NTF - SOLDERING TECHNOLOGY DEVELOPMENT FOR CRYOGENICS

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ABSTRACT

The advent of the National Transonic Facility (NTF) brought about a new application for an old joining method, soldering. Soldering for use at cryogenic temperatures requires that solders remain ductile and free from tin-pest (grey tin), have toughness to withstand aerodynamic loads associated with flight research, and maintain their surface finishes. Solders are used to attach 347 Stainless-Steel tubing in surface grooves of models. The solder must fill up the gap and metallurgically bond to the tubing and model. Cryogenic temperatures require that only specific materials for models can be used, including: Vasco Max 200 CVM, Lescalloy A-286 Vac Arc, PH 13-8 Mo. Solders identified for testing at this time are: 50% Sn - 49.5% Pb - 0.5% Sb, 95% Sn - 5% Sb, 50% In 50% Pb, and 37.5% Sn - 37.5% Pb - 25% In. With these materials and solders, it is necessary to determine their solderability. After solderability is determined, tube/groove specimens are fabricated and stressed under cryogenic temperatures. Compatible solders are then used for actual models.

INTRODUCTION

NTF - SOLDERING TECHNOLOGY DEVELOPMENTS FOR CRYOGENICS

In an earlier presentation (ref. 1), Firth discussed objectives of the solder research program, problems associated with using solders at cryogenic temperatures, test results, and experiences with recent models.

This presentation will cover soldering methods, testing procedures, and results of solderability tests done on Vasco Max 200. Soldering techniques to install pressure tubes on cryogenic models, physical testing of the solders, surface preparation, and surface analysis will also be discussed.

BASE METALS

- Vasco Max 200 CVM - 18% Nickel Alloy
- 347 Stainless Steel - Tubing
- Lescalloy A-286 Vac Arc
- PH 13-8 Mo

Various metals usable at cryogenic temperatures were selected for testing their solderability. Vasco Max 200, Lescalloy A-286, and PH 13-8 Mo are used in making cryogenic wind tunnel models. Pressure tubes used on these models are made of 347 Stainless Steel. Testing is being done on the Vasco Max 200 and 347 Stainless Steel at this time. Lescalloy A-286 Vac Arc and PH 13-8 Mo will be tested later. The rest of this presentation centers around Vasco Max 200 and 347 Stainless Steel.

SOLDERS TO BE TESTED

- Indalloy #119 50% Sn, 49.5% Pb, 0.5% Sb 420° F Liquidus
- 95% Sn, 5% Sb 464° F Liquidus
- Indalloy #7 50% In, 50% PB 480° F Liquidus
- Indalloy #5 37.5% Sn, 37.5% PB, 25% In 358° F Liquidus

The solders listed above were selected for testing. At this time, 50% Sn - 49.5% Pb - 0.5% Sb and 95 Sn - 5 % Sb solders are being tested. Once tensile and lap shear specimens are completed, testing will begin on 50% In - 50% Pb and 37.5% Sn - 37.5% Pb - 25% In. The liquidus temperatures for each solder are included. This list is not intended to exclude other solders. The 93% Pb - 5.2% Ag solder has been mentioned as another possible solder for investigation (ref. 1).

FLUXES TESTED

- Eutectic's Eutector Flux 157 - Contains Fluoride
- All-State Duzall Flux - Contains Zinc Chloride
- Ruby's Stainless Steel Soldering Flux - Contains Muriatic Acid

The base metals selected are considered very difficult to solder. Therefore, corrosive fluxes are needed to break the surface oxides and promote good wetting by the solders. Selection of the fluxes listed above was based upon their availability and their recommended use on the base metals mentioned earlier. Testing of these three fluxes was conducted on Vasco Max 200. Vasco Max samples were fluxed, placed on a pre-heated hot plate, and allowed to heat up to soldering temperature. Solder was then applied to the fluxed surface. After the solder finished flowing the sample was removed from the hot plate, air cooled to room temperature, and washed in water.

Visual inspection of each sample showed the fluoride flux promoted better wetting of the solder. The zinc chloride flux and muriatic acid flux had to continually be added during the heatup and soldering operation. The fluoride flux was applied to the surface before heating up and again when the solder was applied. Samples of 347 Stainless Steel tubing were tinned using each of the fluxes previously mentioned. Again the fluoride flux worked better than the other two. The fluxes will be tested on samples of the other base metals at a later date. Other fluxes are being looked at for possible use on these base metals.

SOLDERABILITY TEST

- o SPREAD SPECIMENS
- o TINNING SPECIMENS
- o TUBE/GROOVE SPECIMENS
- o WING SPECIMENS

A solderability test will be conducted on each base metal and solder. At this time only Vasco Max 200 has been tested using 50% Sn - 49.5% Pb - 0.5% Sb. Listed above are four tests to be used for evaluation. Spread/wettability specimens were done on various abraded surfaces to determine which surface gave the best wettability of the solder. Abrading the surface is necessary because Vasco Max 200 oxidizes when heat treated. Fluxes cannot remove this heavy oxide so mechanical abrading methods are used. After determining which surface finishing methods give the best percent spreading/wettability these surfaces were used in subsequent tests to evaluate their tinning qualities.

Tube/groove specimens were fabricated to determine the best method for soldering 347 Stainless-Steel tubing in the grooves of a Vasco Max specimen. Once a satisfactory method was determined, tubes were soldered in grooves on both the upper and lower surfaces of a wing specimen for the fatigue/flex test. Chemical or plated surface preparations were not considered due to the lack of practical application to the model's partial assembly and the intricate nature of hardware design.

FORMULAS FOR EVALUATING PERCENT SPREADING

$$D = 0.3937 \sqrt[3]{\frac{\text{Weight}}{\text{Specific Gravity}} \left(\frac{6}{\pi}\right)}$$

$$\frac{D-H}{D} \times 100 = \text{Percent Spreading}$$

D = Diameter of the Hypothetical Sphere

H = Height of the Solder Droplet After Spreading Over the Metal

The formulas above (ref. 2) were used to calculate the percent spreading/wettability of the solder. Calculate the diameter of the hypothetical sphere (D) using the first formula. Weight (gms) and specific gravity (gms/cm^3) of the solder are known values, and the value 0.3937 converts centimeters to inches. Measure the height of the solder droplet (H) after spreading over the metal surface. Plug the values for D and H into the second formula and calculate the percent spreading. The lower the height of the solder after spreading the higher the percent spreading. The ideal percent spreading would be one that reaches 100%.

Spread Test Data

Vasco Max 200
50% Sn, 49.5% Pb, 0.5% Sb

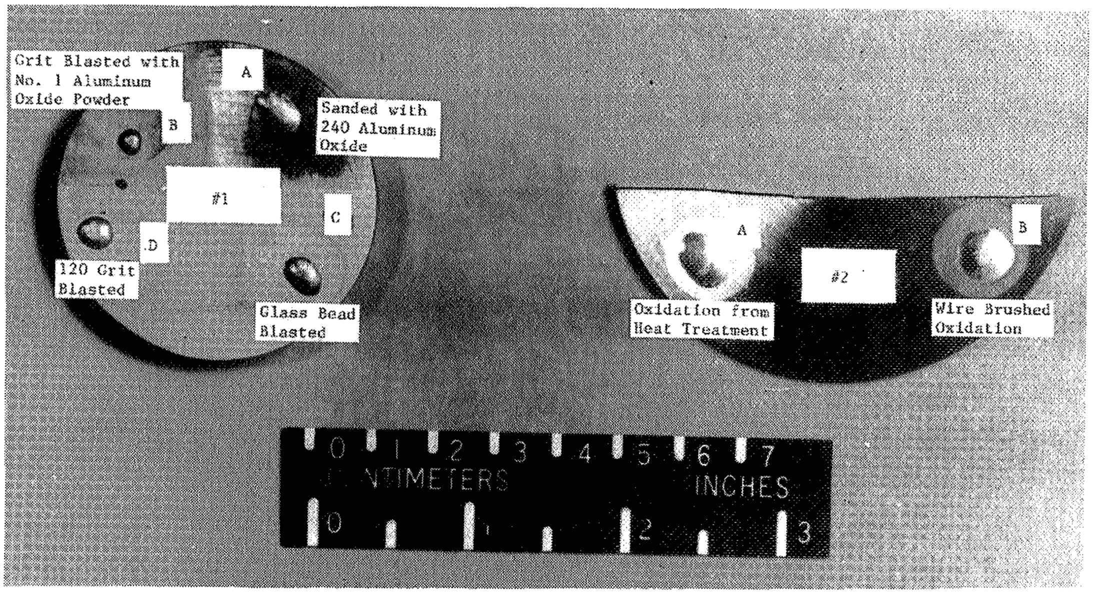
Specimen Number	Surface	Ra	Solders Specific Gravity (gm/cm^3)	Solders Weight (grams)	H (inches)	D (inches)	% Spreading	Description
1A	Sanded w/240 Alum. Oxide	15	8.85	0.0918	0.0228	0.1065	78.6%	Adequate
1B	No. 1 Alum Oxide Powder	34	8.85	0.1095	0.0581	0.1330	48%	Very Poor
1C	Glass Bead Blasted	34	8.85	0.1175	0.0299	0.1157	74.1%	Adequate
1D	120 Grit Blasted	40	8.85	0.1044	0.0350	0.1112	68%	Poor to Fair
2A	Brown Oxide Heat Treated	13	8.85	0.1299	0.0193	0.1196	83.8%	Good
2B	Brown Oxide Wire Brushed	13	8.85	0.1206	0.0164	0.1167	85.9%	Very Good
3	Sanded with 120 Alum. Oxide	19	8.85	0.5	0.0214	0.1875	88.6%	Very Good
4	Steel Wooled Heavy Oxide	22	8.85	0.5	0.1045	0.1875	44.3%	Very Poor
5	120 Grit Blasted	51	8.85	0.5	0.0375	0.1875	80%	Adequate/Good
6	Glass Bead Blasted	34	8.85	0.5	0.0285	0.1875	84.8%	Good +
7	Machined Surface(Lathe)	16	8.85	0.5	0.0230	0.1875	88%	Very Good
8	Machined Surface (Mill)	15	8.85	0.5	0.0145	0.1875	92%	Excellent

Spread test data on Vasco Max 200, 50% Sn - 49.5% Pb - 0.5% Sb solder, and the floride flux are shown in the chart above. All Vasco Max 200 specimens have been heat treated. Solder of a known weight and flux was placed on the specimen surface. The specimen was placed on a hot plate and heated to approximately 525° F which allowed the specimen to reach the solder's flow temperature. After the solder stopped flowing the specimen was removed from the hot plate, air cooled to room temperature, and cleaned of flux residue. The height of the solder, after spreading over the specimen, was measured. Calculations were performed to determine percent spreading. This procedure was followed on each spread test specimen. Specimen #1 (see photos that follow) was divided into four areas for preliminary investigation of various surfaces. Area #1B, abraded with No. 1 Aluminum Oxide (27 microns), had the worst percent spreading of the four surfaces. Because of low percent spreading on surface 1B no more tests were done for this type of surface. Percent spreading on the other three was not as high as expected. Therefore, the surfaces of specimens #3 through #8 were prepared for soldering as shown in the chart above. Each specimen was soldered with 0.5 grams of solder using the same soldering procedures as specimen #1. Specimen #3, sanded with 120 aluminum oxide, had 88.6% spreading. Specimen #4, oxide rubbed with coarse steel wool, had 44.3% spreading. Specimen #5, grit blasted with 120 grit, had 80% spreading. Specimen #6, glass bead blasted, had 84.8% spreading. Specimens #7 and #8 were machined by lathe and milling machine, respectively, to remove oxidation.

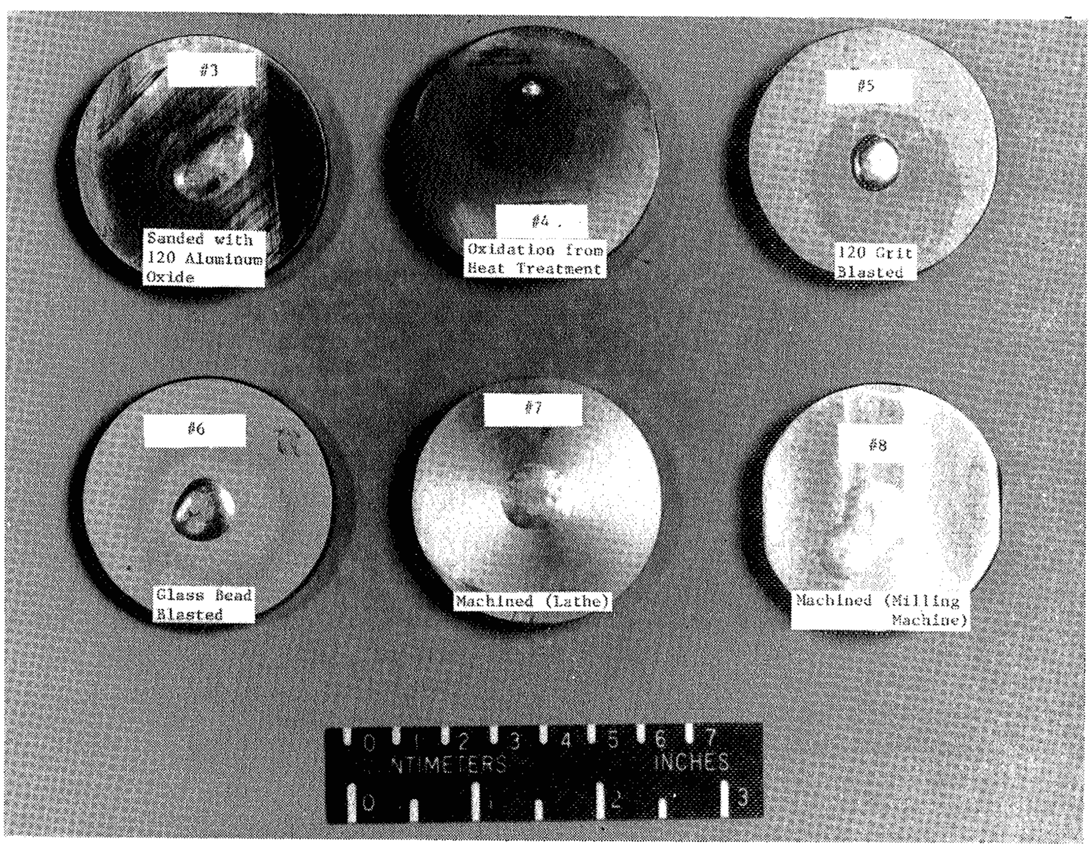
Specimen #7 had 88% spreading and specimen #8 had 92% spreading. Specimen #8 was inadvertently tilted while being removed from the hot plate which resulted in the solder flowing and wetting more of the surface. Therefore, the spreading percent is slightly higher. Surface finishes were measured for each specimen to determine if any relationship exists between it and percent spreading. There did not appear to be any distinct relationship between the two.

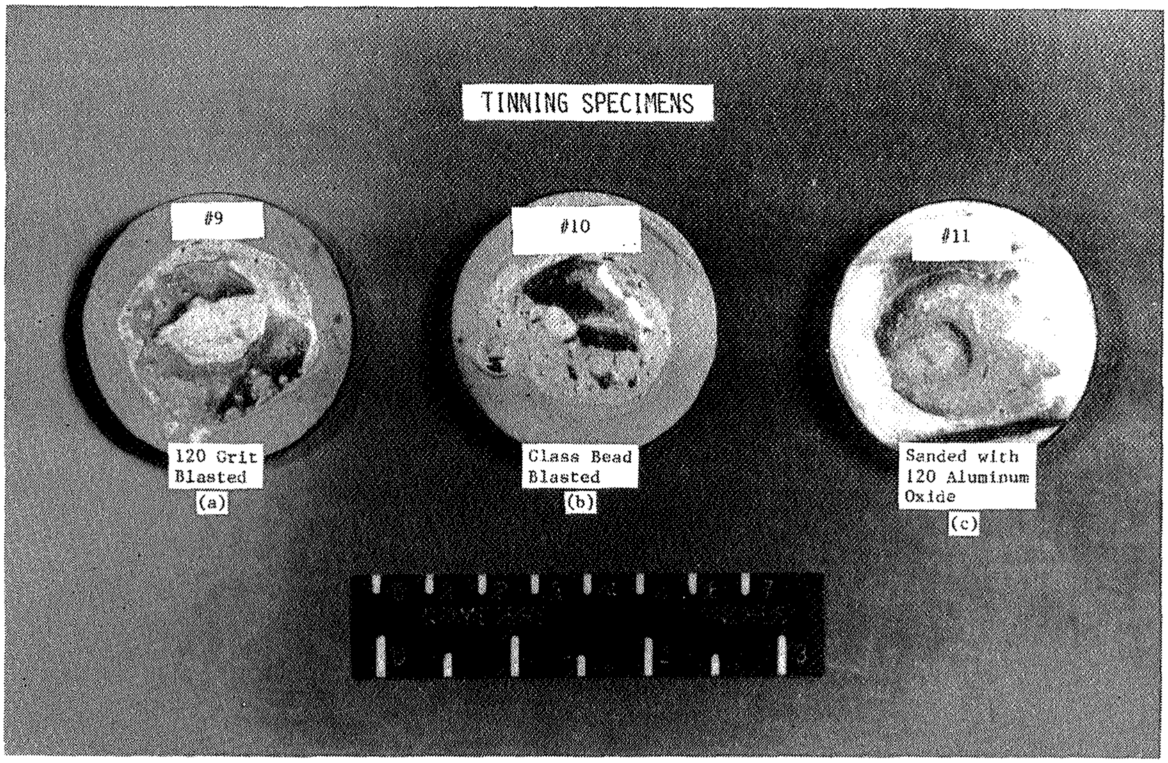
Specimen #2 was heat treated in an argon atmosphere retort to eliminate oxidation. If oxidation was eliminated there would be no need to abrade the surface. A golden brown oxide that formed on the specimen was the result of a leaky retort. Prior to spread testing, #2B was wire brushed while nothing was done to #2A. Wire brushing did not remove the oxide. The flux broke the oxide allowing the solder to spread over the surface. Surfaces #2A had 83% spreading and #2B had 85.9%.

SPREAD TEST SPECIMENS

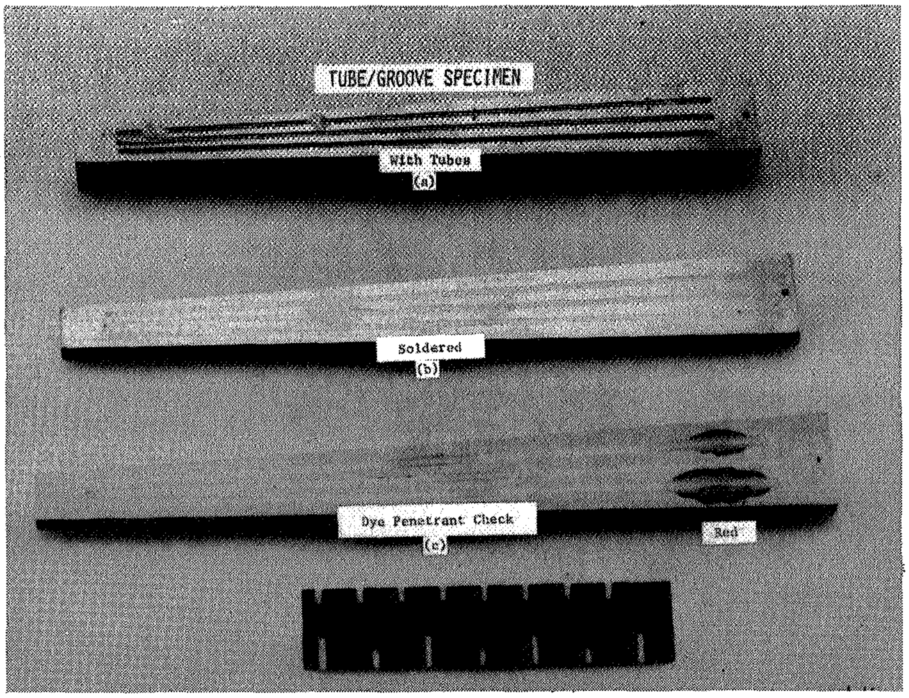


SPREAD TEST SPECIMENS





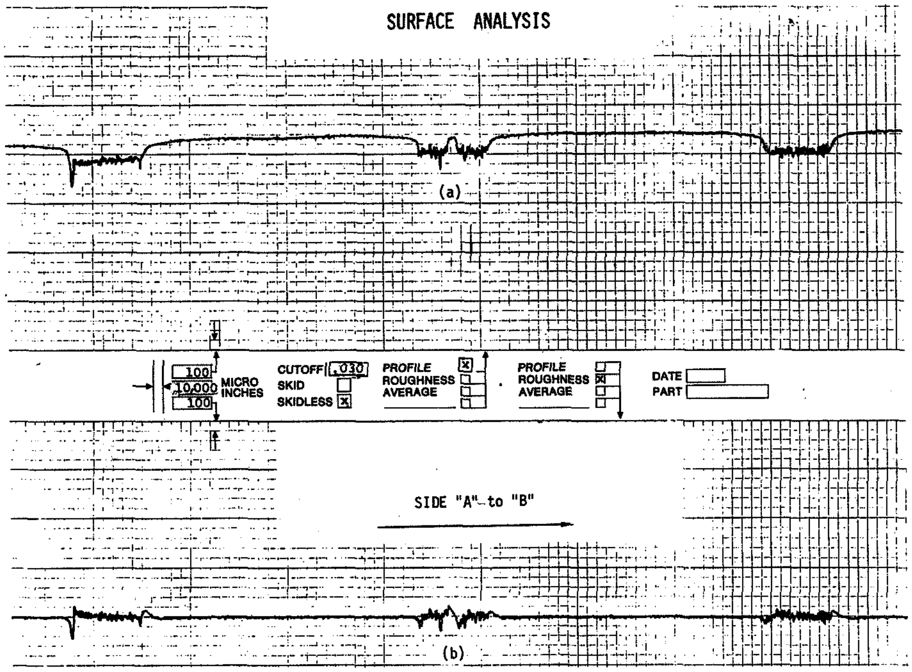
Tinning specimen surfaces were prepared as shown in the picture above. These specimens were heated the same way as for spread testing. Once the specimens reached soldering temperature a soldering gun was used to tin the surface. Specimens #9 (a) and #10 (b) had areas of dewetting whereas #11 (c) tinned excellently with total wetting. A machined surface was not tinned because from past experience machined surfaces in excellently. Grit and bead blasting, which do not promote capillary action, are not recommended methods of surface preparation for soldering.



Now that a method of removing heat treatment oxides has been found, tube/groove specimens have been fabricated. The purpose of these specimens was to develop a method for soldering tubes in surface grooves. The specimens were then subjected to cryogenic temperatures.

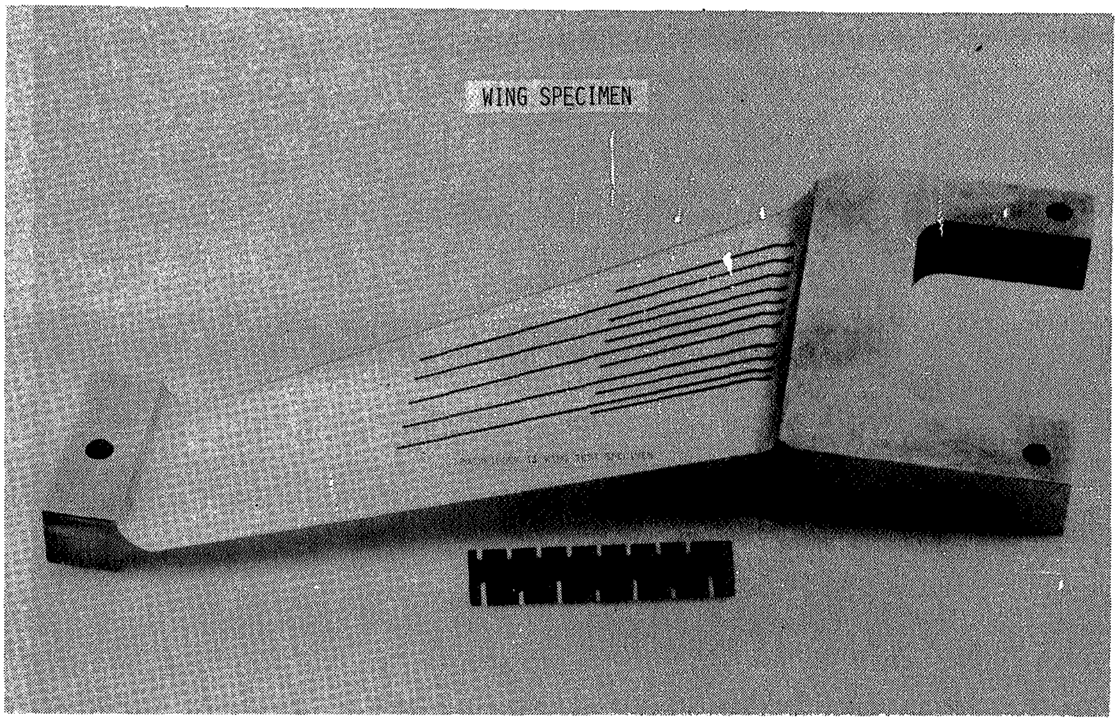
Grooves were sanded with 120 aluminum oxide and cleaned of all contaminants. Tubes were straightened as much as possible, tinned, and placed in the groove. To insure that the tubes remain below the surface during soldering the following two methods were used: staked in place or tacked in place with solder (a). Staking the tubes in place was done before heating. Tacking in place was done at approximately 100°F below the solder's liquidus temperature. A soldering gun was used to heat the tack area to the solder's flow temperature. A small amount of flux was applied to the tube/groove prior to heating. At the high points of the tube, it was pushed to the bottom of the groove and tacked in place. Starting at one end of the groove the tube was soldered in place using a soldering gun. Solder flowed into the groove and around the tube to fill the groove so that excess solder built up above the specimen surface. When soldering was completed and the specimen cooled down, it was cleaned in soapy hot water to remove flux residue. Excess solder was machined to within .002" to .003" of the surface, then hand finished (b). Since solder is softer than the base metal there was a tendency to undercut the solder. This is normal, and care must be taken to keep undercutting to a minimum.

The finished surface was dye penetrant checked for metallurgical bonding between solder and specimen (c). Dark, wet looking areas are where flux is weeping from a debond area. Red areas also indicate debonding. Debonding may be the result of: edge contamination, specimen not being hot enough, or poor technique. After the specimens were cryo cycled they were dye penetrant checked again for any growth of debond areas. This would indicate whether flux left in the joint between solder and specimen or brittleness of the solder affected the metallurgical bond.



A surface analysis of the previous tube/groove specimen was done before and after cryo cycling. One cross section of the analysis is shown above. Solder at each groove has been undercut during finishing. The center groove of (a) has a raised spot in the middle because the tube was not on the groove bottom when soldered.

The surface roughness of the solder and specimen is shown above in (b). Because of the solder's softness it was rougher than the base metal surface. A surface typical of the one shown above would be usable on a conventional wind tunnel model. However, on cryogenic wind tunnel models this surface is not acceptable. Air flow over the grooves would not be smooth, resulting in lower Reynolds numbers. One way to correct this problem would be to paint the surface with a cryogenic compatible coating and finish to a uniform finish.



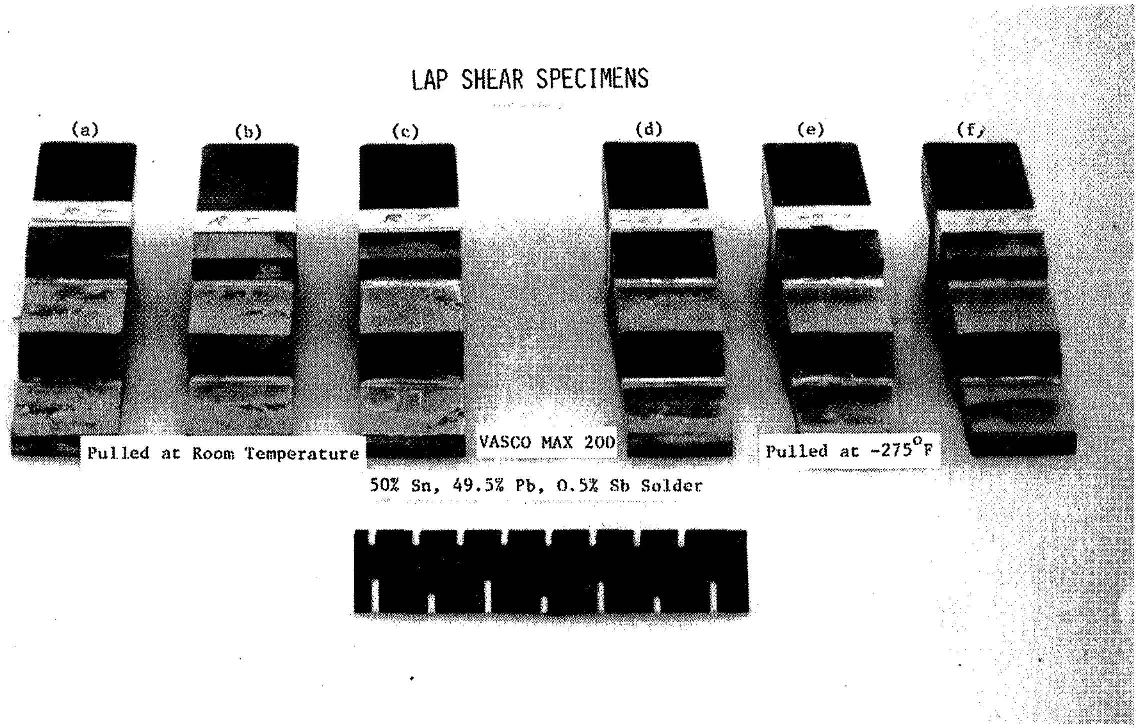
Flex tests on the Vasco Max 200 wing shown above were done to test the solder's ability to withstand aerodynamic loading in cyrogenic environments. Grooves are located on both upper and lower surfaces. Tubes tinned with 95% Sn - 5% Sb were placed in the first four grooves on the upper surface. The wing was placed on a hot plate that had been preheated to approximately 100°F below the liquidus temperature of 95% Sn - 5% Sb. The tubes/grooves were soldered and finished by the same method used for the tube/groove specimens. After the upper surface was hand finished, the first four grooves of the lower surface were tubed, soldered and hand finished. This process was repeated for the next four grooves on upper and lower surfaces using 50% Sn - 49.5% Pb - 0.5% Sb solder. The wing was cleaned in soapy, hot water and dried. A filler material was used to bond tubing and fill the groove in the last 3 grooves on upper and lower surfaces.

A surface analysis and dye penetrant check were done on both surfaces before and after testing. The 50% Sn - 49.5% Pb - 0.5% 5b solder wet and flowed better than the 95% Sn - 5% Sb solder. Test results showed 95% Sn - 5% Sb to be too brittle, whereas 50% Sn - 49.5% Pb - 0.5% Sb remained ductile. Results concerning the filler material are given in reference 1.

PHYSICAL TESTING OF SOLDER

- Lap Shear Specimens
  - Room Temperature
  - -275° F
- Tensile Specimens
  - Room Temperature
  - -275° F

Vasco Max specimens were tested using single lap and butt joints to determine lap shear and tensile strengths. Lap shear specimens were 1" wide and with a 1" overlap at the joint and gapped to .003". Tensile specimens were 1" in diameter and gapped to .003". After soldering, the specimens were cryo cycled from room temperature to -275°F five times. Half the specimens were pulled at room temperature while the others were pulled at -275°F. Testing was done at a cross-head rate of 0.05 in/min. Lap shear specimens were tested on Vasco Max 200 using 95% Sn - 5% Sb and 50% Sn - 49%.5% Pb - 0.5% Sb solders. At the present time testing on the tensile specimens is not complete.

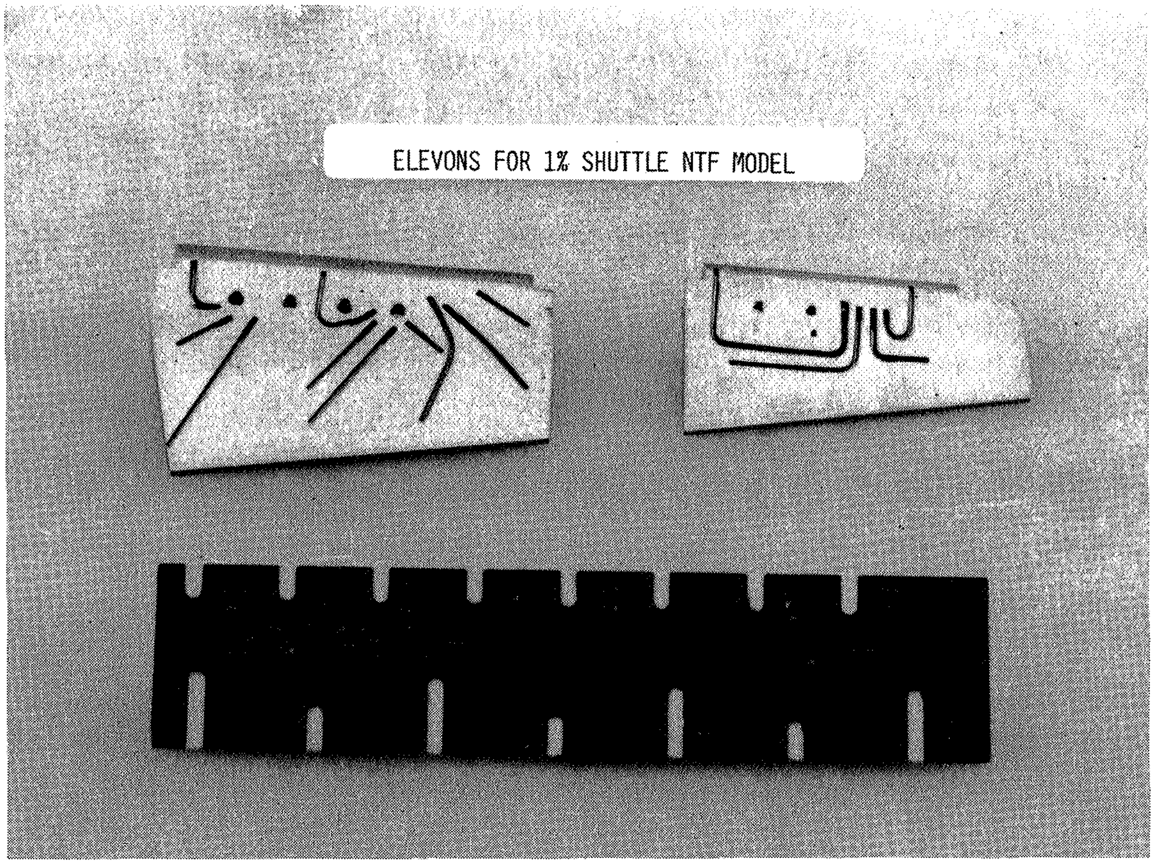


Specimens for the lap shear test are shown above. A fixture was made to hold the specimens during soldering so that the gap would be .003". Prior to soldering each mating surface was tinned and cleaned. The fluoride flux was brushed on the tinned surfaces. The specimen was then clamped in the fixture. The fixtured specimen was turned upside down so that the specimen's top rested on the hot plate. The hot plate had been preheated to approximately 100°F above the solder's liquidus temperature. After the specimen reached temperature, solder was applied to the joint. The fixtured specimen was then removed from the hot plate and air cooled to room temperature. Flux residue was washed off in hot, running water.

Specimens (a), (b), and (c) failed in the solder. Solder pulled away from the specimen surface in (d) and (e), while in specimen (f) 1/3 of the joint failed in the solder and the rest pulled away from the surface. Lap shear strengths of the specimens are listed below:

Specimen (a) - 4395 psi	Specimen (d) - 3030 psi
Specimen (b) - 4200 psi	Specimen (e) - 240 psi
Specimen (c) - 4400 psi	Specimen (f) - 4850 psi

The strengths were not as high as expected. More specimens will be fabricated and tested later. Flux inclusions were seen on all lap shear specimens. From all observations some amount of flux inclusion is normal.



An application for soldering pressure tubes in surface grooves is found in the elevons for the 1% Shuttle Accent NTF Model. Grooves were machined on both sides to accept 0.020" O.D. Stainless-Steel tubing. Sharp edges were chamfered slightly to prevent the solder's tendency to pull away from sharp edges. After soldering is complete and the elevons surface have been finished a 0.010" diameter hole will be drilled into the tube normal to the surface. This will allow pressure readings to be gathered from the elevon's surface during tunnel test model.

Concluding Remarks

1. Testing is almost complete on Vasco Max 200.

2. A method and materials for soldering pressure tubes on both upper and lower surfaces of a Vasco Max 200 wing specimen have been determined.

3. The flux containing fluoride promotes better wetting and flow of 50% Sn - 49.5% Pb - 0.5% Sb solder on Vasco Max 200 and 347 stainless steel.

4. The ductility of 50% Sn - 49.5% Pb - 0.5% Sb solder appears to be very good at cryogenic temperatures.

5. Oxidation from heat treating Vasco Max 200 has to be removed before soldering or eliminated in the heat treatment.

6. Mechanically abrading with a course aluminum oxide paper or remachining the surface are the recommended methods of surface preparation prior to soldering or tinning.

7. Surfaces prepared by grit or glass bead blasting do not tin well and inhibit capillary action.

8. The 95% Sn - 5% Sb solder is too brittle at cryogenic temperatures to withstand aerodynamic loads.

9. Soldered surfaces when finished are rougher than the base metal; therefore, model surfaces that have had pressure tubes soldered in place will need cryogenic resistant coating to maintain a smooth, aerodynamic surface.

10. The reason for lower than expected lap shear strengths for both 50% Sn - 49.5% Pb - 0.5% Sb and 95% Sn - 5% Sb solders is not known so additional tests will be conducted.

11. Tensile specimens have been completed at this time using 50% Sn - 49.5% Pb -0.5% Sb and 95% Sn - 5% Sb solders.

Acknowledgement

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