$\bar{\mathbf{v}}$

 \bullet

 $\frac{1}{2}$

 $\mathfrak i$

 $\mathring{\|}$ \bar{r}

 $\|$

 $\bar{1}$ $\mathord{\downarrow}$ $\bar{1}$ $\hat{\boldsymbol{\beta}}$ $\hat{\boldsymbol{\theta}}$

 \mathbf{I}

 $\hat{\mathbf{r}}$

 $\overline{}$

 $\bar{1}$

 \bar{z}

 $\hat{\boldsymbol{\gamma}}$

 $\ddot{}$

 $\ddot{}$

 \bar{t}

 $\frac{1}{2}$

 \mathbb{C}

REPORT NO. P65--117

RE-ORDER NO. 65-7/8

)

 \sim $\lambda\phi$

This work was performed for the Jet Propulsion Laboratory, **California Tnsdt_te of Technology, sponsored by the** National Aeronautics and Space Administration under Contract NAS7-100.

 $\mathcal{A}_{\mathcal{A}}$

FINAL REPORT **- PART I** _Contract **No.** q51069)

Q

DETERMINATION OF THE EFFECTS OF A THERMAL STEP,ILIZATION PROCESS ON THE MECHANICAL AND ELECTRICAL PRO?ERTIES OF SOLDERED AND WELDED **JOINTS**

Prapared for.

let Projection Laboratory ",_i_io,,, ,_ . _. J_e **of I'echnology** P csode..n. Calitornia

9 OCTOBER 1965

AEROIPACE GROUP **-t HUGHES :** **I ._** i **_,_ ,R** .RAFT **COMPANY •.-, ".E_ -" CAt,.IFORNIA**

FINAL REPORT - PART I

Contract No. 951069

DETERMINATION OF THE EFFECTS OF A THERMAL STERILIZATION PROCESS ON THE MECHANICAL AND ELECTRICAL PROPERTIES OF SOLDERED AND WELDED JOINTS

> r. Z. Neister, Hughes Project Engi by 9 October 1965

For

Jet Propulsion Laboratory California Institute of Technology Pasadena, *California*

Approved:

 \therefore L. Shinn, manager $\frac{1}{2}$ valentals Technology Department

Materials Technology Department $\overline{}$

Hughes Aircraft *Company* **-** Culver City, *California*

CONTENTS

Ţ

Page

ILLUSTRATIONS \overline{a}

 $\begin{array}{c} \mathbf{1} \\ \mathbf{1} \end{array}$ $\mathbf{1}$

 $\widehat{}$

Figure

Figure

ţ

 \sim \sim

i,

TABLES

ABSTRACT

12854

The purpose of this investigation was to determine the effects of a **thermal sterilization** process on **the** mechanical and electrical properties of soldered and welded joints. An additional scope of work under this **same** contract was for the determination of the effects of **thermal** bake-out, heat sterilization, and ethylene oxide decontamination on **the** solderability of component leads.

This investigation involved the preparation and testing of **1362** soldered joints of 11 different types, including both stranded and solid conductors joined **to** connector cups and bifurcated **terminals.** A **total** of 834 cross-wire weld joints of 7 different material combinations were also prepared and tested. All **solder** and weld joint **types** were tested and examined before and after thermal sterilization. Testing consisted of:

- a) Ultimate strength
- b) Electrical **resistance**
- c) Electrical **testing** during vibration
- d) Ultimate strength after vibration
- e) Stress-rupture strength
- f) Metallurgical examination
- g) Electron probe microanalysis

Based on a statistical analysis of the test results, it was concluded that there was no **significant** change in the ultimate strength or electrical resistance of either **the** soldered or welded joints due **to** the effects of a thermal sterilization process. Neither the metallurgical examination nor the electron probe microanalysis **showed** any degradation in metallurgical structure or in the extent of gold diffusion which could be attributed to thermal **sterilization.** However, **the stress-rupture** strength tests on the connector cup soldered joints pointed out that steady-state loads of only 10% of the ultimate strength of the joint were enough to cause short-term solder joint failures under thermal sterilization \mathbb{R} . In **ITP** conditions.

1. PURPOSE AND SCOPE

1. I PURPOSE

The purpose of this investigation was to determine the effects of a thermal sterilization process on the mechanical and electrical properties of soldered and welded joints. An additional scope of work under this same contract was for the determination of the effects of thermal bake-out, heat sterilization, and ethylene oxide decontamination on the solderability of component leads.

This work was done for the *let* Propulsion Laboratory under Contract No. 951069 (subcontract under NASA *Contract* NAS 7-100). Mr. A.G. *Fitak* was the ffPL Project Engineer on this program. During the final stages of this program, Mr. R.F. Holtze became the new JPL Project Engineer on this program, since Mr. *Fitak* was transferred to other duties.

This final report has been divided into three separate parts. Part I, which is this part, covers the thermal sterilization effects on solder and weld joints. Part II contains the weld schedule isoforce diagrams, raw data sheets, photomicrographs, and electron probe microanalysis charts for the solder and weld joints under Part I. Part III covers the solderability studies. It is therefore possible while reading the test results of Part I to turn to the applicable raw data sheet or photomicrograph in Part II and consult both pieces of information at the same time. Part III, being a separate study and not as lengthy as Part I, is allinclusive with its own data sheets and photomicrographs included under the same cover.

1. Z SCOPE

Part I of this final report is a determination of the effects of a thermal sterilization process on the mechanical and electrical properties of soldered and welded joints. This involved the preparation and testing of 136Z soldered joints of ll different types including both stranded and solid conductors joined to connector cups and bifurcated terminals. A

 \mathbf{I}

total of 834 cross-wire weld joints of 7 different material combinations were prepared and tested. All of the different types of soldered and welded joints were tested and examined before and after thermal sterilization. Testing consisted of ultimate strength, electrical resistance, electrical testing during *vibration,* ultimate strength after vibration, and stress-rupture strength. In addition certain solder and weld joint types were submitted to electron probe microanalysis. All solder and weld joint types were examined metallographically and 48 photomicrographs were taken. The test results were analyzed statistically. Photographs were taken of each type of solder and weld.joint specimen prior to testing and of each piece of test equipment used with at least one of each type of test specimen installed prior to test.

2. MATERIALS AND EQUIPMENT

2. 1 MATERIALS FOR SOLDER JOINTS AND WELD JOINTS

The materials used in this program for the preparation of the solder joints and weld joints are listed in Table I. Vendor certifications were required for all materials. Incoming inspection for the materials listed in Table 1 was done by metallographic sectioning, tensile strength tests, spectrographic analysis, and magnified examination. Inspection was primarily aimed at correct plating thicknesses, proper base metal or alloy, and proper material dimensions.

Table II is a tabulation of the plating thickness measurements made on the materials listed in Table I. Certain of the materials were not plated and therefore are not included in Table II. The thickness measurements in Table II are averages of five readings with the minimum-maximum range given whenever possible.

2.2 RAW MATERIALS FOR SOLDERED JOINTS

- 2.2.1 Solvent for removal of flux residue 1, 1, 1-Trichlo per Federal Specification O-T-620
- **2.2.2** Protective coating for *connector* cups and bifurcated terminals - Lonco Sealbrite No. 230-10.
- 2.2.3 Flux for tinning stranded conductors Alpha No. I00 per MIL-F-14256 C, Type W.
- 2.2.4 Flux for tinning all gold plated and solder coated conductors -**Alpha** No. 611 per MIL-F-14256C, Type **A.**
- **2.2.5** Flux for tinning bare nickel conductors Alpha No. 90 Stain Steel Flux.
- **2.2.6** Cored solder for making all solder joints Kester plastic rosin core solder, per QQ-S-571d, 0.032^c diameter, Composition Sn63, Form W, Type R, Core **P3.**

 $\overline{\mathbf{3}}$

Table I. List of materials for solder joints and weld joints.

- 1. Stranded conductor, #24 AWG, 19/36, per MIL-W-16870, Type *E,* silver plated, Teflon insulated.
- Z. *Cinch* connector cup per JPL 20045/Z00-E.
- 3. Bendix connector cup per JPL ZPH-ZZ45-0300-B, JPL DS31?, and MIL-C-26482C.
	- 4. Bifurcated terminal, solder coated, per JPL DS167-7.
	- 5. Bifurcated terminal, solder coated, per JPL DS167-3.
	- 6. Bifurcated terminal, gold plated, per JPL DS99-7.
	- 7. Conductor, copper, OFHC, 0.020" diam., gold plated per MIL-G-45204, Type I, Class 1.
	- 8. *Conductor,* copper, OFHC, 0.020" diam., tin-lead coated, tin 10-70%, 0.0001" average min. thickness -0.001" average maximum thickness per Revision A to MIL-STD-lZ76. Preferred tin-lead alloy to be $63-37$ or $0-40$.

** 9. Conductor, Dumet, 0.020" diam., per MIL-STD-1276, Type D, gold plated per MIL-G-45204, Type I, Class I.

- 10. Conductor, Kovar, 0.018" diam., per MIL-STD-IZ76, Type K, gold plated per MIL-G-45204, Type I, Class I.
- II. Conductor, Nickel, 0.025" diam., per MIL-STD-IZ76, Type N-Z, gold plated per MIL-G-45204, Type I, Class I.
- 2. Conductor, Nickel, bare, 0.025" diam., per MIL-N-460
- ^{3.} Conductor, Nickel, bare, 0.016" diam., per MIL-N-460
- ¹. Conductor, Nickel, 0.016" diam., per MIL-STD-1276, Type N-2. old plated per MIL-G-45204, Type I, Class L.
- 15. Conductor, Kovar, ribbon, per MIL-STD-IZ76, Type K, 0.005" x 0.016", gold plated per MIL-G-45Z04, Type I, Class I.
- 16. Conductor, Nickel, Inco 200 ribbon, bare, 0.010" x 0.031" (rolled from wire & annealed).
- 17. Printed circuit board material, 0. 062" thick, per MIL-P-13949C, Type FL-GE, glass-base epoxy laminate.

* Supplied by JPL

a: Durnet wire supplied without a nickel strike between the copper sheath and the outer gold electroplate.

्र २,

Table II. Plating thickness measure

;',-'Terminals were *centrifuged* s,,ldcr coated using an Electroverl "ACTA" Automatic Centrifugal Tinning Appar

**Analysis showed the tin-lead composition of the solder plating to be 55%-65% tin with the balance lead.

2.2.7 Solder in solder pot for tinning conquetors - Kester bar solder per QQ-S-571d, Type Sn 63-B-S.

2. 3 EQUIPMENT FOR PREPARING SOLDERED JOINTS

- 2.3.1 Soldering iron used for making all soldered joints **-** Weller Model W-TCP (60 watts) thermally controlled soldering pencil with a Type PT-A6 600° F iron clad screwdriver tip.
- 2.3.2 Insulation stripper for stranded conductor wire Pioneer Magnetics Thermal Stripper.
- 2.3.3 Suction method for removing excess solder from tinned connector cups - Zeva 70 watt soldering iron equipped with a Bazooka solder gobbler.
- Z. 3.4 Solder pot for tinning conductors **-** Dee Melting Pot, Model 13.

2.4 EQUIPMENT FOR PREPARING WELDED JOINTS

- 2.4. l Hughes Aircraft Company Model VTW-30B Stored Energy Power Supply (100 watt-seconds), Serial No. 30B-299, calibrated on 6-14-65.
- 2.4.2 Hughes Aircraft Company Model VTA-60 Welding Head, Serial Number 60A-364
- 2.4. 3 RWMA-1 copper *cadmium* alloy welding electrodes and RWMA-2 copper-chromium alloy welding electrodes.

2. 5 TEST EQUIPMENT

The equipment used for conducting the various tests on the solder and weld joints is listed at the beginning of each section describing the applicable test procedure.

3. PREPARATION OF TEST SPECIMENS

3. 1 BAKE-OUT OF CONDUCTORS

Conductor materials for solder and weld joints were baked-out prior to tinning and joining. The following conductor materials were baked-out at 200°C for 168 hours in an inert nitrogen atmosphere using a National Appliance Co. Vacuum Oven, Model 58402, Serial No. B59:

- a) Gold plated copper wire
- b) Gold plated Dumet wire
- c) Gold plated Kovar wire
- d) Gold plated *nickel* wire
- e) Bare nickel wire
- f) Gold plated Kovar foil

Originally these conductors were baked-out at 200°C for 168 hours in air; however the gold plated *copper* and *gold* plated Dumet wires turned black (severe oxidation} as a result of this bake-out schedule and were in an unsolderable condition. It was therefore decided to conduct all bake-outs in an inert atmosphere.

All solder coated copper wires and the leads of 1/4-watt axial lead resistors were baked-out at 150°C for 168 hours in an inert argon atmosphere using a National Appliance Co. Vacuum Oven, Model 58301, Serial No. C58.

The only wires not baked-out were the teflon insulated stranded condnctors and the Inco 200 ribbon. None of the connector cups or bifurcated terminals were baked-out.

3.2 TINNING OF CONNECTOR CUPS

Prior to their use for solder joints, the Cinch and Bendix connector cups were tinned. Tinning was in accordance with *JPL* Interim Procedure "Procedure for Soldering Wire to Connector Pin Solder *Cups."* This procedure essentially calls out filling the cup cavity with solder using a soldering iron and then removing the excess solder by means of sucking or wicking. Suction (using a solder gobbler} was used to remove

excess solder from the cup cavities. Flux residues were then removed by agitation in trichloroethane solvent. All connector cups were then dip coated with a protective coating of Lonco Sealbrite No. Z30-10.

The solder coated bifurcated terminals were centrifuged solder coated by the vendor (Lyn-Tron, Inc.) but had not been protectively *coated.* Therefore these terminals were *protectively* dip coated with Sealbrite No. 230-10 at Hughes Aircraft Company.

3. 3 TINNING OF CONDUCTORS

All conductor wires used in the preparation of solder joints were tinned prior to joining. Stranded conductors had the Teflon insulation stripped back for a distance of 5/3Z" - 3/16" using a Thermal Stripper. All wires used in the preparation of solder joints were precut to a length of Z" prior to tinning. Tinning of the conductor wires, stranded conductors, and resistor leads was done by first dip fluxing the end of the wire and then dipping the wire in a molten 63/37 solder pot at 500° F for 5 seconds. Alpha 611 activated rosin flux was used for all conductor wires except:

a) Stranded conductor - used Alpha 100 nonactivated rosin flux.

b) Bare nickel wire - used Alpha No. 90 stainless steel flux. Flux residues remaining after tinning were removed by an agitated rinse in Trichloroethane, except for Alpha No. 90 flux residues which were removed by three successive hot water rinses followed by a distilled water rinse and a hot air dry.

None of conductors used in the preparation of welded joints were tinned prior to welding.

3.4 FABRICATION OF SOLDER *5OINTS*

Soldering of all solder joints was done in accordance with JPL Process Specification ZPE-1081-0002-A "Soldering of Electronic Equipment," dated 9 Dec., 1964 using a Weller Model No. W-TCP (60 watt) soldering iron with a PT-A6 600°F iron clad tip. A non-activated rosin cored solder (Sn 63-WRP3) was used for all joints. All solder joints were made by one operator who was certified for soldering per

NASA Electrical Assembly Specification MSFC-PROC-158B "Procedure for Soldering of High Reliability Electrical Connections."

Prior to making any solder joints, the soldering iron was tested to see if it would meet the temperature requirements of JPL Spec. ZPE-1081-000Z-A. This specification states that "the temperature of the soldering iron shall be maintained within 475° F to 610°F during the soldering operation. The test results showed that during the soldering operation, the tip temperature is maintained between 530° F and 580°F using the PT-A6 tip.

Soldering techniques for specimen standardization were established by soldering samples of all joint types and material combinations and metallographically sectioning typical joints. Inspection of the metallographic mounts was done to insure that, as far as practical, all joints of one type are similar (e. g. , same appearance, same mass of solder, etc.).

All solder joints were 100% inspected. No repairs were allowed. Only joints visually acceptable under 20X magnification were tested.

Flux residues from all solder joints were removed by I, I, l-Trichloroethane.

The number and types of solder joints fabricated for this program are listed in Table III. Figures l and 2 are photographs showing all of the eleven types of solder joint specimens prepared for this program. The specimens in Figure I are labeled in accordance with the specimen identification used in Table III. This same identification code has been used throughout the entire program. The specimens in Figure 2 are of gold plated Kovar wire soldered to solder coated bifurcated terminals (identification I. 3.4).

Figures 3, 4, and 5 are close-up photographs of the eleven types of solder joint specimens. The ten specimens shown in Figures 3 and 4 have been photographed from two views. The solder joint specimens on the left show the side view and the specimens on the right show the top view of the same type of solder joint. The specimens in Figure 5 are I/4-watt axial lead resistors soldered to solder coated bifurcated terminals. The terminals have been staked in a $1/16"$ thick glass-base

Table III. *Fabrication* of solder joints.

Figure 1. Photograph showing ten types of solder joint specimens (R104841).

Figure *3.* Photograph of five types of solder joints-1. 1.1, 1. 1.2, 1. 2, 1. 3. 1, 1. 3. 2 (R105350).

Figure 4. Photograph of five *types* of solder joints-1. *3.3,* 1. *3.* 4, 1. *3.* 5, 1. *3. 6,* 1.4 (R105349).

Figure 5. Solder joint specimens of 1/4-watt axial lead resistors soldered to solder-coated bifurcated terminals. Specimen No. 1. 5 (R105005).

epoxy laminate printed circuit board and soldered to the gold plated etched circuit pad areas on the underside.

3.5 FABRICATION OF WELD JOINTS

0

0

The number and types of weld joints to be fabricated for this program are those listed in Table IV. Note that leads materials 1. 1, 1. 2, 1. 3, 1. 4, 1. 5, and 2 are baked at 200^oC for 168 hours in nitrogen. Lead material 1.6 was baked at 150 ^oC for 168 hours in argon. In addition to the weld joints listed in Table IV, JPL welded ten (10) each of joint types 1. 1 thru 1. 6 as specified in Table IV using material baked-out and furnished by Hughes Aircraft Company. JPL did not prepare any weld joint specimens for joint 2. (Kovar foil to Kovar foil). All Hughes & JPL weld joints were made using material from the same spool of wire.

Table IV. Fabrication of welded joints.

The configuration of the weld joints is shown in the sketches (Types A and B) on Figure 6. The weld joint shown as Type A will be prepared in accordance with Figure 4 of JPL Spec. No. GPO-30995- GEN. Type B weld joint was used for electrical tests during vibration (before and after heating) and ultimate strength tests after vibration (before and after heating).

Ħ.

Weld joints were made in accordance with *JPL* Spec. No. GPO-30995-GEN "General Specification for the Design and Fabrication of Resistance Welded Electrical Connections." Welding will be done using a Hughes Model VTW-30B Stored Energy Power Supply (100 wattseconds) with a Hughes VTA-60 weld head. This unit is a capacitance discharge resistance welder having a force fired head with controllable welding energy. The weld electrode material was dependent upon the materials being joined. Weld schedule determination was by the isoforce diagram method whereby weld energy (watt-sec.) is plotted against weld breaking strength for constant clamping forces. All welding was done by one certified operator.

Figure 6. Weld joint configurations.

Typical specimens of all weld joint types were metallographically sectioned and examined for internal defects and specimen standardization. All weld joints were 100% inspected at 20X magnification. Weld schedule verification was per paragraph 3. 3.6 of JPL Spec. GPO-30995-GEN.

In order to prepare weld joints per JPL specification No. GPO-30995-GEN it was necessary to first establish optimum schedules. This required from 100 to 200 weld joints of each type to be prepared and pulled apart while varying parameters such as weld energy, electrode force, electrode material, and polarity and observing percent setdown, percent lead deformation, interface spitting, mean pull strength, etc. Six weld specimens for each joint type were also metallographically examined per paragraph 3. 3.5 of JPL Spec. GPO-30995-GEN for weld schedule defects. Weld schedule verification was done by preparing fifty (50) weld joints of Type A in Figure 6 for pull testing. Forty (40) of these fifty (50) weld joints were used to obtain ultimate strength data for another testing portion of this program. Those weld joints giving the most trouble were:

- I. l Bare nickel to Inco 200 ribbon
- 1.3 Gold plated Kovar to Inco 200 ribbon

2.0 Gold plated Kovar ribbon to itself.

Attachment 1 in Part II of this final report contains an isoforce diagram for each of the seven types of weld joints. All pertinent data regarding determination of weld schedules has been included on the isoforce diagrams.

Table V is a summation of weld schedule data showing the various weld schedules established by Hughes and JPL for the weld joints. All JPL weld joints were fabricated using a Weldmatic Model I065 power supply with electrodes having 0.050" tip diameters.

Figure 7 is a photograph showing all of the weld joint specimens (Type A and Type B) prepared for this program. The Type A specimens are on the lower bench top and the Type B specimens are on the upper shelf. The identification numbers for the specimens are coded in accordance with Table IV. The Type B specimens of one type are shown

n_ **•** ۲ **°.-I °p.,l 0** o **c_** c \bm{u} m n_ **,.-i** ,.Q **c_**

 $\begin{array}{c} \n\cdot & \cdot \\
\cdot & \cdot \\
\cdot & \cdot\n\end{array}$

 $\frac{1}{\sqrt{2}}$

Ţ

/.

Ţ

 \hat{q}

Figure 7. Photograph of all seven types of weld-joint specimens (R105346).

directly above the Type A specimens of that same type. Note that certain weld joint specimens have already been labeled with small tags. All specimens were eventually labeled, either before or after destructive testing.

Figure 8 is a photograph showing two each of the seven types of weld joint specimens, all of which were fabricated per configuration A of Figure 6. The weld joints on the left show the Inco ribbon on the bottom and the weld joints on the right show the Inco ribbon on the top. Weld joint 2.0 (Kovar foil to Kovar foil) of course has the same material on the top and bottom.

Figure 8. Photograph of the seven types of weld-joint specimens $(R105348)$.

4. TEST PROCEDURES

4. 1 GENERAL TEST INFORMATION

Testing of solder and weld joints was in accordance with Table VI. A more detailed breakdown of the number of joints for testing is given in Table VII.

Table VIII shows a flow diagram of the test sequence and the number of soldered and welded joints which were submitted to each particular test. The numbers (1 through 13) in the circles above the testing blocks refer back to that particular test outlined in Table VI.

All solder and weld joints were separately identified by special labels and code numbers. This was especially important during the electrical resistance tests so that the same joint could be identified and electrically tested before and after heat sterilization. After testing, all joints (separately identified) were bagged for delivery to JPL. Joints which were destroyed by virtue of ultimate strength tests had their mating members taped back together, so that JPL can, if necessary, refer back to this particular specimen at a later date.

Photographs of each type of test equipment used with a typical joint type installed were taken prior to testing.

Calibration data, where applicable, for the test equipment used was recorded in order to allow traceability of test result data to the original recordation made during testing.

Separate data sheets were used for each particular type of test. A typical data sheet is shown in Figure 9. Original raw data sheets for all tests conducted during this program have been included in Part II, Attachment 2, to this final report.

4. 2 DRY HEAT STERILIZATION TEST PROCEDURES

A National Appliance Co. Vacuum Oven, Model 58402 (Serial No. B59), was used to accomplish the dry heat sterilization cycling in the following portions of the program:

- (I) Ultimate strength testing.
- (Z) Metallographic examination.

Table VI. Test outline.

I

 $\ddot{}$

 $- - -$

 $\frac{1}{2} \sum_{i=1}^n \frac{1}{2} \sum_{i=$

1. ULTIMATE STRENGTH TE 2. METALLURGICAL EXAMINA $3₁$. HEAT STERILIZ . ELECTRICAL TEST DURING VIBRAT , STRESS RUPT . ELECTRON PROBE MICRO-ANA 1100 solder joints (100 each of 11 types) 748 weld joints (100 each of 7 types + 48 JPL welds) 1848 joints 110 solder joints (10 each of 11 types) 82 weld joints (10 each of 7 types $+$ 12 JPL welds) 192 joints ELECTRICAL RESISTANCE TEST 800 solder joints (40 each of 10 types tested **Z** times) 560 weld joints (40 each of 7 types tested **2** times) 1360 joints A. 440 solder joints (40 each of 11 types) [BEFORE ULTIM 280 weld joints (40 each of 7 types) I STREN B. C_{\star} D. E. 55 solder joints (5 each of II types) 41 weld joints (5 each of 7 types plus 6 JPL welds) 110 solder joints(10 each of 11 types) | BEFORE 94 weld joints (10 each of 7 types | VIBRAT plus 24 JPL welds) 72 solder joints (36 each of -2 types) } STRESS-R 4 solder joints (2 each of -2 types) [ELEC. 2 weld joints (2 each of 1 type) | MICRO-J METALLUI 1098 joints 220 solder joints (10 each of 11 types tested 2 times) 188 weld joints (10 each of 7 types tested 2 times + 24 JPL welds tested 2 times) 408 joints 144 solder joints (72 each of 2 types) 8 solder joints (4 each of **Z** types) 4 weld joints (4 each of 1 type)

Table VII. Number of joints for testing.

-v-I **4-)** 0 **C** °r-. **3** r-4 **n_** o **U]** 0 **td n_** 0 **> E_**

Z6

تبيعي
TEST Ultimate Strength DATE July 12, 1965

TYPE OF JOINT: Solder Joint

MATERIAL: Gold plated copper to solder coated bifurcated terminal

per 1.3.1

TEST PERFORMED BY: D. L. Teter

Figure 9. Typical data sheet for recording test results on solder and weld joints.

- (3) Vibration testing.
- (4) Electron probe microanalysis.

During heat cycling specimens were mounted in such a manner that the space between them precluded any synergistic effects that might occur. Only specimens of good quality and workmanship were selected for testing and all necessary steps were taken to assure maximum cleanliness of samples prior to test.

To achieve the purity of nitrogen atmosphere required in the test chamber during the heat exposure, a mechanical vacuum pump and nitrogen source (bottled commercial grade nitrogen) was used. Using an appropriately mounted thermocouple, a record was made of temperature as a function of time during the entire cycle.

Heat sterilization of solder joint specimens for stress-rupture testing was carried out in a large Conrad Temperature-Altitude Chamber Model No. FH8-3-3 (Serial No. 7268). This chamber is capable of being evacuated and backfilled with nitrogen. Automatic recording devices were used to keep a constant record of the time-temperature data. Periodic sampling and analysis of the atmosphere within the test chamber was performed to insure that no air leakage has occurred.

All dry heat cycling will be carried out in accordance with JPL Specification XSO-30275-TST-A "Environmental Test Specification Compatibility Test for Planetary Dry Heat Sterilization Requirements" dated 24 May 1963. The thermal sterilization compatibility test conditions are 145° C $\pm 2^{\circ}$ C for 36 hours in a dry nitrogen environment. This test is performed three (3) times with stabilization to room conditions between heating cycles.

In stress-rupture testing, however, a single 108 hour exposure was used since the three 36 hour exposure cycles per the specification would be extremely difficult to accomplish with the test configuration to be used. The test rig cannot be conveniently removed from the chamber once exposure has begun and the large mass of the chamber would require excessively long cooling periods before room temperature is **achieved.**

A flow diagram for heat sterilization was previously given in Table VIII. Heat sterilization of solder joint 1.5 (components between pairs of bifurcated terminals) was done with the joint still mounted on the printed wiring board. After heat cycling the terminal was removed from the printed wiring board for further testing. To facilitate removal from the circuit board without damage to the solder

joint, the bifurcated terminal was not staked, but was soldered directly to the gold plated etched circuit pad (see Figure 5). Removal was accomplished by clipping the leads from the resistor bodies and quickly de-soldering the terminals from the etched pads using heat sinks in order not to disturb the solder joint between the component lead and the tines of the bifurcated terminal. $\frac{\text{not}}{\text{d} \text{f} \text{se}}$

Figure 10 is a photograph showing the vacuum oven used for the dry heat sterilization tests with the weld joints inside the vacuum oven in separate Petri dishes. Figure 11 is a close-up of this vacuum oven with the solder joint specimens inside. Also shown in Figure 11 are certain of the solderability specimens.

4. 3 ULTIMATE STRENGTH TEST PROCEDURES

4.3. 1 Equipment

4.3. 1. 1 Hunter Spring Tensile Tester, Model TJH (Serial No. 175) equipped with a Hunter Mechanical Force Gage, Model D-50-T

Figure 10. Vacuum oven used for dry heat sterilization tests with weld joints inside (R105391).

specimens ready for dry heat sterilization (R105259).

(Serial No. 261). Calibrated on 26 April 1965. This tensile tester was used for all ultimate strength tests on solder joints.

4. *3.* 1. 2 Hunter Spring Tensile Tester, Model TJH (Serial No. 190) equipped with a Hunter Mechanical Force Gage, Model D-50-T (Serial No. 365). Calibrated on 17 March 1965. This tensile tester was used for all ultimate strength tests on welded joints.

4. *3.* 2 Procedures

All ultimate strength tests on solder joints were made by one technician using one tensile tester and all ultimate strength tests on welded joints were made by one technician using one tensile tester. Ultimate strength tests of both solder and weld joints were done at room temperature and were in accordance with Tables VI and VIII. **A** total of 1100 solder joints and 748 weld joints were tested for ultimate

strength. Weld joints and solder joints of similar types were clamped as nearly alike as *possible* using the same tensile tester jaw attachments and were **pulled** at the same rate (i. e. *,* identical air pressure).

Weld joints were all pulled in the torsion-shear mode as **shown** in Figure 12 and as **required** in JPL Spec. GPO-30995-GEN. Figure **13** is a close-up **photograph** of **weld joint 1.** 3 **being pulled** apart. These **same** jaw attachments **were used for** all **weld** joints.

Solder joints **to** connector cups and **to bifurcated ter_ninals were tested for ultimate strength** in **the** direction of **pull shown** in **the sketches** on Figure **12.** Special **jaw** attachments **for the Hunter** Tensile Tester made **it** *possible* **to the** grasp **the** many different joint configurations. Figure **14 shows the Hunter** Tensile Tester with **solder joint 1.1. 1 (stranded** conductor **to** CINCH connector cup) **in** position **ready for ultimate strength testing.** Figure **15 is** a close-up of **the** jaws of **the** Tensile Tester **showing solder** joint **1.3.3 (Dumet** wire **to bifurcated terminal)** in *position* for ultimate **strength testing.**

After **ultimate strength testing** of **the solder** and **weld** joints, **the** specimen **halves** were taped together and **labeled** accordingly. All strength values were **recorded** in pounds on **separate** Raw Data Sheets. These data **sheets** contained the strength values before and after thermal sterilization for each joint type. In addition each tested joint was examined for the failure **mode** and this was also recorded on the applicable data sheet. All labeled joints were placed in plastic bags and **saved** for **later submittal** to **JPL.**

4. 4 ELECTRICAL RESISTANCE TEST PROCEDURES

4.4. 1 Equipment

J

- 4.4. I. 1 Hewlett Packard Model 425A D.C. MicroVolt- Ammeter, Serial No. 399-01060. Calibrated on 18 June 1965.
- 4.4. I. 2 Daystrom Model 931 D.C. Milliammeter, Serial No. 28020. Calibrated on 18 May 1965.

 \mathcal{A}_J

Figure **13.** Weld joint being tested for ultimate strength in tensile tester **(R105347).**

Figure 14. Hunter tensile tester with solder joint 1. 1. 1 in position for ultimate strength testing **(R104840).**

Figure 15. Solder joint 1.3.3 mounted in jaws of tensile tester ready for ultimate strength testing (R105341).

4.4. **1.** *3* Kepco Model SM-36-5M Voltage Regulated D. C. Power Supply, Serial No. C-28791.

4.4. 1.4 Ohmite No. 0528 Rheostat, Series **A.**

4.4. 1. *5* Various Hughes-designed test fixtures for clamping solder and weld joints during electrical resistance tests.

4.4. 2 Procedures

Electrical resistance tests of soldered and welded joints before and after heating were done in accordance with the schedules shown in Tables VI and VIII. A total of 800 resistance tests on soldered joints and 560 resistance tests on welded joints were made. Solder joint 1. 5 was not given the electrical resistance test since it must remain mounted in the printed wiring board during thermal sterilization without being separated from the body of the 1/4-watt resistor. For purposes of a final statistical analysis of the test results, each individual joint was separately identified and tested for electrical resistance before and after heating. All electrical resistance tests were made by one technician.

The solder joint configuration for **resistance** testing was **per** Figure IZ. The welded **joint** configuration for resistance testing was per Type A on Figure 6.

Electrical resistance tests on solder joints were done using the upper test setup shown in Figure 16. Electrical resistance tests on welded joints will be done using the lower test setup shown in Figure 16. Special clips were used to **insure** that **all** joints of one type **are** tested in **an identical** manner. Precautions were taken to insure that contact resistance was minimized, so that only the resistance of the soldered or welded joint would be read. The test set-up shown in Figure 16 essentially **consists** of **applying a** current of I **ampere** dc through the **joint and** measuring the resultant voltage drop - which is equal to the joint resistance.

Figure 17 is **a** photograph of the electrical test set-up used for resistance measurements **of** both soldered and welded **joints.** All measurements were made after 1 minute of 1 ampere current flow. Readings were **then recorded on** the applicable Raw Data Sheets.

Figure 16. Test setup **for** electrical **resistance** (i. e. *,* voltage drop) tests of soldered and **welded** joints.

Figure 17. Test set-up for electrical resistance measurements of soldered and welded joints (R 105342).

Figure 18 is a close-up photograph of a solder joint (gold plated Dumet wire to solder coated bifurcated terminal) being tested for electrical resistance. **A** 200 gram weight was used, where applicable, for uniform contact pressure.

Figure 19 is a close-up photograph of a different type of solder joint (stranded conductor to Bendix connector cup) being tested for electrical resistance. The voltage probes are the two inside contacts and the current probes are the two outside contacts.

Figure 20 is a close-up photograph of a weld joint (gold plated Kovar to Inco) being tested for electrical resistance.

4.5 STRESS-RUPTURE STRENGTH TEST PROCEDURES

4. 5. 1 Equipment

4. 5. 1. 1 Conrad Temperature - Altitude Chamber, Model No. FH 8-3-3, Serial No. 7268. Calibrated on 14 May 1965. This chamber is equipped with a Honeywell Electronik temperature recorder which was last calibrated on 14 July 1965.

Figure **18.** Solder joint (wire to bifurcated terminal) in test fixture for electrical resistance measurement **(R105343).**

Figure 19. Solder joint (stranded conductor to connector cup) in test fixture for electrical resistance measurement **(R** 105 **344).**

Figure 20. Weld joint in test fixture for electrical resistance measurement (R105345).

- 4.5. 1. 2 Hughes Aircraft Company specially designed stress-rupture test fixtures. Two test fixtures were constructed. Each test fixture consisted of the following:
	- a) One specimen rack capable of holding *9* solder joints.
	- b) Nine Unimax Precision Microswitches, Type 2OGMXW-1, SPDT, 15 amps at 125 vac.
	- Nine buckets each loaded with shot to provide dead weights. c)
	- Nine panel-mounted Aero Instruments Type 61134-1 Running Time Meters (120 v, $60 \sim$) each with a neon indicator light. d)

4. 5. 2 Procedures

Stress -rupture strength was determined under conditions of actual heat sterilization (i.e., 145° C for 108 hours in a nitrogen atmosphere) and also at room temperature for 108 hours. Stress-rupture tests were performed on solder joints 1. 1. 1 and 1. 1. 2 (stranded

conductor to *CINCH* and BENDIX connector cups) in accordance with the schedule given in Tables VI and VIII. A total of 144 solder joints (72 each of joints 1. 1. 1 and 1. 1.2) were tested for stress-rupture strength. In addition **two** small ink marks were made on each solder joint prior to stress-rupture loading. Any change in axial position of these marks during or after the 108 hours of testing would show evidence of creep in the solder joint.

Testing was done by suspending weights from joint specimens within the test chamber so they will be under constant tensile load. The time to rupture (at a particular stress loading) was recorded. *Figure* 21 is a diagram of the stress-rupture test setup for a single soldered specimen. *When* the joint fails, the micro switch will close. Closing of the microswitch stops the time meter and turns off the neon light. The weights were selected so as to cause those joints of questionable strength to fail within the 108 hours of testing, provided that their stress-rupture strength is degraded by thermal sterilization. Since 9 joints were tested simultaneously, the initial selection of weight

Figure 21. Diagram of stress-rupture test setup.

loadings was from 10% to 90% of the ultimate strength of the solder joints being tested--approximately 15 *pounds.* After the 108 hour testing period, the joint was examined for the failure mode and for any indication of creep. Each joint was then labeled and bagged. The time meter readings were recorded on the applicable Raw Data Sheets.

Stress-rupture tests at room temperature conditions for both the Cinch and Bendix connector cup solder joints were connected using weights ranging from 10% to 90% of the ultimate strength of the joints. However during stress-rupture testing under sterilization time and temperature conditions, all of the Cinch connector cup solder joints failed even at the 10% loading. All failures occurred within 5. Z hours after the start of testing. The majority of joints failed within 1 hour after the 145°C temperature was reached, whereas identical joints would last the full 108 hours at **25°C.**

J

New stress loadings were therefore adopted for the remaining stress-rupture tests at sterilization time and temperature conditions for the Bendix connector cup solder joints. These new loadings are shown in Table IX and were designed to insure that some joints would fail and some joints would not fail after the 108 hours at 145°C in a nitrogen atmosphere. The ultimate strength of these joints is approximately 15 pounds.

Specimen Number	Old Loads (lbs)	% Ultimate Strength	New Loads (lbs)	% Ultimate Strength	Alternate New Loads (lbs)	% Ultimate Strength
	1.625	10.7	0.15		0.30	\mathbf{z}
\mathbf{z}	3.125	20.8	0.30	2	0.45	3
$\overline{\mathbf{3}}$	4.625	-30.7	0.45	3	0.60	4
$\overline{4}$	6.125	40.7	0.60	4	0.75	5
5	7.625	50.7	0.75	5	1.125	7.5
6	9.125	60.6	1.50	10	1.50	10
7	10.500	70.0	2.25	15	2.25	15
8	12.063	80.4	3.00	20	3.00	20
9	13.563	90.3	3.75	25	3.75	25

Table IX. Old and new stress-rupture test loadings.

Figure 22 is a photograph of the stress-rupture test set-up under room temperature conditions. Nine Cinch connector cup solder joints are shown under test. The front three joints have already failed as indicated by the weight buckets having fallen to the floor.

Figure 23 is a close-up photograph showing a Bendix connector cup solder joint suspended from the specimen rack. The metal cross-piece (not the joint itself) depresses the roller on the snap switch.

Figure 24 shows 18 Bendix connector cup solder joints (1.1.2) in the Temperature-Altitude Chamber ready for stress -rupture testing under conditions of heat sterilization time and temperature. Note the two panels of running time meters outside the chamber.

4.6 ELECTRICAL TEST DURING VIBRATION TEST PROCEDURES

4. 6. 1 Equipment

4. 6. 1. 1 Ling Electronics Vertical Vibration System, Model C-P 3/4, Serial No. 45. Calibrated on 16 Aug. 1965.

 $-$ Figure 22. Stress-rupture test set-up under room temperature conditions (R 1 0 5 **3** *39).*

Figure 23. Close-up photograph showing a Bendix contractor cup solder joint suspended from the specimen rack for stressrupture testing (R105340).

Figure 24. Stress-rupture test set-upfor 18 solder joints under conditions of heat sterilization time and temperature (R105736).

- 4.6. 1.2 Endevco Accelerometer, Model 2211, Serial No. R5641. Calibrated on 14 May 1965.
- 4.6.1.3 Hewlett Packard Wave Analyzer, Model 302A, Serial No. 149-00127. Calibrated on 28 May **1965.**
- 4.6. 1.4 Hewlett Packard Electronic Counter, Model **5ZZB,** Serial No. 1897. Calibrated on 21 June 1965.
- 4. 6. I.5 Hewlett Packard Audio Signal Generator, Model **205AB,** Serial No. 6965. Calibrated on 16 June 1965.
- 4. 6. 1.6 Hewlett Packard Vacuum Tube Voltmeter, Model 400 H, Serial No. 1197. Calibrated on 2 June 1965.
- 4.6. 1.7 Hewlett Packard A. C. Current Probe, Model 456A, Serial No. 103-0939. Calibrated on 22 May 1965.
- 4.6. 1.8 Chadwick-Helmuth Sweep Synchronizer, Model 201, Serial No. 103AR.
- 4. 6. 1.9 Chadwick-Helmuth Strobex, Model 121R.
- 4.6. I. I0 Dynac Sweep Oscillator, Model DY-2200, Serial No. 167. Calibrated on 6 August 1965.

4.6.2 Procedures

The purpose of this test is to detect electrical variations in joint resistance during vibration and to compare these variations with other specimens. These electrical variations in joint resistance are assumed to be directly related to joint deterioration.

Electrical tests during vibration were done on all solder and weld joint types per the schedules outlined in Tables VI and VIII. A total of Z20 solder joints and 188 weld joints were electrically tested during vibration before and after thermal sterilization. Weld joint specimens for this particular test were Type B as shown in *Figure* 6.

The electrical test setup as originally proposed (see Figure 25) was found unsatisfactory for joints involving magnetic materials. The lines of flux in the magnetic field created by the shake table would be cut by the vibrating joint generating a false display on the oscilloscope. This variation in scope output, **although** only in the microvolt region, was of a magnitude approximately equal to that of any expected joint variation and therefore it was impossible to tell with any certainty whether the joint or the noise was responsible for the change in waveform. Adequate magnetic shielding of the joint was not practical due to the heavy flux density. A new electrical test setup was developed which proved satisfactory. This setup is shown in Figure 26.

In the new set-up the d-c current source is replaced by **an** a-c signal **and** a tuned microvoltmeter is used as a detector. The **a-c** signal was set **at 5** kilocycles **and** the microvoltmeter was tuned to maximum sensitivity at that frequency. This technique shows considerable improvement over the previous technique **and** will detect **a** change in joint resistance (i.e., voltage drop or rise) of less than 5%. A 5 KC

Figure 25. Old electrical test setup **for** electrical measurements of soldered and welded joints during vibration.

Figure 26. New electrical test **setup for** electrical measurements of soldered and welded joints during vibration.

signal (ac current as opposed to dc current) is used since this is above the shaker frequency and will eliminate any harmonics. This technique will reject any signal except that through the joint under test. Both good joints and bad joints (loose connections) were tested with this technique to insure that the measured signal was due only to the change in joint resistance. At a current input of only 15 milliamperes, it was possible to get a voltage change of 250 microvolts for bad joints. Good joints showed no change in resistance.

Vibration testing was done by running the joint under test from 60 to Z000 cycles per second at a 15-G acceleration level and noting the resonant frequency. The joint was then allowed to dwell at that resonant frequency for 5 to 10 minutes and any change in joint resistance (i.e., microvolt signal output to the wave analyzer) was noted at resonance. The current probe was used to monitor the current through the joint and the electronic counter was used to measure the resonant frequency. An a-c current input of 100 milliamperes was used for all weld joints and solder joints. All joints of one type were identically mounted on the shake table and were measured at the identical resonant frequency. All joints were mounted taut without slack to prevent different resonant

frequencies for joints of the same type. Using a constant table displacement, weld joints at resonance exhibited a maximum amplitude of about 0. 1155 inch. During vibration testing physical resonance was observed by watching the joint displacement and electrical resonance was observed by monitoring the scope display.

0

0

Figure 27 is a photograph showing one weld joint and three solder joints of different types mounted on the vibration platform. photograph was taken to illustrate the different methods of mounting used for each type of weld joint and solder joint. During actual vibration testing, either four or **two** joints of one type in series were vibrated together. Weld joints and solder joints or joints of different types were not mixed together. This

Figure 28 is a close-up photograph of these same four joints. The vibration plane was perpendicular to the plane of the photograph. Before vibration the series circuit of either four or two joints was measured for electrical resistance. The current input, resonant frequency, series joint resistance, and any change in resistance during vibration were all recorded on the applicable Raw Data Sheets.

Figure 27. Weld joint and solder joint specimens mounted on vibration table in preparation for the electrical tests during vibration (R105473).

Figure 28. One weld joint and three solder joints mounted on vibration table (RlO5474).

4.7 METALLURGICAL EXAMINATION

4. 7. 1 Equipment

- 4. 7. 1. 1 Zeiss Inverted Metallurgical Microscope, Model No. B-5000. Used for examining unetched mounts of all metallographic specimens at 500X magnification.
- 4. 7. 1. 2 Reichert Metallograph, Model MeF. Used for examining etched mounts of all metallographic specimens at magnifications up to 1000 X. The Reichert Metallograph is equipped with a builtin camera and was also used for taking photomicrographs. The film used was Kodak Super Panchro Press.
- 4. 7. 1. **3** Buehler Model 67-1509 Vibromet Polisher.
- 4. 7. 1.4 Buehler No. 1330AB Mounting Press.
- 4.7. 1.5 Precision Scientific "Precisionite" mounting powder for all hot mounts.
- 4.7. 1.6 Fulton Metallurgical "Quickmount" mounting powder for all cold mounts.

4.7.2 **Procedures**

Metallographic **examination was** done on all **joint types** as outlined in Tables VI and VIII. A **total** of **110 solder joints** and 82 **weld joints** were metallographically mounted, **sectioned** and examined. This total does not include the additional n_etallographic mounts which were made for material incoming inspection and for specimen standardization. Photomicrographs were made of 26 weld joints at a magnification of 150X. The total of 26 weld joints included:

- a} One each of 7 weld joint types before heat sterilization.
- b) One each of 7 weld joint types, after heat sterilization.
- c) One each of 6 JPL weld joint types before heat sterilization.
- d) One each of 6 JPL weld joint types after heat sterilization.

Photomicrographs were made of 2Z solder joints (2 each of II solder joint types - one before and one after heat sterilization). Photomicrographs of stranded conductors soldered to connector cups (joint types 1.1.1 and 1.1.2) were taken at 60X. Photomicrographs of solid wire conductors soldered to bifurcated terminals were taken at 100X. All solder joints were cold mounted in Quickmount and all weld joints (except joint type i. 6 - solder coated copper to Inco) were hot mounted in Precisionite. Weld joint !. 6 was cold mounted in Quickmount.

Metallographic mounts of solder joints (bifurcated terminal and connector cup types) were prepared by multiple mounting, as shown in Figure 29. Five joints which were not submitted to thermal sterilization were mounted directly above and adjacent to five joints which had been thermally sterilized. This enabled a quick visual comparison between specimens without switching mounts. Weld joints were individually mounted in separate mounts as shown in Figure 29. This was necessary since it is very difficult to polish into the exact center of two weld joints on the **same** mount.

48

"1

 \mathcal{A}

All weld joints and solder joint metallographic specimens were examined for any degradation which could be attributed to **the** thermal sterilization cycle.

The various etchants used to define the metallurgical structure of the solder and weld joints are listed at the bottom of each photomicrograph.

4.8 ELECTRON PROBE MICROANALYSIS

4.8. 1 Equipmeht

4.8. 1. 1 Applied Research Laboratories Model **21000** Electron Microprobe X-RayAnalyzer, Serial No. EMX-39.

4.8.2 Procedures

Electron probe microanalysis was subcontracted to the Materials Testing Laboratories, Division of Magnaflux Corp. , Los Angeles, *California.* A total of 8 solder joints and 4 weld joints were analyzed as shown in Table X.

The specimens were primarily examined for any signs of gold diffusion into the copper wire, brass terminal, or Inco (nickel) ribbon. Figure 30 is a *photograph* of the ARL Electron Microprobe X-Ray Analyzer.

All joints were mounted in standard 1" diameter metallographic mounts and final polishing was accomplished with diamond abrasive. All joints were sectioned in such manner as to expose the cross section of the copper wire.

The Applied Research Laboratories Microprobe was used for these analyses. Monochromatic crystal detectors were peaked out on suitable portions of the specimens, using the following radiation lines.

5O

Specimen Number	Sterilized	Type of Joint	Joint Description
1.4.111 1.4.112 1.4.113 1.4.114	No. No. Yes Yes	Solder Solder Solder Solder	Gold Plated (approx. 88 micro- inches) Copper Wire (0.020" dia.) to Gold Plated Bifurcated Terminal. Terminal was 1/2 Hard Brass with Copper Strike, Silver Strike, Sil- ver Plate $(0.0002" - 0.0003")$ and gold Plate (70 microinches)
1.3.1.111 1.3.1.112 1, 3, 1, 113 1, 3, 1, 114	No. No. Yes Yes	Solder Solder Solder Solder	Gold Plated (88 microinches) Copper Wire (0.20" dia.) to Solder Coated Bifurcated Terminal. Ter- minal was 1/2 Hard Brass with Solder Coating (160-500 micro- inches).
1.5.111A 1.5.112A 1.5.113A 1.5.114A	No No. Yes Yes	Weld Weld Weld Weld	Gold Plated (88 microinches) Copper Wire (0.029" dia.) to Nickel (Inco 200) Ribbon $(0.010" \times 0.031")$

Table **X.** Solder and weld joint specimens submitted to electron probe microanalysis.

Figure 30. Electron microprobe x-ray analyzer.

The specimen in each case was set so that the beam travel would be in a direction perpendicular to the joint interface and **as** close as possible **to** its centerline. Traversing was in **all** cases by one micron increments. At each **step** the elements in question were **read** out after a fixed time integration (i. e. **simultaneous** counting into each recording channel). The attached charts are identified as to **the** elements run. A record of sample current was also made, **this** figure being inversely related to average atomic number. The units in all cases are arbitrary.

The following conditions were used for the analyses:

5. TEST RESULTS

5..I ULTIMATE STRENGTH TEST RESULTS

5. i. 1 Solder Joints

Pages 1 through 6 in Part II, Attachment 2, of this final **report** contain the Raw Data Sheets for the ultimate strength tests on the eleven types of solder joints. For each type of joint, numbers 1-40 were tested before heating (control specimens) and numbers 41-80 **were** tested after thermal sterilization. All ultimate strength values are given in pounds. After each strength value the mode of failure **was** noted:

W *=* Wire failure J *=* Joint failure

Wire and joint failures for bifurcated terminals and connector cups are illustrated in the sketches shown in Figure 31. Wire failures for bifurcated terminals usually occurred from 1/4" to 1/Z" away from the solder joint. Connector cup solder joints (1. 1. 1 and 1. 1.2) all failed at the wire. Certain bifurcated terminal solder joints (i.e., 1.3.4, 1.3.5, and 1.5) were predominantly joint failures and other bifurcated terminal solder joints (i.e., 1.3.6) were all joint failures. Joint failures (see *Figure* 31) were of two types: l) the wire broke right at the joint; and 2) the wire was pulled out of the bifurcated terminal without breaking.

Table XI presents a statistical analysis of variance for the ultimate strength test data on solder joints. A single factor analysis of variance was required since this was a destructive test. In view of the nature of the test data the significance level was determined using both the *F* test and *Chi-Square* test for significance. *For* example, a significance level of 0.01 means that there is only 1 chance in 100 that the analysis was wrong and a significance level of 0.001 means that there is only 1 chance in 1000 that the analysis was wrong. The 95% confidence intervals of means was calculated for both the control specimens and the sterilized. Taking the first solder joint in Table XI (1. 1. 1

 \sim \sim

BIFURCATED TERMINAL JOINT FAILURE

Figure 31. Failure modes for solder joints.

Table XI. Analysis of variance - ultimate strengths of soldered joints.

55

 $\frac{1}{2}$

Stranded conductor to Cinch cup), the control specimens have a lower limit of 14.6 pounds and an upper limit of 14.7 pounds. We are 95% confident that the arithmetic mean lies within this interval. The same reasoning applies to all specimens. Four of the solder joint types (i.e., I. 1.2, 1.2, i. 3.2, and 1.4) showed a decrease in ultimate strength due to sterilization and one type (I. 3. i) showed an increase in ultimate strength. Thermal sterilization had no significant effect on any of the other joints or, putting it another way, any change in strength values before or after sterilization was due to chance or random error.

Figure 32 is a series of bar diagrams of the ultimate strength values for soldered joints. This is simply a different way of presenting the statistical values of Table XI. The control and sterilized specimen strength values are plotted side-by-side with the shaded area at the top of each bar graph being the 95% confidence intervals of means. When the shaded areas overlap each other, the conclusion is that there is no effect on strength due to thermal sterilization. It is also easy to see which of the solder joint types are strongest. Joint types I. 3.5 and 1.3.6 have higher ultimate strengths than any of the others, both being nickel wire.

Taking as an example joint type i. 1.2 which shows a decrease in ultimate strength after sterilization, this decrease (although significant statistically) is actually only a decrease of 1% . A 1% change in the strength of a solder joint is, for all practical purposes, relatively unimportant in view of the many other variables involved.

5. 1.2 Welded Joints

Pages 7 through 10 in Part II, Attachment 2, of this final report contain the Raw Data Sheets for the ultimate strength tests on the seven types of weld joints. For each type of joint, numbers 1-40 were tested before heating and numbers 41-80 were tested after thermal sterilization. All ultimate strength values are given in pounds. At the right of each strength value is noted the mode of failure:

> W2 = Wire broke adjacent to weld R2 *= Ribbon* broke adjacent to weld WS *=* Weld separation

مأما

Figure 33 contains sketches illustrating these three failure modes for the weld joints. Certain of the weld joints (i.e., I. l, 1.2, 1.5) failed predominantly in the W2 mode, while other types of weld joints failed predominantly in the R2 mode (i.e., 1.3, 1.4, 2.0) or the WS mode **(i.e.,** 1.6).

Table XII presents a statistical analysis of variance for the ultimate strengths of welded joints. One weld joint type (1.5 gold plated copper to Inco) showed a significant decrease in strength after sterilization. The "gold plated Kovar foil to itself" weld joint showed a slight effect due to sterilization primarily due to the comparative increase in standard deviation. Although joint type I. 5 exhibited an effect with a significance level **of** 0. 001 by the F test, the actual change in ultimate strength was only 5% . This change is attributed to a shift in the distribution **of** the test result data, as illustrated by the histogram shown in Figure 34.

Figure 35 is a series of bar diagrams for the ultimate strengths of weld joint presenting, in a more graphical manner, the statistical data given in Table XII.

5.2 ELECTRICAL RESISTANCE TEST RESULTS

5.2. 1 Solder **Joints**

Pages II through 15 in Part II, Attachment 2, of this final report contain the Raw Data Sheets for the electrical resistance tests on ten types of soldered joints. For each joint type, specimens 41-80 were tested before thermal sterilization and the identical specimens were again tested after thermal sterilization. All resistance values are given in milliohms (ohms $x 10^{-3}$).

Table XIII presents a two factor statistical analysis of variance for the electrical resistance test data on solder joints. Four of the solder joint types (i.e., i. I. I, I. 1.2, 1.3.5, and I. 3.6) exhibited a slight decrease in electrical resistance as a result of thermal sterilization and two of the solder joint types (i.e. , 1.2 and 1.3.2) showed a slight increase in electrical resistance due to thermal sterilization.

58

 \mathbf{I}

R 2 = RIBBON BROKE ADJACENT TO WELD

SEPARATION W S $=$ WELD

Figure 33. Failure modes for welded joints.

 λ

Table XII. Analysis of variance - ultimate strengths of welded joints.

į.

 $\begin{array}{c} \hline \end{array}$

 $\mathring{\|}$

60

Ġ,

 $\ddot{}$ $\frac{1}{2}$

Figure 34. Histograms **of** strengths of gold-plated copper to Inco weld joints showing shift due to heat sterilization.

The F test was used throughout for significance level calculations and all shifts in the 95% confidence intervals of means had a significance level of 0.001.

Figure 36 is a series of bar diagrams for the electrical resistances of soldered joints presenting, in a more graphical manner, the statistical data given in Table XIII. Note the extremely high resistance of the gold plated Kovar solder joint (1.3.4) compared to the others. The average resistance of this joint is 18 times greater than the average resistance of a stranded conductor (1.2} soldered to the same bifurcated terminal. It is also interesting to **note** that the Bendix connector **cup** joint (1.1.2) had more than twice the resistance of the Cinch connector cup joint (1. 1. 1).

Although joints 1.2 and 1.3.2 showed an increase in resistance after thermal sterilization, this increase was only 4.7% in the case **of** joint 1.2 and 5.4% in the case of joint 1.3. Z.

 $\frac{1}{\sqrt{2}}$

 62

 γ
Table XIII. Analysis of variance - electrical resistance of soldered joints.

 $\tilde{\mathbb{F}}_q$

 $\label{eq:3.1} \frac{d\mathbf{y}}{dt} = \frac{1}{\sqrt{2\pi}}\left[\frac{d\mathbf{y}}{dt} + \frac{d\mathbf{y}}{dt} + \frac{d\mathbf{y}}{dt}\right] + \frac{d\mathbf{y}}{dt} + \frac{d\mathbf{y}}{dt}$

 $\ddot{}$

 $\ddot{}$

 $-- -$

 63

 $\ddot{}$

 $_3$ $\lambda _i$

5.Z.? Weld Joints

Pages 16 through 19 in Part If, Attachment Z, of this final report contain the Raw Data Sheets for the electrical resistance tests on the seven types of weld joints. For each joint type specimens 41-80 were tested before thermal sterilization and the identical specimen was again tested after thermal sterilization. All resistance values are in micro- -6 ohms (ohms $x\,10$). A negative sign in front of the resistance reading indicates that the needle of the microvoltmeter indicated a negative polarity for that point. The lack of a sign in front of the resistance reading indicates a positive polarity reading on the microvoltmeter. Note that weld joints 1.1, 1.2, 1.5 and 2.0 have negative polarities, while weld joint I. 3 had a positive polarity. Joints 1.4 and 1.6 had both "+" and "-" polarities. This is a phenomenon observed by others for which no satisfactory explanation is known. Of course a resistance reading has no polarity and therefore the values on the Raw Data Sheets should be interpreted as absolute readings.

Table XIV presents a statistical analysis of variance for the electrical resistances of welded joints. Three weld joint types (i.e., I. 3, 1.5, and 1.6) showed an increase in resistance after sterilization and one weld joint type (i. e. , I. 2) showed a very slight decrease in electrical resistance after thermal sterilization. It is interesting to note that the variance of the resistance readings for the weld joints was much greater than the variance for the solder joints and also that the joint resistances for weld joints were a magnitude lower than the solder joint resistances. The gold plated Kovar foil weld joint had a resistance almost 73 times greater than the solder coated copper wire weld joint.

Figure 37 is a series of bar diagrams for the electrical resistances of weld joints presenting, in a more graphical manner, the statistical data of Table XIV. In the case of both solder and weld joints the Kovar materials exhibited high resistances, From Figure 37 one also notes that the nickel weld joints (1.1 and 1.2) had less shaded areas on the bar diagrams indicating the electrical resistances of these materials had less spread (i. e. , the joints were more consistent and predictable).

65

Table XIV. Analysis of variance - electrical resistance of welded joints.

 $\hspace{1.6cm} - \hspace{1.4cm}$

 \tilde{z}

 $\overline{}$

66

Ŷ,

Bar diagrams of electrical resistances of welded joints before and after
sterilization. 95 percent confidence intervals of means are shown shaded. Figure 37.

5. 3 STRESS-RUPTURE STRENGTH TEST RESULTS

Pages 20 through 23 in *Part* II, Attachment 2, of this final report contain the Raw Data Sheets for the Stress-Rupture Strength tests on two types of solder joints (i.e., stranded conductor to *Cinch* connector cup and stranded conductor to Bendix connector cup). Both types of solder joints were tested for the full sterilization time (108 hours) at room temperature $(74^{\circ}F)$ and also for the full sterilization time at sterilization temperature (145°C) in a *nitrogen* atmosphere. Each test was repeated 4 times with 9 Specimens loaded during each test. For each joint type specimens 111-146 were tested at room temperature and specimens 147-182 were tested at sterilization temperature. Failures were of two types: 1) wire failures; and 2) joint failures. These failure modes are illustrated in Figure 38. Wire failures were predominant at the 80% and 90% of ultimate strength loadings (i. e. , 12 lb. - 1 oz. and 13 lb. -9 oz.) due to untwisting of the wire strands under load. Joint failures occurred at the lower loadings with the wire pulling right out of the connector cup.

Creep was not observed on any specimen. A small amount of creep could have occurred and would not have been detected, since the test was basically for stress-rupture strength. Creep is usually observed over a period of 1000 hours with axial deformation being read periodically.

JOINT FAILURE

Figure 38. Failure modes during stress-rupture strength tests.

In view of the nature of the data and since the loads were not left on the wires until all joints failed, a meaningful statistical analysis was not possible. However a graph of the results is presented in *Figure* 39. Considering first the room temperature results, one notes that the Bendix cup joints up to and including 70% of ultimate strength lasted the full 108 hours. The Cinch cup joints up to and including 50% of ultimate strength lasted the full 108 hours. *Again* from Figure 39 consider the results of sterilization temperature. None of the Cinch joints--even at only 10°70 of ultimate strength--lasted through 108 hours. *Failures* occurred within the first 5 hours.

The Bendix cup joints for 1% , 2% , 3% , 4% , and 5% of ultimate strength lasted the full I08 hours. Unfortunately none of the Cinch cup joints were tested at such low loads. Even at 10% of ultimate strength the Bendix cup joints lasted an average of 27 hours. Figure 39 certainly points out that the Bendix cup joints are stronger than the Cinch cup joints in stress-rupture strength. Figure 39 also shows that the stress-rupture strength of a connector cup solder joint decreases considerably at elevated temperatures.

Taking the Bendix cup joint as an example, the strength dropped from 70% of ultimate strength to only 5% of ultimate strength at 145° C. In other words, the strength at sterilization temperature was only 14% of the strength at room temperature.

By way of explaining the higher stress-rupture strength of the Bendix cup, the Bendix cup had a copper + silver + gold outer electroplate while the Cinch cup had only a copper + gold outer electroplate. The Bendix cup was also deeper (0. 134" as compared to 0. 100") and had a larger inside diameter (0.048" as compared to 0.0445").

5.4 ELECTRICAL TEST DURING VIBRATION TEST RESULTS

5.4. 1 Solder Joints

Pages 24 through 27 in Part II, Attachment 2, of this final report contain the Raw Data Sheets for the electrical tests during vibration **of** the eleven types of solder joints. For each joint type, specimens 91-100

69

i

LOAD, PERCENT ULTIMATE STRENGTH

Figure 39. Stress-rupture test results at room temperature and at sterilization time and temperature.

> $\hat{\mathbf{z}}$ Ą.

were the control specimens and numbers 101-110 were vibrated after having been submitted to thermal sterilization. For each set of either 4 or 2 joints, the a-c joint resistance is given (at 100 milliamperes current input) together with the percentage change in joint resistance at resonance.

In no instance, with either the control specimens or the heated specimens, was any detectable change in joint resistance observed during vibration. Figure 40 summarizes the results showing the various resonant frequencies of each joint type and the series electrical resistances. The Cinch connector cup joints had an unusually high joint resistance--approximately 225 micro-ohms per joint--as compared to the other joint types. However if we compare this resistance value with Figure 36, which contains the electrical resistance test results, we note that the dc electrical resistance of the Cinch connector cup joint was about 250 micro-ohms. Since this was an extremely sensitive test which was capable of picking up resistance changes as small as 5% , it must be assumed that the solder joints were made so well that they, in fact, did not change electrically under vibration conditions.

5.4.2 Weld Joints

Pages 28 through 31 in PartII, Attachment 2, of this final report contain the Raw *Data* Sheets for the electrical tests during vibration of the seven types of weld joints. For each joint type, specimens 91-100 and JPL specimens 3-6 were tested without prior treatment, while specimens 101-110 and JPL specimens 7-10 were tested after thermal sterilization.

In no instance, with either the control or the sterilized specimens, was any detectable change in joint resistance observed at resonance during *vibration* testing. Figure 41 summarizes the test results for weld joints in a similar manner as was done for solder joints in Figure 40. One interesting thing to note is that the resonant frequencies of the weld joints are almost twice that of the solder joints. The joint resistances of the Hughes and JPL weld joints are of a similar magnitude. In two joint types (i.e., 1.3 and 1.5) the Hughes joints had **lower**

71

 $\ddot{}$

Summation of results for electrical tests during vibration of soldered joints. Figure 40.

 $\overline{}$

 \cdot

 $\hat{\boldsymbol{\beta}}$

Summation of results for electrical tests during vibration of welded joints. Figure 41.

*Questionable

 $\hat{\gamma}$

73

 $\overline{}$

resistances and in two joint types (i.e., 1.4 and 1.6) the JPL joints had lower resistances.

5.5 ULTIMATE STRENGTH AFTER VIBRATION TEST RESULTS

5.5. 1 Solder *Joints*

Pages 32 and 33 in Part II, Attachment 2, of this final report contain the Raw Data Sheets for the ultimate strength after vibration test results for the eleven types of solder joints. All strength values are given in pounds. After each strength value, a "W" or "J" has been recorded denoting the failure mode for that particular joint.

Table XV is a statistical analysis of variance of the ultimate strengths of solder joints after vibration. None of the joints showed any significant effect due to thermal sterilization. Figure 42 contains a series of bar diagrams which present the statistical data of Table XV in a more graphical manner. Comparing Figure 42 with Figure 32 (ultimate strengths of solder joints without vibration) one notes that the strengths of the joints are approximately the same regardless of whether they have been vibrated or not. Comparing the 95% confidence intervals of means (shaded areas), it is noted that vibration has obviously caused a marked change in the variance about the mean of the strength data. Remembering the difference in sample sizes, it is possible that this difference in variance could be attributed to the fact that only 25% as many specimens were tested for ultimate strength after vibration as were tested for ultimate strength without being vibrated first.

5.5.2 Weld Joints

Pages 34 and 35 in Part II, Attachment 2, of this final **report** contain the Raw Data Sheets for the ultimate strength after vibration test results for the seven types of weld joints. After each strength value (in pounds) is noted the mode of failure as described earlier in Figure 33.

Table XV. Analysis of variance - ultimate strengths of soldered joints after vibration.

 $\ddot{}$

*Within round-off limits

 $\frac{1}{2}$

 $\frac{1}{2}$

Ñ

 75

Table XVI is a statistical analysis of variance of the ultimate strengths of the weld joints after vibration. None of the joints showed any significant effect due to thermal sterilization. Figure 43 contains a series of bar diagrams which present the statistical data of TableXVI in a more graphical manner. *Comparing* Figure 43 with Figure 35 (ultimate strengths of weld joints not vibrated) one notes that the strength values are quite similar in value. As with the solder joints, the variance about the mean was greater with the weld joints which had been pulled apart after being vibrated. Certain of the weld joint types $(i.e., 1.1, 1.2, 1.4, 1.6, and 2.0)$ even exhibited slightly higher average ultimate strengths after being vibrated.

Figure 44 demonstrates the consistencies of the ultimate strengths of the weld joints made by Hughes and JPL using different equipment, different weld schedules, and different operators. The values plotted as bar diagrams are the arithmetic means taken from the Raw Data Sheets (Pages 34 and 35) and using the "before heat" readings in all cases. Even the weld joint with the greatest difference of means (number 1.4) shows only a difference of 16% .

5.6 METALLURGICAL EXAMINATION TEST RESULTS

5.6. 1 Solder Joints

Figure 1 through 22 in Part II, Attachment 3, of this final report contain the photomicrographs of the metallographic mounts of the 11 types of solder joints. The solder joint in the photomicrograph at the top of each page was not sterilized, while the solder joint in the photomicrograph at the bottom of each page had been thermally sterilized. A potassium dichromate etchant was used for all solder joints to bring out the metallurgical structure. By visually comparing the top and bottom photomicrographs, the observer can examine the solder joints for any metallurgical effect due to thermal sterilization.

Although the photomicrographs are only at magnifications of $60x$ and 100x, all mounted solder joint specimens (110 in total) were examined at 500x in both the polished and etched condition. None of the

77

Table XVI. Analysis of variance - ultimate strengths of welded joints after vibration.

 $\ddot{}$

,一个人的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,
第2012 年 - 我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时候,我们的时

 $\overline{}$

 $\frac{1}{2} \left(\frac{1}{2} \right) \frac{1}{2} \left(\frac{1}{2} \right) \frac{1}{2} \left(\frac{1}{2} \right)$

 \sim \sim \sim

78

ŀ,

 Q

79

 $\Phi_{\vec{P}}$

Figure 44. Comparison of the average ultimate strengths of the Hughes and **JPL** weld joints.

eleven types of solder joints showed any degradation in the metallurgical structure of the solder joint which could be attributed to thermal s te r ilization.

5.6.Z Weld Joints

Figures 23 through 48 in Part II, Attachment 3, of this final report contain the photomicrographs of the metallographic mounts of the 7 types of weld joints. The weld joint in the photomicrograph at the top of each page was not sterilized, while the weld joint in the photomicrograph at the bottom of each page had been thermally sterilized. For each of the weld joint types (except joint 2.0 - Kovar foil to itself) a set of photomicrographs showing the before and after sterilization structure has been prepared for the weld joints made by 5PL. These joints are identified by the letters JPL 1 or JPL Z following the weld joint number.

80

• r.

All JPL 1 joints were not sterilized, while the JPL 2 joints were thermally sterilized. The etchants used for the weld joints were either: a) nitric-acetic; b) Carapella; or c) Carapella + nitric-acetic. All weld joint photomicrographs are at 150x magnification.

/

All of the mounted weld joint specimens (82 in total) were examined at 500x in both the polished and etched condition. None of the seven types of weld joints showed any degradation in the metallurgical structure of the weld joint which could be attributed to thermal sterilization. The only weld joint showing any **unusual** characteristic was the gold plated copper to Inco joint (joint no. 1.5). Figures 39-42 in Part II, Attachment 3, show this particular weld joint. An internal structural change was present within the middle of the Inco ribbon in both the Hughes and JPL joints. Looking back at the original weld schedules (Table V), it is noted that this particular joint required the highest weld energy (33 watt-seconds for the Hughes weld joint) of any of the weld joints. This energy has apparently penetrated deeply within the Inco ribbon. However this defect has not seemed to detract **from** the ultimate strength of this joint, since the ultimate strength bar diagrams (Figure 35 and Figure 43) appear **normal.**

5.7 ELECTRON PROBE MICROANALYSIS TEST RESULTS

Pages 1 through 8 in Attachment 4, Part iI, of this final report contain the electron probe microanalysis charts for both the wire end and terminal end of solder joint 1.3. l (gold plated copper wire to solder coated bifurcated terminal). Two charts are included for each of the four specimens examined.

Pages 9 through 16 in Attachment 4, Part II, of this final report contain the electron probe microanalysis charts for both the wire end and terminal end of solder joint 1.4 (gold plated copper wire to gold plated bifurcated terminal). Two charts are included for each of the four specimens examined.

Pages 17 through 24 in Attachment 4, Part II, of this final report contain the electron probe microanalysis charts for both the wire side and ribbon side of weld joint 1.5 (gold plated copper wire to Inco (nickel)

81

 Ω

ribbon). Two charts are included for each of the four specimens examined.

The major consideration in these analyses was the comparative extent of gold penetration into the copper for the sterilized and the unsterilized specimens. To this end the charts were read in terms of the width of the apparent diffusion zone.

For specimens 1.4. **lll** through 1.4. ll4 there were two interfaces of interest. The charts show no diffusion of gold into the copper wire or the terminal. The gold coating on the brass appeared to be absent. It should be noted that the 0% Gold point (i.e. background) is higher for the heavier metals than for copper.

Specimens 1.3.1.111 through $1.3.1.114$ showed no gold diffusion into the copper. It should be noted that the gold layer did not appear on these specimens.

Specimen 1.5. 111 and 1.5. 112 showed no evidence of gold in the copper at the point examined. Specimen 1.5. 113 showed no penetration at the center and 6 microns of gold diffusion into the copper near one edge of the weld. Specimen 1.5. 114 showed 6 microns of gold diffusion into the copper. In these cases there were very large **local** variations in composition in the weld metal. It appeared that the gold had been caused to migrate to the edges, away from the point of maximum pressure.

Although the results on the weld **specimens** were somewhat inconclusive, it is concluded that no gold diffusion into the copper or copper alloy was caused by the sterilization treatment. This is particularly borne out by the lack of any indication of diffusion of gold into copper in the gold to copper interface in the soldered joints, where the interface was very **sharp.** There is no logical reason to expect more gold diffusion in one specimen than another. It is believed, therefore, that all the observed variations resulted from the joining conditions.

The *Materials* Testing Laboratories has recommended that if further work on this problem is contemplated, it is suggested that the same specimen be examined before and after sterilization, so that

82

other variables would be blanked out. The use of such tiny weld specimens would involve treating it in the mount. If it is necessary to avoid this, it would seem that more massive specimens would serve the purpose.

6. CONCLUSIONS

Table XVlI presents a summary of the test results on soldered and welded joints. A more detailed discussion of the results of each particular test is included in Sections 5. 1 through 5.7 of this final report. Analyses of variance, bar diagrams, and histograms of the test results are included in Tables XI-XVI and *Figures* 31-44.

In general both the welded and soldered joints show little or no change in ultimate strength or electrical resistance as a result of thermal sterilization. This conclusion applies to ultimate strength before and after vibration and to both static electrical resistance **tests** and the percentage change in dynamic electrical resistance during vibration. From Table XVII it is noted that two solder joints (1.3.3 gold plated Dumet to solder coated bifurcated terminal and 1.3.4 gold plated Kovar to solder coated bifurcated terminal) showed no significant changes for all tests. In the case of the welded joints, two joints (1. 1 bare nickel to inco and 1.4 gold plated *Dumet* to Inco) showed no significant changes for all tests.

Where significant shifts occurred (increase or decrease), these shifts, although significant statistically, were actually minor in magnitude. For example, in the case of ultimate strengths, the random errors were such that the use of 80 specimens (40 sterilized and 40 unsterilized) permitted detection of strength shifts of considerably less than 5% with ease. Comparing changes in strength or electrical resistance before and after sterilization, these changes were, on the whole, usually less than 5%. Since such small shifts are not likely to be of importance in actual applications, it is concluded that, for all practical purposes, there was no significant change due to the effects of thermal s terilization.

Neither the metallurgical examination nor the electron probe microanalysis showed any degradation in metallurgical structure or in the extent of gold diffusion which could be attributed to the thermal sterilization process.

85

ጓ እ

Table XVII. Summary of test results on soldered and welded joints.

86

 $\frac{1}{2}$

Only the stress-rupture tests demonstrated that thermal sterilization times and temperatures can seriously affect the strength of soldered joints under load. These tests (refer to Section 5.3) pointed out that steady-state loads of only 10% of the ultimate strength of a connector cup solder joint were enough to cause short-term joint failures under sterilization conditions. However these same joints would withstand loads of 50%-70% of the ultimate strength for the full i08 hours at room temperature conditions. It is therefore recommended that solder joints be under little or no stress during the thermal sterilization treatment.

/

ACKNOWLEDGMENT

This investigator wishes to express his appreciation to the following people for their valuable assistance in the *various* phases of this program:

Mr. D. Teter for soldering all of the solder joints.

 \sim . The set of the s

- Miss E. Manning for welding all of the weld joints and for ultimate strength testing of all weld joints.
- Mr. J. Holberton for ultimate strength testing of all solder joints and for assistance in stress-rupture testing.
- Mr. D. *Cranmer* for electrical resistance testing of all solder joints and weld joints.
- Mr. R. Kassebaum for *vibration* testing of all solder joints and all weld joints.
- Mr. N. Ferguson for all metallographic mount preparation and phctomicrographs.
- Mr. R. Rydelek for conducting all thermal sterilization and lead bake-out tests.
- Mr. D. Hirsch for the design and construction of the various special test fixtures required for ultimate strength testing and stress-rupture strength testing.
- Mr. *C.* Bahun for statistical analysis of variance calculations.
- Mr. G. Dreyer for assistance in weld schedule determination.
- Mr. J. Fraser and Mr. R. Bays for the design of the electrical resistance test fixtures and for assistance in conducting the vibration and stress-rupture tests.

FINAL REPORT - PART II Contract No. 951069

DETERMINATION OF THE EFFECTS OF A THERMAL STERILIZATION PROCESS ON THE MECHANICAL AND ELECTRICAL PROPERTIES OF SOLDERED AND WELDED JOINTS

by

r. Z. Keister, Hughes Project Engin 9 October 1965

For

Jet Propulsion Laboratory California Institute of Technology Pasadena, *California*

Approved:

Smith, Manager Materials Technology Department

Materials Technology Department AEROSPACE GROUP Hughes Aircraft *Company* • *Culver City, California*

CONTENTS

Introduction to Part **II**

Attachment 1. Weld Joint Schedule Isoforce Diagr

Attachment 2. Raw Data She

Attachment 3. Photomicrographs of Metallog ${\tt Specir}$

Attachment 4. Electron Probe Microanalysis Cha for Soldered and Welded Joi

INTRODUCTION TO PART II

Part II of the final report on the determination of the effects of a thermal sterilization process on the mechanical and electrical properties of soldered and welded joints is intended to supplement Part I. Part II contains four basic sections identified as follows:

Attachment 1 - Weld Joint Schedule Isoforce Diagrams

Attachment 2 - Raw Data Sheets (Pages 1 - 35)

Attachment 3 - Photomicrographs of Solder Joint and Weld Joint Metallographic Specimens (Figures I - 48}

Attachment 4 - Electron Probe Microanalysis Charts (Pages 1 - 24} Attachment 1 is intended for use with Section 3.5 "Fabrication of Weld Joi 's" in Part I.

At.achment 2 is intended for use with Section 5 "Test Results" in **Part I.**

Attachment 3 is intended for use with Section 5.6 "Metallurgical Examinatio., Test Results" in Part I.

Attacl.-]ent 4 is intended for use with Section 5.7 "Electron **Probe** Microanalysls Test Results" in Part I.

 \mathfrak{d}_{i}

ATTACHMENT 1

WELD **JOINT SCHEDULE** ISOFORCE **DIAGRAMS**

 $\frac{1}{4}$ í

 $\boldsymbol{\gamma}'$

 $\sum_{i=1}^n$

 $\begin{array}{c} \n\mathbf{1} \\
\mathbf{2} \\
\mathbf{3}\n\end{array}$

Å
ATTACHMENT **2** RAW DATA SHEETS

- 1. Ultimate Strength Before and After Thermal Sterilization
	- a) Solder Joints: Pages 1 6
	- b) Weld Joints: Pages 7 10
- 2. Electrical Resistance Before and After Thermal Sterilization
	- a) Solder Joints: Pages 11 15
	- b) Weld Joints: Pages 16- 19
- 3. Stress Rupture Strength of Connector Cup Solder Joints
	- a) Room Temperature: Pages 20 21
	- b) Sterilization Temperature: Pages 22 23
- 4. Electrical Test During Vibration Before and After Thermal Ste rilization
	- a) Solder Joints: Pages 24 27
	- b} Weld Joints: Pages 28 31
- 5. Ultimate Strength After Vibration Before and After Thermal Sterilization
	- a) Solder Joints: Pages 32 33
	- b) Weld Joints: Pages 34 35

0

Page 1

PROJECT ENGR.

F.KSISTIR

STEANDED COUDICTOR TO SOLDER CONTED PUBLICATED TERMINAL (1.2) TYPE OF JOINT TESTED BY JOHN HOLSESTON

WEWIES PAILLE J-ION PALULE

ALL VALUES IN POUNDS

PROJECT ENGR. **F.KEISTER**

Page 2

ř.

F.KEISTER PROJECT ENGR.

 μ_{t}

Page 3

PROJECT ENGR. F.KEISTER

Page 4

 $P_{20}e5$

F. KEISTER

 $\langle \mathcal{I}_\lambda \rangle$

PROJECT ENGR.

 $\frac{1}{2}$

controls in the proposed of the company of the control of

I

PROJECT ENGR. F.KEISTER

 \bar{z}

a sa mga bayang pangalang ng pan
Mga pangalang ng pa

a mata ya Kingi

 $\mathcal{A}^{\mathcal{A}}$

— *—* — —

 ϵ and ϵ . ϵ

ĺ

 $\ddot{}$

PROJECT ENGR. F.KEISTER

t

 \sim

 $\frac{1}{4}$

l,

PROJECT ENGR. **F.KEISTER**

 $\sqrt{6}$

JPL CONTRACT 951069 Page 9 TEST WLIIMATE STRENGTH - WELD JOINTS Gold plated copper to Inco (1.5A) and Solder coated copper to Inco (66A) TYPE OF JOINT DATE $x - 3 - 45$ TESTED BY E. Manning W2 = Wire broke adjacent to weld. $ws = \text{weld separation}$ سنتهيم بالمراج 8-24-65 - 7 7, **STRENGTH** $T^{\prime\prime}$ $\frac{1}{2}$ JOINT VO. 5171467 " JOINT NO. STRENGTH JOINT NO. (AFTER HEAT) (Lefore heat) Chefre hosts $(fFIRHH)$ 785 in peurde. بمن - 4 <u> 1.6 A</u> $1.5A$ **C** حفصيع $\ddot{}$ ∠ 8. $\overline{\mathscr{Y}'}$ W2 $\overline{\mathcal{L}}$ $h\!\!\!/\mathcal{S}$ 12.0 W2 \mathscr{Q} W \mathbf{I} \mathbf{r} $\overline{\mathscr{D}}$ $\overline{2}$ -2 ፕ **WS** $\overline{\mathbf{2}}$ <u>W2</u> $\bar{\mathscr{D}}$ $\overline{\mathscr{L}}$ $h/2$ \mathcal{R} 10.0 2 غىنا 20 $\overline{}$. The $\overline{}$ w. <u>WS</u> $\overline{\mathcal{L}}$ $\overline{25}$ 3 3 10.0 W2 \angle 112 $\overline{25}$ WS $\overline{\mathbb{R}^2}$ $\frac{\partial f}{\partial \Omega}$ \mathcal{R} $2M$ 4 -74 4 \mathcal{R} $l\gamma$ $\sqrt{ }$ <u>W2</u>

5

 $W³$

 $Z\hat{z}$

 $\overline{^{7/5}}$

46

╱

 τ

 λ

W2

D

 100 W₂

7. 5

 $7₅$

<u>WS</u>

 M^S

 $\mathbf i$

73

ی کر

 $2M$

 $2M$

Z

 $\overline{\mathcal{Z}}$

L

 \mathcal{R}^{\parallel}

F.KEISTER PROJECT ENGR.

 Δ

 \sim .

PROSECT ENGR.

F.KEISTER

フ

PROJECT ENGR,

F.KEISTER

Page 11

 \mathcal{L} 5

 $\mathcal{L}_{\mathcal{L}}$

Ķ $\frac{1}{2}$ ີ້.
ອ້າ

 \mathcal{Y}_0

Hewlet Packard Notel 435A **BC** Hicro WH-Ammelte $B = 13$ $5e$ _{vil} 344-01666 colib. Grift-of

Daystrom Holel 931 b.c. Hillianneler

TEST <u>Electrical</u> Resistance - Solder Joints TYPE OF JOINT Suder cooked Cu to solder coated terminal (1.3.2) by strong Model 131 b.c. Millionweb.
TESTED BY D. Cranmer Kepco hold SH-36-5N Voltage Republied B.C. Power Supply After Before Service C-28791 $After$ Refore heating heating heating heating Resistance J *oint No* Resistance Resistance Resistance J _{oi} $n + No$ -3 <u>ohm × 10</u> $ohm X10$ $1.3.3$ $ohm X10$ $1.3.2$ $ohmX10$ $.74$ 78 41 235 235 <u>41</u> .70 70 42 21 22 42 68 70 43 $.235$ 43 $.23$ 66 68 44 24 23 44 70 20 45 $20\bar{5}$ 45 76 $76'$ 46 215 46 $20₁$ $7l$ $74\,$ 47 <u> 21^{5} </u> 215 47 $\overline{2}$ ماما 49 225 225 48 <u>74</u> $72 4.9$ 23 $2/$ 49

 50

 51

52

 53

 54

 55

56

 52

 58

 59

 $\overline{6}$

ا ما

 62

 $\overline{43}$

 64

 65

<u> 46</u>

 $\sqrt{2}$

 68

 69

 70

 $7/$

 72

 73

 74

 75

 76

77

 78

 29

 $8Q$

 22

 $.24$

 $2^{\frac{1}{5}}$

 23

 235

 25

 235

 25

 245

 26

 74

<u>کرم</u>

 765

20

 235

 215

 215

 27

 24

ىۋە.

 22

 23

21

 74

22

 23

 $.24$

 24

 22

<u> 225</u>

<u>ذ 15</u>

 $2l$

 23

 23

 -23

 22

 $.245$

 235

 26

335

 24

 215

<u> محمد</u>

 $.22$

 12

 21

 $.20$

 22

 22

 $2l$

235

. 21 5

 $2\overline{O}$

 $\overline{22}$

79

 205

 $2²$

 $\overline{24}$

 $.95$

 215

50

 51

52

 53

 54

55

 56

 52

 58

 59

ں ما

<u>61</u>

 42

 $\overline{63}$

 64

 65

<u> 46</u>

 67

 68

 69

 $7\circ$

 $7/$

72

 73

 $\overline{74}$

 75

 76

77

<u>78</u>

 79

80 $\ddot{}$

 $\begin{tabular}{|c|c|c|c|} \hline \rule{0pt}{3ex} \rule{$

 $7 - 14 - 65$ \sim $-$

 $73.$

 84

 72

 $8₄$

 \mathcal{I} \mathcal{I}

 $7L$

 81

 68

 74

<u>7/</u>

 $\overline{27}$

76

 78

 74

76

 78

 25

 $.74$

 $2\overline{6}$

 73

<u>76</u>

70

 $\overline{2}\overline{2}$

<u>74</u>

<u> 77</u>

 $\overline{2}\overline{2}$

76

 74

76

 $.80$

 $\overline{27}$

66

91

80

 89

 $\overline{\mathcal{L}}$

<u>71</u>

72

عاها

<u>73</u>

 73

70

70

 $\overline{69}$

 74

<u>72</u>

 $7₂$

 82

 84

 70

 $7⁵$

<u>7/</u>

 74

<u> 27</u>

 60

<u>2.Z</u>

<u>74</u>

 76

<u>71</u>

 $\overline{73}$

 82

 $7\,\prime$

WALLET ENGR. F.KEISTER

 ρ

 $7 - 14$

F.KEISTER

Page 14

γ

 $\mathcal{L}^{\text{max}}_{\text{max}}$

Ť.

 $7 - 14 - 65$

 \mathcal{F}_c

 $7 - 16 - 65$

 γ

Pone 15

PROJECT ENGR, **F.KEISTER**

G.

 \bar{z}

 $\tilde{\star}$

 \mathbf{I}

 $\frac{1}{2}$

 $\begin{array}{c}\n\mathbf{r} \\
\mathbf{t} \\
\mathbf{t} \\
\mathbf{t}\n\end{array}$

 \cdot

> $\ddot{\cdot}$ $\ddot{}$ \ddot{i}

 $\frac{1}{\frac{1}{2}}$ and $\frac{1}{2}$ are $\frac{1}{2}$ and $\frac{1}{2}$ are $\frac{1}{2}$ and $\frac{1}{2}$ and $\frac{1}{2}$ are $\frac{1}{2}$ and $\frac{1}{$

 $\sum_{\substack{p \in \mathcal{P} \\ p \in \mathcal{P}}} \frac{1}{p} \sum_{\substack{p \in \mathcal{P} \\ p \in \math$

المستخدم ال
المستخدم المستخدم ال

 $\sigma_{\rm c} \propto \omega^2$

 $\mathcal{O}^{\frac{1}{2}}$

 \mathcal{J}

k.

میں
کیسیم PROJECT ENGR. **F.KEISTER**

 \sim 1

Ż

PROJECT ENGR. <u>F.KEISTER</u>

ጥ
ነር የ

财产

STRESS-RUPTURE @ man temperature (140F) TEST TYPE OF JULIT Stranded conductor to BENDIX connector CUP DATE TESTED CY J. Holberton

PINAL TOTAL TINE CREEP INITIAL SPECINEN WEIGHT Remorks TINÊ TIME **COBSERVED ?** ٧ **NUMBER** $(\tau_{\epsilon} - \tau_i)$ (pounds) (τ_{ϵ}) (T_i) hours 1.1.2 $\boldsymbol{\hat{z}}$ \sqrt{W} $\overline{\mathsf{N}}$ 630.6 No Farver 518.9 111.1002 \mathbf{W} STARTED No FAILVEE 630.6 No \mathfrak{m} \mathfrak{n} 112 311.202 **518 A** 11.7 485.6 Ŋе No FAILULE 373.9 113 4 the than Ŋр No FAILLEL 630.1 $\mathbf{111.7}$ ¥ 61.202 518.4 114 $\mathsf{r}.m$) oll No Farult 630.4 TEST
S:30 $\overline{115}$ $7 - h$ bes 51 5.7 \mathcal{N}° 267.2 N_{b} \neq N_{c} ⊥ய⊐ 916.202 155.5 116 Þ \mathcal{M}^{ν} 1259.7 $\pm u\alpha$ No FAWLE $10 \text{ L} \text{ S}$ 148.0 $\overline{11}$ **JOINT FAILVER** $\overline{21.}$ 63.0 ÷ 42.0 118 12 H Lat. Wire Pailore ! **NO** 0.5 0.1 $13 - 11$ $9₀₁$ 0.4 119 *ING FAILIRE* 124.7 7553 **No** 116.10 oz 630.6 120 129.7 σ U i No FAILLE 755.3 Ä. 316.20216306 $|2|$ 124.7 No FAILUIS \mathbf{f} 60.3 $\mu_{\mathbf{z}}$ 4856 (416.10 o2) 122 NO FAILLE 129.7 No 1.0301 945 1 616.202 123 No FAILRE 724.7 N. 716.1001 755.1 630.4 124 No FAILVE 24.7 N, 391.9 916 202 2672 125 No Ferces $\sqrt{24.7}$ h• 384.4 259.7 $1016.802.1$ $12L$ WIRE FAILVER $\overline{\mathcal{N}_{\Omega}}$ $\overline{\mathfrak{o}}$. 63.1 63.0 $9L$ 1261 127 WILE EARNE 0.1 ما \mathbf{v}_α $, 5$ $1516.992.$ 12^c No FOILLER $83/3$ 1360 \mathcal{U} 116 1002 1553 124 μ^{o} NOFAILVEE $1891,2$ 135.9 316 202 1755.3 130 **NO FEMILIANS** 360 ₩ 746.3 4161002615.3 131 **IS FAILVEE** $\overline{\mathscr{C}}$ 155.9 V^{σ} <u> 202 754.8</u> d/Δ 132 $\overline{(\cdot)}$, τ 18910 $135,9$ 1551 LU フト 133 910 201 891.9 $135,9$ 527.8 μ o **NO PERSONAL** 134 \mathbb{N} N FALUES 10152 and 38414 520.2 1358 135 Is Fried $\sqrt{2}$ $|oz|$ $|o3|$ 1132.2 4151 126 USWE FAILW ω ⊝.೧ $|S|$ \ge S_{cs} $|S|$ σ $\sqrt{ }$ 137 M_0 Fairvers 135.7 Йs $1610e100$ 135.7 $\overline{13}$ 135.7 135.7 \vee N_{ω} = + 16026 316202100 139 Now to encor 557 μ 735.7 4161002100 140 ł 125.7 **VIL FAILURE** 135.7 10.5 202 0.0 141 Daniel E W -5.7 $\sqrt{25}$ N 17160230 192 *N FALAS* $\overline{\mu}$ 135.7 $35.$ 91620200 143 $\mathcal{O}(\mathcal{O})$ $\mathcal{N}^{\mathcal{O}}$ $\sqrt{35.7}$ $\sqrt{2}$ $\overline{\mathcal{O}}$ \overline{a} $\Delta = 12$ 144 $W \subset \mathbb{R}$ y P ∴.Ì $1214.352.00$ 21.5 21.5 145 JUST TELVIE \mathcal{N} Ò. $\frac{13k}{13k}$ 202 00 -1 \circ . 146 ł

 $\frac{1}{2}$

PROJECT ENGR.

EXCISTER

id, Jenit S

PROJECT EMGR.

 $\overline{}$

 \sim

 \sim $\bar{\beta}$ $\frac{1}{2}$

 $\frac{1}{2}$

F.KEISTER $-$ 30 $\frac{x}{4}$

 $\tilde{\mathcal{N}}$

TEST STRESS-RUPTURE @ Sterilization Time & Temperature Stranded conductor to TYPE OF JOINT

TESTED DY 7.8Cml

b

j.

y

 $DATE = 1.2 - 4.5$ $+L$

 $9 - 13 - 6$

 P_{age} 23

OIMPEN 705MIG

F.KEISTER

₹

 $\mathcal{L}^{\mathcal{L}}$

 \mathcal{A}

ļ

Iŧ

PROJECT ENGR.

 $\sim 10^{-1}$

 \sim \sim

 $\overline{1}$

F.KEISTER

eneg# $\overline{}$ $\mathcal{L}_{\mathcal{A}}$

 $\frac{1}{\sqrt{2}}$

error in

√.

PROJECT ENGR. F.KEISTER

133

PROJECT ENGR. F.KEISTER

 λ

المالية المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات الم
المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات المستشربات

 \mathbf{I}

 $\ddot{\mathbf{z}}$

PROJECT ENGR.

 ϕ

 ϵ

ERSISTER ansa. PRODECT

Page 29

 $P₂₅$ 30

EKTISTER <u>po</u> aman. ここにくて

TEST Glectrical Test During Vibratuin-Weld Joints Weld Joint - Solder coated supper to Inco (1.6) TYPE OF COLLY and Kover fill to Kover full (2.) ザックダ \mathcal{K} TESTED BY $\overline{\mathcal{L}}$. The contract of $\overline{\mathcal{L}}$

No heat STERIUSED SPECIMEN SPECIMEN \overline{J} 0/NT % σ J WIOU **TO CHANGE IN** RESISTANCE RESISTANCE $\mathcal{L}% _{M_{1},M_{2}}^{\alpha,\beta}(\varepsilon)=\mathcal{L}_{M_{1},M_{2}}^{\alpha,\beta}(\varepsilon)$ **NUMBER** CHANGE $#$ VISEATION $101B$ $1.6.918$ ما29 200 \mathcal{O} $00028 102R$ $1.6,928$ \rightarrow $103B$ $1.6.938$ 10 Y B 1.6.94B 105870001460 1.6.958 000581 $106B$ ∕∂ $1.6.90B$ 107 \mathcal{B} $1.4.97B$ 100028 D l B σ $1.6.98B$ $109R$ OLC I 4 L6.99B $\sqrt{}$ $40B$ $1.6.100B$ $\sqrt{\rho_{L}}$ \overline{z} Γ $1.6.19L3$ $JPLI$ 00024 -j(L_2 20 $\hat{\mathcal{Q}}$ LG JPLY $TPL9$ - 5 $1.6.0925$ $TPL10$ J. $1.6.0$ PL6 RESONANT FREQUENCY : 478 EAS ŧ STERILIZED NO HEAT ' Y CHANGE IN **SPECINEN** $308M$ \overline{M} **OCO/ELITAGE**

 \mathcal{X} . $\frac{1}{2}$

 $\frac{1}{2}$.

 \mathcal{L}^{max}

FIREISTER PROJECT ENGR.

Page 31

FENDIN CUP

Standed conductor

PROJECT ENGR.

F.KSISTER

Page 32

TEST WITHATE STRENGTH PIETER VIREATION

TYPE OF JOINT SOLDER JOINTS - VARIOUS THES

DATE Sept 7,1965

TESTED DY JOHN C. HOLBERTON SENECH LAB AWALUST C RF

THE UPPE

PROJECT ENGA. F.KEISTER

 λ

 P_{PQ} e 34

PROJECT ENGR. **F.KEISTER**

 $\frac{1}{2}$

 $\frac{1}{2}$

 \cdot [']

ì.

Susierse _{. .} <u>PAOJECT ENGR.</u>

N
ATTACHMENT 3

PHOTOMICROGRAPHS OF METALLOGRAPHIC SPECIMENS

I. Solder Joints: Figures I **-** ZZ 2. Weld Joints: Figures 25 **-** 48

 M^{λ}

Figure **1.** Solder joint **1. 1. 1. 83** stranded conductor to cinch connector cup; not sterilized; **60x** magnification; potassium dichromate etch **(02344).**

Figure **2.** Solder joint **1. 1. 1.89** stranded conductor to cinch connector cup; sterilized; **60x** magnification; potassium dichromate etch **(02345).**

Figure 3. Solder joint **1.** 1. 2.83; stranded conductor to Bendix connector cup; not sterilized; 60x magnification; potassium dichromate etch (02346).

Figure **4.** Solder joint 1. 1. 2. 88; stranded conductor to Bendix connector cup; sterilized; 60x magnification; potassium dichromate etch (02347).

Figure 5. Solder joint 1.2.82; stranded conductor to solder coated bifurcated terminal; not sterilized; 100x magnification; potassium dichromate etch (02348).

Solder joint 1.2.87; stranded conductor to solder Figure 6. coated bifurcated terminal; sterilized; 100x magnification; potassium dichromate etch (02349).

Solder joint 1.3.1.84; gold plated copper conductor to Figure 7. solder coated bifurcated terminal; not sterilized; 100x magnification; potassium dichromate etch (02350).

Solder joint 1.3.1.86; gold plated copper conductor to Figure 8. solder coated bifurcated terminal; sterilized; 100x magnification; potassium dichromate etch (02351).

Figure 9. Solder joint **1.3.** 2.81; solder coated copper conductor to solder coated bifurcated terminal; not sterilized; lOOx magnification; potassium dichromate etch (02352).

Figure 10. Solder joint **1.3.** 2. **87;** solder coated conductor to solder coated bifurcated terminal; sterilized; 1 **OOx** magnification; potassium dichromate etch (02353).

Figure 11. Solder joint 1.3. 3. 83; gold plated Dumet conductor to solder coated bifurcated terminal; not sterilized; 100x magnification; potassium dichromate etch (02354).

Figure 12. Solder joint 1.3.3.86; gold plated Dumet conductor to solder coated bifurcated terminal; sterilized; 100x magnification; potassium dichromate etch (02355).

Figure 13. Solder joint **1.3.4.83;** gold plated Kovar conductor to solder coated bifurcated terminal; not sterilized; lOOx magnification; potassium dichromate etch (02356).

Figure 14. Solder joint 1.3. 4. 89; gold plated Kovar conductor to solder coated bifurcated terminal; sterilized; 100 magnification; potassium dichromate etch (02357).

Figure 15. Solder joint 1.3. 5.84; gold plated nickel conductor to solder coated bifurcated terminal; not sterilized; 100x magnification; potassium dichromate etch (02358).

Figure 16. Solder joint 1.3. 5.88; gold plated nickel conductor to solder coated bifurcated terminal: sterilized 100x magnification; potassium dichromate etch (02359).

Figure 17. Solder joint 1. 3.6. **84;** bare nickel conductor to solder coated bifurcated terminal; not sterilized; 1 **OOx** magnification; potassium dichromate etch (02360).

Figure 18. Solder joint 1. 3.6. 87; bare nickel conductor to solder coated bifurcated terminal; sterilized; 1 **OOx** magnification; potassium dichromate etch (02361).

Figure 19. Solder joint 1.4. 83; gold plated copper conductor to gold plated bifurcated terminal; not sterilized; 1 **OOx** magnification; potas sium dichromate etch (02362).

Figure 20. Solder joint 1.4.90; gold plated copper conductor to gold plated bifurcated terminal; sterilized; 1 **OOx** magnification; potassium dichromate etch (02363).

L. .-

Figure 21. Solder joint 1.5. 85; resistor lead to solder coated bifurcated terminal; not sterilized; 1 **OOx** magnification; potassium dichromate etch (02364).

Figure 22. Solder joint 1.5. 89; resistor lead to solder coated bifurcated terminal; sterilized; 100x magnification; potassium dichromate etch (02365).

 $\mathcal{L}^{\mathcal{A}}$

Figure **23.** Weld joint 1. 1. 81; bare nickel wire to Inco ribbon; not sterilized; 150x magnification; nitric-acetic etch (02316)

Figure **24.** Weld joint 1. 1.86; bare nickel wire to Inco ribbon; sterilized; 150x magnification; nitric-acetic etch *(023* 19).

Figure 25. Weld joint 1. 1. JPL 1; bare'nickel wire to Inco ribbon; not sterilized; 150x magnification; nitric-acetic etch (02317).

Figure 26. Weld joint 1. 1. JPL 2; bare nickel wire to Inco ribbon; sterilized; 150x magnification; nitric-acetic etch **(023** 18).

Figure 27. Weld joint 1.2. 81; gold plated nickel wire to Inco ribbon; not sterilized; 150x magnification; nitric-acetic etch (02320).

Figure 28. Weld joint 1. 2. 87; gold plated nickel wire to Inco ribbon; sterilized; 150x magnification; nitric-acetic etch (02323).

Figure 29. Weld joint 1. 2. JPL 1; gold plated nickel wire to Inco ribbon; not sterilized; **150x** magnification; nitric-acetic etch (02321).

Figure 30. Weld joint 1.2. JPL 2; gold plated nickel wire to Inco ribbon; sterilized; **150x** magnification; nitric-acetic etch (02322).

Figure **3** 1. Weld joint 1. 3. 85; gold plated Kovar wire to Inco ribbon; not sterilized; 150x magnification; Carapella **t** nitric-acetic etch **(02324).**

Figure **32.** Weld joint 1. 3.86; gold plated Kovar wire to Inco ribbon; sterilized; 150x magnification; Carapella **t** nitric-acetic etch **(02324).**

0

e

Figure 33. Weld joint 1.3. JPL 2; gold plated Kovar wire to Inco ribbon; not sterilized; 150x magnification; Carapella + nitric-acetic etch (02325).

Figure **34.** Weld joint 1. **3.** JPL 2; gold plated Kovar wire to Inco ribbon; sterilized; 15 Ox magnification; Carapella **t** nitric-acetic etch (02326).

Figure 35. Weld joint 1.4.84; gold plated Dumet wire to Inco ribbon; not sterilized; 150x magnification; Carapella etch (02328).

Figure 36. Weld joint 1.4. 87; gold plated Dumet wire to Inco ribbon; sterilized; 150x magnification; Carapella etch (02331).

Figure 37. Weld joint 1.4. JPL 2; gold plated Dumet wire to Inco ribbon; not sterilized; 150x magnification; Carapella etch (02324).

Figure 38. Weld.joint 1.4. JPL 2; gold plated Dumet wire to **Inco** ribbon; sterilized; 150x magnification; Carapella etch (02330).

Figure **39.** Weld joint 1.5.81; gold plated copper wire to Inco ribbon; not sterilized; **150x** magnification; nitric-acetic etch **(02332).**

Figure 40. Weld joint 1.5. 88; gold plated copper wire to Inco 'ribbon; sterilized; **150x** magnification; nitric-acetic etch (02335).

, Figure 41. Weld joint 1.5. **JPL** 1; gold plated copper wire to Inco ribbon; not sterilized; **150x** magnification; nitric-acetic etch (02333).

Figure 42. Weld joint 1.5. JPL 2; gold plated copper wire to Inco ribbon; sterilized; 150x magnification; nitric-acetic etch (02 334).

Figure 43. Weld joint 1.6.82; solder coated copper wire to Inco ribbon; not sterilized; 150x magnification; nitric-acetic etch (02336).

0

Figure **44.** Weld joint 1.6.90; solder coated copper wire to Inco ribbon; sterilized; 150x magnification; nitric-acetic etch (02339).

Weld joint 1.6. JPL 1; solder coated copper wire to Inco
ribbon; not sterilized; 150x magnification; nitric-acetic Figure 45. etch (02337).

ribbon; sterilized; **150x** magnification; nitric-acetic etch (02338).

0'

Figure 47. Weld joint **2.** 83; gold plated Kovar foil to itself; not sterilized; 150x magnification; Carapella etch (02340).

Figure 48. Weld joint 2.86; gold plated Kovar foil to itself; sterilized; **150x** magnification; Carapella etch **(02343).**

ATTACHMENT 4

ELECTRON PROBE MICROANALISIS CHARTS FOR SOLDERED AND WELDED JOINTS

 $\gamma\phi$

 γ

N ż,

 $, 1$

ήÕ

 $\hat{\Psi}_n$

 γ

مای

 $\ddot{}$

 $\sum_{i=1}^{n}$

 $\hat{\delta}^{(n)}$ \mathbf{x}

4 Y

REPORT NO. P65-117

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Accenautics and Space Administration under Contract NAS7-100.

FINAL REPORT - PART III (Contract No. 951069)

DETERMINATION OF THE EFFECTS OF THERMAL BAKE-OUT, HEAT STERILIZATION, AND ETHYLENE OXIDE DECONTAMINATION ON THE SOLDERABILITY OF **COMPONENT LEADS**

Prepared for: Jet Propulsion Laboratory California Institute of Technology Pasadena, California

9 OCTOBER 1965

HUGHES HUGHES AIRCRAFT COMPANY

AEROSPACE GROUP

CULVER CITY, CALIFORNIA

FINAL REPORT **-** PART **III Contract** No. **951069**

DETERMINATION OF THE EFFECTS OF THERMAL BAKE-OUT, HEAT STERILIZATION, AND ETHYLENE OXIDE DECONTAMINATION ON THE SOLDERABILITY OF COMPONENT LEADS

by

F. Z. Keister, Hughes Project Engin **9** October 1965

For

Jet Propulsion Laboratory California Institute of Technology Pasadena, California

This work was performed for the Jet Propulsion Laboratory, California Institute of Technology, sponsored by the National Aeronautics and Space Administration under **Contract** NAS7-100.

Approved:

Smith, Manager Materials Technology Department

Materials Technology Department AEROSPACE GROUP Hughes Aircraft Company · Culver City, California

ABSTRACT

The purpose of this investigation was to determine the **effects** of thermal bake-out, heat sterilization, and *ethylene* oxide decontamination on the solderability of component leads.

This program involved the preparation and testing of 320 *specimens,* of which 64 were metallographically examined and 256 were solderability tested. The specimens for this program included nine different **solid** and stranded wire conductor materials, two types of connector cups, and two types of bifurcated terminals. Specimens were solderability tested (by dipping in a solder pot) and examined metallographicalIy before and **after** three treatments: I) thermal bake-out; **2)** heat sterilization; and 3) ethylene oxide decontamination. **Photomicrographs** at i000X magnification were taken of all specimen types before and after treatment. All solderability test results were analyzed statistically.

Based on the test results, it was *concluded* that thermal bake-out, heat sterilization, and ethylene oxide decontamination had very little effect on the solderability or metallurgical integrity of the **specimens** tested. Although a few specimen types did exhibit certain **effects** due to either thermal bake-out or heat sterilization, these **effects** were minimal and could be attributed to either unrelated causes or to factors which could be corrected in production usage.

 $-\alpha$

CONTENTS

Page

V

ILLUSTRATIONS

to k

vii

 $\frac{1}{2}$

Figure

ľ

 δ_1

TABLES

ix

I. PURPOSE AND SCOPE

1. 1 PURPOSE

The purpose of this investigation was to determine the effects of thermal bake-out, heat sterilization, and ethylene oxide decontamination on the solderability of component leads. The primary purpose of this contract (refer to Parts I and II) was to determine the effects of a thermal sterilization process on the mechanical and electrical properties of soldered and welded joints.

This work was done for the Jet Propulsion Laboratory under Contract No. 951069 (subcontract under NASA *Contract* NAS 7-100). Mr. A. G. Fitak was the JPL Project Engineer on this program. During the final stages of this program, Mr. R. F. Holtze became the new JPL **Project** Engineer on this program, since Mr. Fitak was transferred to other duties.

This final report has been divided into three **separate parts.** Part I covers the thermal sterilization effects on solder **and** weld joints. **Part** II contains the weld schedule isoforce diagrams, raw data sheets, photomicrographs, and electron probe microanalysis charts for the solder and weld joints under Part I. Part *iiI,* which is this part, covers the solderability studies and is complete in itself with all its own data sheets and photomicrographs included under the same C **ore** r.

I. Z SCOPE

Part HI of this final report is a determination of the effects of thermal bake-out, heat sterilization, and ethylene oxide decontamination on the solderability of component leads. This portion of the program involved preparing and testing 320 specimens of which 64 were metallographically examined and 256 were solderability tested. The specimens for this program included 9 different conductor materials, **2** types of connector cups, and **2types** of bifurcated terminals. Specimens were solderability tested (by dipping in a solder pot) and examined

 \mathbf{I}

 $\lambda_{\rm G} \lambda$

before and after three treatments: 1) thermal bake-out; **2)** heat **ster**ilization; and 3) ethylene oxide decontamination. Thirty-six photomicrographs were taken (32 of them at 1000 x magnification) of typical specimens before and after treatment. The test results were analyzed statistically. Photographs were taken of typical specimens of **each** type after being solder dipped and before and after thermal bake-out, heat sterilization, and ETO decontamination. Photographs were also taken of each type of test equipment used.

 $\sum_{k=1}^{n}$

2. MATERIALS AND EQUIPMENT

2. 1 MATERIALS FOR SOLDERABILITY SPECIMENS

The materials used in this program for the solderability specimens are listed in Table I. These materials (i.e., conductors, terminals, and connector cups) are identical to those used in Part I of this program, except for two new materials - items 12 and 13. Both of these conductors had a nickel underplating beneath the outer gold plating. Metallographic and spectrographic examination of these two new materials showed them to be as ordered with the thickness of the nickel strike being 80-92 microinches and the thickness of the gold plating being 100-112 microinches.

2. 2 MATERIALS FOR FLUXING AND SOLDER DIPPING SPECIMENS

- 2.2.1 Alpha No. 611 activated rosin flux per MIL-F-14256C, Type A. This flux was used for fluxing all gold plated and solder coated solid wire conductors, connector cups, and bifurcated terminals.
- 2.2.2 Alpha No. I00 unactivated rosin flux per MIL-F-14256C, Type W. This flux was used for fluxing stranded conductors.
- 2.2.3 Alpha No. 90 stainless steel flux. This flux was used for fluxing bare nickel solid wire conductors.
- 2.2.4 Kester bar solder per QQ-S-571d, Type Sn 63-B-S. This solder (63% tin, 37% lead) was used in the dip solder pot.
- 2.2.5 I, I, 1 Trichloroethane per Federal Specification O-T-620. This solvent was used to remove flux residues from all specimens fluxed with Alpha No. 100 and Alpha No. 611 flux. Water was used to remove residues of Alpha No. 90 flux.

3

Table I. List of materials.

 $\dot{\mathbf{o}}$

2. 3 MATERIALS FOR PREPARING METALLOGRAPHIC **MOUNTS**

- 2. 3. I Precision Scientific "Precisionite" mounting **powder for** all hot mounts.
- 2. 3.2 Fulton Metallurgical "Quickmount" mounting powder for all **cold** mounts.

2.4 EQUIPMENT

- 2.4.1 Dee Melting Pot, Model 15. Used for solder dipping test
- 2.4.2 Leitz Wetzlar (German) binocular microscope, Serial N 505106 with an American Optical Illuminator, Model 11144. Used for visual examination of solder dipped specimens **at** 30x magnification.
- 2.4.3 National Appliance Co. Vacuum Oven, Model 58402, **Serial** No. B59. Used for bake-out of all specimens at 200°C in **a** nitrogen atmosphere and also used for dry heat sterilization of all specimens.
- 2.4.4 Hughes Aircraft Company specially-designed antechamber and apparatus for exposure of specimens to ethylene oxide dec ontamination.
- 2.4.5 Zeiss Inverted Metallurgical Microscope, Model No. B-5 and Reichert Metallograph, Model MeF. Used for examining metallographic mounts at 500x to 1000x magnifications and also used for taking photomicrographs since the Reichert Metallograph is equipped with a built-in camera. Photomicrographs were taken with Kodak Super Panchro Press film.
- 2.4.6 Buehler Model 67-1509 Vibromet Polisher and Buehler Model 1330AB Mounting Press. Used for preparing metallographic specimens.

5

% */*

3. TEST PROCEDURES

3. I GENERAL TEST INFORMATION

Figure 1 **outlines** the test program plan for **the** seven (7) **wire** types (i.e., A, B, C, D, E, F, G) listed in (ii) of the Statement of Work. The Statement of Work is included as Table II. Twenty (20) specimens of each of these seven (7) wire types were prepared. Eight (8) each were solderability tested before and after bake-out and two (2) each were metallographically examined before and after bake-out. Thus a total number of 140 specimens were required, of which 28 were metallographically examined and I12 were solderability tested.

Figure 2 outlines the test program plan for the six (6) specimen types (i.e., A, B, C, D, E, F) listed in (iii) and (iv) of the Statement of Work (Table II). Thirty (30) specimens each of types A, B, C, D, E, and F were prepared. Eight (8) each were solderability tested before sterilization and two (2) each were metallographically examined before sterilization. Eight (8) each were solderability tested after ethylene oxide decontamination and two (2) each were metallographically examined after ethylene oxide decontamination. Eight (8) each were solderability tested after heat sterilization and two (Z) each were metallographically examined after heat sterilization. Thus a total number of 180 specimens were required of which 36 were metallographically examined and 144 were solderability tested.

In summation the total program involved 320 specimens of which 64 were metallographically examined and 256 were solderability tested.

3.2 BAKE-OUT TEST PROCEDURES

In accordance with the Test Program Plan (Figure I) and the Statement of Work (Table II), bake-out tests of all (ii) specimens (i.e., A, B, C, D, E, F, G) were done ina vacuum oven and consisted of exposure to 200°C for a period of 168 hours in an inert nitrogen atmosphere. Figure 10 in Part I of this final report shows a photograph of the vacuum oven used for bake-out tests.

 $\overline{7}$

فتتسبب

 ~ 10

 Ω ⁰

 $\sim 10^{-1}$

أمفهتم والقاليمان الداريد

Figure 1. Test program plan per (ii) wire specimens.

Figure 2. Test program plan per (iii) and (iv) specimens.

8

 $\hat{\boldsymbol{\beta}}$

 $\mathcal{L}_{\mathcal{A}}$

Table II. Statement of work for solderability test program.

 \mathcal{A} $\ddot{\Omega}$ $\mathcal{C}^{\mathcal{A}}$

3.3 DRY HEAT STERILIZATION TEST PROCEDURES

In accordance with the Test Program Plan **(Figure** 2) and the Statement of Work (Table II), heat sterilization of all (iii) specimens (i.e., A, B, C, D, E, F) was done in accordance with JPL Specification XSO-30275-TST-A "Compatibility Test for Planetary Dry Heat Sterilization Requirements." Heat sterilization consists of three 36 hour cycles at 145°C in a dry nitrogen environment. Dry heat sterilization testing is described completely in Section 4.2 in Part I of this final report. Figures 10 and Ii in Part I of this final report show views of the vacuum oven used for dry heat sterilization testing. In the photograph in Figure Ii can be seen certain of the solderability test specimens in Petri dishes within the vacuum oven on the lower shelf.

3.4 ETHYLENE OXIDE DECONTAMINATION TEST PR OCEDURES

In accordance with the Test Program Plan (Figure 2) and the Statement of Work (Table II), ethylene oxide decontamination of all (iv) specimens (i.e., A, B, C, D, E, F) was done in accordance with JPL Specification GMO-50198-ETS-A "Compatibility Tests for Ethylene Oxide Decontamination Requirements" dated 3 September 1964. Exposure is accomplished by subjecting specimens **to** 12_0 ethylene oxide - 88% Freon 12 gas mixture by weight at 35% relative humidity and 24[°]C for 24 hours, followed by a similar exposure at 40[°]C if specimens show no deterioration of properties. The concentration of the ethylene oxide in the decontamination chamber is 500 \pm 50 milligrams of ethylene oxide per liter of gaseous atmosphere. The gas composition and relative humidity were determined by gas chromatographic analysis. The test chamber is subjected to a vacuum to remove sterilant gas atmosphere within 30 minutes after the exposure period ends. Only specimens of good quality were selected for **testing** and all necessary steps were taken to assure maximum cleanliness of the samples prior to testing.

 10

Figure 3 is a photograph of the special antechamber used for ethylene oxide decontamination. This antechamber is capable of being evacuated or pressurized.

Figure 4 is a close-up photograph showing specimens A; B, C, D, E, and *F* in Petri dishes ready for ethylene oxide decontamination.

3. 5 SOLDERABILITY TEST **PROCEDURES**

In accordance with the Test Program Plan (Figures 1 and 2) and the Statement of Work (Table II), all specimens were solderability tested per MIL-STD-202C, Method 208A "Solderability" dated IZ September 1963. Essentially this test method consists of dipping a fluxed wire or other applicable specimen in a **pot** of molten **solder.** The dipped specimen is then cleaned and visually examined for **solder** coverage. Operator manual dipping was used, as opposed to machine dipping, in order to simulate more closely actual production conditions. One operator performed all dipping tests on all specimens. This was a deviation from MIL-STD-202C which calls out mechanical dipping. The temperature of the molten solder was maintained at 450° $\pm 10^{\circ}$ F. The composition of the solder was 63% tin - 37% lead. This was a deviation from MIL-STD-202C which calls out 60-40 **solder.** The fluxes used (see Paragraphs 2.2.1, 2.2.2, and 2.2.3 under the Materials section) for the solder dipping tests were mutually agreed upon between JPL and Hughes.

Following is a step-by-step procedure:

Step 1. Dip specimen in appropriate flux for 2-5 seconds.

- Step 2. Skim dross from top of molten solder and dip specimen in solder to a depth approximately one-half the specimen length. The immersion and emersion rates were approximately 1-2 inches per second and the dwell time in the solder bath was $5 \pm 1/2$ seconds for all specimens.
- Step 3. Allow the specimen to cool in air and then remove flux residues by immersion in the applicable solvent followed by gentle wiping with a clean cloth.

11

 \mathcal{V}_f

Figure 3. Ethylene oxide decontamination antechamber.
(R105390)

Figure 4. Specimen (iv) types **A,** B, C, D, E, and F in ethylene oxide decontamination antechamber. (R 105260)

- Step 4. Examine the specimen under 30x magnification to determine:
	- (a) That the specimen is 95-percent covered by a continuous new solder coating
	- (b) That pinholes or voids are not concentrated in one area and do not exceed 5-percent of the total area.

Figure 5 is a photograph showing the equipment used in the solderability tests.

All results of the specimen examinations were recorded on Raw Data Sheets in terms **of** percentage coverage **of the** specimen **by** ^a continuous new solder coating. All **examinations** of the **before** and after bake-out specimens were done by one operator and all examinations of the before and after heat sterilization and ETO decontamination specimens were done by one operator.

3. 6 METALLOGRAPHIC EXAMINATION TEST PROCEDURES

In accordance with the Test Program Plan (Figures I and 2) and with the Statement of Work (Table II), two each of all specimens before and after bake-out, before and after heat sterilization, and before and after ethylene oxide decontamination (ETO) were metallographically examined. A total of 64 specimens were mounted and examined. Specimens A, B, C, D, E, F, and G per Figure **1** were all hot mounted in Precisionite. Specimens A, B, C, D, E, and F per Figure Z were all cold mounted in Quickmount.

Thirty-two photomicrographs at 1000x magnification of typical specimens before and after bake-out, heat sterilization, and ETO decontamination were taken. In addition, four photomicrographs at 50x magnification were taken of a typical connector cup, typical bifurcated terminal, typical solid wire conductor, and typical stranded comductor specimen in the mounted condition.

All mounted specimens were examined **in** both the polished and etched conditions at magnifications from 500x to 1000x. Specimens were examined primarily for grain boundary diffusion of the plating

13

 $14\,$

alloy in the parent metal as well as any interdiffusion of the surface plating. By comparing treated and untreated specimens at high magnification, any degrading effects of the thermal and/or sterilization treatments on the electroplated or solder coated surfaces of the specimens should be revealed.

Metallographic mounts of all specimens were prepared by multiple mounting so that the "before" and "after" specimens could be compared side-by-side without the necessity of switching mounts. Figure 6 illustrates the various multiple mounting methods employed. Figure 6A shows the mounting method used for specimens A, B, C, D, E, F, and G which were all solid wire conductors untreated and baked-out. *Figure* 6B shows the mounting method used for specimens A, B, C, D, E, and F which were conductors, connector cups, and bifurcated terminals untreated, heat sterilized, and ETO decontaminated. Specimens A and D were in one mount, B and C in another mount, and E and F in the third mount since these pairs were similar types of specimens.

Figure 6A. Mounting configuration for specimen types A, B, F, and G before and after bake-out.

- Figure 6B. Mounting configuration for specimen types C and B before and after heat sterilization and ETO decontamination.
- Figure 6. Mounting configurations for treated and untreated specimens.
4. RESULTS

4. 1 SOLDERABILITY TEST RESULTS

Figure 7 is a photograph showing a typical example of each type **of** specimen before bake-out (on the left) and after bake-out (on the right). These specimens in Figure 7 have all been solder dipped. The solder dipped ends of the wires are towards the center **of** the photograph.

Table III is the Raw Data Sheet giving the test results for the solderability tests on the specimens before and after bake-out at 200°C for 168 hours in nitrogen.

Table IV presents the results of a statistical analysis **of** the data shown in Table III. The statistical analysis was done using the binomial distribution with an arc sine transformation of the percentage data. The gold plated Kovar C (ii) specimens showed a slight effect due to bake-out; however they still pass the requirements of MIL-STD-202C. The 95% confidence interval of the mean was 96.0% - 99.9%, the lower limit of which is greater than the 95% minimum solder coverage. The gold plated Dumet specimens F (ii) with the nickel undercoat failed to pass the solderability requirements of MIL-STD-202C after bake-out. In this case the 95% confidence interval of the mean was 92.3% - 97.4%. In other words we are 95% confident that the mean falls within this range. This same wire type, before bake-out, showed 100% solder coverage for all eight specimens examined.

Figure 8 is a photograph showing a typical example of each type of specimen as follows:

- a) Not **solder** dipped
- b) Solder dipped untreated
- c) Solder dipped after heat **sterilization**
- d) Solder dipped after ethylene oxide decontamination.

The solder dipped portions of the connector cups and bifurcated terminals point towards the right, while the solder dipped **portions of** the conductors point towards the center.

17

 \mathcal{N}

HUGHES AIRCRAFT CO.

FORM 703-C

 \sim \sim

 $\hat{\mathbf{v}}$

19

 $\mathbb{R}^{\frac{1}{2}^{\frac{1}{2}}}$

Table IV. Bake-out effects on solderability.

Tables V and VI are Raw Data Sheets giving the test results **for**the solderability tests on the specimens outlined in (iii) and (iv) of the Statement of Work (Table II) and shown in *Figure* 8. Note that specimens A and C were especially good with 100% solder coverage thr ou ghout.

Table VII presents the results of a statistical analysis of the data shown in Tables V and VI. In only one case (solder coated bifurcated terminal) was there any significant decrease in solderability. The significance level was 0.001 indicating that there is only 1 chance in 1000 that the analysis is wrong. With this same specimen, the effect of ETO decontamination was not significant.

Solderability test specimens which have been solder dipped before and after heat sterilization and ethylene oxide decontamination. (R105895) Figure 8.

 $\frac{1}{2}$

l,

l,

 $\overline{21}$

HUGHES AIRCRAFT CO.

FORM 703-C

HUGHES AIRCRAFT CO.

 \overline{a}

 $\sigma_{\bm{r}}$

FORM 703-C

23

Effects of thermal sterilization and ethylene oxide decontamination on the solderability of connector cups, bifurcated terminals, stranded wire, and solid wire. Table VII.

 \mathcal{P}

4. 2 METALLOGRAPHIC EXAMINATION TEST RESULTS

4. 2. I General

Figure 9 and Figure i0 are photomicrographs at 50X magnification showing a typical metallographically mounted specimen of:

Figure 9 (top) - Solid conductor Figure 9 (bottom) - Stranded conductor *Figure* 10 (top) - *Connector* cup *Figure* 10 (bottom) - Bifurcated terminal

These photomicrographs at low magnification (50X) were included to give a reader an idea of the mounting configuration used for each type of solderability specimen. The photomicrographs which follow (Figures 11 through 23) are all at much higher magnification (1000X) and show only a very small area of the mounted specimen.

4.2.2 Before and After Thermal Bake-Out

Figures Ii through 17 are photomicrographs at 1000X magnification of Specimens A, B, *C,* D, E, F, and G as outlined in Figure 1 and Table II. Each figure *contains* two photomicrographs. The **top** photomicrograph is of that particular specimen before thermal bakeout. The bottom photomicrograph is of a similar type specimen which has been thermally baked-out at 200°C for 168 hours in a nitrogen atmosphere. The different etchants used were selected to bring out the metallographic structure of that particular type of wire and electroplating. All of the specimens in Figures 11 through 17 are solid wire conductors.

Although Specimen F(ii), as shown in Figure 16, failed the solderability test per MIL-STD-202C, Method 208A (refer to Table IV), there was no evidence of any grain boundary diffusion of the nickel plating into the outer copper sheath or of any interdiffusion of the gold and nickel electroplated layers. None of the specimens which were metallographically mounted showed any signs of degradation which could be

25

Figure 9. Photomicrographs of a typical solid conductor (top picture) and a typical stranded conductor (bottom picture) as metallographically mounted. Magnification at 50X. Potassium dichromate etchant. The solid conductor **(Neg.** 02407) is a solder coated copper wire and the stranded conductor (Neg. 02409) cpnsists of 19 strands of 36 **AWG** silver plated copper wire.

Figure 10. Photomicrographs of a typical connector cup (top picture) and typical bifurcated terminal (bottom picture) as metallographically mounted. Magnification at **50X.** Potassium dichromate etchant. Connector cup **(Neg.** 02406) **is** the Bendix type and the bifurcated terminal (Neg. 02408) is the gold plated type.

Before Bake-Out (Neg. *02392)*

After Bake- Out (Neg. **02393)**

Figure 11. Photomicrographs at lOOOX of Specimen A(ii) (gold plated copper wire) before and after thermal bake-out. Potassium dichromate etchant.

Before Bake- **Out** (Neg. **02394)**

After Bake- Out (Neg. **02395)**

Figure 12. Photomicrographs at **lOOOX** of Specimen B(ii) (gold plated Dumet wire) before and after thermal bake-out. Aceticnitric etchant.

Bake-Out (Neg. 02396)

Figure 13. Photomicrographs **at lOOOX of Specimen C(ii) (gold plated** Kovar wire) before **and after thermal bake-out. Carapella** etc hant .

Before Bake-Out (Neg. **02398)**

After **Bake-** Out (Neg. *02399)*

Figure **14.** Photomicrographs at lOOOX of Specimen D(ii) (gold plated nickel wire) before and after thermal bake-out. Aceticnitric etchant.

Aft e r Bake- Out (Neg. 02401)

Figure 15. Photomicrographs at **lOOOX** of **Specimen E(ii) (bare nickel** wire) before and after **thermal bake- out. Acetic-nitric** etchant.

Before Bake-Out (Neg. **02402)**

After Bake- Out (Neg. 02403)

Figure 16. Photomicrographs at 1000X of Specimen F(ii) (gold plated Dumet with nickel undercoat) before and after thermal bake- out. Acetic-nitric etchant.

Before Bake- **Out** (Neg. 02404)

After Bake- Out (Neg. 02405)

Figure 17. Photomicrographs at 1000X of Specimen G(ii) (gold plated copper wire with nickel undercoat) before and after thermal bake- out. Potassium dichromate etchant.

and after heat sterilization and ETO decontamination. Potassium dichromate Figure 18. Photomicrographs at 1000X of Specimen A(iii-iv) (stranded conductor) before Photomicrographs show only one or two of the silver plated copper etchant. strands.

Figure 19. Photomicrographs at 1000X of Specimen B(iii-iv) (brass, copper plated, gold plated Cinch connector cups) before and after heat sterilization and ETO decontamination. Potassium dichromate etchant.

(Neg. 02379)

(Neg. 02373)

(Neg. 02367)

Figure 20. Photomicrographs at 1000X of Specimen C(iii-iv) (brass, copper plated, silver
plated, gold plated Bendix connector cup) before and after heat sterilization and
ETO decontamination. Potassium dichromate etchant.

After ETO Decontamination

After Heat Sterilization
(Neg. 02374)

No Treatment (Neg. 02368)

(Neg. 02380)

After ETO Decontamination (Neg. 02381)

After Heat Sterilization (Neg. 02375)

> No Treatment (Neg. 02369)

Photomicrographs at 1000X of Specimen E(iii-iv) (solder coated bifurcated
terminal) before and after heat sterilization and ETO decontamination. Potassium dichromate etchant. Figure 22.

After ETO Decontamination (Neg. 02382)

After Heat Sterilization
(Neg. 02376)

No Treatment (Neg. 02370)

Figure 23. Photomicrographs at 1000X of Specimen F(iii-iv) (brass, copper strike, silver
plated, gold plated bifurcated terminal) before and after thermal sterilization
and ETO decontamination. Potassium dichromate etchant

attributed to thermal bake-out. The nickel undercoat on Specimens F and G did not appear to enhance the solderability of the gold plated copper or gold plated Dumet wires. Since the effect was not observable metallographically, it is assumed that the nickel underplating formed a type of barrier layer hindering a wetting or alloying reaction between l the tin in the solder and the copper basis metal. It has been report that nickel undercoats will form a duplex intermetallic compound layer with tin which, although assisting initial solderability of freshly plated coatings, can decrease solderability after storage if the thickness of the coating is not adequate. Nickel plating, by itself, is not very solderable (as compared to tin, solder, gold, copper, etc.). If the outer gold electroplate was porous, the nickel underneath would therefore inhibit optimum wetting. By similar reasoning it would then be expected that the gold plated copper wire (with the nickel undercoat) would also experience decreased solderability. However, such was not the case. Further testing of these questionable wire types would be necessary in **order** to **obtain** a full understanding **of** this **phenomenon.**

4. 2.3 Before and After Heat Sterilization and Ethylene Oxide Dec ontamination

Figures 18 through 23 are **photomicrographs** at 1000X magnification of Specimens A, B, C, D, E, and F as outlined in **Figure** 1 and Table II. Each figure contains three photomicrographs of the same type of specimen which has been submitted to three different environments. The specimen in eachleft-hand photomicrograph has received no treatment. The specimen in each middle photomicrograph has undergone heat sterilization. The specimen in each right-hand photomicrograph has undergone ethylene oxide **(ETO)** decontamination. All

¹Thwaites, C.J. "The Solderability of Some Tin, Tin Alloy and Other Metallic Coatings," Trans. Inst. Met. Finishing, vol. 36, 203 (1959).

metallographic mounts were etched in potassium dichromate. $\;{\rm Th}\;$ different specimens are as follows:

Figure 18 Figures 19 and 20 - Connector cups Figure 21 - Stranded conductors - Copper wire solid conductors *Figures* 22 and 23 - Bifurcated terminals.

Only two of the specimens exhibited any apparent metallurgical change as a result of the exposures. Specimens B, C, D, and F showed no evidence of any grain boundary diffusion of the plating layer into the basis metal or any interdiffusion of the surface plating layers. Specimen C (Figure 20) shows a slight change in the metallic copper and silver plated layers, but this phenomenon was not observed on all heat sterilized specimens and did not appear to affect the solderability (refer to Table V). A noticeable change appears on the stranded conductor specimen (Figure 18 - middle picture) which has undergone heat sterilization. Although this might appear to be a grain boundary diffusion of the silver plating, it could also be caused by unequal etching or polishing of the mount and improper focusing. This effect was not as pronounced during examination of the actual mount. This change was not observable on all specimens and did not appear to affect the solderability (see Table V). During the soldering operation, the thin silver electroplate becomes soluble in the tin of the tin/lead solder alloy and may cause silver scavenging from the plated wire strands. For this reason it is common to use a solder alloy containing a small percentage of silver (i.e., 1.5% -2%) when soft soldering silver plated parts.

Specimen E (solder coated bifurcated terminal) was the other specimen showing what appears to be a marked difference in metallurgical structure after heat sterilization. Note that the middle photomicrograph in Figure 22 shows an intermediate layer between the brass basis metal of the bifurcated terminal and the outer solder coating. Metallographic examination showed this layer to be a thin gold electroplate approximately 0.000016 inch thick. This gold plated layer is not present on the left-hand or right-hand photomicrographs. Further checking

42

revealed that the vendor of the bifurcated terminals apparently shipped a mixed lot of terminals, some of which were solder coated directly over brass and some of which were solder coated over gold plated brass. This is proven by examining Figures 14, 20, and 22 in Attachment 3, Part II, of this final report. The gold plated intermediate layer is also shown in these photomicrographs of actual solder joints, while other *photomicrographs* of solder joints to solder coated bifurcated terminals do not exhibit the gold electroplate. This mixed lot of terminals could possibly account for some of the poor solderability results of this type of specimen (refer to Tables VI and VII). It should also be kept in mind that the solder coated bifurcated terminals were all coated with a layer of Sealbrite No. 230-10. Overheating of this *protective* coating during the heat sterilization cycle could possibly inhibit good solderability. The gold plated bifurcated terminals were not coated with Sealbrite, nor were the gold plated connector cups. Connector cups are not ordinarily protectively coated until after tin**ning.**

It must therefore be stated that none of the specimens exhibited significant metallurgical degradation or diffusion **characteristics** which **could** definitely be **attributed** to **either** heat sterilization or **ethylene** oxide decontamination. The **changes appearing** in the **photomicrographs could** logically be **assigned** to other unrelated **causes.** The heat sterilization temperature **(145°C)** is **about** 70°F below the melting **point** of 63/37 tin/lead solder and the nitrogen **atmosphere** prevents the formation of any metallic oxides which would otherwise form **in** air. The ETO decontamination exposure had even less **effect** on the metallurgical structure than the heat sterilization **exposure.**

43

外ン

5. CONCLUSIONS

Table VIII presents a summary of the test results concerning the effects of thermal bake-out, heat sterilization, and ethylene oxide decontamination on the solderability of component leads. A more detailed discussion of the results is given in Sections 4. 1 and 4.2 of this report, including raw data sheets, a statistical analysis of the solderability tests, and thirty-six photomicrographs.

Of the seven different types of solid wire conductors tested before and after thermal bake-out, only the gold plated Kovar wire and the gold plated Dumet wire (with the nickel undercoat) showed any decrease in solderability after thermal bake-out. Of these two wire types, the gold plated Dumet wire (with the nickel undercoat) would be the only one actually failing to meet the 95% minimum solder coverage required by MIL-STD-202C, Method 208A. Metallurgical examination at 1000X magnification of these seven types of solid wire conductors showed no signs of degradation, grain boundary diffusion, or interdiffusion of the electroplated layers which could be attributed to thermal bake- out.

Of the six types of specimens (i.e., stranded and solid conductors, connector cups, and bifurcated terminals) tested before and after heat sterilization and ETO decontamination, only the solder coated bifurcated terminal exhibited a significant drop in solderability as a result of thermal sterilization. A metallographic examination of the solder coated bifurcated terminals later revealed that certain of the specimens had a thin gold electroplate beneath the outer solder coating while others did not. These terminals also had a protective lacquer overcoating. It is felt that these two factors (not present on any of the other specimen types) could have contributed to some of the poor solderability results. Metallurgical examination of the six types of specimens (A, B, C, D, E, and F) in the lower half of Table VIII revealed that two of the specimen types showed a slight change in metallurgical structure after heat sterilization. However, these slight changes were not evidenced on all

45

\

Summary of test results.

Table VIII.

 $\ddot{}$

Metallurgical
Examination ETO Decontamination None None None None None None \mathbf{I} \mathbf{I} \mathbf{i} \mathbf{I} \mathbf{I} \mathbf{I} \mathbf{I} Solderability None None None None None None \mathbf{I} $\bar{1}$ \mathbf{I} \mathbf{I} \mathbf{I} \mathbf{I} $\pmb{\mathsf{l}}$ Metallurgical
Examination $\text{Slight}^{\star\star}$ S^{1} ight $\stackrel{***}{_{\sim}}$ Effect of Different Exposures None None None None \mathbf{I} Heat Sterilization $\mathbf{1}$ $\mathbf i$ $\pmb{\cdot}$ $\mathbf i$ $\mathbf i$ \mathbf{I} Solderability
Test Significant
drop None None None None None \mathbf{I} \cdot \mathbf{I} \mathbf{I} \mathfrak{t} \mathbf{I} \mathbf{I} Metallurgical
Examination None None None None None None None \mathbf{I} $\mathbf{1}$ \mathbf{I} \mathbf{I} \mathbf{I} \mathbf{I} Thermal Bake-Out Solderability
Test Slight,
passes* Fails* None None Noue None None \mathbf{I} \mathbf{I} \mathbf{I} \mathbf{l} \mathbf{I} \mathbf{I} Gold plated bifurcated terminal Brass, copper plated, silver
plated, gold plated connector
cup (Bendix) Gold plated copper wire with
nickel undercoat Gold plated Dumet wire with
nickel undercoat Brass, copper plated, gold
plated connector cup (Cinch) Solder coated copper wire Solder coated bifurcated
terminal Gold plated copper wire Gold plated Dumet wire Gold plated Kovar wire Gold plated nickel wire Stranded conductor Type of
Specimen Bare nickel wire .
ز Ľ. a. \vec{a} $\vec{\omega}$ ن $\vec{\mathbf{r}}$ ö ż, á \vec{a} \vec{E} k,

**Structural change questionable; could be caused by other factors; and was not 100% repeatable. *Refers to solderability specification which requires minimum of 95% coverage.

 $\frac{1}{2}$

46

specimens nor could they be attributed definitely to heat sterilization. Metallurgical examination of the ETO decontaminated specimens showed no signs of degradation, grain boundary diffusion, or interdiffusion of the electroplated layers which could be attributed to *ETO* exposure.

As a whole it can be *concluded* that thermal bake-out, heat sterilization, and ethylene oxide decontamination had very little effect on the solderability or metallurgical integrity of the specimens tested. Although a few specimen types did show certain effects due to either thermal bake-out or heat sterilization, these effects were minimal and *could* be attributed to either unrelated causes or to factors which *could* be *corrected* in production electronic systems.

ACKNOWLEDGMENTS

This investigator wishes to express his appreciation to the following people for their valuable assistance in the various phases of this part of the program:

The Second Act of Act