MASTER SELENIDE ISOTOPE GENERATOR *for the* GALILEO MISSION

TELEDYNE ENERGY SYSTEMS

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Pictured on the cover is Galileo's drawing of the solar system, which includes the four satellites of Jupiter he discovered in the 1600's. A Renaissance professor, inventor and astronomer, Galileo perfected the telescope with which he made his Jupiter discoveries. The 1982 NASA mission to Jupiter is named in his honor. Like Galileo and his telescope, the NASA mission to the far reaches of outer space will be contributing to Mankind's never ending quest for knowledge.

SELENIDE ISOTOPE GENERATOR

for the GALILEO MISSION

HEAT PIPE ATTACHMENT TECHNOLOGY

FINAL REPORT

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HEAT PIPE ATTACHMENT TECHNOLOGY - PHASES I & IIBackground

On April 1, 1976 Battelle Columbus Laboratories were subcontracted by Teledyne Energy Systems to develop an optimum joining method for attaching copper/water heat pipes to an aluminum alloy (6061-T6) radiator and housing for the SIG/Galileo Program. Specifications required that the joint between the copper heat pipe and the aluminum must have a sufficient thermal conductivity such that there is no more than a 2°C temperature drop at a nominal operating temperature of 125°C (257°F). Further, any type of joining technique which is used must take into account the thermal stability of both the heat pipe and the heat-treated 6061-T6 temper aluminum alloy hardware. Accordingly, a maximum temperature of 177°C (350°F) during joining is desirable, however, this condition was increased to 221°C (430°F) per solder alloy selection. In addition, the resultant joint must be capable of a minimum of 1,500 psi shear strength which was a result of a cursory analysis by TES.

It first appeared that either organic adhesive technology or metallurgical joining technology could both be useful in developing a copper-to-aluminum bond to meet the specifications outlined above. Both approaches were evaluated during Phase I of the research program. The organic adhesive technology was dropped because it failed to meet some of the requirements.

Research Plan

The research plan was divided into two phases. Phase I was concerned with the selection and screening of candidate joining materials using simplistic hardware. On the basis of these tests, final selection of the optimum materials and methods were made. Phase II was concerned with the application of the optimum materials and methods of joining to configured hardware supplied by TES. Finally, specifications were to be prepared in Phase II for the assembly of prototype flight hardware.

To implement the research plan, each phase was split into the following tasks associated with either metal joining or adhesive bonding.

PHASE I: PRELIMINARY SELECTION, SCREENING, AND OPTIMIZATIONTask 1. Metallurgical Joining of Copper to Aluminum

There were several technical aspects which had to be accounted for when developing a metal joining system for attaching copper heat pipes to aluminum alloy, 6061-T6. Some of them were:

- The effect of thermal joining procedures on the mechanical properties of both the aluminum alloy and the copper. For the aluminum alloy proposed, this fixed the joining temperature at a maximum, in the order of 232°C (450°F). The joint fabrication time also had to be limited for the same reason.
- The joinability of aluminum with suitable low-temperature joining procedures and filler materials (solders) and the necessity to prepare the surface of the aluminum with an appropriate solderable metal coating.
- The need for a joint with high integrity capable of uniform heat conduction at minimum ΔT across the joint. Joints which are too thick or those which contain voids are undesirable.

Task 1 was designed to show the feasibility of using the soldering process to join copper heat pipes to aluminum. Certain decisions by Battelle were made on the basis of experience and judgment. The selection of the solder and the base metal preparation technique, followed the experience and judgment criterion.

The solder was chosen on the basis of the melting temperature in order to reduce undesirable metallurgical effects on the base metal properties and on the knowledge that no corrosive exposure would be expected. A Sn-Pb solder containing about 60% tin which begins melting at 182°C (360°F) and is completely molten below 205°C (400°F) was the suggested candidate along with a 60 Sn-36 Pb-4 Ag 170°/172°C (339°/342°F), 63 Sn-37 Pb 183°/185°C (362°/364°F), 61.9 Sn-38.1 Pb, 185°C (362°F) and a 91 Sn-9 Zn eutetic solder melting at approximately 198°C (388°F).

The aluminum had to be carefully prepared for soldering because of the tenacious oxide which forms on its surfaces. Further, the use of some solders directly on magnesium-containing aluminum alloys cause grain boundary penetration. Therefore, the aluminum was prepared by electroplating with copper, followed by a 60 Sn-40 Pb solder plating.

Task 1.1. Development of Soldering Procedure

Initial procedure development work and joint evaluation studies were carried out on flat surfaced specimens (lap shear joints) of copper bar and 6061 aluminum bar. The following experiments were undertaken:

- (a) Studies as to whether joints could be made in air without a flux or if a protective atmosphere must be used.
- (b) Ultrasonic excitation of the joint parts as they reach soldering temperature.
- (c) Evaluation of several heating methods which included direct flame heating of the aluminum or the copper, or both, radiant heating in an oven, infrared heating, and thermal conduction by heating platens. These tests were limited to finding the fastest method for the type of joint desired.
- (d) Tests on flat surfaced joints to show whether or not dewetting would be of concern.
- (e) Evaluating various heating rates and times at soldering temperature to ascertain their effect on base metal, dissolution of the solderable copper coating on the aluminum, faying surface dewetting, and joint strength.
- (f) Radiographic testing to determine the joint quality.
- (g) Cursory mechanical properties and the thermal conductivity testing of the joints.

Task 1.2. Studies of Simulated Joints

Under this task, the objective was to produce and evaluate joints which were long enough and complex enough to show any problems which may arise when fabricating an actual heat exchanger of the proposed design and to indicate ways to overcome these problems.

Task 2. Adhesive Bonding of Copper to Aluminum

Because of the low thermal conductivity of polymeric materials, organic adhesives considered for bonding copper to aluminum were filled with metallic powders or fibers in sufficient quantity to provide particle-to-particle contact. The adhesives selected for evaluation should not require temperatures above 182°C (360°F) for application or curing. Several metal-filled adhesives selected from those commercially available were evaluated using simplified procedures. In addition, BCL's polymer chemists carried out a limited adhesive-formulation study to develop an adhesive with high conductivity and high shear strength at temperatures up to 182°C (360°F).

Under this task, static bond strengths were evaluated on several adhesives by using an Instron tensile machine. A vibration test was supposed to be devised during this task, whereby the vibration spectrum to be encountered during launch would be determined and a "mock-up" provided to simulate anticipated conditions.

PHASE II: APPLICATION TO HARDWARE AND SPECIFICATION DESIGN

The task efforts under Phase II study were engineered to demonstrate the applicability of the most promising metallic and adhesive joining method ascertained as a result of Task 1 and 2 in Phase I. However, the final results of Task 2 concluded that adhesive bonding was not a feasible method of joining copper to aluminum to achieve high shear strength and high thermal conductivity at a nominal operating temperature of 125°C (275°F).

Task 1. Metallurgically Joined (Soldered) Copper to Aluminum

Under this task, BCL was subcontracted to develop two (2) procedural specifications for the electroplating of copper on aluminum (PS0300037) and the electroplating of tin-lead (60Sn.40Pb) on copper (PS0300038). In addition, BCL was to conduct experimental copper-to-aluminum soldered (60Sn.40Pb) joints on "mocked-up" detail components. As a result of this work BCL would prepare the initial procedures to metallurgically join axially-grooved copper/water heat pipes and copper auxiliary cooling tubes to flight hardware.

TASK 1.1 - EXPERIMENTAL WORK CONDUCTED BY BCL - PHASE IITask 1.1.1 Materials and Equipment

The initial procedure development effort was carried out on flat specimens of 6061 aluminum and OFHC copper. Simple lap joints, 0.75 (19 mm) long, 0.75-inch by 6.00-inch wide and 0.041-inch (19 mm by 152 mm by 1.04 mm) thick were made from sheet stock. Tests were limited to finding a technique which would produce uniformly continuous joints in a minimum time at a temperature of 232°C (450°F) or below.

The specimens were prepared for soldering by electroplating with the 60 tin-40 lead solder alloy after masking all but the joint area using the following procedures:

Aluminum

1. Degrease - trichloroethylene
2. Etch in E-14*, 10 oz/gal (75 g/l), 27°C (80°F), 30 secs
3. Rinse
4. Nitric acid (70%) dip, 50/50, 27°C (80°F), 15 secs
5. Rinse
6. Zincate coat 35°C (95°F), 5-10 secs
 - Sodium hydroxide - 120 g/l
 - Zinc oxide - 20 g/l
 - Sodium cyanide - 5 g/l
 - Copper cyanide - 1 g/l
 - Nickel cyanide - 1 g/l
 - (A specific mixing procedure required)
7. Rinse
8. Copper strike (cyanide bath), 30 A/ft² (323 A/m²), 54°C (130°F), 15 min
9. Rinse
10. Fluoboric acid dip, 3 vol.%, 15 secs
11. Tin-lead plate, 50 A/ft² (538 A/m²), 27°C (80°F), 15 min

*Proprietary aluminum etchant made by Enthone, West Haven, Conn.

Tin fluoborate concentrate (19% Sn)-4 liters

Lead fluoborate concentrate (27.5% Pb)-1 liter

Fluoboric acid (48%) - 1 liter

Distilled water containing 250 g dissolved boric acid - 4 liters

Kenvert Reflect Tin-Lead ALPHA* - 0.7 liter

Kenvert Reflect Tin-Lead GAMMA* - 0.1 liter

Dilute total solution 20 liters with distilled water

Use 60 tin-40 lead alloy anodes.

Copper

1. Degrease - trichloroethylene
2. Scrub, Shipley's Scrub Cleaner, #11**
3. Rinse
4. Soak in copper strike solution, no current, 54°C (130°F), 1 min
5. Copper strike (cyanide bath) 30 A/ft² (323 A/m²), 54°C (130°F), 5 min
6. Rinse
7. Fluoboric acid dip 3 vol.%, 15 secs
8. Tin-lead plate, 50A/ft² (538 A/m²), 27°C (80°F), 15 min.

The plating procedure as outlined resulted in tin-lead coating thicknesses of 0.001 inch (0.025 mm). During initial experimental soldering, two thicknesses were evaluated; 0.0010 and 0.0015 inch (0.025 and 0.038 mm). The thicker coating was judged most suitable for the application.

The heating method first evaluated was a combination of hot platens and heated weights. This method provided most of the desired features. It was simple and could be readily expanded for larger specimens. Note: This method of heating was based upon a preliminary design as shown in Figure 1. The hot platens were laboratory fabricated from 0.5 inch (12.7 mm) aluminum plate and heated with electrical resistance strip heaters. The temperature was controlled by several thermocouples inserted in the platens and the loading

*Proprietary brighteners made by Conversion Chemical Corp., Rockville, Conn.

**Proprietary cleaner; acidic, pumice/detergent mixture.

weights. The weights were steel blocks sized to give either a 1 or 5 psi (0.7 or 3.5 g/mm^2) load on the joint area. The lighter unit weight was judged best for the technique utilized.

Task 1.1.2 Development of Technique

As previously indicated, the technique developed was carried out on lap shear joints. This work is summarized by the following:

1. Successful void-free joints were not made by melting the as-plated parts in contact with each other. Neither fluxing with a rosin base flux nor vibrating (60 Hz) while the solder was molten improved the joint quality.
2. The solder coating on both the aluminum and the copper must be premelted before joint assembly as to assure that the coating is gas free.
3. The preferred joint loading technique was to place a hot 1 psi (0.7 g/mm^2) weight on the joint after the solder melted and then vibrate the weight for 1 to 1.5 minutes. This aided in removal of voids from the joint interface.
4. The necessity for fluxing the joint before assembly was not verified. Essentially void free joints were made both with and without flux.
5. The temperature of the hot platen for best soldering conditions was $200^\circ\text{--}204^\circ\text{C}$ ($390^\circ\text{--}400^\circ\text{F}$). The weights are preheated to $204^\circ\text{--}210^\circ\text{C}$ ($405^\circ\text{--}410^\circ\text{F}$). The average time at these temperatures was 3 minutes. The time above the solder melting temperature was less than 10 minutes.

Task 1.1.3 Fabrication of Simulated Production Joints

Aluminum details and copper tubes having joint configurations approximating those for the intended application were furnished by TES. The soldering procedure used to join the copper tube to the aluminum detail for the simulated production joints required construction of suitable hot platens and hot loading weights which could be vibrated during soldering. The platens were constructed from 0.5 in. (12.7mm) aluminum plate. The

loading weights were made up of 6 in. (152. mm) long segments having the cross section shown in Figure 2. The weights provided a load of approximately 1 psi (0.7 g/mm^2) on the joint area during soldering. Four segments were loosely attached to a holding bar to permit vibration while soldering the long leg of the "L" shaped detail. A 1000-watt resistance heater ran through all the blocks. Figure 3 shows one end of the assembled blocks with the loose attachment and holes for the vibrating tool. A short block of the same design but without a built-in heater was used for the short leg of the "L". This block was heated on a separate hot plate.

The procedure used for soldering the simulated production joints duplicated, as nearly as possible, that outlined for the preliminary lap-shear joints. Figure 4 shows an "L" type specimen in place for soldering the long leg of the "L". The short leg was soldered first. As shown, two 60 Hz vibrators were used and each being moved from contact hole to contact hole for short periods during the vibrating cycle. Temperature was controlled by four thermocouples distributed in the hot platen and the loading weights. The data collected while soldering the six joints made are given in Table 1. After vibrating the joint it was immediately loaded with additional weights as shown in Figure 5. The added weight provided a total of about 3.3 psi (2.3 g/mm^2) on the joint area during the solder/freezing period.

Task 1.2 Results and Discussion

The simulated production joints showed that the procedure developed was a viable method of attaching the copper tubes to the aluminum detail. A finished "L" specimen is shown in Figure 6.

All of the simulated production joints were radiographed to learn the extent of porosity in the joint area. The results are summarized in Table 1. No joints were made which were porosity free. This was expected because the necessary assembly techniques do not permit flow of solder through the joint area and because of the desired low soldering

temperature and minimal time at temperature. The radiographs showed the usefulness of vibrating the joint while the solder is molten. All pores were quite small and evenly scattered in the joint because of vibration.

Task 1.2.1 Metallographic Study of Aged Copper-Aluminum Soldered Joints

It was reasonable to assume that the thin copper coating placed on aluminum components to promote solderability would be consumed by diffusion into the tin-lead solder due to elevated temperature exposure. A question then arose: would the solid $\text{Sn/Pb/Cu}_x\text{Sn}_x$ alloy continue to adhere to the aluminum base metal? Naturally, the requirement for long term exposure deterred specific answers to these questions, however, a short time aging/metallographic study was undertaken to ascertain the amount of depletion of copper from the aluminum-solder interface.

One "L" shaped detail was cut into several pieces which were aged in an air oven at 120°C (248°F) for periods of 200, 400 and 760 hours. Cross sections of the joints which were aged for different time periods, were examined metallographically. The results are shown in Figures 7 through 10.

The changes in joint area microstructure occurring with time at 120°C (248°F) are described as follows:

- The unreacted copper thickness on the aluminum is reduced from about 0.0004 in. (0.01 mm) on the as-soldered joint to about 0.0003 in. (0.008 mm) on the joint which was aged 760 hours.
- The "compound layers" increased from about 0.00008 in. (0.002 mm) to about 0.0002 in. (0.005 mm) during aging 760 hours at 120°C (248°F).
- The solder structure is altered from a fine 2 phase, nearly eutectic, structure to a coarse 3 phase structure. No attempt was made to identify these structures. It is believed that the darker phases are high in lead (Pb) content.

These observations are consistent with the literature. There are no available data which permitted the prediction as to when all of the copper coating on the aluminum would go into solid solution with the Sn/Pb. A quotation from the Tin Research Institute, Publication 517, "The Growth of Intermetallic Compounds on Common Basis Materials Coated with Tin and Tin-Lead Alloys" seems to adequately conclude this work:

"The precise way in which the transformation of the ductile solder alloy, progressively, to the more brittle intermetallic compounds and a more lead-rich alloy affects a joint's physical and mechanical properties is not known, but experience has shown that their presence is generally deleterious."

In an effort to develop an indication of the influence of elevated temperature aging on the mechanical strength of the joint system under study, lap shear tensile tests were conducted.

Task 1.2.2 Shear Strength Study of Unaged and Aged Copper-Aluminum Soldered Joints

Lap shear tensile test specimens were prepared from strips of copper 6.0 in. x 0.75 in. x 0.125 in. (152 mm x 19 mm x 3.2 mm) and the same size but thinner (0.04 in. -1.02 mm) 6061 aluminum. One end of each joint was electroplated with 60 tin-40 lead solder. The electroplated solder was fused and cleaned before assembly in the soldering fixture. The soldering fixture was designed to produce a constant lap joint 0.25 in. (6.4 mm) long and having a clearance of 0.003 in. (0.08 mm). A total of 24 joints were made. Slight variations in fluxing techniques were used. This information plus the actual joint clearances obtained are given in Table 2. Specimens receiving different treatments were grouped for aging such that each group contained specimens from each of the different treatments in Table 2. After soldering, four of the groups were aged at $150^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for the times indicated in Table 2. The aging temperature selected was higher than the anticipated nominal operating temperature in order to obtain a qualitative effect of longer times at 120°C . Test results are given in Table 3.

The following statements are made on the basis of the data:

- Heating to 120°C without aging reduced the joint tensile strength about 44 percent.

- Aging at 150°C for 830 hours reduces the 120°C strength about 16 percent and the room temperature strength about 12 percent.
- The greatest loss in strength occurred during the first 200 hours of aging; little difference in joint strength at 120°C was found between 200 and 800 hours.
- The lack of a strength change after 200 hours would indicate that to learn the true effect of elevated temperature aging on the joint system, longer aging times would be required.
- The data collected on these tests appear to be consistent with information found in the literature.

Task 1.3 BCL Conclusions

1. Metallurgical joining by soldering will give a bond between the copper heat pipe and aluminum alloy fin which will have the necessary strength at a nominal operating temperature of 120°C and also possess good thermal conductivity.
2. Based on the limited studies undertaken, aging the solder joint for up to 830 hours at 120°C will give average strengths at 120°C of > 3000 psi. Therefore, failure of the joint, due to intermetallic compound precipitation or consumption of the copper plate spike, does not appear likely.

NOTE: Preliminary stress analysis performed by TES indicated a maximum shear stress requirement of 1500 psi.

PHASE III. CONTINUED HEAT PIPE ATTACHMENT TECHNOLOGYBackground

As a result of the initial work conducted by Battelle Columbus Laboratories, TES prepared SIG 110015, Auxiliary Cooling Tube Installation Procedure, to implement BLC procedures to join a copper tube to aluminum hardware. As shown in Figure 11, the Ground Demonstration System's (GDS) configuration was cylindrical in design and required the design of SIG 111003, Bonding Tool-Auxiliary Cooling System, as shown in Figure 12. The problems encountered were long (≥ 1.0 hr) heat-up rates to achieve soldering temperature of $> 232^{\circ}\text{C}$ (450°F) for the 100% tin solder employed for attaching cooling tubes and the enamelizing and degradation of the rosin flux (Kester 1544) due to the long durations at elevated temperatures. To achieve the auxiliary cooling tube attachment, torch soldering was resorted to with minimal success due to flux degradation.

To achieve adequate procedures to join not only copper auxiliary cooling tubes to cylindrical generator housing but copper/water heat pipes to various configurations, TES sponsored a continued joining technology program at BCL. Under this subcontract Statement of Work, BCL would redefine the electroplating procedures and electroplate at least 0.005 inch thick copper on full-scale assembly saddles. In addition, BCL would perform an evaluation of various solder fluxes and provide technical support to TES during the soldering of the electroplated assemblies.

TASK 1.1 EXPERIMENTAL WORK CONDUCTED BY BCLTask 1.1.1 Electroplating

Modifications to the electroplating procedure described previously were made to incorporate plating of 5 mils of copper on the saddle portions of full-scale aluminum assemblies. A brief discussion of some of the difficulties in the development work will explain why earlier procedures had to be changed.

Major problems encountered in work conducted while checking procedures and solutions prior to actual plating of full scale parts were as follows:

- Plating through pinholes in 3 coats of stop-off lacquer.
- Infrequent, small blisters in the copper on the upper inside curve of the saddle which was plated in the horizontal position.
- Excessive copper thickness at extreme ends of copper plated saddle.
- Inadequate thickness of copper deposited in the bottom of the saddle accompanied by excessive copper at the top of the lands which prevented fit-in of the heat pipe or auxiliary cooling tube.

Three coats of one kind of stop-off lacquer recommended by the coating manufacturer did not fully protect those portions of the details which were not to be plated. Long exposure times (up to 4 hours) of the details to the highly acid bright copper plating solution resulted in nodules of copper on the masked surface and breakaway of stop-off during handling after copper plating. Two coats of the recommended coating (Microstop) plus two coats of a tougher, more flexible coating, Microflex, provided an adherent film which was resistant to penetration and peeling.

Some small blisters were present in the thick copper coatings on one preliminary test detail. These blisters originated at the aluminum/copper interface. Three factors were believed to be possible causes of blistering due to incomplete activation of the aluminum substrate:

- (1) Aluminum inadequately cleaned.
- (2) Incomplete zinc coverage, and
- (3) Incomplete initial coverage in either copper plating solution.

Therefore, procedures were modified as follows to better insure that no defects would be obtained in the plated copper.

- (1) The time in the etching solution was increased from 30 seconds to 4 minutes.
- (2) Entrapment of excessive water in the saddles presents the possibility of diluting the treating solutions. Therefore, the saddles were drained before each step.
- (3) A double zincating procedure was also adopted to better insure aluminum activation before copper plating.
- (4) High current density treatments were used at the beginning of each copper plating step to insure complete coverage over the entire area of the saddle in shorter times. Thereby, the bottom of semicircular saddles would be protected from attack by the plating solution.

Nodular electroplated copper deposited at the ends of the long (up to 30 inches) saddles resulted in uneven distribution of copper along the entire saddle and required excessive work in reshaping. Therefore, acrylic plastic current shields were designed and fabricated to minimize edge build-up. One configuration which prevented the edge effect but still allowed good activation and copper coverage of the aluminum is shown in Figure 13.

The final problem to be resolved was the unequal current distribution in the saddle and at the tops of the saddle lands. This problem is a result of the projecting lands which draw more current and thus more copper is deposited on the lands than in the internal portion. Figure 14 shows a photomicrograph of a typical section of a copper plated aluminum alloy saddle. The copper thickness inside the saddle averages about 5 mils while the copper

at the tops of the saddle lands is about 20 mils thick. In order for the copper heat pipe or auxiliary cooling tube to fit the saddle with a minimum of reworking, three current densities for acid copper plating were evaluated, namely 8, 25, and 40 amperes/square foot. Test details were plated with an average thickness of 7.9 mils. After plating, the details were sectioned, polished, and etched and the copper thicknesses were measured with an optical digital micrometer. The results were as follows:

<u>Current Density, amperes/square foot</u>	<u>Copper at Bottom of Saddle (mils)</u>	<u>Plating Time, (hours)</u>
8	5.4	16
25	4.7	5.7
40	4.1	3.6

The above tabulation shows that the current density of 8 A/sq ft was the most desirable for copper plating because more copper was deposited in the bottom of the saddle and 16 hours is a convenient plating time for an overnight operation. The copper deposit produced in the saddle was matte and smooth and proved to be an excellent surface on which to solder.

Plating at 8 A/sq ft gave the best distribution but copper build-up at the lands prevents a 0.312 inch diameter tube to enter the saddle. Therefore, some minor reshaping of the saddle groove was accomplished by removing some of the copper according to the sequence shown in Figure 15. Figure 15 shows that about 4 mils of copper must be removed from the top inside of the channel by mechanical means. A broaching tool, shown in Figure 16, was designed to remove the copper equally from both sides of the saddle. Several cutting points with varying degrees of rake were tried for ease of broaching. The best cutting edge had a 5° positive rake and was fabricated from flame hardened drill rod. The broach was used with a smooth pulling motion, only. With the tool, a 30 inch groove could typically be reshaped in about 5 to 10 minutes.

Task 1.1.2 Evaluation of Soldering Fluxes

A comparative study of commercial and other non-corrosive soldering fluxes was undertaken to find the one most suitable for solder joining of various configured hardware. The objective was to find the flux having the best fluxing capability, thermal stability, and the most innocuous residue for use when soldering 100% Tin or 60 Tin . 40 Lead electroplated details.

Task 1.1.3 Procedure

All fluxes were initially evaluated on clean bare copper with heating times from 1/4 to 2 hours. A 0.25 inch diameter wire ring was constructed from 60 Sn-40Pb solder, 0.036 inch in diameter and placed on a small copper coupon with 3 drops of flux. The test coupons were heated on a hot plate until the solder melted and then raised 5°C (40°F). Fluxes were rated on the basis of solder flow and the type of residue remaining. The initial tests on bare copper were then followed by a similar test on copper/tin-lead plated aluminum coupons.

Task 1.1.4 Fluxes Evaluated

In the following tabulation of fluxes, commercially available fluxes Numbers 1 through 6 and Number 13 were furnished by TES. The remaining fluxes, Numbers 7 through 12, were obtained or formulated by BCL.

(1)	Multicore	PC26
(2)	Multicore	366A
(3)	Ersin	5381
(4)	Multicore	366
(5)	Kester	1571 HT
(6)	Kester	1645
(7)	Isopropyl alcohol + Kester 1571 HT (3 to 1 by volume)	
(8)	Commercial castor oil + 5% oleic acid USP, by volume	
(9)	Commercial castor oil + 10% oleic acid USP, by volume	

- (10) Commercial castor oil + 20% oleic acid USP, by volume
- (11) Oleic acid USP, 100%
- (12) Commercial castor oil, 100%
- (13) Ersin Multicore Solder, 60 Sn-40Pb, 177/1.

Task 1.2 Results

When the commercially available fluxes (Numbers 1-7 and 13) were tested with long heating times on bare copper, only Numbers 3 and 5 appeared worthy of further consideration. With Numbers 3 and 5, solder flow occurred to the extent that the solder ring disappeared. The ring retained its shape under the remaining commercial fluxes. The residue from all the fluxes in the commercial group was hard and varnish-like. The residue from flux No. 5 coalesced into several small globules while all the other residues formed a uniform varnish-like coating over the test coupon. Tests conducted on commercial fluxes on plated aluminum coupons gave the same results as the bare copper tests except that in all cases the solder flowed uniformly over the coated coupon.

Several tests using shorter heating times with the commercial fluxes did not alter the conclusion that flux No. 5 was the only flux meriting further consideration. Dilution of flux No. 5 with isopropyl alcohol (Flux No. 7) in an effort to minimize the hard globules of residue was not successful. The globules were eliminated but the resultant residue left a thin uniform varnish coating. In addition, the fluxing capability was reduced.

Mixtures of castor oil and oleic acid as represented by fluxes No. 8, 9, and 10 all produced good solder flow both on bare copper and plated aluminum. Heating times were shortened for these tests to less than 1 hour at the suggestion of TES. Solder flow was best with Numbers 9 and 10 indicating the proper composition was in the range of 10 to 20 percent oleic acid. The residue from these fluxes was easily wiped from the surface indicating it was fluid as expected and should flow more readily from the joint area during the soldering operation.

Flux No. 11 (oleic acid) was too active as boiling and fuming were excessive. Solder flow was very good but there were indications that the plated solder was stripped from the copper plated aluminum.

Flux No. 12 (castor oil) had no fluxing capability as demonstrated by a complete lack of solder flow on bare copper.

The final evaluation of fluxes involved soldering of configured details with Fluxes No. 5 and 10. The resulting joints were evaluated by radiographing each joint from two angles. The joint soldered with No. 5 flux showed some porosity. The joint made with Flux No. 10 was pore free.

Task 1.3 BCL Conclusions

Based on plating of four prototype assemblies, the developed procedures (PS0300037 and PS0300038) will produce good quality electroplated aluminum and copper details. Heat pipes and auxiliary cooling tubes can be fitted to over-plated and reshaped saddles for subsequent soldering. The development of a solder flux containing 10 to 20 percent oleic acid in castor oil resulted in pore free solder joints.

TASK 1.4 APPLICATION OF BCL ATTACHMENT TECHNOLOGY

To verify the attachment procedures developed by BCL, TES prepared LCP 10037, Soldering Procedure Heat Pipe Attachment, Fin/Housing Specimens. This procedure permitted trial solder attachments with dummy copper heat pipes (LCP10011-001) to SIG Fin-Housing Specimens (LCP10009-009). Subsequently, heat pipes (LCP 10079-009 and LCP 10023-009) were soldered to Fin-Housing Specimens (LCP 10009-009) for life testing at 125°C (257°F). The required tooling (LCP 10051) is similar to that shown in Figure 18. Figure 17 shows a completed fin-housing specimen with emissivity coating applied. The major problem encountered during soldering was tool warpage which resulted in unbonded areas along the length of the heat pipe.

The next undertaking was the joining of GDS heat pipes (SIG30042) to the fin assembly (SIG 30026). TES prepared a soldering procedure, SIG 30029, and designed a bonding tool, SIG 30030 (as shown in Figure 18) to accomplish this task. Figure 19 shows the bonding tool in place and Figure 20 shows the completed assembly. Because of the shorter length of this bonding tool (approximately thirteen (13) inches) as compared to the fin-housing specimen tooling (LCP 10051) which was approximately thirty (30) inches in length, warpage was judged not to be a problem. However, noticeable warpage did occur especially for the attachment of the last two (2) heat pipes, Nos. 15 and 16. Results of this work concluded the requirements for redesign of the tooling and the necessity for trial runs on prototype hardware, especially in light of the fact that the SIG/Galileo design was of a different configuration as illustrated by the Generator Housing (SIG 30001) as shown in Figure 21.

PHASE IV. CONTINUED HEAT PIPE ATTACHMENT TECHNOLOGY

During Phases I and II of the subcontract activities conducted by Battelle Columbus Laboratories, cursory shear strength studies (tensile tests) were performed on unaged solder (60 Sn-40 Pb) joints at room temperature and at 120°C. In addition, tensile tests were also performed at room temperature and 120°C on joints aged up to 830 hours. Although these tests were encouraging based upon the requirement of 1500 psi, the test program was inconclusive from the standpoint of configured hardware and an in-depth analysis to ascertain the maximum shear strength requirement resulting from such items as mismatch of thermal expansion coefficients and forces resulting from various operational conditions. Therefore, BCL was subcontracted to investigate the thermal mismatch condition by mechanical creep studies. In addition, a new task was designed to obtain additional data on the effects of elevated temperature aging on the shear strength of the 60 tin-40 lead soldered joints and the shear strength at elevated temperatures after aging.

On November 15, 1978, a meeting was conducted at TES with representatives of TES and BCL to discuss the merits of the aforementioned subcontract statement of work. The objective of this meeting was to discuss the comments received by TES from Fairchild, Sandia and DOE relative to the proposed statement of work.

Opening discussions were centered on the recent structural analysis conducted by TES which indicated that the most severe stresses in the heat pipe/saddle solder joint occur during elevated temperature operation. This worst case condition would be the results of thermal stresses introduced during the localized heating/soldering technique planned for making the heat pipe-to-saddle joint. This most recent cursory analysis

indicated that a maximum shear stress of approximately 17,000 psi could be achieved in the solder joint at the 104°-121°C (220°-250°F) generator operating temperature range.

This analytical result was contrary to the analysis performed earlier by TES which indicated that a stress of approximately 400 psi would be achieved in the solder joint following cooldown to room temperature from the 183°-188°C (361°-371°F) soldering temperature. Since the BCL statement of work for the solder evaluation was predicated on earlier analysis, the meeting centered on trying to determine the reasons why the two analyses differed so significantly. The most obvious difference in the two analyses was the assumptions made relative to the exact temperatures of the copper heat pipe and aluminum saddle during the solder operation. The most recent analysis assumed that only a small portion of the aluminum in the vicinity of the saddle reached the 183°-188°C bonding temperature.

However, the entire aluminum saddle was not free to expand to the same extent as the much hotter copper heat pipe, thereby resulting in a larger residual thermal stress due to the restraint of the surrounding aluminum. Because of the assumptions made, the worst case shear stress occurred in the solder joint at elevated temperatures where the solder joint strength is reduced. The earlier analysis assumed that the heat pipe and the saddle were at a uniform temperature during the soldering operation and would result in a residual shear stress which reaches a maximum at room temperature. Since the 60 Sn - 40 Pb solder joint has its maximum strength at room temperature, this condition appeared to be most favorable.

Several variations in the bonding process were discussed, the most notable being:

- Preheating the entire generator housing and radiator skin to some intermediate temperature (perhaps 135°C (275°F)) prior to adding the localized heat to the heat pipe and saddle to effect the solder bond.

- Restraining the thermal expansion of the copper heat pipe during the localized heat technique to reduce the severity of the thermal stresses.

Having identified the incongruities of the existing statement of work, considerable time was spent discussing the value of the test program. It was agreed that the scope of the present program had to be expanded to provide answers to the many questions. A two (2) phase test program was entertained which would initially provide basic data on the strength of 60 Sn - 40 Pb soldered joints, and would later serve as a test program to verify the design of the actual heat pipe/saddle solder joint. Double-lapped shear specimens were to be used to generate the data base for characterizing the strength of the soldered joint. This newly generated data would have provided a firmer data base for analyzing the heat pipe/saddle joint. This data would have then been factored into an analytical structural model to establish the most severe operational condition for the soldered joint. The second phase of the new test program would have subjected test specimens, representative of the actual heat pipe/saddle configuration, to selected environments in order to qualify the integrity of the soldered joint design. Further, Battelle would prepare an unsolicited proposal for a parallel research program on the "Analysis of Heat Pipe Soldered Joint." This proposal would include a stress analysis of the soldered joint together with recommendations on how the bonded joint could be improved.

On January 29, 1979, DOE issued a "Stop Work Order" which concluded all attachment technology studies at BCL and TES.

Spec. Type	Length (inches)	Joint Sides	Spec. Leg.	Flux	Coating Thickness Inch	Hot Platen Temp. °F	Hot Block Temp. °F	Max. Temp. °F	Vibra- tion Time min.	Solder Added	Time Above 350° F min.	Weight After Vibra- tion	Soldering Notes	Radiographic Notes
Straight	6	Equal	---	Yes	0.0015	400	405	400	1.25	No	2	No	Weight removed before frozen, joint opened; re-melted to same cycle & weight on to below 300° F	X-ray, 1 large void near end, scattered fine porosity; good fillets
Straight	18	Equal	---	Yes	0.0015	400	405	400	1.25	No	2	Yes	Weight on to below 300° F; Plate bowed up 1/8"	Uniformly scattered fine porosity; good fillets
Straight	4	Unequal	---	Yes	0.0015	395	450	400	1.00	No	2	Yes	Solder on Al hard to melt; wgt. on to below 300° F plating too low on long side to permit good fillet	Scattered fine pores; good fillet deep in groove
Straight	20	Unequal	---	Yes	0.0015	400	410	400	1.25	Yes	8	Yes	Power on longer because of flux bubbles; wgt. on to below 300° F, 6" pc. solder wire placed in long side groove, bow up 1/8"	Scattered fine pores, more than others; evidence of no fillet deep in groove except where wire ended
L	24 x 5	Equal	Short	No	0.0015	410	420	420	1.00	Yes	10	Yes	No problems	
L	24 x 5	Equal	Long	No	0.0015	410	420	420	1.25	No	12	Yes	At L angle fillet not complete, probably caused by fit up on short leg; bow up 5/32"	Large void at L angle; uniformly scattered fine porosity; few pores to 1/32" diameter
L	24 x 5	Unequal	Short	Yes (Al)	0.0015	420	420	420	1.00	Yes	11	Yes	Extra clamp near angle; no other clamp; flux cut 50% with isopropyl alcohol	Scattered porosity; more than usual; none over 1/32" diameter
L	24 x 5	Unequal	Long	Yes (Al)	0.0015	400	420	420	1.25	Yes	12	Yes	Could not get flux under tube near angle (4-5 inch) solder wire laid in long side groove; bow up 1/2"	First 10 inches from angle scatter pores, some as big as 1/4: diameter, pores in angle and may be due to lack of flux, remaining 14 in., fine scatter

TABLE 2. TENSILE/AGING SPECIMEN PRODUCTION RECORD

No.	Jt. Area Fluxed Yes/No	Solder Added at end Yes/No	Measured Jt. Thick inch	Notes
1	Y	Y	.003	Some dewetting when Al coat was fused; rejigged to get clear, right
2	Y	Y	.003	ditto
3	Y	Y	.005	"
4	N	Y	.003	"
5	N	Y	.002	Cu blister noted by plater
6	N	Y	.003	
7	N	N	.003	Not a good joint, no fillets
8	N	N	.002	ditto
9	Y	N	.002	"
10	Y	N	.002	"
11	Y	N	.003	"
12	N	N	.003	Not a good joint, no fillets
13	Y	N	.002	
14	Y	N	.003	
15	Y	N	.002	
16	Y	N	.003	
17	Y	N	.005	
18	Y	N	.003	
19	Y	Y	.003	
20	Y	Y	.002	
21	Y	Y	.002	
22	Y	Y	.004	
23	Y	Y	.003	
24	Y	Y	.003	

TABLE 3. TENSILE STRENGTH OF AGED COPPER-ALUMINUM SOLDERED JOINTS

Aging Temperature 150°C
Testing Speed 1.0 in/min (0.42 min/5)

Specimen Number	Aging Time-Hr.	Testing Temp. °C	Breaking Load lbs	Average Tensile Strength psi (M Pa)		Notes
1	0	RT	1000	----	----	Tensile machine problems
4	0	RT	1110	----	----	Failed in solder
13	0	RT	1270	----	----	ditto
19	0	RT	1270	----	----	ditto
Average	0	RT	1217	7200	49.6	
3	0	120	430	----		Void equal to 1/3 joint area
5	0	120	675	----		Failed in solder
14	0	120	610	----		ditto
20	0	120	750	----		ditto
Average	0	120	678	4018	27.7	
	193	120	570	----		Failed in solder
6	193	120	545	----		ditto
17	193	120	500	----		ditto
21	193	120	605	----		ditto
Average	193	120	555	3289	22.7	
9	504	120	395	----		Large unbonded area
16	504	120	550	----		Failed in solder
22	504	120	600	----		ditto
Average	504	120	575	3407	23.5	
10	830	120	540	----		ditto
15	830	120	565	----		ditto
23	830	120	600	----		ditto
Average		120	568	3365	23.2	
11	830	RT	1140			ditto
18	830	RT	1010			ditto
24	830	RT	670			Failed at Cu bar/solder interface
Average		RT	1075	6370	43.9

FIGURE 1. OVERALL VIEW OF 100 We THERMOELECTRIC MODULE

FIGURE 2A. CROSS SECTION OF JOINT LOADING WEIGHTS

FIGURE 2B. CROSS SECTION OF SIMULATED PRODUCTION JOINTS

FIGURE 3. HEATED SOLDERING BLOCK SEGMENT - ONE SEGMENT

FIGURE 4. SETUP FOR SOLDERING THE LONG LEG OF AN
"L" SPECIMEN - VIBRATING TOOLS IN POSITION

FIGURE 5. SOLDERING SETUP WITH POST-SOLDERING WEIGHTS
IN PLACE FOR COOLING JOINT

FIGURE 6. AS-SOLDERED "L" SPECIMEN SHOWING BOWING OF THE LONG LEG

FIGURE 7. AS-SOLDERED JOINT, COPPER TUBE TO 6061 ALUMINUM

SPECIMEN #3 SPECIMEN 4A

200 HRS AT 120°C
AS POLISHED; 250X

NOTE - EUTECTIC PATCHES HAVE
NEARLY DISAPPEARED FROM
SOLDER

FIGURE 8. COPPER TUBE TO 6061 ALUMINUM SOLDERED JOINT
AFTER AGING AT 120°C FOR 200 HOURS

SPECIMEN #1

400 HRS. AT 120°C
AS POLISHED; 250X

0J556

SPECIMEN #1

400 HRS. AT 120°C
AS POLISHED; 750X

Cu

Cu PLATE

Cu₃Sn

Cu₆Sn₅

SOLDER

HIGH Pb PHASE IN SOLDER APPEARS
TO BE AGGLOMERATING

Cu₆Sn₅

Cu₃Sn

Cu PLATE

AL

0J577

FIGURE 9. COPPER TUBE TO 6061 ALUMINUM SOLDERED JOINT
AFTER AGING AT 120°C FOR 400 HOURS

OJ581

SPECIMEN C-2760 HRS. AT 120°C
AS POLISHED; 250X

Cu

PROBABLY AN ETCH/
POLISH EFFECTCu₃SnCu₆Sn₅SPECIMEN C-2760 HRS. AT 120°C
AS POLISHED; 750X

SOLDER - Pb PHASE WELL AGGLOMERATED

Cu₆Sn₅Cu₃SnCu PLATE - SHOWS RELIEF PATTERN OF
STRUCTURE CAUSED BY ETCH POLISH

Al

OJ582

FIGURE 10. COPPER TUBE TO 6061 ALUMINUM SOLDERED JOINT
AFTER AGING AT 120°C FOR 760 HOURS

FIGURE 11. HOUSING DETAIL

FIGURE 13. SCHEMATIC AND DIMENSIONAL DRAWING OF ACRYLIC PLASTIC END SHIELD FOR ELECTROPLATING ALUMINUM ALLOY ASSEMBLIES

4J035

10X

FIGURE 14. CROSS SECTION OF TYPICAL SADDLE ELECTROPLATED WITH ABOUT 5 MILS OF COPPER

FIGURE 15. SCHEMATIC SHOWING REQUIRED SEQUENCE FOR ACHIEVING DIMENSIONAL TOLERANCE OF ELECTROPLATED SADDLES

FIGURE 16. BROACHING TOOL - FULL SCALE

TES-33009-45

45.

FIGURE 17. FIN-HOUSING SPECIMEN (LCP10009)

FIGURE 18. FIN/HEAT PIPE BONDING TOOL

FIGURE 19. FIN ASSEMBLY-BONDING TOOL IN PLACE

FIGURE 20. FIN ASSEMBLY-HEAT PIPES ATTACHED (SOLDERED)

FIGURE 21. SIG/GALILEO GENERATOR HOUSING (SIG30001)