate</u> (g/m <sup>2</sup> /hr) |
|---------------|----------------------------------------------------------------------------|-------------------------------|-------------------------------------------------------------------|
| 150           | 8.0                                                                        | 67.0                          | 0.402                                                             |
| 200           | 15.0                                                                       | 12.40                         | 0.749                                                             |

Further calculations showed that the dissolution rate of Cu at 250 C was 0.43 g/m<sup>2</sup>/hr, and the corrosion rate based on gas volume changes was 1.94.

As the authors point out, this data is surprising, in that the copper appears to liberate hydrogen from the water. Thermodynamically this does not appear feasible. A suggestion is made that this is a "catalytic" effect or an "impurity" effect in the copper alloys used. It is perhaps conceivable that the copper removes the oxygen level below that for the equilibrium value for disproportionation of water to H<sub>2</sub> and O<sub>2</sub>, or that it catalytically decomposes the water. Certainly more information is needed on this subject.

#### Task 2. Destructive Analysis of Heat Pipe

Because of the limitation in funding, only parts 1 and 2 of this task, analysis of gas in pipes, and chemical analysis of water, have been performed. The pipes available for analysis are:

- . 2A12 - life tested for 6680 hrs at 270 F
- . 2A14 - life tested for 6680 hrs at 360 F
- . 3A10 - fabricated as above types, not life tested.

#### Mass Spectrographic Results

The heat pipes 2A12 and 2A14 were connected into a gas mass spectrograph and the gases present in each pipe were analyzed. The results are given in Table 1.

TABLE 1. CONTENT AND COMPOSITION OF GAS FROM HEAT PIPES

| Heat Pipe No. | Total Gas<br>(cc, SP, 25 C) | CO <sub>2</sub> | Composition of Gas (Vol.%) |      |                |                |      |                | CH <sub>4</sub> |
|---------------|-----------------------------|-----------------|----------------------------|------|----------------|----------------|------|----------------|-----------------|
|               |                             |                 | ETOH                       | A    | N <sub>2</sub> | O <sub>2</sub> | He   | H <sub>2</sub> |                 |
| 2A12          | 0.00827                     | 2.3             | 7.98                       | 24.2 | 49.6           | < 0.003        | 10.6 | 5.32           | N/D             |
| 2A14          | 4.06                        | 0.13            | N/D                        | 0.15 | 11.5           | 2.90           | N/D  | 85.2           | 0.07            |



The most significant feature is the large volume of gas present in pipe 2A14. This corresponds well with the inactive condenser length of 4 inches present on this pipe at the end of the life test. The composition of the gas was also important: greater than 85%  $H_2$ , 11.5%  $N_2$ , 2.9%  $O_2$ . At first sight, this would appear to indicate a large amount of corrosion of the copper with liberation of  $H_2$ . In view of the thermodynamics of this situation discussed in the Literature Review in Task 2, which seem to preclude such a reaction, this may well be an erroneous assumption. The presence of 11.5%  $N_2$ , or 0.46cc, suggests that at least 0.55cc air was present when this heat pipe was closed.

The results of analysis from 2A12 indicate essentially no hydrogen buildup, and a complete depletion of oxygen from the air originally present. Thus a very low corrosion rate would be expected in this situation.

#### Chemical Analysis of Water

The water present in the life-tested heat-pipes was removed after gas mass spectroscopy had been performed. The volume was roughly 1 ml; such a small volume precluded complete analysis. It was originally planned to run checks on conductivity, pH,  $Cl^-$ , and  $SO_4^{2-}$  concentration and  $Cu^{2+}$  content. pH and  $Cl^-$  checks were run; no information was obtained on  $SO_4^{2-}$  because of the size of specimen, and the method of choice for  $Cu^{2+}$  in such a small volume (atomic absorption spectrometry using a graphite furnace) was not available because of malfunction of the instrument. The samples have been saved, however, and will be run when the instrument is functional again.

The results of the analytical tests are given in Table 2. pH's were run on  $N_2$ -saturated samples.  $Cl^-$  determinations were made by a coulometric technique. It is evident that all three samples contain significant amounts of  $Cl^-$ -ion; the pH of water from the untested pipe (3A10) is more than 1 pH unit more alkaline than that from the life-tested pipes. Since the  $Cl^-$  values are roughly comparable, to within a factor of 2, this suggests that the pH of the water in the life-tested pipes was decreased during the time spent at temperature.

TABLE 2. CHEMICAL ANALYSIS OF LIQUID FROM HEAT PIPES

| Heat Pipe No. | pH  | C <sup>-</sup> (ppm) |
|---------------|-----|----------------------|
| 2A12          | 8.5 | 31                   |
| 2A14          | 8.5 | 18                   |
| 3A10          | 9.6 | 40                   |

## SUMMARY AND CONCLUSIONS

The literature survey is as yet incomplete. However, it is apparent that, although there is a large amount of data on the corrosion of Cu in relatively low-temperature ( $< 100$  C) aqueous environments, there is a scarcity of data at higher temperatures. No data has been found on the effect of different oxide films on corrosion. It may be that literature yet to be received may alleviate this shortage. This emphasizes the necessity of conducting further theoretical and experimental work in this area.

The survey thus far has shown that, at temperatures to 90 C, there are significant effects of pH and  $\text{Cl}^-$  on the corrosion of copper, although the rationalization of these effects has not been attempted in thermodynamic terms. There does appear to be contradictory data on the effects of  $\text{Cl}^-$  on dissolution and film thickening, and it may be that pH is the controlling variable in this regard. Increased temperature and  $\text{Cl}^-$  ion concentration lead to an increased pitting tendency.

Since most natural waters contain some carbonate species, the carbonate/bicarbonate couple has played a major role in the solutions used by previous investigators. It is unlikely that this situation pertains to the water in the heat-pipe, because of the purity of water used, and the method of filling and degassing the pipe.

In summary, while the literature surveyed to date has not lead to definitive conclusions, it has given general trends for the temperature,  $\text{Cl}^-$  ion concentration, and pH on the corrosion of copper in water.

It is too early at this stage to draw firm conclusions from the results of Task 2. The results of copper ion concentration determinations, and of the nature and quantity of the films present on the inside of the pipes, will greatly aid in rationalizing the corrosion behavior of copper in the heat pipe environment. However, the increased pH and  $\text{Cl}^-$  levels of water from the 3A10 pipe show that present rinsing procedures after the Ebonal process are probably less than adequate. The reduction in pH and, to a lesser extent, Cl levels in water from the after-test pipes suggests that these factors have played an as-yet unidentified role in the corrosion process.

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APPENDIX G

Analysis of Wetting of Heat Pipe Surfaces,  
Research Triangle Institute, June 1975.

RESEARCH TRIANGLE INSTITUTE

June 1975

ANALYSIS OF  
WETTING OF HEAT PIPE SURFACES

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## 1.0 SUMMARY AND CONCLUSIONS

To aid in the proper design of copper heat pipe surfaces capable of long-term reliable operation, an analysis of the wetting of copper substrates by water was performed. Included in this analysis were thermodynamic analyses of various copper/water systems. From these analyses, it is concluded that, at the working temperatures anticipated for the system, the stable surface condition expected for the inside of a copper heat pipe is a mixture of solid cupric oxide and cupric hydroxide.

A review of previous studies of wetting revealed that contact angle measurements, a measure of the degree of wetting, have been performed for the wetting of only metallic copper surfaces. Also, it has been shown that wetting of copper substrates is decreased by the introduction of either gaseous oxygen or hydrocarbon contamination into the system. Based on these findings, it is recommended that contact angle measurements be performed for metallic copper, cuprous oxide and cupric oxide surfaces prepared under various conditions and exposed to various gaseous environments similar to those expected in realistic heat pipe designs. In addition, it is recommended that the stability of the wetting property of the cupric oxide/cupric hydroxide surface be determined experimentally.



## 2.0 INTRODUCTION

The analysis of the wetting of copper heat pipe surfaces requires consideration of the materials and assembly techniques that determine the nature of the surfaces in contact with the heat transfer fluid.

In the heat pipe fabrication design analyzed in this work, four types of materials are employed. The tubular body of the pipe is a phosphorus-deoxidized copper alloy 122 with end caps and fill tubes of oxygen-free copper alloy 102. An electrolytic copper square-mesh wick covers about one-third of the inner circumference of the tube and is held in position by a wire spring of beryllium-copper alloy 25. The wick and spring are assembled into the alloy 122 tube and the end caps with fill tubes are welded onto the ends.

During assembly, the inside surfaces are treated with various solvents and acids. Finally, a proprietary formulation, Ebonol, is used to generate a film of cupric oxide on the inside surfaces.

The assembly operation is completed by sealing one end, introducing the proper volume of high purity water, degassing the water and, finally, sealing the other end. The entire inside wall of the pipe is grooved longitudinally to facilitate water transport to the wick which, in turn, transports the water from the condenser to the evaporator.

Achievement of a design for heat pipe surfaces displaying optimum wetting by water requires analysis of the effects of several closely related design parameters:

- Effect of different copper substrates on wetting.

For example, does pure copper wet better than cuprous oxide or cupric oxide?

- Effect of various surface cleaning procedures on wetting. For example, following the surface treatment process, what quantity of chemical residue can be tolerated?
- Effect of purification of the working fluid, water, on wetting. For example, should water be triply or doubly distilled? What effect does dissolved oxygen or carbon dioxide have on wetting?
- Effect of atmospheric gases on wetting. For example, should the enclosed heat pipe be completely free of gaseous oxygen or carbon dioxide to obtain complete wetting? Should heat pipe surfaces be prepared in a carbon dioxide free environment?
- Effect of temperature on wetting. For example, does water wet better in a vapor atmosphere at 500°F or at room temperature?
- Effect of different copper alloys on wetting.
- Effect of capillary action on wetting.
- Effect of pH (buffers), wetting agents, and corrosion inhibitors on wetting.
- Effect of handling procedures on wetting.

The approach chosen was to use the principles of equilibrium chemical thermodynamics to assess the importance of the effects of the above parameters. Then using the results of the thermodynamic analysis, experiments were designed which were directed at determining the surface conditions necessary for optimum surface wetting of copper heat pipe surfaces.

### 3.0 THERMODYNAMIC ANALYSIS

Thermodynamic calculations were performed to determine the stable copper substrates expected both in oxygen-free and in contaminated aqueous environments. Characterization of the nature of the stable copper specie would permit establishment of improved fabrication techniques and procedures in the manufacturing of heat pipes. In heat pipes constructed of copper, four possible systems exist. These are:

The convention used in these equations is explained by describing equation (3). Equation (3) represents a copper surface with a cuprous oxide layer on it and, over that oxide, a layer of cupric oxide which is in contact with water. It is important to know which one of these systems is stable, both when the water used for heat transfer is pure and when it is contaminated with oxygen and carbon dioxide from the atmosphere. Determination of the stable substrates for these environments was required prior to analysis of wetting of these surfaces. Thermodynamic and wetting analyses were performed for systems which were assumed to be uncontaminated by chemical residue and to contain pure copper substrates.

The assumption of systems uncontaminated by chemical residue is valid since residue can, in principle, be eliminated through properly designed fabrication, assembly, handling and purification procedures. The assumption of pure copper substrates should be considered in the context of the purities listed in Table 1.

Table 1. Purity of Copper Substrates

| Substrate                              | Percent of Copper by Weight | Reference |
|----------------------------------------|-----------------------------|-----------|
| Electrolytic Copper                    | 99.92                       | 1         |
| Oxygenfree Copper Alloy 102            | 99.95                       | 1         |
| Phosphorus Deoxidized Copper Alloy 122 | 99.9+                       | 1         |
| Copper Alloy 25                        | 97.65 - 98.00               | 2         |

### 3.1 Oxygen/Water Environments

An analysis of two oxygen/water environments was performed. One environment was assumed to contain water contaminated with dissolved oxygen; the other was assumed to contain only pure water. The solid phase was assumed to consist of pure copper in contact with either of the oxygen/water environments. Two questions were posed:

1. Does copper dissolve in pure water to form cuprous or cupric oxide?
2. What is the stable form of copper in contact with oxygen/water environments at 25°C and 260°C?

Several equations can be written for possible reactions of copper with water to form oxides and hydroxides. These equations are listed in Table 2. Also listed in this table are the calculated values of the standard free energy changes for reactions at 25°C and 260°C. The latter temperature was selected to correspond to that expected inside the heat pipes during operation. These calculations were made using the standard free energies of formation listed in Table 3.

A flow diagram, shown in Figure 1, of possible copper reactions summarizes the reactions listed in Table 2. From this flow diagram, it is concluded that in pure water, metallic copper is the stable species at both temperature extremes. Therefore, in pure water, wetting of metallic copper should be analyzed.

In the system

wherein water is contaminated with dissolved oxygen, the thermodynamic

Figure 1. Flow Diagram of Possible Copper Reactions.

The numbers represent the standard free energies of reaction at 25°C for one gram-atom of copper. Note that in the absence of oxygen, metallic copper (inside the dashed box) is the stable state in an aqueous environment; while in the presence of oxygen, cupric hydroxide (inside the dotted box) is the stable specie in an aqueous medium.

\*These values depend on the stoichiometry of the reactions shown in Table 2.

Table 2. Reactions of Copper with Water

| Reaction                                                                                                                                         | $\Delta G^\circ_{298.15^\circ\text{K}}, \text{ kcal(g-atom Cu)}^{-1}$ | $\Delta G^\circ_{533.15^\circ\text{K}}, \text{ kcal(g-atom Cu)}^{-1}$ |
|--------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------|-----------------------------------------------------------------------|
| 1. $\text{Cu(c)} + 1/2 \text{H}_2\text{O}^* \rightleftharpoons 1/2 \text{Cu}_2\text{O(c)} + 1/2 \text{H}_2(\text{g})$                            | 10.7                                                                  | 10.436                                                                |
| 2. $\text{Cu(c)} + \text{H}_2\text{O}^* \rightleftharpoons \text{CuO(c)} + \text{H}_2(\text{g})$                                                 | 25.2                                                                  | 26.461                                                                |
| 3. $\text{Cu(c)} + \text{H}_2\text{O}^* \rightleftharpoons \text{CuOH(c)} + 1/2 \text{H}_2(\text{g})$                                            | 30.88                                                                 |                                                                       |
| 4. $\text{Cu(c)} + 2\text{H}_2\text{O}^* \rightleftharpoons \text{Cu(OH)}_2(\text{c}) + \text{H}_2(\text{g})$                                    | 27.65                                                                 | 29.192                                                                |
| 5. $\text{Cu(c)} + 1/4 \text{O}_2(\text{g}) \rightleftharpoons 1/2 \text{Cu}_2\text{O(c)}$                                                       | -17.45                                                                | -41.542                                                               |
| 6. $1/2 \text{Cu}_2\text{O(c)} + 1/4 \text{O}_2(\text{g}) \rightleftharpoons \text{CuO(c)}$                                                      | -13.55                                                                |                                                                       |
| 7. $\text{Cu(c)} + 1/2 \text{O}_2(\text{g}) \rightleftharpoons \text{CuO(c)}$                                                                    | -31.0                                                                 | -77.496                                                               |
| 8. $1/2 \text{Cu}_2\text{O(c)} + 1/2 \text{H}_2\text{O}^* \rightleftharpoons \text{CuOH(c)}$                                                     | 20.20                                                                 |                                                                       |
| 9. $1/2 \text{Cu}_2\text{O(c)} + 3/2 \text{H}_2\text{O}^* \rightleftharpoons \text{Cu(OH)}_2(\text{c}) + 1/2 \text{H}_2(\text{g})$               | 16.97                                                                 | 18.756                                                                |
| 10. $\text{CuO(c)} + \text{H}_2\text{O}^* \rightleftharpoons \text{Cu(OH)}_2(\text{c})$                                                          | 2.39                                                                  | 2.731                                                                 |
| 11. $\text{CuOH(c)} + \text{H}_2\text{O}^* \rightleftharpoons \text{Cu(OH)}_2(\text{c}) + 1/2 \text{H}_2(\text{g})$                              | - 3.23                                                                |                                                                       |
| 12. $\text{Cu(c)} + 1/2 \text{O}_2(\text{g}) + \text{H}_2\text{O}^* \rightleftharpoons \text{Cu(OH)}_2(\text{c})$                                | -28.61                                                                | -74.764                                                               |
| 13. $\text{Cu(c)} + 1/4 \text{O}_2(\text{g}) + 3/2 \text{H}_2\text{O}^* \rightleftharpoons \text{Cu(OH)}_2(\text{c}) + 1/2 \text{H}_2(\text{g})$ | - 0.48                                                                | -22.786                                                               |
| 14. $\text{CuOH(c)} \rightleftharpoons 1/2 \text{Cu(c)} + 1/2 \text{Cu(OH)}_2(\text{c})$                                                         | -17.06                                                                |                                                                       |
| 15. $\text{Cu(OH)}_2(\text{c}) \rightleftharpoons \text{Cu}^{++}(\text{aq}) + 2 \text{OH}^-(\text{aq})$                                          | 26.26                                                                 |                                                                       |

\* Liquid at 298.15°K and gas at 533.15°K.

Table 3. Values for Free Energies of Formation

| Substance                                      | $\Delta G^\circ_{298.15^\circ\text{K}}$ , kcal mole <sup>-1</sup> | Reference | $\Delta G^\circ_{533.15^\circ\text{K}}$ , kcal mole <sup>-1</sup> | Reference |
|------------------------------------------------|-------------------------------------------------------------------|-----------|-------------------------------------------------------------------|-----------|
| Cu(c)                                          | 0                                                                 | 7         | 0                                                                 | 8         |
| Cu <sub>2</sub> O(c)                           | -34.9                                                             | 3         | -31.090                                                           | 3         |
| CuO(c)                                         | -31.0                                                             | 3         | -25.502                                                           | 3         |
| CuOH(c)                                        | -25.38                                                            | 4         |                                                                   |           |
| Cu(OH) <sub>2</sub> (c)                        | -84.87                                                            | 5         | -74.734                                                           | 8         |
| H <sub>2</sub> O*                              | -56.259                                                           | 6,7       | -51.963                                                           | 6         |
| H <sub>2</sub> (g)                             | 0                                                                 | 6         | 0                                                                 | 6         |
| O <sub>2</sub> (g)                             | 0                                                                 | 6         | 103.987                                                           | 6         |
| Cu <sup>++</sup> (aq) + 2 OH <sup>-</sup> (aq) | 26.26                                                             | 8         |                                                                   |           |

\* Liquid at 298.15°K and gas at 533.15°K.



analysis indicates that the cupric oxide displays marginal stability, while metallic copper should be transformed spontaneously into cupric hydroxide. The standard free energy change associated with the transformation of cupric oxide into the hydroxide is so weakly prohibitive that, for practical purposes, it should be assumed that cupric oxide reacts with water to form cupric hydroxide.<sup>9</sup> Revelation of cupric oxide as an intermediate product in the formation of cupric hydroxide leads to the conclusion that, in an oxygen limited environment such as is expected in heat pipes, the stable surface condition expected is a mixture of solid cupric oxide and cupric hydroxide.

### 3.2 Experimental Verification of Thermodynamic Analysis

Following the previous analysis, experiments were performed to verify the conclusions derived from the calculations. Several possible processes can be written for the dissolution of cupric oxide in water. The following equations describe these processes:

Equation (7) can be viewed as the sum of equations (6) and

The expression for the solubility products for these equations are, respectively,

$$K_{\text{SP}} = [\text{Cu}^{++}][\text{OH}^-]^2 = 4S^3 \quad (11)$$

$$K_{\text{SP}} = [\text{Cu}(\text{OH})^+][\text{OH}^-] = S^2 \quad (12)$$

$$K_{\text{SP}} = [\text{HCuO}_2^-][\text{H}^+] = S^2 \quad (13)$$

$$K_{\text{SP}} = [\text{CuO}_2^{=}] [\text{H}^+]^2 = 4S^3 \quad (14)$$

where S is the solubility of cupric oxide in water. Table 4 lists calculated and reported values of the solubility products of these reactions. Except for the cupric hydroxide specie, the calculated solubility products do not agree with those reported.

From equilibrium thermodynamics, the more energetically favorable reaction can be ascertained from consideration of standard free energy changes.<sup>9,12</sup> When comparing different reactions, the reaction with the

Table 4. Cupric Oxide/Water Systems

| $K_{SP} =$            | <u>Calculated*, 20°C</u> |     | Reported, 25°C          | Reference |
|-----------------------|--------------------------|-----|-------------------------|-----------|
|                       | $K_{SP}$                 | pH  | $K_{SP}$ at 25°C        |           |
| $[Cu^{++}][OH^-]^2$   | $2.7 \times 10^{-17}$    | 8.7 | $1.6 \times 10^{-19}$   | 4         |
| $[Cu(OH)^+][OH^-]$    | $3.6 \times 10^{-12}$    | 8.4 | $1.6 \times 10^{-25}^+$ | 4,10      |
| $[HCuO_2^-][H^+]$     | $3.6 \times 10^{-12}$    | 5.7 | $1.479 \times 10^{-19}$ | 11        |
| $[CuO_2^{=}] [H^+]^2$ | $2.7 \times 10^{-17}$    | 5.4 | $1.047 \times 10^{-32}$ | 11        |

\*Calculated from the value of solubility of cupric oxide reported to be  $1.9 \times 10^{-6} M$ .<sup>2</sup>

<sup>+</sup>This value is the product of the solubility constants for equation (6)  $1.6 \times 10^{-19}$ , and equation (11),  $1 \times 10^{-6}$ .

more negative standard free energy change is the more energetically favorable reaction. The standard free energy change  $\Delta G^\circ$  for oxide dissolution is given by

$$\Delta G^\circ = -RT \ln K_{SP} \quad (15)$$

where  $R$  = gas constant, and  $T$  = temperature. Therefore, the reaction with the larger solubility product will be energetically more favorable.

Using the reported values of the solubility products listed in Table 4, the thermodynamic analysis indicates that the most favorable reaction of cupric oxide with water is that of equation (6).

For comparison with the cupric oxide reaction with water, the reaction of cuprous oxide with water was analyzed. According to Latimer,<sup>4</sup> cuprous oxide dissolves in water to produce cuprous and hydroxyl ions according to the following equation,

The solubility product  $K_{SP}$  of cuprous oxide in water can be calculated from the solubility of the oxide,  $S$ , by the expression

$$K_{SP} = [\text{Cu}^+][\text{OH}^-] = 4S^2 \quad (17)$$

For  $S$  equal to  $6.99 \times 10^{-7} \text{M}$  at  $20^\circ\text{C}$ ,<sup>13</sup> the solubility product of cuprous oxide is  $1.95 \times 10^{-12}$ . Therefore, according to the expression

$$\text{pH} = -\log_{10} ([\text{Cu}^+]K_W/K_{SP}) \quad (18)$$

where  $K_W$  is the ionization constant of water ( $K_W = 6.81 \times 10^{-15}$  at  $20^\circ\text{C}$ ), the pH of the cuprous oxide/water system is 8.3.

From the analysis of pH, dissolution of cupric oxide, as well as that of cuprous oxide, should produce a basic solution. It was decided to measure the pH of a solution of cupric oxide dissolved in water at 25°C to determine if the results agree with the thermodynamic analysis that cupric oxide dissolves to yield cupric hydroxide. Table 4 lists the pH values calculated from the solubility products and solubility of cupric oxide at 20°C. If the oxide dissolves to yield the hydroxide according to equation (6), the measured value of pH should be close to the calculated value of 8.7.

The pH measurement was performed by boiling distilled, deionized water for 90 minutes to ensure degasification. Then, cupric oxide was added to the degassed water which was subsequently boiled for 30 minutes. A drying tube containing calcium sulfate sealed the solution of aqueous cupric oxide from the atmosphere. The pH of the solution was measured at 25°C under a nitrogen atmosphere. The value of pH at this temperature is 8.61. It is concluded that the measured value of pH verifies the thermodynamic conclusion that cupric oxide reacts with water to yield cupric hydroxide.

### 3.3 Effect of Oxygen and Carbon Dioxide in the Aqueous Environment

Based upon the calculation of a large, negative Gibb's free energy of formation, it was concluded that copper has a tendency to dissolve in water containing oxygen and carbon dioxide.<sup>5</sup> This reaction, proceeding according to the following equation, is accompanied by a favorable (negative) free energy change of nearly 20 kcal mole<sup>-1</sup>,

$$\Delta G^\circ_{298} = -19.88 \text{ kcal.}$$

From the equation,<sup>5</sup>

$$-\log K = 14.59 + 2 \log P_{\text{CO}_2} + \frac{1}{2} \log P_{\text{O}_2} \quad (20)$$

it follows that if the partial pressure of dissolved oxygen were  $10^{-10}$  atm, the partial pressure of dissolved carbon dioxide required for spontaneous formation of cupric bicarbonate would have to be less than  $10^{-12}$  atm. Therefore, it is concluded that in the presence of a small concentration of dissolved carbon dioxide, oxygen is required for metallic copper to react with water to yield cupric bicarbonate.

Alternatively, from this same equation, it follows that if the partial pressure of dissolved carbon dioxide were  $10^{-10}$  atm, the partial pressure of dissolved oxygen required for dissolution of metallic copper to form cupric bicarbonate would have to be only about  $10^{-19}$  atm.

On the other hand, consideration of the dissolution of metallic copper

with an aqueous medium containing dissolved oxygen, proceeding according to the expression,

for which  $\Delta G^\circ = -28.6 \text{ kcal mole}^{-1}$ , and

$$\Delta G^\circ = -RT \ln [\text{Cu}^{++}][\text{OH}^-]^2 + \frac{1}{2} RT \ln P_{\text{O}_2} \quad (22)$$

leads to the conclusion that, for the value of the solubility of cupric oxide equal to  $1.90 \times 10^{-6}$  molar at  $20^\circ\text{C}$ , the partial pressure of dissolved oxygen would have to be only  $10^{-77}$  atm for the formation of cupric hydroxide.

Similar analyses of the minimum partial pressures of oxygen and carbon dioxide were performed for reactions involving cuprous oxide, and the cupric oxide and hydroxide. These equations and the results of the analyses are presented in Table 5. As seen from Table 5, in an aqueous medium containing oxygen and carbon dioxide, dissolution of copper constitutes the main problem. However, copper dissolution is unlikely to be fully effective because the cupric bicarbonate formed can react further, as seen below, to produce solid products which are likely to deposit on the metal and exert some protective action,

Table 5. Analyses of Minimum Partial Pressures of Carbon Dioxide and Oxygen for Some Selected Reactions at 25°C.

| Reactions                                                           | Partial Pressure Expression, i.e.<br>$\Delta G^\circ / (2.303)RT = -\log K_{SP} + x \log P_{O_2} + y \log P_{CO_2}$ | Minimum Partial Pressure                                                                                                                             |
|---------------------------------------------------------------------|---------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------------------------------------------------------------------------------------------------|
| $1/2 Cu_2O + H_2O + 2CO_2 + 1/4 O_2 \rightleftharpoons Cu(HCO_3)_2$ | $-14.57 = 1.75 + 2 \log P_{CO_2} + 1/4 \log P_{O_2}$                                                                | $P_{CO_2} = 10^{-1.50} \text{ atm for } P_{O_2} = 10^{-2} \text{ atm}$                                                                               |
| $1/2 Cu_2O + H_2O + 2CO_2 \rightleftharpoons Cu(HCO_3)_2 + Cu$      | $11.07 = -11.07 + 2 \log P_{CO_2}$                                                                                  | $P_{CO_2} = 10^{11.07} \text{ atm}$                                                                                                                  |
| $Cu(OH)_2 + 2CO_2 \rightleftharpoons Cu(HCO_3)_2$                   | $6.40 = -6.40 + 2 \log P_{CO_2}$                                                                                    | $P_{CO_2} = 10^{6.40} \text{ atm}$                                                                                                                   |
| $CuO + H_2O + 2CO_2 \rightleftharpoons Cu(HCO_3)_2$                 | $7.71 = -7.71 + 2 \log P_{CO_2}$                                                                                    | $P_{CO_2} = 10^{7.71} \text{ atm}$                                                                                                                   |
| $Cu + H_2O + 2CO_2 + 1/2 O_2 \rightleftharpoons Cu(HCO_3)_2$        | $-14.57 = 14.59 + 2 \log P_{CO_2} + 1/2 \log P_{O_2}$                                                               | $P_{CO_2} = 10^{-12.08} \text{ atm for } P_{O_2} = 10^{-10} \text{ atm}$<br>$P_{O_2} = 10^{-19.04} \text{ atm for } P_{CO_2} = 10^{-10} \text{ atm}$ |
| $Cu + 1/2 O_2 + H_2O \rightleftharpoons Cu(OH)_2$                   | $21.32 = -17.17 - 1/2 \log P_{O_2}$                                                                                 | $P_{O_2} = 10^{-77} \text{ atm at } 20^\circ\text{C}$                                                                                                |

## 4.0 ANALYSIS OF WETTING

### 4.1 Wetting of Heat Pipe Surfaces

The operation of any heat pipe is strongly dependent on two properties of the internal structure. The first of these is the wettability of the surface by the working fluid, and the second is the geometry of the wick structure. This latter is fairly well under the control of the design engineer, while the former is subject to many factors ranging from the base material to the handling procedure and atmosphere of the room before the heat pipe is sealed. It is also affected by impurities in the working fluid.

The heat transport capability of a conventional heat pipe is determined by geometry of the wick, effect of gravity, geometry of the pipe, vapor viscosity, and the surface tension, heat of vaporization and kinematic viscosity of the working fluid. For good performance, the liquid must wet the wick well and also have a high value of surface tension.

The most widely used method of determining the extent of wetting of a surface by a liquid is by measurement of the contact angle of the liquid on the surface. Looking at the profile of a sessile liquid drop on a smooth, solid surface, the contact angle  $\theta$  is defined as the angle between the solid surface and the tangent to the surface of the drop at the point of contact.

The magnitude of this angle is given by the Young<sup>15</sup> equation which describes the balance of surface forces holding the drop in its equilibrium configuration,

$$\gamma_{LV} \cos \theta = \gamma_{SV} - \gamma_{SL} \quad (28)$$

where

$\gamma_{LV}$  = surface tension of the  
liquid vapor interface,

$\gamma_{SV}$  = surface tension of the  
solid vapor interface, and

$\gamma_{SL}$  = surface tension of the  
solid liquid interface.

This equation states that the sum of the horizontal component of the surface tension of the liquid equals the difference between the surface tensions of solid-vapor and solid-liquid interfaces. The righthand side of this equation is a wetting parameter, describing the tendency of the liquid to adhere or cling to the solid. The lefthand side of equation (28) consists of experimentally measurable quantities, the surface tension of the liquid and its contact angle. A liquid is said to wet a solid surface completely if its contact angle value is zero; the liquid does not wet the surface if the angle is 90° or greater.

Wetting is also related to the surface tension of the solid substrate. The beading of various liquids on a surface forms the basis of assigning that surface a characteristic parameter called the critical surface tension  $\gamma_c$ . The critical surface tension is found by measuring the values of contact angles of various liquids on the surface, plotting the cosines of the contact angles versus the surface tensions of the respective liquids, and extrapolating the resulting curve to  $\cos \theta = 1$ .

Therefore, the critical surface tension of a solid is the value of the surface tension where a liquid would just spontaneously completely wet that surface if applied as a drop. This phenomenon illustrates what is probably the most important single factor in dealing with wetting: that liquids of lower surface tension will spontaneously wet solids of higher surface tension to reduce the total surface free energy of the system. The extent of this wetting is given by the value of the contact angle. Application of critical surface tension phenomenon to the wetting of heat pipes reveals that the copper substrate with the higher critical surface tension will be most readily wet.

Experience with metal surfaces has shown that, in the absence of a contaminating organic layer, all metals are wet completely by water.<sup>16</sup> However, "real" metal surfaces investigated contain combined oxygen (ranging in nature from a monolayer of chemisorbed oxygen to a thick layer of surface oxide) as a result of their exposure to the atmosphere. These surface metal oxides are capable of strong hydrogen bonding interaction with water.

Some data on the wetting of copper substrates, shown in Table 6, appears in the literature. It can be seen that, in the absence of dissolved oxygen and carbon dioxide, water completely wets metallic copper.

Table 6. Contact Angle of Water on Copper Substrate

| Substrate | Contact Angle, deg | Conditions                           | Reference |
|-----------|--------------------|--------------------------------------|-----------|
| Cu(c)     | 0                  | Water thoroughly degassed.           | 30        |
| Cu(c)     | 20                 | Water containing dissolved oxygen.   | 30        |
| Cu(c)     | 72                 | 1 atm, 100°C.                        | 31        |
| Cu(c)     | 0                  | Water containing dissolved hydrogen. | 30        |

#### 4.2 Factors Affecting Wetting

Layers of grease or any hydrocarbon not more than one molecular layer thick can increase the contact angle considerably; it may not even be necessary for the layers to have their molecules closely packed on the surface. Langmuir<sup>14</sup> showed that a glass plate brought up through a water surface covered by a monomolecular film of some hydrocarbon substance had its contact angle very much increased. Blodgett<sup>17</sup> found that the wetting of glass covered by a layer of stearic acid three molecules thick is no better than that of paraffin wax. Devaux<sup>18</sup> also found a considerable diminution of wetting by layers one molecule thick.

The importance of surface cleanliness on wetting has been demonstrated by measurements of contact angles of water on gold surfaces. In 1964, White<sup>19</sup> reported results of observations of the wetting of gold surfaces by water under atmospheric conditions. He obtained dropwise condensation from air saturated with water vapor under conditions of cleanliness during surface preparation and wettability measurement which were adequate to render base metals hydrophilic, i.e., able to spread water spontaneously with a zero contact angle. He ultimately reported the contact angle as  $60 \pm 5^\circ$ .<sup>20</sup> In 1965, Erb<sup>21</sup> reported continuous dropwise condensation of water vapor in a gold-plated still at atmospheric pressure. Fused quartz and nonnoble metals were observed to yield filmwise condensation under these same conditions. Shortly thereafter, Bewig and Zisman<sup>22</sup> reported a water contact angle of  $0^\circ$  on a gold disk which had previously been heated to near melting in a flowing atmosphere of hydrogen (at atmospheric pressure) purified of organic contaminants. When the precautions against contamination of the flowing hydrogen

were relaxed, the contact angle was no longer zero. The authors concluded that non-zero contact angles, as cited previously, for water on gold were a result of hydrophobic organic contamination. White,<sup>19</sup> on the other hand, claimed that the zero contact angles observed by Bewig and Zisman resulted from hydrophilic contamination introduced during heating of the sample and by the embedding of alumina abrasive during previous sample polishing. In 1970,<sup>23</sup> Bennett and Zisman reported the results of measurements of the contact angle of water on gold made under conditions designed to eliminate the possibilities of hydrophilic contamination which had been suggested by White and Erb. To avoid surface segregation of metallic impurities, the surface was not heated during preparation. The surface was polished with magnesia followed by leaching with hydrochloric acid to remove any residual embedded abrasive. A contact angle of  $0^\circ$  was found for water on this gold surface.

If the surfaces are markedly rough, the contact angle observed will not be the true angle representing the work of adhesion of the liquid to the solid. The effect of roughness is to increase the apparent angle if the true (advancing) angle is greater than  $90^\circ$ , but to decrease it if the true angle is less than  $90^\circ$ . This is because the liquid penetrates into the hollows if the true angle is less than  $90^\circ$ ; part of the supposedly plane surface consists not of the solid but of the liquid, which has a zero contact angle against itself. If the true angle exceeds  $90^\circ$ , no liquid penetrates into the hollows, the apparently plane surface of solid consists therefore partly of solid and partly of air, for which the liquid has practically no adhesion. Coghill and Anderson<sup>24</sup> noted an equilibrium angle of about  $145^\circ$  and an advancing angle of  $160^\circ$  for powdered galena thickly sprinkled on a plate; a plane surface of galena has a much smaller angle. A similar phenomenon has been noticed with

plates covered with fine mineral powders on which a layer of stearic acid had been deposited. The true contact angle of the solid was probably about  $100^\circ$ ; the observed angle on the rough granular surface was about  $160^\circ$ .

The existence of contact angle hysteresis as a result of contamination or heterogeneity on a smooth surface has been discussed by a number of authors.<sup>32</sup> The hysteresis observed in the work on gold can be attributed to the presence on these surfaces of both hydrophilic and hydrophobic areas, or site clusters. Increased surface activation resulted in an increased ratio of hydrophilic to hydrophobic areas, with an accompanying decrease in both advancing and receding angles, until a zero receding angle was obtained. At this point the average surface free energy less that of the liquid-solid interface was sufficient to overcome that of the water and keep it spread. The advancing angle was nevertheless still relatively high due to the inability of the drop periphery to advance across hydrophobic regions. The situation may be pictured on the basis of hydrophilic "islands" in a hydrophobic "sea." As the surface was activated further, the hydrophobic area continued to diminish with an accompanying decrease in the advancing angle until the hysteresis disappeared as the advancing angle reached zero.

It is clear from a comparison of the conventional high-vacuum and ultrahigh-vacuum experiments that the gradual increase in ratio of hydrophilic to hydrophobic area which occurs upon heating can be interpreted in terms of a gradual removal of hydrophobic organic contamination from the real surfaces. For the case of measurements in conventional high vacuum, the activation procedure of high temperature oxidation and



evacuation gradually cleans the surface, until there are sufficient uncontaminated hydrophilic areas to yield, in the case of gold, a low or zero receding angle, and an advancing angle of 20-30°. A point is reached at which additional activation is ineffective and may even result in additional contamination. This is due to the fact that the conventional high vacuum is not an ultraclean system. Once the system is evacuated, the residual organic vapors have ready access to the surface which is partially cleaned, and a steady state is established at which the rate of contamination equals the rate of vacuum clean up. However, in the ultrahigh-vacuum system which consists solely of metal and glass, there are no organic vapors to recontaminate the surface. The high temperature oxidation-evacuation procedure consequently continues to clean the surface until the hysteresis disappears and the advancing as well as receding contact angle is zero.

The inability of the conventional high-vacuum system to clean the gold surface is not observed for the case of a polished fused-silica surface. In the latter case, preliminary heating to 200° is adequate to yield a zero contact angle with water at room temperature. Using water contact angles as a measure of cleanliness, it is apparent that it is far more difficult to remove organic contamination from a gold than from a fused-silica surface. It is clear, then, that a set of conditions which is sufficient to decontaminate one particular surface will not necessarily succeed for a different type.

For the case of any metal other than gold, an uncontaminated real surface would contain chemically combined oxygen, either as a built up surface oxide or as a chemisorbed monolayer. The attainment of a surface

which is clean (oxygen free) as well as uncontaminated with organic material, would then entail a procedure such as ion bombardment to remove any chemically combined oxygen already present, followed by maintenance of a suitable ultrahigh vacuum to prevent oxygen from recombining with the surface before the measurement is completed. Where water vapor is introduced for the measurement, extensive degassing of the water source is not necessarily sufficient to avoid introduction of a sufficient number of oxygen molecules to chemisorb to a few square centimeters of an active metal surface. For the case of metals in general, therefore, a special gettering technique<sup>33</sup> would have to be devised to ensure measurement on an oxygen-free surface.

#### 4.3 Preparation of Pure Water

The problem of providing very pure water for wetting of heat pipes deserves mention. High purity water is an exceedingly rare commodity. Furthermore, water obtained from ion exchange resins has been of considerable value for much electrolytic conductance work, but such water is not necessarily useful for heat pipes. It is believed that ion exchange resins tend to contaminate the water with organic substances and, furthermore, a test of the specific conductivity of water is obviously far from a satisfactory test for the presence of nonionic, surface-active impurities. Franks<sup>25</sup> has measured the surface tension of water at 25°C obtained from tap water which had been passed through Elgastat deionizers. In spite of the fact that this water--from the point of conductivity studies--was of extremely good quality (10 megohm resistivity, i.e., conductivity  $10^{-7}$ /ohm cm), its surface tension was as low as 68 to 70 dynes/cm at 25°C. Franks showed that it decolorized alkaline potassium permanganate.

Other authors have doubted the permissibility of using water obtained from ion exchange resins for surface wetting.<sup>26</sup> Thus, it seems fair to insist that water for surface wetting must be obtained by distillation. Suitable apparatus for this purpose has been described by various authors. The apparatus devised by Franks<sup>25</sup> seems very practical. A somewhat similar device has been described by J. E. Taylor<sup>27</sup> while a more laborious scheme for purifying water has been described by Butler.<sup>28</sup> The essential element of Butler's method is the recycling in a vacuum of each batch of water.

The water obtained with Butler's apparatus has extremely good resistive properties and is also of excellent quality for wetting phenomena. The disadvantage of the system described by Butler is the rather low rate of production (say 25 to 50 ml per hour). A very sophisticated apparatus for production of extremely good water has been described by Eigen and DeMaeyer.<sup>29</sup> The water obtained in this manner has a specific conductance of only  $5.7 \times 10^{-8}$ /ohm cm at 25°C. The authors do not state any value for the surface tension of this water; however, from the description of the apparatus, it appears that the water would be excellently suited for surface studies. Again, the limitations are the low rate of production and the complicated apparatus.

#### 4.4 Measurement of Wetting

The experimental determination of contact angles is complicated by two factors: first, that the angle depends on the surface tension of the liquid surface; second, that the angle is rarely definite, but may have any value between two extremes, according as the liquid is tending to advance over a dry surface, or to recede from a previously wetted one.

The effect of contamination of the liquid surface is to diminish the contact angle. This is clear from the equation:

$$W_{SL} = \gamma_{LA} (1 + \cos \theta) \quad (29)$$

The value of the adhesive energy,  $W_{SL}$ , will not be affected by contamination on the liquid-air surface such as insoluble grease on water, but  $\gamma_{LA}$  will be diminished. Hence the value of  $\cos \theta$  must be greater if the liquid surface tension is diminished by contamination, and  $\theta$  must be less. The importance of a clean liquid surface for determining contact angles has not always been recognized; some the the results in the literature are probably considerably too low owing to no provision having been made for cleaning the surface.

The second complication, that the angle depends on whether the liquid is advancing over or receding from a solid surface, has also been often overlooked. It may, however, be observed without any apparatus with almost any rain drop travelling down an ordinarily dirty window pane; these appear to stick somewhat in their downward path and the lower edge has a much larger angle than the upper. The angle is much larger, usually, if the liquid is advancing than if it is receding; forces insufficient to move the line of contact of liquid and solid raise or lower the angle anywhere within

the limits of the "advancing" and "receding" angles, beyond which the line of contact moves along the surface. This difference between advancing and receding angles is often called the "hysteresis" of the contact angle. It is well known to those who have measured surface tension by the rise in a capillary tube, for the rise is often less if the liquid is pushing its way up from below than if it is falling from a point above its final equilibrium height.

The cause of the "hysteresis" of contact angles is still obscure. It looks superficially as if there was a frictional resistance to motion of the liquid edge over the solid. Sometimes, if sufficient time is allowed, the liquid settles down to an equilibrium, stationary angle, the same after either advancing or receding motion.

The main experimental problem in measuring the wettability of clean copper consists of admitting water vapor without any accompanying trace of oxygen. The extreme precautions taken to accomplish this consist of the following: (1) Degassing the liquid water into the sorption pump by momentarily opening a valve and then closing it as the top water layer starts to freeze from evaporation. This operation should be performed repeatedly. (2) The water should be exposed to a chamber evacuated to ultrahigh vacuum by means of an ion pump. This operation should be performed several times. The water reservoir remaining after treatment according to this and the previous step should serve as the original source of water vapor throughout all the experiments with copper. (3) Prior to admittance to the sample chamber for contact angle measurement, water vapor from the degassed liquid should be adsorbed onto clean (oxygen-free) germanium powder in an intermediate chamber and allowed to equilibrate for at least one hour. The germanium should be previously cleaned by heating in vacuo

at 700°, and be regenerated after each run. Clean germanium rapidly chemisorbs a monolayer of oxygen. The efficiency of the germanium powder should be monitored in a separate experiment by deliberate adsorption of oxygen (after one of the 700° cleanings) which could be measured by pressure difference with a thermistor pressure sensor. The amount of oxygen chemisorbed should approximately be equal to that which would have been dissolved in the entire water reservoir if it were in equilibrium with the atmosphere. Since the reservoir should be thoroughly degassed and since, furthermore, only a small portion of it should vaporize into the vacuum chamber for contact angle measurement, the capacity of the germanium powder should far exceed that necessary to completely free the water vapor of any possible oxygen residue.

From the description of obtaining pure, oxygen-free water, it is clear that this quality of water should not be expected to be obtained in the fabrication procedures used in heat pipes. For this reason, the contact angle experiments recommended in the next section will utilize water degassed in a conventional vacuum system ( $10^{-6}$  torr) rather than an ultrahigh vacuum system ( $10^{-10}$  torr).

## 5.0 RECOMMENDATIONS AND PROGRAM PLAN

### 5.1 Recommendations and Statement of Proposed Work

To aid proper design of copper heat pipe surfaces capable of providing long-term reliable operation, it is recommended that the wetting of various types of copper substrates--metallic copper, cuprous oxide or cupric oxide--be determined. It is also recommended that the cupric oxide surfaces be prepared by two different techniques to determine how surface preparation affects wetting and that cuprous oxide surfaces be prepared at various partial pressures of oxygen to determine the effect of surface condition on wetting. In addition to wetting, stability of heat pipe surfaces is equally important for long-term reliable operation. In accordance with the results of the thermodynamic analysis, which revealed that copper heat pipe surfaces can be expected to consist of a mixture of solid cupric oxide and cupric hydroxide, it is recommended that the stability of the wettability of this surface be determined in accelerated tests employing conditions of elevated temperature and larger partial pressures of oxygen than expected in normal operation.

The minimum proposed work recommended for aiding in the proper design of heat pipe surfaces capable of providing long-term reliable operation is summarized in the following work statement.

#### STATEMENT OF WORK

A statement of work for the proposed program is as follows:

1. Prepare cupric oxide substrates by the techniques of gaseous oxygen oxidation and of passing gaseous oxygen through steam.
2. Prepare cuprous oxide substrates by the technique of passing gaseous oxygen through steam.



3. Measure contact angles of water on cupric oxide, cuprous oxide and pure copper substrates under conditions permitting variations in the partial pressures of carbon dioxide and oxygen--performing these measurements at room and elevated temperatures.
4. Determine the stability of cupric oxide substrates in an accelerated test employing conditions of elevated temperature and high partial pressure of oxygen.
5. Prepare a final report.

## 5.2 Program Plan

| Surface Wetting                                                               | Months |   |   |
|-------------------------------------------------------------------------------|--------|---|---|
|                                                                               | 1      | 2 | 3 |
| 1. Prepare metallic copper, cuprous oxide and cupric oxide surfaces.          | →      |   |   |
| 2. Measure contact angles of water on copper surfaces.                        |        | → |   |
| 3. Interface with Teledyne Energy Systems and Battelle Columbus Laboratories. |        |   | → |
| 4. Prepare a final report.                                                    |        |   | → |

### 5.3 Program Mangement

The Research Triangle Institute's Engineering Division, under the direction of Dr. J. J. Wortman, will have overall responsibility for the proposed program. The Laboratory Supervisor for this program will be Dr. J. W. Harrison, Manager, Engineering Physics Department. Dr. D. E. Gilbert will serve as Project Leader and will coordinate the program. Dr. D. E. Gilbert has experience in surface wetting, corrosion, and thermodynamic analysis. He will conduct the preparation of copper surfaces, measurements of contact angles, determination of the stability of the cupric oxide surface condition, and prepare a final report. Technical support will be supplied by Mr. R. T. Pickett.

The man-hour commitments to the proposed program are as tabulated below:

Gilbert - 101 hours - Physical Chemist

Pickett - 84 hours - Technician

Straube - 40 hours - Clerical Support.

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TES-SNSO-3-30

APPENDIX H

Technical Summary Report for Axially-Grooved Heat Pipe  
Study, Dynatherm Corporation, DTM-75-3, June 30, 1975.

DTM-75-3

TECHNICAL SUMMARY REPORT  
for  
AXIALLY-GROOVED HEAT PIPE STUDY

June 30, 1975

Prepared under Purchase Order B-70605

for

Teledyne Energy Systems  
Timonium, Maryland

by

Dynatherm Corporation  
Cockeysville, Maryland

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## I. INTRODUCTION

This report describes the work done by Dynatherm Corporation under Teledyne Energy Systems Purchase Order Number B-70605. The program was initiated on May 16, 1975 and completed on June 30, 1975.

The objective of the program was to conduct a study on axially-grooved copper/water heat pipes as applied to the requirements of RTG's. Toward this objective, a detailed parametric computer analysis of the performance of such heat pipes was completed. It was found that relatively small diameter heat pipes (e.g., 1/4 inch O.D.) are sufficient to meet most of the specified heat transport requirements. The data presented in Section II of this report are general enough to make them applicable to a variety of geometries and RTG systems.

For applications where the operating temperature is below the useful temperature range of water as working fluid, alternate fluid/material combinations were studied. This is the subject of Section III. It was concluded that ammonia in an extruded aluminum heat pipe provides the best alternate choice below 40°C.

Section IV provides a technical discussion of heat pipe performance under "g" effects due to spacecraft spin. Closed form analytical expressions were derived for heat pipes located either parallel to the spin axis or in a plane normal to the axis.

In Section V, an assessment of the fabricability of various axial groove geometries is made. Several manufacturers of grooved tubing were contacted and cost estimates were obtained. The indicated limitations should be used in conjunction with the parametric results of Section II to select an optimum groove geometry for a particular application.

Finally, a small experimental effort was undertaken to improve the wetting characteristics of copper surfaces without chemical treatment. Part of this work was done under Purchase Order B-70269; however, for convenient reference, it is included in this report.



## II. PERFORMANCE DATA BASE OF AXIALLY-GROOVED COPPER/WATER HEAT PIPES

### II-1 Introduction

The data base presented in this section provides a background for the selection of a heat pipe design for a specified requirement. Emphasis has been placed on heat transport capability and weight for the range of heat pipe geometries studied. Axial grooves, as shown in Figure II-1, was the common wick geometry studied. The data base was generated by using the HPAD computer program. This program is described in Volume 2 of Dynatherm's Heat Pipe Design Handbook (HPDH). The HPAD program uses an approximate closed form solution of the pressure balances in a heat pipe to determine its maximum heat transport capability. The program can generate performance data for a large number of geometries and temperatures. A check comparison between predictions of the HPAD program and those of a more complicated computer program indicated a very good agreement. The latter program employs a more rigorous analysis and has been verified repeatedly by experimental results. Because of this satisfactory comparison between these programs, the data base generated by HPAD should be sufficiently accurate to meet the requirements of design calculations.

### II-2 Heat Pipe Performance Requirements

This part of the study is restricted to axially-grooved copper/water heat pipes. The ranges of parameters under investigation are listed in Table II-1.

Table II-1

|                        |   |                                         |
|------------------------|---|-----------------------------------------|
| Heat Pipe Diameters    | : | 3/16 to 3/4 inch                        |
| Operating Temperatures | : | 40 <sup>0</sup> C to 150 <sup>0</sup> C |
| Heat Transport         | : | 800 to 3000 Watt-inch                   |
| Heat Pipe Length       | : | up to 3 feet                            |

FIGURE II-1  
TYPICAL AXIALLY-GROOVED HEAT PIPE

### II-3 Heat Transport Capability

In many cases, the axial heat transport capability of a heat pipe can be conveniently expressed in terms of the Heat Transport Capability Factor  $(\dot{Q}L)_{\max}$ . According to the HPDH where  $(\dot{Q}L)_{\max}$  is derived and discussed,  $(\dot{Q}L)_{\max}$  is completely defined by the wick properties and the heat pipe's orientation with respect to an acceleration field. Once the  $(\dot{Q}L)_{\max}$  capability is known for a particular design, it can be compared directly to the specified requirement which is expressed in terms of the Heat Transport Factor  $\dot{Q}L$ . Note that  $\dot{Q}L$  is a requirement and  $\dot{Q}L_{\max}$  is a capability. In order to meet the specified requirement, the following relation must hold:

$$\dot{Q}L \leq (\dot{Q}L)_{\max} \quad \text{II-1}$$

The Heat Transport Factor  $\dot{Q}L$  is defined by the integral:

$$\dot{Q}L = \int \dot{Q}(x) dx \quad \text{II-2}$$

where  $\dot{Q}(x)$  is the axial heat flow rate.

For a simple heat pipe with evaporator and condenser at opposing ends, the integration is extended over the length of the heat pipe. In this case,  $\dot{Q}L$  becomes:

$$\dot{Q}L = \dot{Q}_{\text{total}} \left( \frac{1}{2} L_e + L_a + \frac{1}{2} L_c \right) \quad \text{II-3}$$

where  $\dot{Q}_{\text{total}}$  is the total heat throughput, and  $L_e$ ,  $L_a$ ,  $L_c$  are evaporator, adiabatic, and condenser lengths, respectively. The expression in parenthesis in Equation II-3 is usually referred to as the "Effective Length,  $L_{\text{eff}}$ ".

In a heat pipe with multiple condensers and/or evaporators, the proper procedure for evaluating  $\dot{Q}L$  is shown in the following example for a "L" shaped heat pipe having a single evaporator and dual condensers. The distribution of  $\dot{Q}(x)$  and  $\int \dot{Q} dx$  is shown schematically in Figure II-2. The two condensers have purposely been chosen of different lengths in order to make the example more illustrative. If the condensers were of equal length, the integral of  $\dot{Q} dx$  over the entire length would be zero. But the heat

FIGURE II-2  
EVALUATION OF HEAT TRANSPORT FACTOR  $\dot{Q}_L$   
FOR DUAL CONDENSER "L" HEAT PIPE

transport requirement is always finite. It is given by the maximum value of the integral between any two points along the heat pipe.

Using a few simple graphs, the concept of  $\dot{Q}L$  and  $(\dot{Q}L)_{\max}$  permits the representation of an infinite number of heat pipe lengths and input/output distributions. However, there are some restrictions to the applicability of  $(\dot{Q}L)_{\max}$  which are discussed in the HPDH. This reference should be consulted whenever an acceleration vector aids the liquid circulation in the heat pipe. Also,  $(\dot{Q}L)_{\max}$  is only applicable if the vapor flow is laminar. For turbulent vapor, the actual heat pipe length must be specified.

#### II-4 Presentation of Data

The analysis was performed for the following heat pipe diameters and operating temperatures:

Heat Pipe Diameters : 3/16, 1/4, 3/8, 1/2, 3/4 inch

Temperatures : 40, 80, 120, 150°C

The wall thickness for each diameter was selected on the basis of fabricability rather than pressure containment. Table V-1 of Section V lists the wall thicknesses used in this study. A nominal heat pipe length of 24 inches (of which 6 inches constitute the evaporator, 12 inches an adiabatic section, and 6 inches the condenser) was used. Thus, the effective length of this representative heat pipe is 18 inches. All data are presented in terms of  $(\dot{Q}L)_{\max}$  and are, therefore, applicable to any heat pipe length. However, as pointed out in the previous subsection, the  $(\dot{Q}L)_{\max}$  representation is strictly valid only for the range of laminar vapor flows. For the turbulent range, divide  $(\dot{Q}L)_{\max}$  by  $L_{\text{eff}}$  (0.457 meter) to obtain the achievable  $\dot{Q}_{\text{total}}$  for this representative heat pipe:

$$\dot{Q}_{\text{total}} \text{ (Watts)} = (\dot{Q}L)_{\max} \text{ (W-m)} \frac{1}{L_{\text{eff}} \text{ (m)}} \quad \text{II-4}$$

But, as a first approximation, the  $(\dot{Q}L)_{\max}$  values can be used for the heat pipes capability in the turbulent range.

The data presented here are plotted as a function of groove aspect ratio (defined as the ratio of depth-to-width). Three groove widths (0.010 inch, 0.0175 inch, and 0.025 inch) and two land thicknesses (0.010 inch and 0.020 inch) were considered. The selection of the three groove widths was based on considerations of fabricability and the results of preliminary computer runs. Data from manufacturers indicated that groove widths of less than 0.01 inch were impractical. Results from initial computer runs indicated that groove widths larger than 0.025 inch did not offer any advantages. Land thickness between 0.01 and 0.02 inch were investigated. As expected, there is no advantage to making the land any thicker than necessary to achieve fabricability. In all cases, increasing land thickness results in a heavier heat pipe with some reduction in transport capability. At the higher aspect ratios, the reduction in transport capability is small or nonexistent. This is because the reduced vapor space results in increased vapor losses until vapor losses become the dominating loss. Because of the relative unimportance of larger land widths, they were considered for only a few representative cases.

The results of the computer study are shown in Figures II-3 through II-28. The first 20 graphs (Figures II-3 through II-22) present  $(\dot{Q}L)_{\max}$  for the zero gravity case and a land thickness of 0.010 inch. The remaining six graphs (Figures II-23 through II-28) present  $(\dot{Q}L)_{\max}$  for the zero gravity case and a land thickness of 0.020 inch. Each graph shows  $(\dot{Q}L)_{\max}$  as a function of aspect ratio for three groove widths and for one temperature, tube O.D., and land thickness. At aspect ratios of 1, 2, 3, and 4, the actual heat pipe weight per unit length (including the weight of the working fluid) and the number of grooves are given. Finally, the graphs indicate the range of turbulent vapor flow as dotted lines.

The performance in a one "g" environment or in an equivalent acceleration field due to rotation can be evaluated with the aid of Figure II-29. It gives the static wicking height for the three groove widths as a function of temperature. Within the laminar range  $(\dot{Q}L)_{\max}$  decreases linearly with elevation of the heat pipe and becomes zero at an elevation equal to the static wicking height:

$$(\dot{Q}L)_{\max} \left[ \text{at } h \right] = (\dot{Q}L)_{\max} \left[ \text{at } 0 \text{ "g"} \right] \left( 1 - \frac{h}{h_{\max}} \right) \quad \text{II-5}$$

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# LEGEND

TEMPERATURE = 40°C  
TUBE OUTER DIAMETER = 3/8"  
LAND THICKNESS = 0.0100"  
TUBE WALL = 0.0300"

GROOVE WIDTH:

- 0.0100 INCH
- △ 0.0175 INCH
- 0.0250 INCH

--- TURBULENT FLOW  
XX GMS/METER  
(XX) NO. OF GROOVES

FIGURE II-5

LEGEND  
 TEMPERATURE = 80°C  
 TUBE OUTER DIAMETER = 3/4"  
 LAND THICKNESS = 0.0100"  
 TUBE WALL = 0.0350"

GROOVE WIDTH:

- 0.0100 INCH
- △ 0.0175 INCH
- 0.0250 INCH

--- TURBULENT FLOW  
 XXX GMS/METER  
 (XX) NO. OF GROOVES

FIGURE II-12

FIGURE II - 16

# LEGEND

TEMPERATURE = 120°C  
 TUBE OUTER DIAMETER = 3/4"  
 LAND THICKNESS = 0.0100"  
 TUBE WALL = 0.0350"

GROOVE WIDTH:

- 0.0100 INCH
- △ 0.0175 INCH
- 0.0250 INCH

--- TURBULENT FLOW  
 XXX GMS/METER  
 (xx) NO. OF GROOVES

FIGURE II-17

TEMPERATURE = 150°C  
TUBE OUTER DIAMETER = 3.16"  
LAND THICKNESS = 0.0100"  
TUBE WALL = 0.0270"

GROOVE WIDTH :

⊙ 0.0100 INCH

△ 0.0175 INCH

00250 INCH

--- TURBULENT FLOW

XXX GMS/METER

(xx) NO OF GROOVES

FIGURE II -18

FIGURE II - 19

FIGURE II - 21

FIGURE II - 27

TEMPERATURE = 150°C  
TUBE OUTER DIAMETER = 3/4"  
LAND THICKNESS = 0.0200"  
TUBE WALL = 0.0350"

GROOVE WIDTH:

○ 0.0100 INCH  
 △ 0.0175 INCH  
 □ 0.0250 INCH

GROOVE WIDTH:

① 0.0100 INCH

△ 0.0175 INCH

0.0250 INCH

--- TURBULENT FLOW

XXX GMS/METER

(xx) NO. OF GROOVES

FIGURE II - 28

In the range of turbulent vapor flow, Equation II-5 is only an approximation.

Table II-2 summarizes the maximum achievable transport capability in zero "g" for each of the diameters and temperatures considered. This table can serve as a quick reference for initial heat pipe selection using the graphs to provide the detailed information.

## II-5 Conductance Coefficients

The equations for calculating heat pipe conductances are delineated in Section T-8 of the HPDH. In order to utilize these equations, the vapor pressure drop, the wall conductances, and the internal film coefficients must be known. From information obtained as a result of computer runs, it was determined that the vapor pressure drop is negligible (i. e.,  $\Delta T_v \lesssim 0.5^\circ\text{C}$ ) for all cases considered. Therefore, the thermal conductance associated with axial vapor flow can be neglected. Calculation of the wall conductances is straightforward. The evaporator and condenser film coefficients are more difficult to obtain. The limited experimental data, available at the present time, are listed in Table II-3. Of these data, only one set was obtained with an axially-grooved copper/water heat pipe. The heat pipes developed for the HPG and the Vuilleumier refrigerator were also copper/water pipes but they contained copper mesh wick and circumferential grooves on the inside wall. The film coefficients derived from the HPG pipes are lower than the other two sets of data. However, in these pipes, heat was applied and removed over only one-half of the circumference; but the coefficients were calculated based on the total circumference. Thus, the listed values could be low by as much as a factor of two. In summary, a value of  $3000 \text{ Btu/hr-ft}^2\text{-}^\circ\text{F}$  for both evaporator and condenser film coefficient for grooved copper/water heat pipes appears to be a reasonable first estimate. Additional experimental work is obviously needed to determine more accurate data.

| Tube<br>O. D. | $(\dot{Q}L)_{\max}$ (Watt-meters) |          |           |           |
|---------------|-----------------------------------|----------|-----------|-----------|
| Inches        | T = 40°C                          | T = 80°C | T = 120°C | T = 150°C |
| 3/16          | 4                                 | 14       | 30        | 38        |
| 1/4           | 20                                | 55       | 88        | 110       |
| 3/8           | 98                                | 178      | 282       | 355       |
| 1/2           | 204                               | 412      | 695       | 850       |
| 3/4           | 638                               | 1255     | 2110      | 2460      |

Range of Groove Widths : 0.01 Inch to 0.025 Inch  
Range of Aspect Ratios : 1 to 4

TABLE II-2  
MAXIMUM HEAT TRANSPORT CAPABILITY FACTOR  
FOR ZERO "G"

| Temperature<br>(°C) | Evaporator<br>(Btu/hr-ft <sup>2</sup> -°C) | Condenser<br>(Btu/hr-ft <sup>2</sup> -°C) | Reference                                                                                                            |
|---------------------|--------------------------------------------|-------------------------------------------|----------------------------------------------------------------------------------------------------------------------|
| 58                  | 2971                                       | 3390                                      | Experimental Data for 1/4 Inch<br>Grooved Copper Tube<br>(Dynatherm)                                                 |
| 46                  | 2839                                       | 4080                                      | DTM-71-10, "Design and Fabrication<br>of an Ambient Temperature Interface<br>Simulator for Vuilleumier Refrigerator" |
| 66                  | 2080                                       | 1269                                      | DTM-73-9, "Development, Fabrication,<br>and Testing of Heat Pipes for High Per-<br>formance Generators"              |
| 177                 | 1987                                       | 1652                                      | DTM-73-9                                                                                                             |

TABLE II-3  
EXPERIMENTAL FILM COEFFICIENTS  
FOR COPPER/WATER HEAT PIPES

### III. LOW TEMPERATURE ALTERNATIVES TO COPPER/WATER HEAT PIPES

In this report, copper/water heat pipes have been evaluated for the temperature range from 40°C to 150°C. 40°C appears to be a reasonable lower limit for water heat pipes, although, in a pinch, acceptable performance down to 20°C can be obtained. The lower temperature limit is dictated not so much by the properties of the liquid, which remain good down to the freezing point, but by the low vapor density. An obvious absolute lower limit is, of course, the freezing point at 0°C.

A number of alternate fluids are available for heat pipe operation below 40°C. An estimate of comparative performance can be obtained from the Liquid Transport Factor  $N_L$  which is defined in the HPDH. This Liquid Transport Factor is shown in Figure III-1 for six alternate fluids in addition to water. These fluids are:

- Methanol
- Acetone
- Ammonia
- Benzene
- Freon-11
- Freon-21

Before selecting a specific fluid, a few comments regarding the container material are in order. For water heat pipes, copper is the logical choice because it is the only material known to be completely compatible with water. Copper can easily be formed into axially-grooved tubing. This is clearly an advantage, but copper is also heavy and not very strong. Also, copper is not compatible with the most promising of the lower temperature fluids; namely, ammonia. All of the low temperature fluids (with the exception of methanol) are compatible with aluminum tubing. Aluminum can also be formed into axially-grooved heat pipes, has low specific weight, and has good strength. Thus, the choice for heat pipe materials below the water range appears to be aluminum.

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The above selection of aluminum eliminates methanol as a working fluid.\* Freon-11 and Freon-21 fall in the same temperature range as ammonia but are inferior in transport properties. Benzene has not been intensively investigated as a heat pipe fluid. That leaves ammonia and acetone as the preferred working fluids below the temperature range of water. The predicted heat transport capability of these two fluids is shown in Figure III-2 for the standard NASA axially-grooved tubing. This groove geometry is similar to the one utilized on OAO-C and ATS-F. A cross-section of this tubing is shown schematically in Figure II-1. The material is produced by hot extruding 6063 Aluminum alloy. The tubing has been fully qualified by NASA for space applications and is currently used for heat pipes on the Air Force RM20B satellite, the GSFC IUE, and the Navy MSD. Figure III-2 shows, in addition to the heat transport capability, the maximum static wicking height as a function of temperature for the two fluids.

In conclusion, ammonia appears to be the logical choice for an alternate working fluid below 40°C. If used with the existing aluminum extrusions, no additional qualification would be required. Other geometries are certainly feasible but would need development. Acetone is an acceptable working fluid up to approximately 150°C. Its performance is clearly much lower than that of water in the same temperature range, but it may be used in aluminum grooved heat pipes. Thus, if the performance requirements are not too stringent, an acetone/aluminum heat pipe system may result in lower weight than an equivalent water/copper heat pipe.

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\*However, the combination of methanol and copper should be further investigated. Copper has lower strength and higher specific weight than aluminum but can more readily be formed into axially-grooved tubing of different geometries.

#### IV. HEAT PIPE PERFORMANCE UNDER "G" EFFECTS DUE TO SPACECRAFT SPIN

Spinning of a spacecraft in zero gravity introduces an acceleration field which has similar effects on heat pipe performance as a gravity field. The local acceleration (a) is always normal to the rotation vector and has the magnitude:

$$a = r \omega^2$$

IV-1

where r is the distance from the axis of rotation and  $\omega$  is the angular frequency of rotation.

Accelerations are equivalent to a body force on liquid and vapor in a heat pipe. Depending on their direction, they may impede or assist the circulation of the working fluid. Because of the large difference in density between liquid and vapor, body forces on the vapor can usually be neglected. With regard to the liquid, one must distinguish between forces parallel and perpendicular to the heat pipe axis. The parallel component of body forces adds or subtracts directly to or from the available capillary force. The perpendicular component has a more subtle effect which will be discussed later.

The influence of body forces due to spinning cannot be defined quantitatively without specifying the geometry of the heat pipe, its orientation with respect to the spin axis, and its heat transport requirement. The following discussion is therefore restricted to some general relations which should enable the user to apply them to a specific case. Furthermore, typical examples for some frequently encountered geometries are given.

For background and reference, the Heat Pipe Design Handbook published by Dynatherm Corporation is being used (in the following referred to as HPDH). Equations T-61 through T-65 in conjunction with Figure T-7 of the HPDH give a closed form solution of the transport capability of a heat pipe in the presence of a gravitational body force. The significant parameter is  $\eta$  (Equation T-62) which is defined as the ratio of the sum of all pressure differences resulting from body forces to the available capillary pressure. For the case of an axially grooved heat pipe in a gravity field, this parameter should be modified as follows (the wetting angle  $\theta$  is assumed to be zero):

$$\eta = - \frac{r_p}{2H} \left( \frac{2w + \delta}{\pi} \right) + \frac{r_p}{2H} L \sin \beta \quad \text{IV-2}$$

where  $w$  is the groove width and  $\delta$  its depth.

If the prevailing body force is due to rotational acceleration (spinning), the effects can be expressed for most cases as a modification to the parameter  $\eta$  as given by Equation IV-2. The following two cases are representative of most geometries encountered in spinning spacecrafts.

#### IV-1 Heat Pipe Axis Parallel to Spin Axis

In this case, the rotational acceleration has no component in the direction of the heat pipe axis. The parameter  $\eta$  becomes:

$$\eta = - \frac{r_p}{2H} \left( \frac{2w + \delta}{\pi} \right) \frac{r_o w^2}{g} \quad \text{IV-3}$$

where  $r_o$  is the distance between the axis of rotation and the heat pipe, and  $g$  is the gravitational constant. Note that the term  $r_o w^2/g$  is the equivalent number of "g's" acting on the heat pipe. Incidentally, this case applies also to a hoop shaped heat pipe located in a plane normal to the axis of rotation.

#### IV-2 Heat Pipe Axis in a Plane Which is Perpendicular to Spin Axis

This case covers the majority of practical applications. The heat pipe axis may or may not intersect the spin axis. Referring to Figure IV-1 for definition of terms, the parameter  $\eta$  becomes for this case:

$$\eta = - \frac{r_p}{2H} \left( \frac{2w + \delta}{\pi} \right) \frac{r_o w^2}{g} - \frac{r_p}{2H} \frac{w^2 L (d + \frac{1}{2} L)}{g} \quad \text{IV-4}$$

Equation IV-4 in conjunction with the other pertinent equations of the HPDH (T-61 through T-65) describe completely the effects of spin rotational acceleration on heat pipe transport capability. However, there are some restrictions which must be recognized.

FIGURE IV-1  
SCHEMATIC OF ROTATING HEAT PIPE

- (1) The heat pipe must be straight and located within a plane normal to the axis of rotation.
- (2) The heat pipe must not extend past the point of minimum distance from the axis of rotation. This restriction implies that the rotational acceleration is in the same direction everywhere along the heat pipe.
- (3) The evaporator of the heat pipe must be closer to the axis of rotation than the condenser.

Some comments to the above restrictions are necessary. The theory presented in the HPDH is general enough to cover all orientations and geometries of heat pipes. But care must be employed in applying it to specific situations. For example, if the rotational body force aids the liquid return (contrary to Restriction 3), the closed form solution of the HPDH does not apply. In this case, one would design the heat pipe for zero "g" conditions and use the additional pumping due to body forces as an added design margin. If the heat pipe is not located in the plane normal to the axis (Restriction 1), the local components of the body forces must be evaluated and applied to the theory given in Section T-61 of the HPDH. The same applies to Restriction 2. However, it should be noted that the majority of practical cases is covered by the equivalent  $\eta$  parameters of Equations IV-3 and IV-4.

V. PROCUREMENT SURVEY AND COST ESTIMATES  
FOR AXIALLY-GROOVED TUBING

Tubing with internal axial grooves has been manufactured by hot extrusion and cold swaging processes. Micro Extrusions, Inc., (LaHabra, California) has manufactured axially-grooved 6063 and 6061 aluminum tubing using the hot extrusion process. Their process is not applicable to copper because of copper's work hardening properties. Noranda Metals Industries, Inc., (Newtown, Connecticut) has manufactured axially-grooved 6061 aluminum tubing for the ATS program by the cold swaging process. They have demonstrated the feasibility of manufacturing this tubing using copper.

Noranda Metals Industries' advice was requested on the fabrication of copper grooved tubing in the size range of 3/16 inch O.D. to 3/4 inch O.D. Relatively thin walls were desired for thermal reasons. The manufacturer recommended that a minimum wall thickness of 0.027 inch would apply to 3/16, 1/4, and 5/16 inch O.D. tubes. This wall thickness would increase progressively and become 0.035 inch for 3/4 inch O.D. tubing. These wall thickness values are greater than are required for pressure containment in copper/water heat pipes (see Teledyne Energy Systems Memoranda LCHP-042a and LCHP-042b).

The cold swaging process is limited in its ability to achieve high fin height/fin width ratios. A maximum ratio of 4.5 can be achieved for configurations where the number of grooves is small (equivalent to large groove widths). For groove widths of about 0.01 inch, a practical ratio is in the range of 2.5 to 3.0. Axial groove configurations which appear practical for copper are given in Table V-1. The Table V-1 data is not all inclusive. By increasing groove and land widths, the groove depth can be increased to achieve a 4.5 ratio. This results because the number of grooves decreases proportionally.

Since none of the grooved tubes listed in Table V-1 are in manufacture, tooling and setup costs would be incurred. Tooling costs for each geometry are estimated to be \$300.00. Because of the trial and error nature of this process, this cost may be repeated until a satisfactory configuration is obtained. Once tooling is established, a production run of tubing is estimated to cost between \$5 to \$8 per linear foot.

| Tube<br>O.D.<br>(Inch) | Wall<br>Thickness<br>(Inch) | Groove<br>Width<br>(Inch) | Groove<br>Depth<br>(Inch) | Land<br>Thickness<br>(Inch) | Number<br>of<br>Grooves |
|------------------------|-----------------------------|---------------------------|---------------------------|-----------------------------|-------------------------|
| 3/16                   | 0.027                       | 0.01                      | 0.025                     | 0.01                        | 18                      |
| 1/4                    | 0.027                       | 0.01                      | 0.035                     | 0.01                        | 23                      |
| 5/16                   | 0.027                       | 0.01                      | 0.035                     | 0.01                        | 30                      |
| 3/8                    | 0.030                       | 0.01                      | 0.035                     | 0.01                        | 38                      |
| 1/2                    | 0.032                       | 0.01                      | 0.030                     | 0.01                        | 57                      |
| 3/4                    | 0.035                       | 0.01                      | 0.030                     | 0.01                        | 87                      |

TABLE V-1  
POTENTIAL GROOVED TUBE CONFIGURATIONS

## VI. DEVELOPMENT OF SURFACE OXIDATION PROCEDURES

Normally, a copper surface which has been exposed to the atmosphere for more than a few minutes will not be readily wet by water. Previously, Dynatherm has used a surface treatment known as Ebonol (trade name) to condition copper surfaces to obtain excellent wetting properties. This process was used for the HPG heat pipes. Life tests on HPG heat pipes resulted in failures after approximately one year. Analysis of these pipes indicated that the cause of these failures was internal corrosion. The exact cause of this corrosion is not known at the present time. Consequently, alternate surface preparations to achieve water wetting of copper were investigated.

The use of superheated steam to oxidize copper and enhance wetting has been used in the past. This process has the advantage that foreign chemicals are not introduced into the oxidized surface. Basically, the process consists of exposing a heated copper surface to superheated water vapor. Typical surface temperatures are  $150^{\circ}\text{C}$  and the water vapor pressure is less than 50 Torr.

A series of tests were conducted to investigate superheated steam oxidation of copper surfaces. Copper screen wicks, which could be separately heated, were suspended above a contained water reservoir. The system could be evacuated and maintained at any specified vapor pressure. Many tests were conducted at various copper surface temperatures, water vapor pressures, and partial air pressures. The results have been inconsistent. Whenever a surface was found which had good wetting properties, a repeat of the same test produced a surface which had poor wetting properties. In most cases, samples did not have uniform wetting properties. It was thought that cuprous oxide was required to obtain good wetting properties. However, most samples that had a red color did not exhibit any wetting. In summary, the wetting properties could be enhanced under certain circumstances but the exact conditions for obtaining reproducibility have not been discovered.

Another series of tests were conducted on six 1-inch square copper coupons. Three cleaning processes were specified and two samples were cleaned using each process. The first process consisted of cleaning the samples using trichloroethane, followed by etching in 50% nitric acid for sixty seconds, rinsing thoroughly in deionized

water, and air drying. The second process was the same as the first process except that, after the water rinse, the samples were rinsed in isopropyl alcohol and then air dried. The third process was the same as the first process except that, after the water rinse, the samples were rinsed in methanol and then air dried.

A drop of water was placed on each of the samples, and the wetting angle was estimated by observing the droplet under a magnifying glass. One sample from each cleaning process was then stored in distilled water, and the other samples were left exposed for various periods of time to room environment. The wetting angle measurements were repeated several times over a month time interval.

The results of the tests indicate that the surfaces rinsed with water and with methanol initially wetted very well (wetting angles of 20 to 30 degrees). The surfaces rinsed with isopropyl alcohol had poorer wetting properties.

After time exposure to air, wetting properties deteriorated; whereas the samples stored under distilled water continue to have good wetting properties. This illustrated the adverse effect of air storage.

The wetting properties of the above samples do not compare with the spreading of water observed on surfaces which were Ebonoled. The final test to determine whether adequate wetting has been achieved is to fabricate a heat pipe using a selected process and to measure its heat transfer and transport characteristics. These results are then to be compared with similar test results obtained with Ebonoled heat pipes. This was done with a heat pipe manufactured using the third of the above processes. To date, this pipe has performed very poorly. However, because of differences in circumferential groove design, direct comparison of test results may not be valid.

Further work to identify a process to improve wetting properties of copper is clearly indicated. In addition to basic investigations of the wetting characteristics of pure and oxidized copper surfaces, testing of candidate surfaces in a heat pipe environment is important.