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ALTERNATE CENTRAL RECEIVER POWER SYSTEM PROGRAM PHASE II

Midterm Technical Report. Volume 3—Materials Experiments

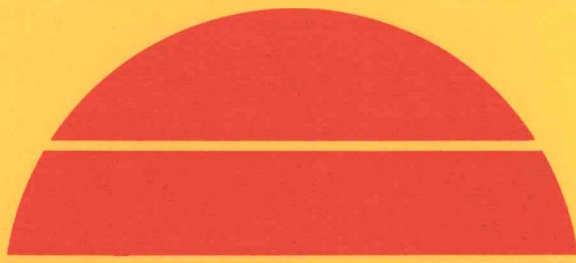
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General Electric Company
Energy Systems Programs Department
Schenectady, New York



U.S. Department of Energy



Solar Energy

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MIDTERM TECHNICAL REPORT
VOLUME III - MATERIALS EXPERIMENTS

**ALTERNATE
CENTRAL RECEIVER POWER
SYSTEM PROGRAM
PHASE II**

July 1980

PREPARED FOR
UNITED STATES DEPARTMENT OF ENERGY
(CONTRACT NO. DE-AC03-79SF10535)

GENERAL ELECTRIC COMPANY
ENERGY SYSTEMS PROGRAMS DEPARTMENT
SCHENECTADY, NEW YORK

GENERAL  ELECTRIC

VOLUME III

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PHASE II PROGRAM OVERVIEW

This Midterm Technical Report documents progress during the first year of the Alternate Central Receiver Power System Program Phase II (DoE Contract No. DE-AC03-79SF10535). The report consists of the following three volumes:

- Volume I - Commercial Plant Design Refinement
- Volume II - Sodium Test Receiver Experiment
- Volume III - Materials Experiments

BACKGROUND

The Phase II program is a follow-on program to the completed Conceptual Design of Advanced Central Receiver Power Systems - Phase I (DoE Contract No. DE-AC03-78ET20500) led by General Electric Corporate Research and Development.

During Phase I, parametric analyses were performed to select the preferred commercial scale (100 MWe) sodium cooled central receiver power plant. The reference concept selected utilizes an external cylindrical receiver with a surrounding field of heliostats. The plant loop schematic is shown in Figure 1 and an artist's concept of the plant in Figure 2. There are approximately three hours of storage, ground level steam generators, and a high efficiency reheat steam power conversion cycle.

A conceptual design was prepared for the reference plant concept and detailed cost estimates were calculated. A number of potential improvements to be examined during Phase II were identified, as were a number of Subsystem Research Experiments (SRE's). The SRE's were selected as those technical steps necessary for advancement of the sodium central receiver technology towards commercialization and addressed critical technical uncertainties.

The Phase II program is a logical extension of the Phase I effort and has as its objective "the near term application of sodium solar central receiver power plants for low cost electric power generation." The specific Phase II activities, shown graphically on Figure 3, include the following efforts:

- Performance of a receiver panel test at the Central Receiver Test Facility (CRTF)

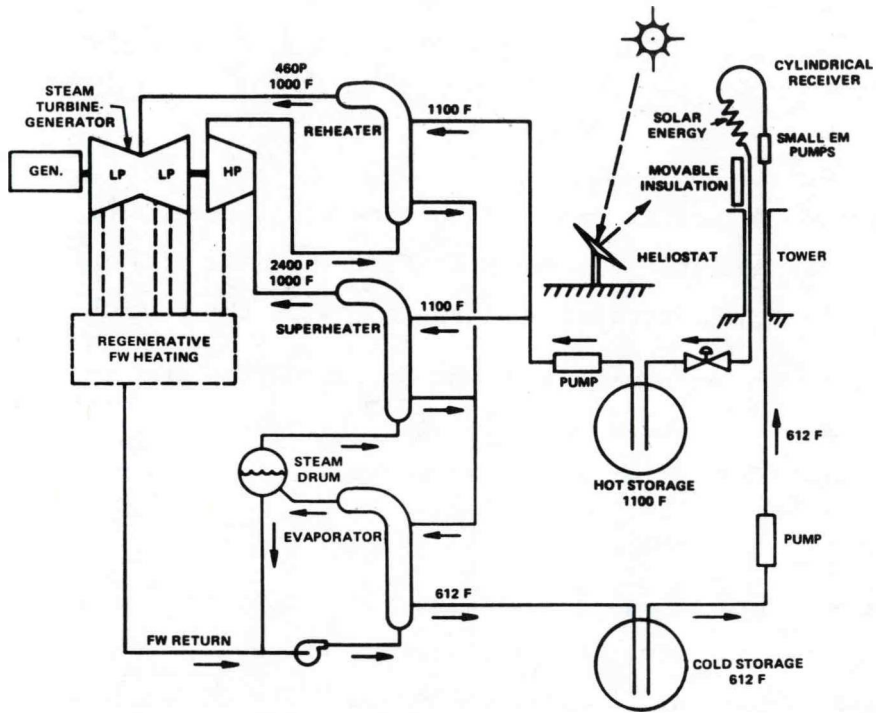


Figure 1. Plant Schematic

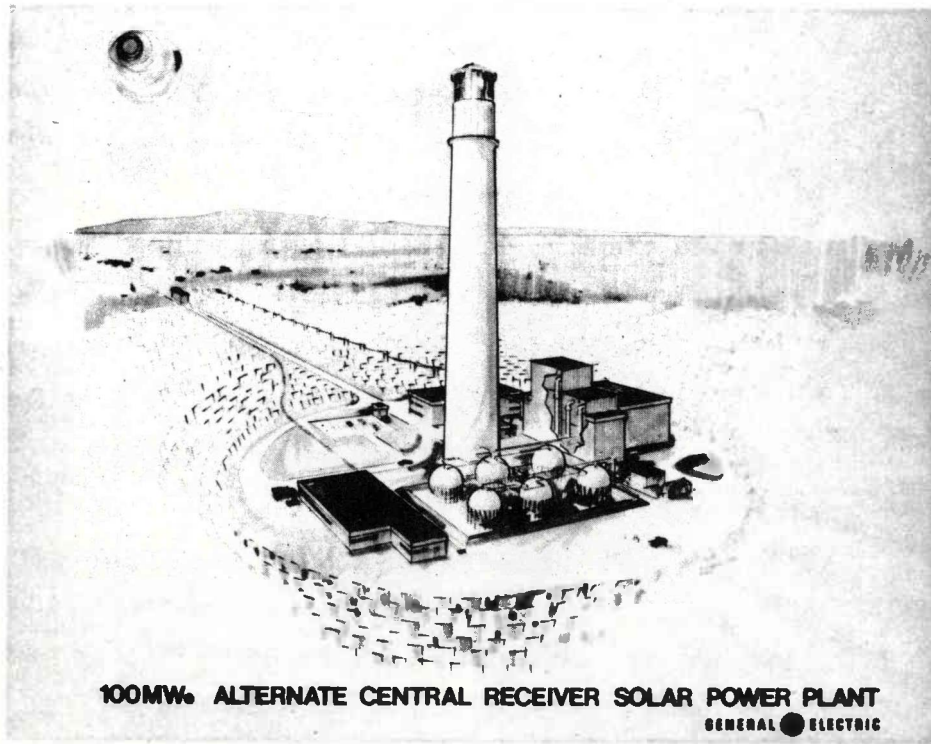
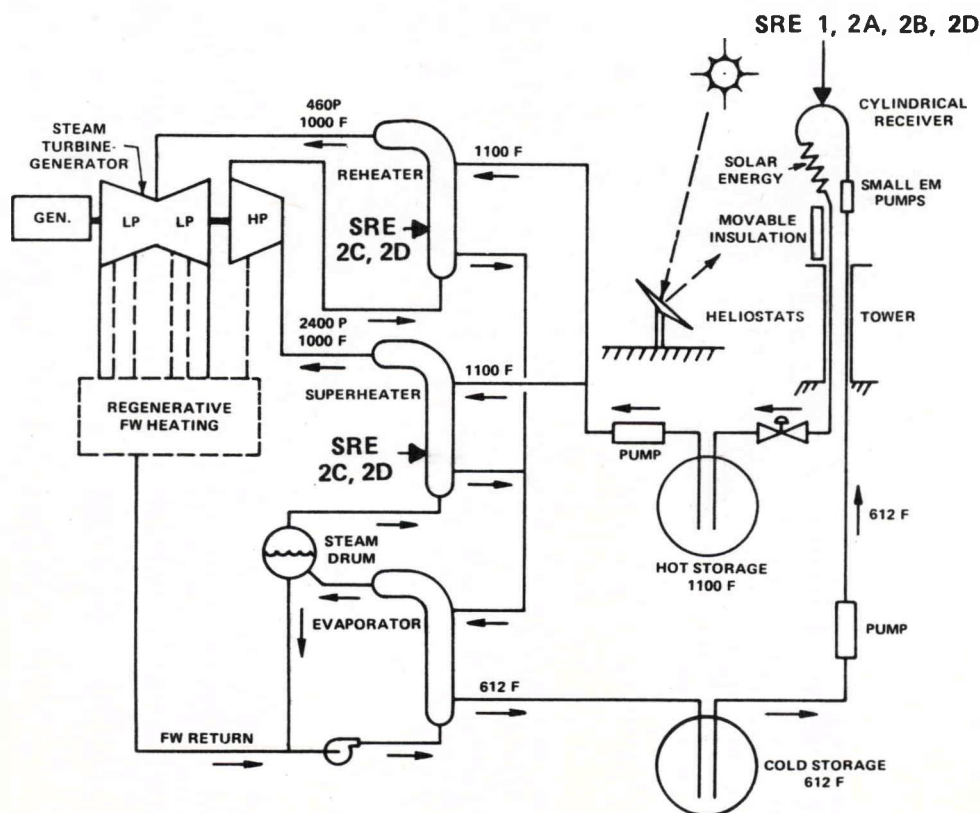


Figure 2. Plant Arrangement

SYSTEM ENGINEERING
AND ANALYSIS

SUBSYSTEM RESEARCH
EXPERIMENTS (SRE'S)

3



● SRE 1	ABSORBER PANEL TEST
● SRE 2	MATERIALS SRE'S
SRE 2A	PANEL FABRICATION DEVELOPMENT
SRE 2B	PANEL INSPECTION AND EVALUATION
SRE 2C	STRESS CORROSION AND FATIGUE
SRE 2D	FATIGUE CRACK GROWTH AND FRACTURE TOUGHNESS

Figure 3. Major Phase II Activities

- Performance of materials experiments and panel fabrication development
- Commercial plant design updates
- Development planning.

PROGRAM WORK PLAN

The Phase II program consists of the five tasks described below that extend over two years.

- Task 1 - Subsystem Research Experiments (SRE's)
Perform the necessary hardware development efforts to move sodium central receiver technology from conceptual design status to commercial demonstration status. Key efforts are design, fabrication, and testing of a Sodium Receiver Test Assembly (SRTA) shown in Figure 4 and the conduct of critical materials experiments.
- Task 2 - Commercial Plant Design
Perform a revivification of the conceptual design, based on improvements identified during Phase I. Near the end of the program, update the design to reflect knowledge gained during Phase II.
- Task 3 - Critical Module Design
Define the next step in plant commercial plant development by conceptualizing a large scale critical module configuration. Update the critical module concept near the end of the program to reflect knowledge gained during Phase II.
- Task 4 - Development Planning
Prepare an update of the Phase I development plan for solar sodium receiver technology near the end of Phase II to reflect the knowledge gained during Phase II.
- Task 5 - Program Management
Perform appropriate program management.

The work flow for accomplishing these tasks is shown on Figure 5 and the related schedule shown on Figure 6.

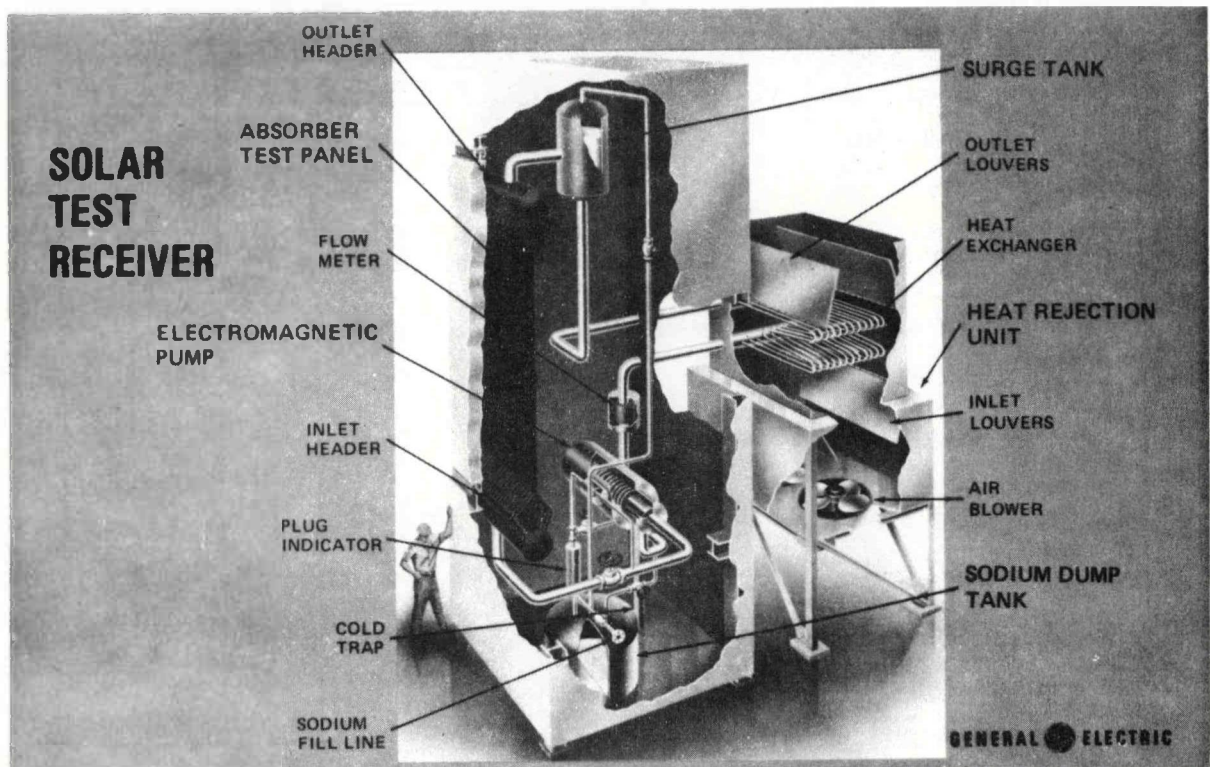
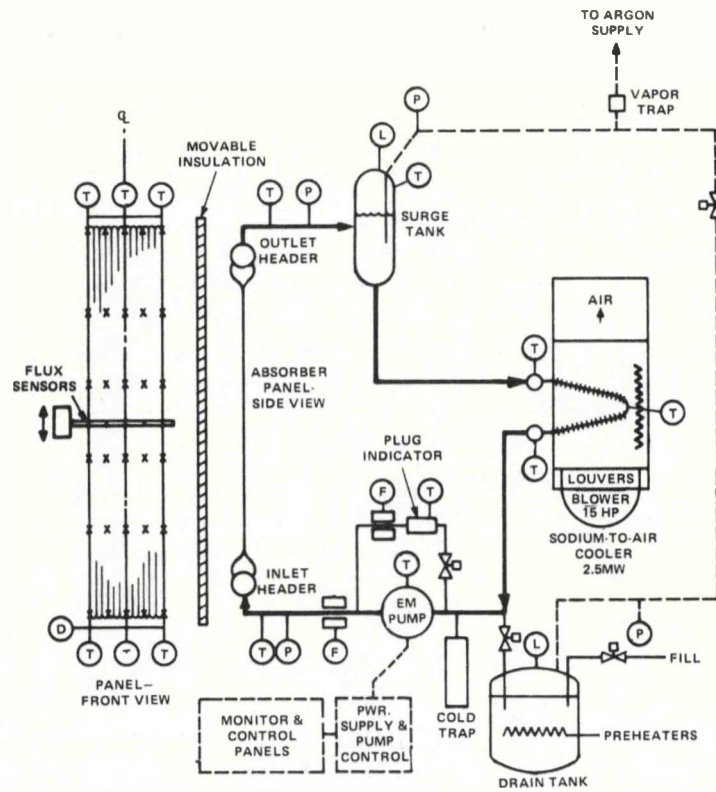


Figure 4. SRTA Schematic and Artist's Concept

ORGANIZATION

The Phase II program is being led by the General Electric Energy Systems Programs Department (ESPD). The transition of leadership from Corporate Research and Development (CRD) to ESPD is a normal activity for General Electric and represents the logical transition of a primarily R&D program into a primarily hardware and commercial application program. CRD played a major role in the plant design refinement task and ensured that a sound technical transition occurred. Kaiser Engineers, Incorporated of Oakland, CA was the Architect Engineer during Phase I and performed the storage tank design refinement described in Volume I of this report. The current organization is shown in Table 1.

TABLE 1

PROGRAM ORGANIZATION

<ul style="list-style-type: none"> ● GENERAL ELECTRIC COMPANY <ul style="list-style-type: none"> - ENERGY SYSTEMS PROGRAMS DEPARTMENT (ESPD) <ul style="list-style-type: none"> Program Management Systems Engineering Plant Integration - ADVANCED REACTOR SYSTEMS DEPARTMENT (ARSD) <ul style="list-style-type: none"> Liquid Metal Engineering Sodium Components Brazing Development ● FOSTER WHEELER DEVELOPMENT CORPORATION (FWDC) <ul style="list-style-type: none"> - Absorber Test Panel Fabrication ● PYROMET INDUSTRIES, INC. <ul style="list-style-type: none"> - Test Panel Brazing - Temporary Brazing Furnace
--

PROGRAM STATUS

As of April 1980, the Phase II program is focused on fabrication of the 2.5 MWth Sodium Receiver Test Assembly (SRTA). The design refinement of the 100MWe commercial plant was completed in October 1979 and the analysis and results are detailed in Volume I of this report.

The SRTA design has been completed and fabrication of the components are underway. The panel fabrication scheme (horizontal furnace braze) has been selected and fabrication of a large temporary brazing furnace is well underway.

The design and fabrication status of the SRTA is reported in Volume II.

Significant progress has been made in the development of the panel fabrication techniques and several materials test efforts are underway. The materials experiments are discussed in Volume III.

SECTION 1

INTRODUCTION

The materials experiments to be conducted during the Phase II Program consist of four Subsystem Research Experiments (SRE's) that address some of the major technology issues identified during the Phase I Program.

1.1 BACKGROUND

Materials performance in critical components has a direct and significant impact on the reliability and lifetime of critical components. In the sodium-cooled central receiver plant two of the most important components are the absorber panels, which are subjected to rather severe thermal transients during cloud passage and daily output cycles, and the steam generators, which will also experience daily load cycles. During Phase I, 593°C (1100°F) was selected as the operating temperature for the absorber panels and for both the reheater and the superheater modules of the steam generators. Incoloy 800 was identified as the reference material for these critical components. The details are reported in Appendix L of Reference 1. In general, Incoloy 800 was selected over 300 series stainless steel alloys for superheaters and reheaters, since those alloys are more susceptible to stress corrosion cracking in both chlorine-containing and caustic media. Incoloy 800 was selected for the absorber panels based upon its low cycle fatigue strength. A backup material (Inconel 617) was also identified.

Although Incoloy 800 has been widely used by industry, there is a need to provide additional data on stress corrosion/corrosion fatigue and fatigue crack growth/fracture toughness at the 593°C (1100°F) temperatures used in the solar plant. Generating this needed data forms an important part of the Phase II program.

The solar receiver is the only new component in the sodium heat transfer loop and fabrication development for the absorber panels was established during Phase I as a fundamental aspect of Phase II. As reported in Reference 1, thin-walled Incoloy 800 tubes are used in the absorber panels. Tube-to-tube joining is needed to provide a protective "light-tight" barrier for the support structure behind the panels and to provide the panels with structural rigidity and strength. Furnace brazing was selected as the fabrication scheme for the tube-to-tube joint. The

details of the panel braze had to be established early in Phase II, in order to allow confident fabrication of the absorber test panel described in Section 3 Volume II of this report.

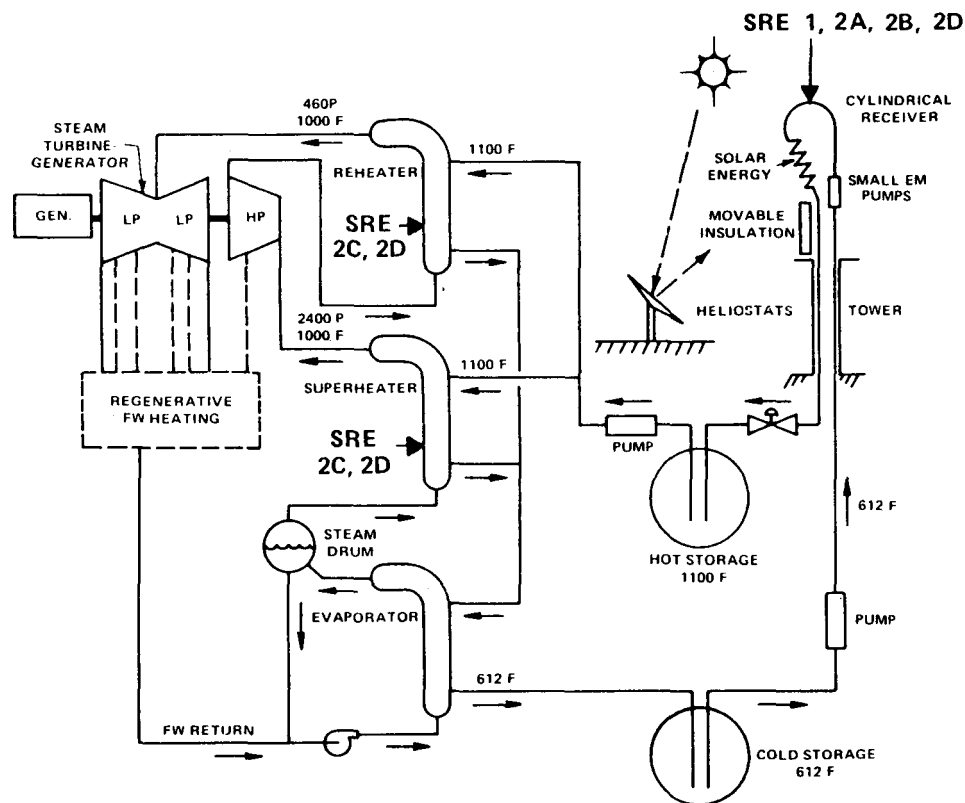
The materials experiments being performed during Phase II, identified as SRE's 2A-2D on Figure 1-1, address the issues described above. The Panel Fabrication Development SRE (2A) has been the largest materials effort during the first year of the program. Section 2 completely documents the brazing development effort. The results of SRE 2A have formed the basis for fabricating the active portion of the absorber test panel (SRE 1). The Panel Inspection and Evaluation SRE (2B) has not yet been performed. Preliminary scoping conducted as part of SRE 2A indicates that ultrasonic inspection techniques offer real promise as a non-destructive examination method for verifying the quality of the tube-to-tube braze.

Both SRE's 2C and 2D are longer term materials experiments that will not be fully documented until the final report of the Phase II program; however, interim progress summaries are provided in Sections 3 and 4, respectively.

SYSTEM ENGINEERING
AND ANALYSIS

SUBSYSTEM RESEARCH
EXPERIMENTS (SRE'S)

1-3



● SRE 1	ABSORBER PANEL TEST
● SRE 2	MATERIALS SRE'S
SRE 2A	PANEL FABRICATION DEVELOPMENT
SRE 2B	PANEL INSPECTION AND EVALUATION
SRE 2C	STRESS CORROSION AND FATIGUE
SRE 2D	FATIGUE CRACK GROWTH AND FRACTURE TOUGHNESS

Figure 1-1. Major Phase II Experiments

Section 2

ABSORBER PANEL FABRICATION DEVELOPMENT

Brazing was selected for the fabrication of solar absorber panels to be used in thin walled tube sodium cooled central receiver plants. The Incoloy 800 tubes used in the absorber panels have a tube wall thickness of 1.27 mm (0.050 inch), making fabrication by welding less reliable than by brazing, without a very extensive welding development program. Thin wall tubes were chosen to reduce thermal transient stresses and increase thermal performance. Incoloy 800 (Specification ASTM B407) was selected as the tube reference material because of its relatively good low cycle fatigue strength and resistance to environmental degradation (oxidation and sodium corrosion) at the maximum tube service temperature of approximately 621°C (1150°F).¹ Nickel and cobalt base brazing filler metals were selected for evaluation due to their relatively low cost, their good properties at the service temperature and their commercial availability. A reducing atmosphere (pure dry hydrogen) was chosen for the brazing furnace, rather than a vacuum atmosphere, due to the large size of both the commercial plant absorber panels and the test panel. The prototype/test panel is approximately 0.98 m by 6.10 m (3.2 feet by 20 feet) as shown in Figure 2-1. Only the central 4.57 m (15 feet) of this panel will be exposed to the solar flux, and so only this region is to be brazed.

2.1 SRE OVERVIEW

The principal objective of the brazing evaluation experiments was to select and develop a reliable brazing procedure with minimum complexity, providing acceptable mechanical properties for the absorber panels. Meeting this objective required that a reference and an alternate brazing filler metal and brazing procedure be selected, and that a reference and an alternate longitudinal tube/tube joint design be chosen.

The second objective addressed braze joint design. The precise joint strength requirements had not been established at the start of the project, necessitating the evaluation of several joint designs. The three joint designs evaluated (Figure 2-2) were tube to tube with no filler strip, triangular shaped filler strip (one side of the joint only), and hourglass (double concave lens) shaped filler strip. The

2-2

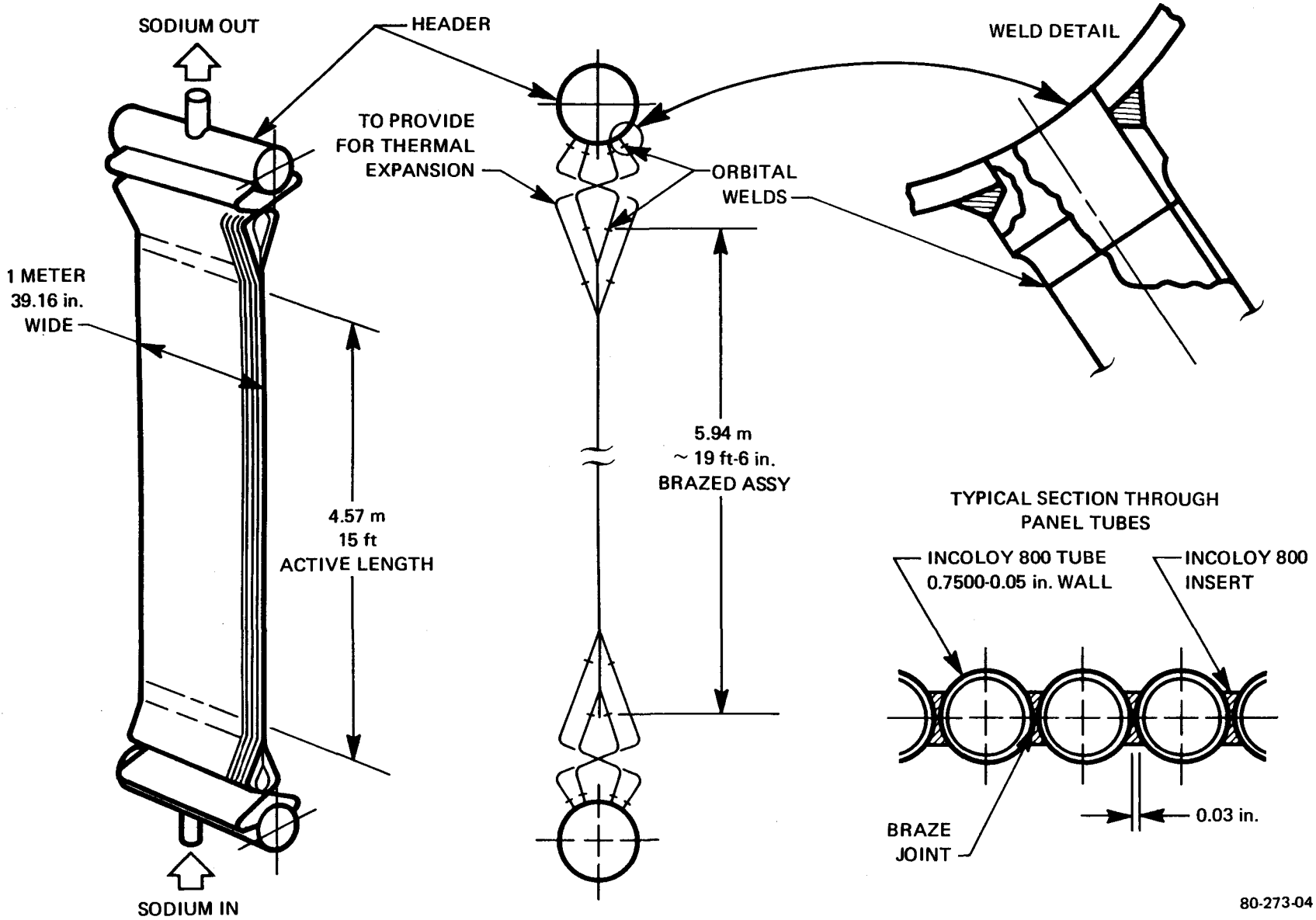


Figure 2-1. Solar Test Panel General Details

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Additional work is in progress to determine the effects of rebrazing and elevated temperature aging on properties for the preferred alloy and joint design. This work is also described.

2.2 EXPERIMENTAL PROCEDURE

2.2.1 HYDROGEN BRAZING FURNACE PARAMETERS

Pure dry hydrogen (99.999%) was selected as the brazing furnace atmosphere due to the 6.1 - 9.1 m (20 to 30 foot) length of the absorber panels to be built for test purposes and larger panels for commercial plants. Construction of vacuum furnaces for such large panels would be extremely difficult. The aluminum and titanium content of Incoloy 800, which are each specified to be between 0.15 and 0.60%, required that the hydrogen dew point at the furnace inlet be maintained at -62°C (-80°F) or lower. This minimized the amount of oxidation on heating since these oxides produced in brazing are not reduced by hydrogen at the brazing temperature. Several bell-type electrically heated braze furnaces, ranging from 0.30 to 1.22 m (1 to 4 feet) in diameter were used to investigate the effects of furnace size. In each case the hydrogen flow rate was controlled to produce at least ten furnace gas changes per hour during the brazing cycle after purging with argon or exothermic gas.

Furnace temperatures were controlled during the brazing cycle by continuously monitoring and recording the tube panel temperatures using contacting thermocouples located on at least three areas of the panel. Thermocouple locations included the thermal mass center and extremities of the panel, and were used to control the minimum heating rates and hold times of the slowest temperature response area.

2.2.2 PREPARATION OF PANEL BRAZING SAMPLE

Both nickel plated and unplated tubes were used, in order to determine whether plating was needed for improved wetting of the base metal by the filler metal. Improved wetting occurs by prevention of the formation of aluminum and titanium oxides on the joint surfaces. From the experimentation it was determined that Incoloy 800 tubing and strip required an electrolytic nickel plating to ensure uniform wetting by the brazing filler metal and consistent joint filling, even when using a high purity hydrogen atmosphere. All tubes and filler strip material were wiped with acetone on a nylon cloth before assembly and application of the filler metal.

Planar tube panels were assembled utilizing one of three types of tube to tube joint configurations:

1. Tube against tube with brazing filler metal added (Figure 2-3).

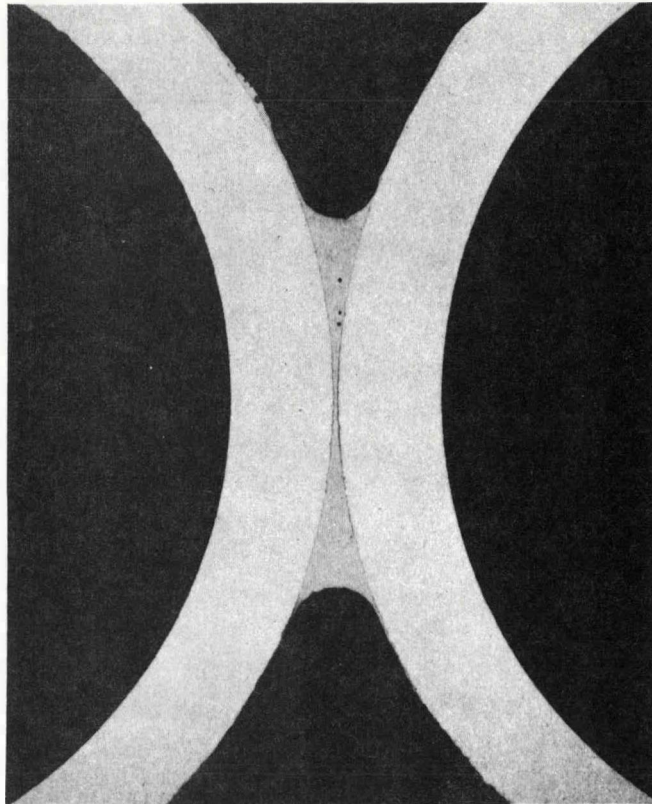


Figure 2-3. Tangent-to-Tangent Braze Geometry

2. Tube against tube with a triangular shaped stainless steel filler strip or powdered non-melting filler added on one side only (Figure 2-4).
3. A joint with a contoured hourglass (double concave lens) shaped Incoloy 800 filler strip between adjacent tubes as shown in Figure 2-5.

Details of the filler strips are shown in Figure 2-6. The side of the panel on which the triangular strip or non-melting powdered metal was added was back of the panel (i.e., the side not exposed to the solar flux).

The principal advantages of a filler strip contoured to fit the tube surface are increased joint area for added load-carrying capacity and decreasing joint gap, promoting the formation of a ductile, soft, single phase continuous microstructure rather than a multiphase microstructure with interconnected, hard, brittle phases. The use of a contoured filler strip such as the hourglass shape also reduces the geometrical stress concentration that exists between tubes, with only a small fillet of filler metal near the line of tube to tube contact. Tube to tube alignment is

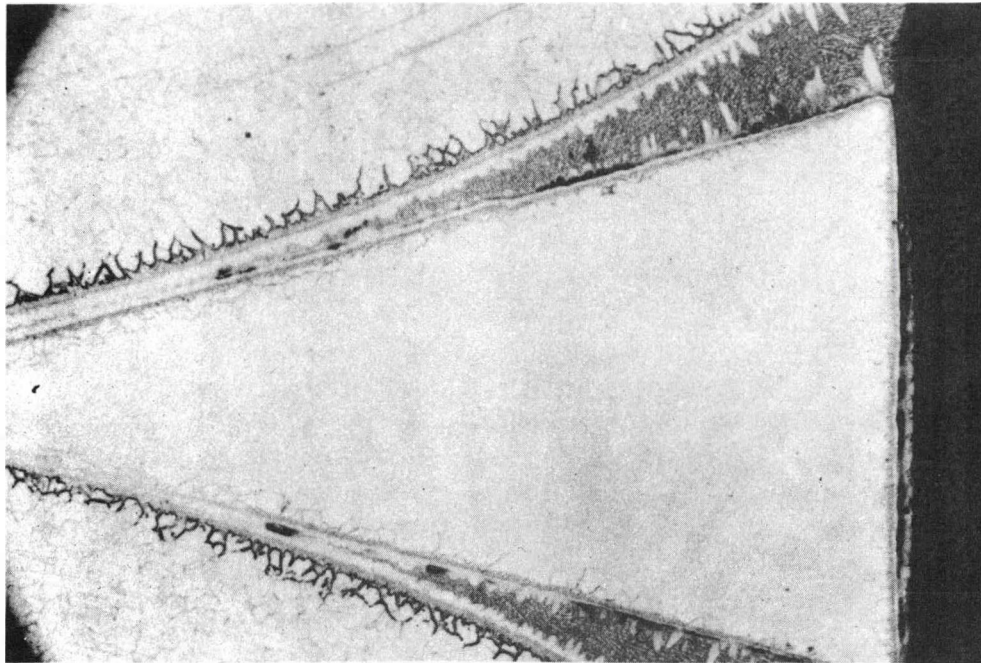


Figure 2-4. Typical Brazed Joint Using A Triangular Filler Strip Between Adjacent Tubes (30X Magnification)

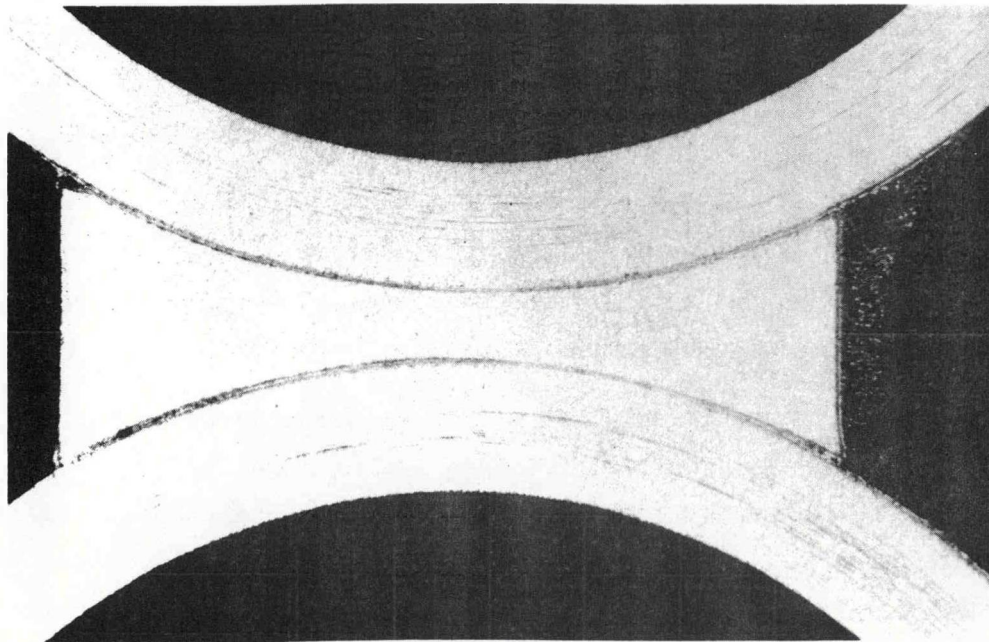


Figure 2-5. Typical Brazed Joint Using An Hourglass Shaped Filler Strip Between Adjacent Tubes (9.5X Magnification)

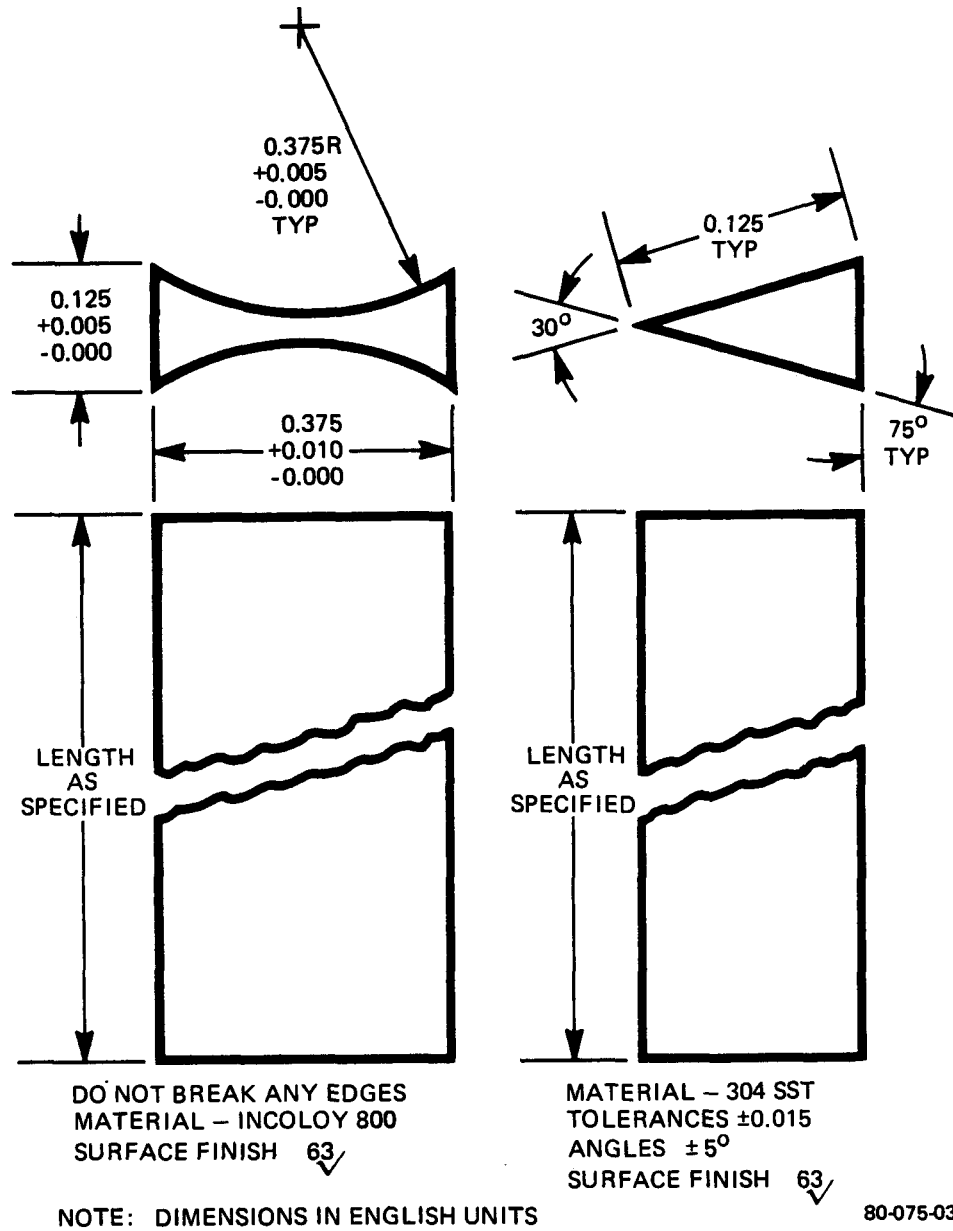


Figure 2-6. Details of the Hourglass and Triangular Shaped Filler Strips Used Between Adjacent Tubes

also simplified during assembly and fixturing. A narrow capillary path for the filler metal to flow through the joint is also provided and excess filler metal runoff is controlled by not having the large fillet present. A major disadvantage of using a filler strip is the added cost for the material, fabrication, plating and handling.

Several methods were used to apply the brazing filler metals, the most favorable methods dependent on the form of the filler metal and the joint configuration. Some of these techniques are discussed below.

Method #1 The panel was assembled horizontally and the brazing filler metal applied as a -140 mesh dry powder fillet to the upper side of the joint. The powder was subsequently cemented in place using a clear acrylic aerosol spray. This method was used both with and without a filler strip.

Method #2 The tubes were assembled using a brazing filler metal foil 0.76 mm (3 mils) thick on each side of an hourglass shaped strip between adjacent tubes.

Method #3 Pressure sensitive adhesive backed filler metal tape 0.76 or 1.52 mm (3 or 6 mils) thick was used, containing 4 to 6 percent binder. Approximately 25 percent excess tape was used in order to compensate for binder burn-out which can result in braze porosity.

Method #4 The tubes were assembled horizontally and water soluble cement, premixed with -140 mesh powder was extruded onto the upper side of the joint to form a fillet. The fillet width was varied from 4.76 to 6.35 mm (3/16 to 1/4 inch) with the use of a filler strip, and from 6.35 to 7.94 mm (1/4 to 5/16 inch) without a filler strip.

Method #5 An alternate to the above approach substituted an organic base cement for the water soluble cement.

Method #6 An organic base cement slurry was used which consisted of 20% by weight brazing filler metal powder and 80% non-melting powder fill. Variations of this method included the addition of a non-melting powder to form the fillet followed by sintering of the composite. Additional filler metal was then applied and allowed to infiltrate the joint during the brazing operation.

2.2.3 BRAZING ALLOYS EVALUATED

Fifteen brazing filler metals were selected for evaluation. Low, intermediate and high temperature braze alloys selected covered a range of braze temperatures from 999° - 1182°C (1830° - 2160°F). The brazing filler metals were evaluated on the basis of:

- wetting of base metal;
- joint penetration;
- joint filling;

- grain boundary attack;
- tube erosion;
- oxidation resistance;
- joint strength, ductility and hardness;
- post braze tubing mechanical properties;
- rebraze characteristics;
- furnace atmosphere;
- joint microstructure;
- filler metal porosity;
- need for flux, plating, and stop-off compounds;
- relative costs;
- product form, including foil, tape, paste, slurry, and dry powder bonded after application.

The excessive grain growth observed for Incoloy 800 at 1121°C (2050°C) and above could result in a reduction in mechanical properties below the specified minimum values. Therefore, all intermediate and high temperature braze alloys (those with braze temperatures of 1121°C (2050°F) and above were evaluated in a screening manner only.

The fifteen brazing filler metals were grouped for evaluation in three brazing temperature ranges, as indicated in Table 2-1. This facilitated initial scoping of brazing temperature effects with fewer furnace heats. The majority of alloys investigated contain chromium to provide an improvement in oxidation resistance. Three cobalt containing alloys were chosen for their high elevated temperature strength, and two manganese containing filler metals were chosen for their improved ductility. The composition of these filler metals is listed in Table 2-2.

Powdered filler metals were investigated to evaluate the possibility of using a non-melting powder to increase the joint width without increasing the size or amount of brittle phases and dendritic grains. The use of a powdered filler would be less costly than a machined filler strip. Two -140 mesh powder alloys were evaluated -- a nickel/chromium alloy, and a nickel/chromium alloy with boron and silicon to aid (subsequent) braze filler metal infiltration. The compositions of these alloys are listed in Table 2-3, along with the compositions of the Incoloy 800 tubing and Incoloy 800 and 304 Stainless Steel filler strips. Type 304SS was used as a filler strip material on initial brazing samples for expediency; however, although satisfactory on short tube samples, the thermal expansion coefficient mismatch with the Incoloy 800 caused the strips to warp when used in longer tube samples.

Table 2-1

BRAZING FILLER METAL CANDIDATES

<u>Alloy Code</u>	<u>Manufacturer Designation</u>	<u>A.W.S. Type</u>	<u>Recommended Brazing Range °C(°F)</u>
<u>Low Temperature Alloys Brazed at 999-1049°C (1830-1920°F)</u>			
NB 50	Microbraz 50	BNi-7	982 - 1093 (1800 - 2000)
NB 51	Microbraz 51	--	982 - 1093 (1800 - 2000)
NB LM	Microbraz LM	BNi-2	1010 - 1177 (1850 - 2150)
AD 770*	Amdry 770	BNi-2	1040 - 1175 (1900 - 2150)
AD 936	Amdry 936	--	968 - 1010 (1775 - 1850)
AD 939	Amdry 939	--	996 - 1038 (1825 - 1900)
<u>Intermediate Temperature Alloys Brazed at 1116-1127°C (2040-2060°F)</u>			
NB 135	Microbraz 135	BNi-4	1135 - 1204 (2075 - 2200)
AD 914	Amdry 914	--	1052 - 1191 (1925 - 2175)
AD 915	Amdry 915	--	1135 - 1204 (2075 - 2200)
AD 915E	Amdry 915E	--	1121 - 1177 (2050 - 2150)
<u>High Temperature Alloys Brazed at 1171-1182°C (2140-2160°F)</u>			
NB 125	Microbraz 125	BNi-1	1066 - 1204 (1950 - 2200)
NB 150	Microbraz 150	--	1066 - 1204 (1950 - 2200)
NB 160	Microbraz 160	--	1149 - 1204 (2100 - 2200)
NB 210	Microbraz 210	BCo-1	1149 - 1232 (2100 - 2250)
AD 100	Amdry 100	BNi-5	1135 - 1204 (2075 - 2200)

*All filler metals were used as -140 mesh powder except AD 770, which was also used as 0.64 mm (0.0025 inch) thick foil and as 0.076 mm (0.003 inch) thick tape.

Table 2-2

COMPOSITION OF EVALUATED SOLAR PANEL

BRAZE FILLER METALS

<u>Alloy Code</u>	<u>Ni</u>	<u>Cr</u>	<u>B</u>	<u>Si</u>	<u>C</u>	<u>P</u>	<u>Cu</u>	<u>Co</u>	<u>Re</u> ¹	<u>W</u>	<u>Mn</u>	<u>Fe</u>
NB 50	Bal.	14.0	--	--	0.08	10.0	--	--	--	--	--	--
NB 51	Bal.	25.0	--	--	--	10.0	--	--	--	--	--	--
NB LM	Bal.	7.0	3.1	4.5	.06	--	--	--	--	--	--	3.0
AD 770	Bal.	7.0	2.8	4.5	--	--	--	--	--	--	--	3.0
AD 936	Bal.	--	0.9	6.0	--	--	4.0	--	.03	--	19.0	--
AD 939	Bal.	--	0.5	6.0	--	--	4.0	--	.03	--	19.0	--
NB 135	Bal.	19.0	1.9	3.5	--	--	--	--	--	--	--	--
AD 914	Bal.	--	3.0	4.5	0.03	--	--	20.0	--	--	--	--
AD 915	Bal.	13.0	2.75	4.0	0.02	--	--	--	--	--	--	4.0
AD 915E	Bal.	13.0	2.8	4.0	0.02	--	--	20.0	--	--	--	4.0
NB 125	Bal.	14.0	3.0	4.5	0.70	--	--	--	--	--	--	4.5
NB 150	Bal.	15.0	3.5	--	--	--	--	--	--	--	--	--
NB 160	Bal.	11.0	2.25	3.5	0.05	--	--	--	--	--	--	3.5
NB 210	17.0	19.0	0.8	8.0	0.40	--	--	Bal.	--	4.0	--	--
AD 100	Bal.	19.0	--	10.0	0.03	--	--	--	--	--	--	--

Table 2-3

COMPOSITION OF SELECTED SOLAR PANELS ALLOYS

NON-MELTING POWDERS FOR FILL

<u>Alloy Code</u>	<u>Manufacturer Designation</u>	<u>Ni</u>	<u>Cr</u>	<u>B</u>	<u>Si</u>	<u>Mn</u>
NG 108	Nicrogap 108	Bal.	15.0	0.2	0.75	7.0
NG 116	Nicrogap 116	Bal.	20.0	-	-	-

TUBE AND FILLER STRIP ALLOYS

	<u>Ni</u>	<u>Cr</u>	<u>Si</u>	<u>C¹</u>	<u>Cu</u>	<u>S</u>	<u>Ti</u>	<u>Al</u>	<u>Mn</u>	<u>Fe</u>
Incoloy 800	30.0 min	19.0 min	1.0	—	0.75 max	0.015 max	0.15 min	0.15 min	1.5 max	39.5 min
	35.0 max	23.0 max		0.10 max			0.60 max	0.60 max		
	<u>Ni</u>	<u>Cr</u>	<u>Si</u>	<u>C</u>	<u>N</u>	<u>S</u>	<u>P</u>	<u>Mn</u>	<u>Fe</u>	
Stainless 304	8.00 min	18.00 min	1.00 max	0.08 max	0.10 max	0.030 max	0.045 max	2.00 max	Bal.	
	10.50 max	20.00 max								

¹ Carbon content of 0.07-0.08% preferred to control grain growth during brazing.

The carbon content of the Incoloy 800 purchased for this investigation was selected to be near the upper end of the allowable composition range in order to minimize grain growth of the tubes during brazing. Grain growth would reduce the mechanical properties, especially ductility, to unacceptable levels. Increasing the carbon content from 0.02 to 0.7% has been shown under certain conditions to increase the recrystallization temperature of Incoloy 800 by approximately 100°C (180°F) in the range of brazing times and temperatures investigated [fifteen minutes at temperatures typically between 982 and 1093°C (1800 and 2000°F)].²

2.2.4 BRAZE SAMPLE CONFIGURATIONS EVALUATED

For the initial screening of the braze alloys, samples 76.2 mm (three inches) long and three tubes wide were used. As a number of alloys were eliminated from the test program, samples 152 and 305 mm (6 and 12 inches) long and ten to twenty-four tubes wide were brazed with NB 51, NBLM and AD 939 to determine the effect of panel warping due to eccentric joints, and the effect of filler metal runoff due to vertical joint orientation. NB 51 was eliminated from further evaluation due to its low strength properties as discussed later in Section 3.2.2. After the two most promising brazing filler metals were selected (NBLM and AD 939), panels 1.2 m (48 inches) long and three tubes wide were brazed with each filler metal, both with and without filler strips, to determine which of the two alloys was most suitable for panel fabrication. After selection of the preferred filler metal (AD 939) two panels, 1.2 meters (48 inches) long and ten tubes wide, were brazed. These panels were fabricated with and without hourglass filler strips.

Joint clearances were relatively easy to control considering the length of samples used. However, in order to evaluate the effects of greater joint clearances that might be encountered during full size panel fabrication, joints with 0.51 and 1.27 mm (0.020 and 0.050 inch) clearances were fabricated and brazed using the preferred AD 939 and NBLM filler metals selected.

2.2.5 METHODS OF FILLER METAL WETTING AND FLOW CONTROL

As discussed in Section 2.2.2 electrolytic nickel plating of both the tube and filler strip was determined to be necessary to obtain uniform wetting of the I800 tubing by the brazing filler metal, and to obtain consistent joint filling. A sulfamate type bath was specified and a plating thickness of 0.0107 to 0.0152 mm (0.0004 to 0.0006 inch) was selected. The specification used was AMS 2424. Specification AMS 2403 could be used as an alternate. A limited number of samples were

also plated using selective electro-deposition, i.e., application locally by means of a brush, according to Military Standard 865A (USAF).

As a possible low-cost alternative to nickel plating a proprietary commercial vaporizing flux (Vapo-Flux from Wall-Colmonoy Corp.) was tried on several unplated samples made with brazing filler metal pastes NBLM and NB 51. The flux was applied to the pre-alloyed joint in amounts ranging from one to three drops per 25 mm (one inch) of joint. The results were reasonably satisfactory, but substantially inferior to joints obtained with plated samples.

In order to prevent excessive wetting of the tube surfaces beyond the joint and, therefore, depletion of the filler metal available to fill the joint, and to prevent inadvertent brazing of the panel to the fixturing, several stop-off compounds were evaluated. The two compounds selected as best suited, boron nitrate or Microbraz Green (Wall-Colmonoy Corp.) were applied by brush painting after filler metal application and masking with plater's tape. Special care was taken to prevent any stop-off compound from coming into contact with the filler metal.

2.2.6 FURNACE BRAZING PROCEDURES

Both vertical and horizontal panel orientation in a brazing furnace were considered and initial brazes were performed in both orientations. After jiggling and application of braze filler metal and stop-off compound, the panel and fixturing were loaded into the furnace muffle. Due to the length of the joint and the consequent large volume of filler metal per joint, leveling of the panel was necessary during horizontal brazing in order to maintain equal size fillets along the length of the panel. During vertical tube brazing, the excess filler metal was allowed to flow to the lower end. Brazing of the panel in a diagonal orientation was performed with the tube axis horizontal and the panel inclined at an angle of 45 to 60 degrees from the horizontal. This orientation also required leveling of the tubes to maintain uniform size fillets.

Two primary types of fixturing were investigated to produce, (1) a constant joint clearance, or (2) a self-closing joint which resulted in no clearance (base metal contact after brazing). The constant joint clearance samples were fabricated from tubes 305 mm (twelve inches) in length or less, and were joined by tack welding the ends of adjacent tubes. The short tube self-closing joints were fixtured by tying the tube bundle together with molybdenum wire. The molybdenum wire draws

the bundle tight since its coefficient of thermal expansion is less than that of Incoloy 800. Longer tube joints were gravity fixtured for self-closing by using an inclined steel plate hearth with a straight bar fastened along the lower edge. The lowest tube of the panel assembly rested against this bar. Weighted bars were placed in contact along the length of the top tube, and also across the tube face of the panel perpendicular to the tubes. For the ten tube panel, the distributed weight of the bars parallel and perpendicular to the tubes were each approximately 4.5 kg per 0.3 m (10 lbs. per foot) of tube length.

After the furnace muffle was sealed and leak-checked, it was purged with argon or exothermic gas. Hydrogen purging was then initiated at a flow rate (depending on muffle size) which would provide between ten and forty volume changes per hour during the brazing cycle, including the heating and cooling periods. The entire volume of gas in the furnace was therefore changed forty times per hour.

As previously stated, the furnace hearth was a steel plate. Several kinds of ceramic plates were evaluated as alternate hearth materials which could be planar reference surfaces for the panel during brazing. Use of these ceramic materials was not adopted because of their excessive outgassing during brazing. Outgassing occurred even when the muffle was given a prior bake out at a temperature below the brazing filler metal solidus temperature, and when the hydrogen flow rate was increased to yield up to ninety-five volume changes per hour. The results of these attempts using ceramics can be seen in Figure 2-7. The filler metal surface has a beaded appearance, indicating poor flow.

During the brazing cycle, a temperature equalizing period was incorporated in order to minimize liquation on heating from the solidus to the liquidus temperature. The equalizing temperature was maintained approximately 6°C (10°F) below the solidus for the particular brazing filler metal tested. This also minimized heating time from the solidus to liquidus.

The panel heating and cooling rates obtained varied with the size of the panel being brazed, which in turn, determined the furnace size used. In order to maintain similar heating and cooling rates, the small furnaces were heated by bringing the heating elements to the equalizing temperature around the muffle, whereas the larger furnaces were heated by lowering the preheated elements over the muffle.

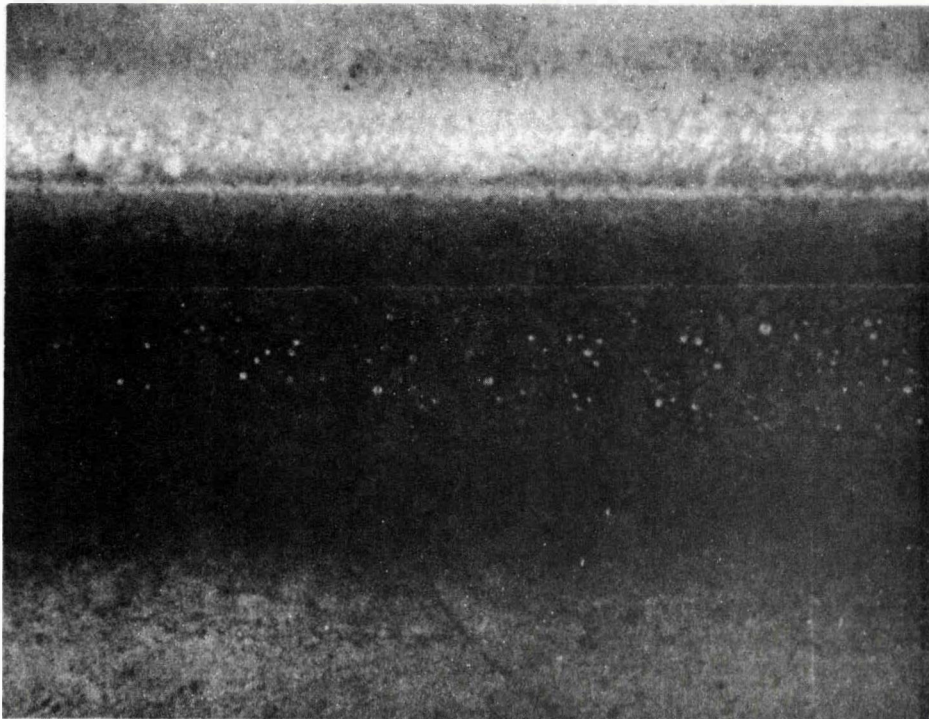


Figure 2-5. Example of Beaded Filler Metal Surface After Brazing with Ceramic Furnace Internals.

Brazing was performed in one of three temperature ranges, depending on the filler metals brazed in the same furnace heat. These basic temperature ranges were 999-1049°C, 1116-1127°C, and 1171-1182°C (1830-1920°F, 2040-2060°F, and 2140-2160°F). The brazing time was considered as starting when the lowest temperature area of the panel was 11°C (20°F) above the minimum temperature for the range. Brazing was continued for 10-12 minutes. One braze assembly was given a twenty-minute duration to evaluate the effect of brazing time on joint strength.

2.2.7 POST-BRAZE EXAMINATIONS AND TESTING

After brazing, each sample was visually examined to establish the surface appearance of the brazing filler metal, including base metal wetting, runoff, flow-through, discoloration, and joint contour (fillet size). Photomicrographs were taken of all typical joints and any unusual aspects of each joint type. Microscopic examination was also made of most combinations in order to determine the general microstructure of the base and filler metals, and to detect possible filler metal porosity, cracking, or incomplete filling. The tube and filler strips were also

examined for erosion, grain boundary penetration and grain growth.

The initial visual and metallographic examination of brazed samples, produced during the extensive screening tests with all alloys, showed that a number of combinations were clearly unacceptable. The remaining samples from this group and subsequent groups were subjected to a series of mechanical tests, including micro-hardness, longitudinal bend, ambient and elevated temperature tensile and shear. Testing still in progress at the time of writing includes fatigue, at ambient and elevated temperature, and tensile, shear and bend tests on thermally aged or re-brazed samples. The loading methods used for the tensile, shear, longitudinal bend, and transverse bend tests are unique, and require special fixtures described later. The pull-pull type (non-reversed loading) fatigue tests in progress utilize the same fixtures and sample configurations as for the tensile, shear and bend tests.

2.3 RESULTS AND DISCUSSION

2.3.1 RESULTS FOR VARIOUS BRAZING PROCEDURES

Test results for various tube-panel orientation, plating/wetting, brazing temperature, fixturing, joint design, and filler metal forms are discussed below.

2.3.1.1. Panel Orientation

For panel sections brazed in the vertical orientation without the use of a filler strip, the fact that only line contact existed between adjacent tubes resulted in unacceptable amounts of excess filler metal runoff. When brazed in the vertical orientation with a filler strip, the excess runoff still existed but was not as severe. An example of the runoff condition is shown in Figure 2-8. The remaining length of the joint, which was then depleted in filler metal as a result of the runoff, had an insufficient filler remaining for adequate thermal fatigue strength. Horizontal brazing was therefore pursued.

A vertical or horizontal furnace large enough to fabricate a prototypic solar test panel of the 6.2 m (20 ft) length was not available. Any of the existing furnaces would have to be modified. Since the horizontal position was considered advantageous, it was decided to continue further development work only in the horizontal orientation. This decision avoided the severe runoff problem with the vertical orientation, and also allowed the use of the preferred self-closing joint with weights for fixturing.

2.3.1.2. Base Metal Plating

Brazing filler metal wetting and joint flow-through characteristics were evaluated on unplated tubing for several of the filler metals which showed acceptable brazability when used with the nickel plating described previously. The results of this evaluation were poor, in that wetting of the Incoloy 800 was sporadic and inconsistent, and flow-through was very minimal. An exception to these results was shown by NB 51, which had satisfactory wetting and flow; however, the strength of this phosphorous-containing filler metal was relatively low, as described later. The visual appearance of unplated tubing joints made with NB LM, which demonstrated the unsatisfactory behavior typical of the majority of the alloys studied, is shown in Figure 2-9 and 2-10. A joint made with NB 51 is included for comparison purposes. Attempts to use a vaporizing flux, as noted previously, in lieu of nickel plating did not enhance wetting of the Incoloy base metal sufficiently to substitute for plating.

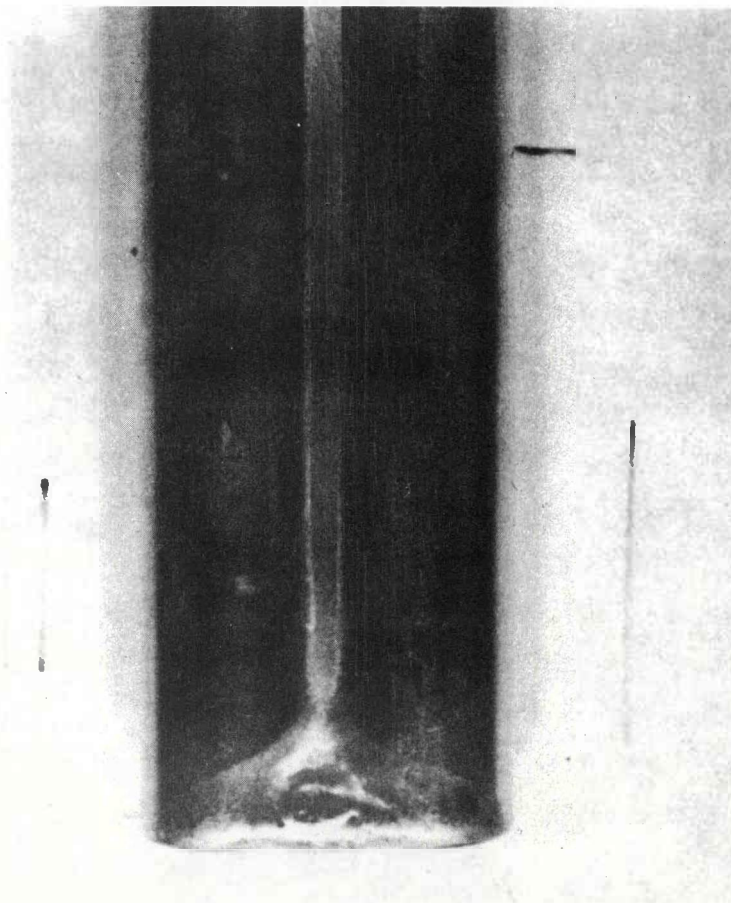


Figure 2-8. Typical Filler Metal Run-Off From Joint Brazed in the Vertical Orientation.

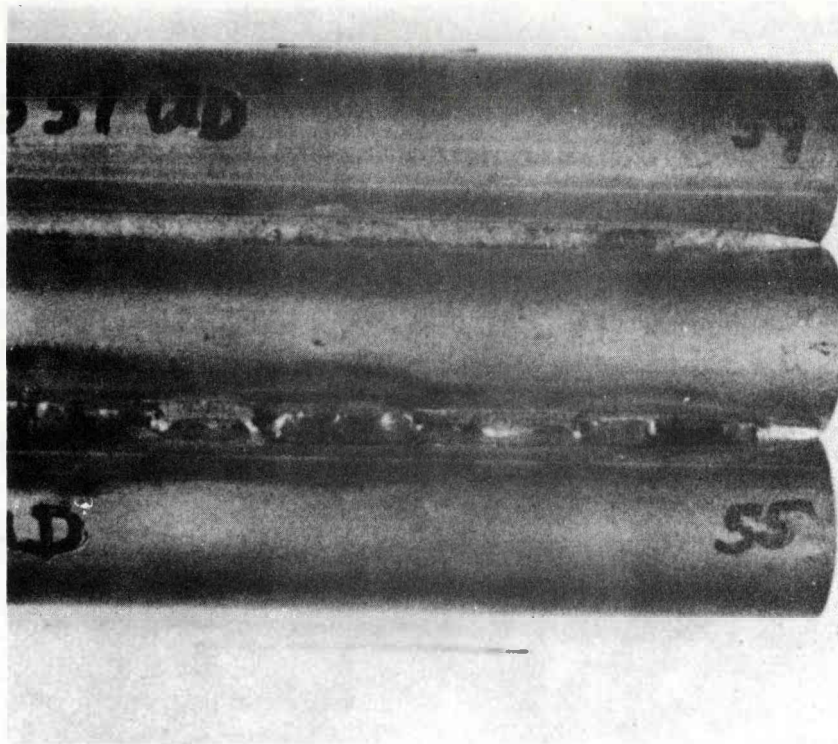


Figure 2-9. Front (Alloyed) Side of Joint Brazed with Unplated Incoloy 800 Tubing Using Filler Metals NB51 (Top) and NB LM (Bottom)

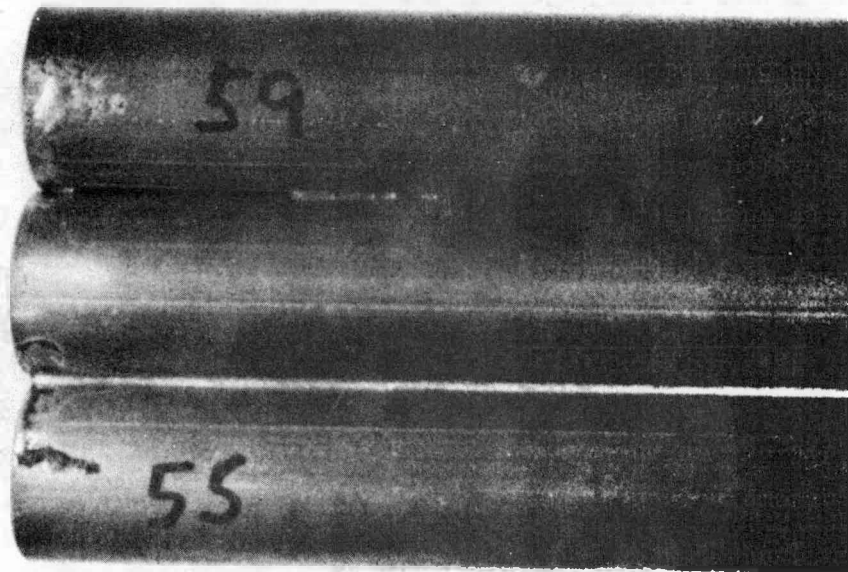


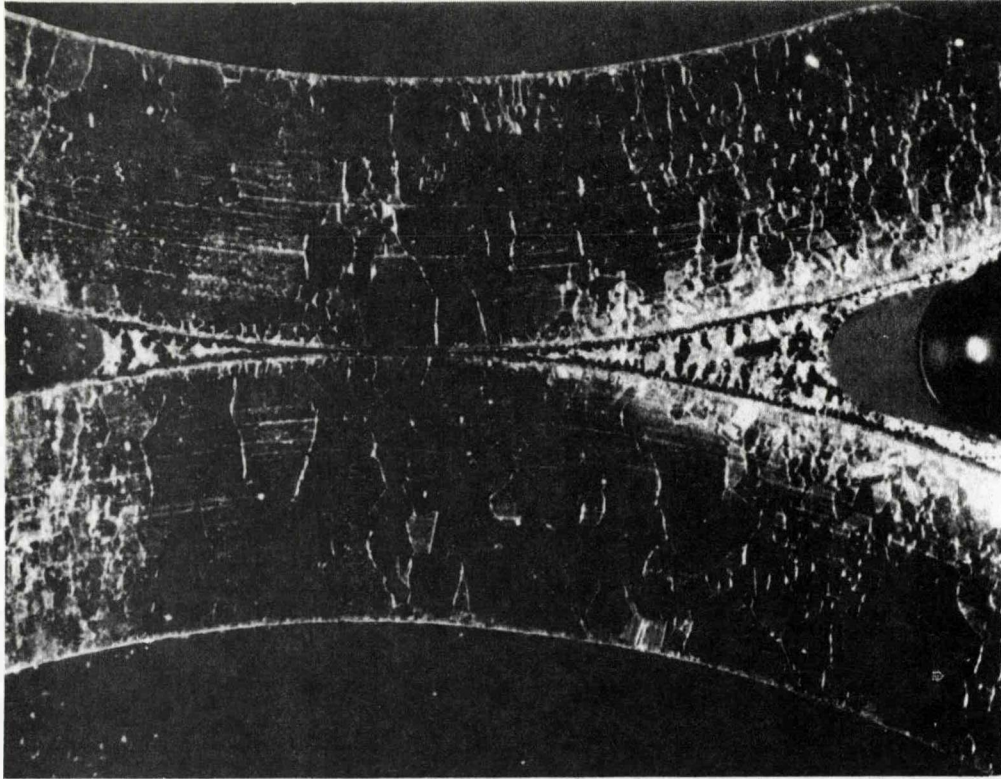
Figure 2-10. Back (Unalloyed) Side of Joint Shown in 3-2(a) Using Filler Metals NB51 (Top) and NB LM (Bottom)

Braze metal wetting, furnace brazing and subsequent mechanical tests of tube specimens were successful when electrolytic nickel plating of all metal surfaces was performed to a controlled thickness of 0.0107 to 0.0152 mm (0.4 to 0.6 mils). A key requirement in nickel plating is the adhesive quality of the nickel plate to the tube material. On selected samples adhesion was determined by scraping the surface, or shearing with a sharp knife or razor through the plating to the base metal. Specimens were also clamped in a vise and the projecting portion bent severely and checked for separation of the plating. Specimens can also be bent back and forth until rupture occurs. If the edge of the ruptured plating can be peeled back or if separation between the plating and the base metal can be seen at the point of rupture when examined at four diameters magnification, adhesion is not satisfactory. Satisfactory nickel plating was obtained under the controlled conditions described above.

2.3.1.3 Brazing Temperature

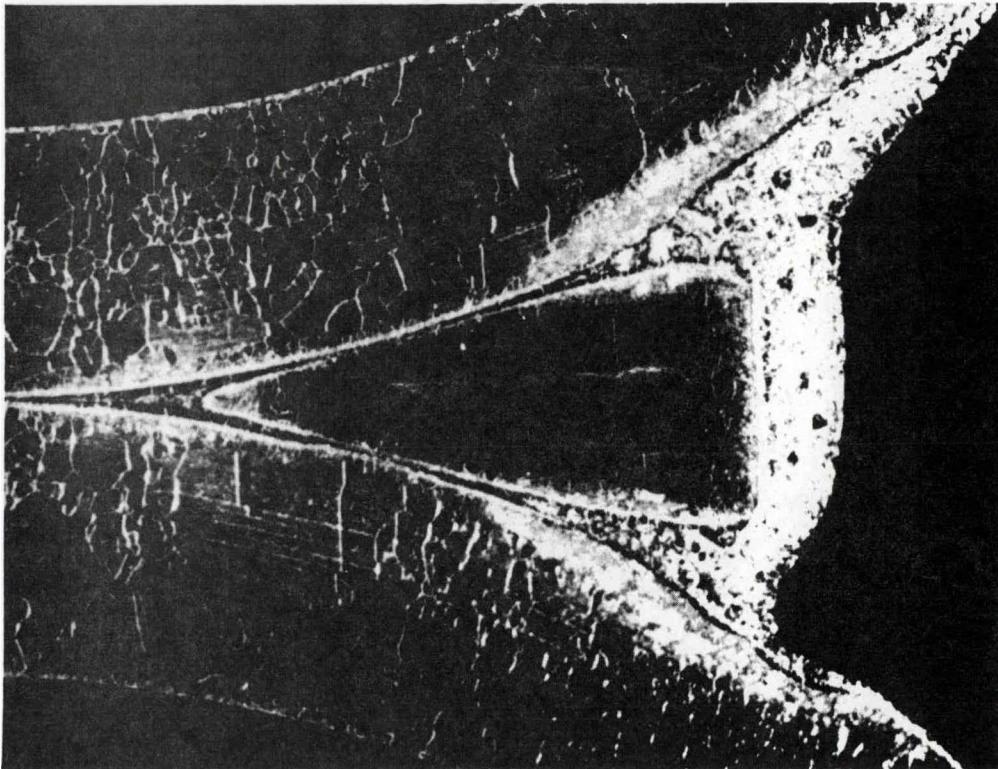
Braze temperature was found to be a critical parameter in obtaining a strong ductile braze. In evaluating the braze samples made with all of the filler metals listed in Table 2-1, it was readily apparent from the metallography of the specimens that the tubing grain growth was excessive after brazing at 1177°C (2150°F). This typical result is shown in Figure 2-11, where it can be seen that there are only two or three grains across the tube wall. [Note: The brazing development work to date has been performed on 1.65 mm (0.065 mil) wall thickness due to the unavailability of prototypic 1.27 mm (0.050 mil) wall thickness tubing, which is being custom drawn for the test panel.] Both tube and filler strip erosion by the filler metal was high for all panel samples brazed at 1177°C, as illustrated typically for NB 125 in Figure 2-12. Erosion or grain boundary penetration more than approximately two grains deep was considered unacceptable for test panel tube wall thickness of 1.27 mm. The effects of erosion and grain boundary diffusion on Incoloy 800 by three of the filler metals evaluated in this work (BNi-1, BNi-2 and BNi-5) have been characterized previously.³ An example of base metal carbide precipitation and grain boundary decoration present after brazing at 1177°C is shown in Figure 2-13.

The filler metals brazed at the intermediate temperature of 1120°C (2050°F) resulted in much less erosion and grain boundary penetration than those brazed at 1177°C. Essentially, no base metal grain growth occurred during brazing at 1120°C.



80-075-08

Figure 2-11. Typical Cross-Section of Joint Brazed at 2150°F (1177°C) for Ten Minutes Showing Excessive Tubing Grain Growth (19X Magnification)



80-075-09

Figure 2-12. Typical Cross-Section of Joint Brazed at 2150°F (1177°C) for Ten Minutes Showing Excessive Tubing Erosion (19X Magnification)

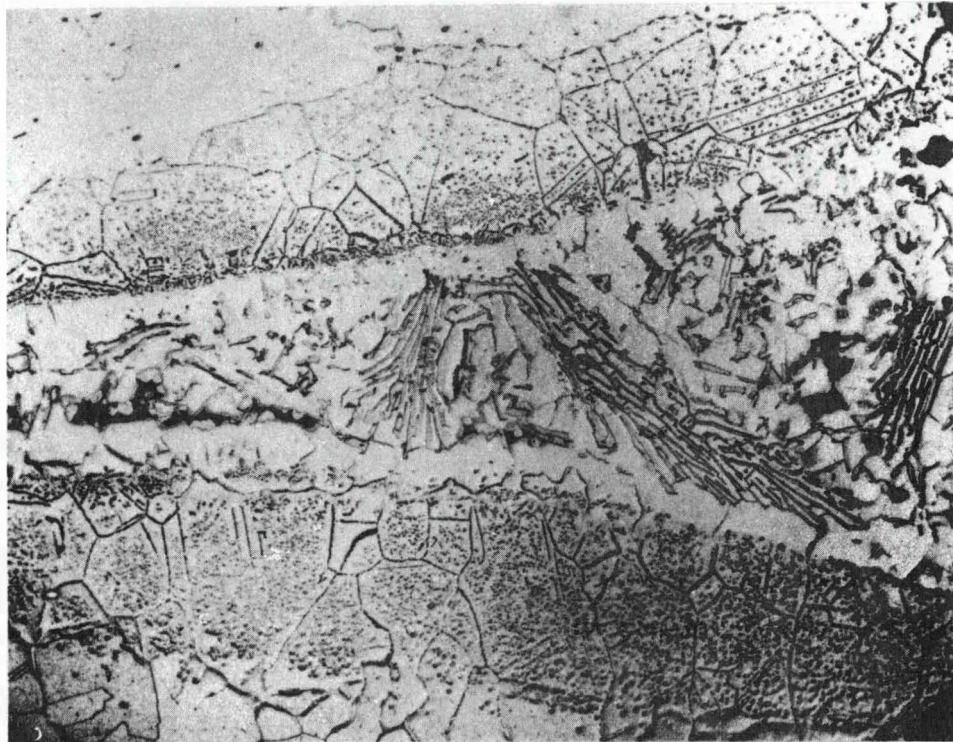


Figure 2-13. Example of Base Metal Carbide Precipitation After Brazing at 1177°C (2150°F) for Ten Minutes (30X Magnification)

Due to the fact that very good brazability results were obtained with at least three of the lower temperature range filler metals (NB LM, NB 51 and AD 939), sample preparation and testing of filler metals requiring the intermediate temperature of 1177°C was discontinued. The elimination of these filler metals was further substantiated by data showing that Incoloy 800 base metal strength decreases rapidly with increased temperature above 1093°C (2000°F). Comparatively modest changes were observed however after brazing at 1016°C to 1038°C (1860 to 1900°F), as shown in Table 2-4. Since rebrazing may be necessary, some margin of base metal strength above the specified minimum was deemed desirable, and tests are planned to determine this effect, which should be smaller than the effect of brazing at an approximate 83°C (150°F) higher temperature. Further brazing was conducted between 1016 and 1038°C with a few specimens brazed at 1049 to 1066°C (1920 to 1950°F) to determine the effects of higher temperatures on joint strength. (Reference Sections 3.3.2 and 3.3.3).

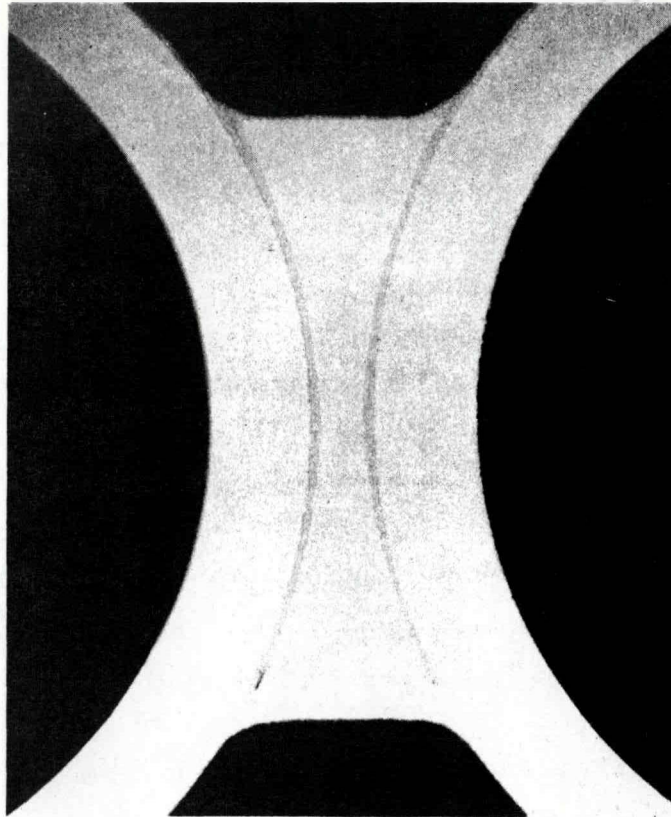
2.3.1.4 Panel Fixturing

Having eliminated the vertical braze panel orientation from the development program, efforts were concentrated on the horizontal fixturing of tubes to retain a flat panel with as narrow a joint gap as possible after brazing. In order to minimize the effects due to thermal expansion of a rigid fixture, a fixture was designed which relied on weights to simultaneously close the joint gap (due to slight bowing of the tubing and filler strips), and keep the tube panel planar during brazing. Typical joints made with this fixture are shown in Figure 2-14. A schematic drawing of this fixture is shown in Figure 2-15. During the development work, this fixture routinely brazed panels ten tubes wide and 1.2 meters long (both with and without hourglass shaped filler strips), flat within 1.6 mm (1/16 inch) along the length, and ± 0.8 mm (1/32 inch) across the width. Before the wide panels without filler strips were brazed, it was thought that the asymmetry of the joint caused by unequal size fillets on the front and back of the panel (due to insufficient or excessive flow-through), would lead to panel transverse warpage. This situation was postulated due to the differential solidification shrinkage of the unequal size fillets. However, the flatness results after brazing with and without filler strips were within the same tolerances.

Table 2-4
INCOLOY 800 TUBING TENSILE TEST RESULTS¹

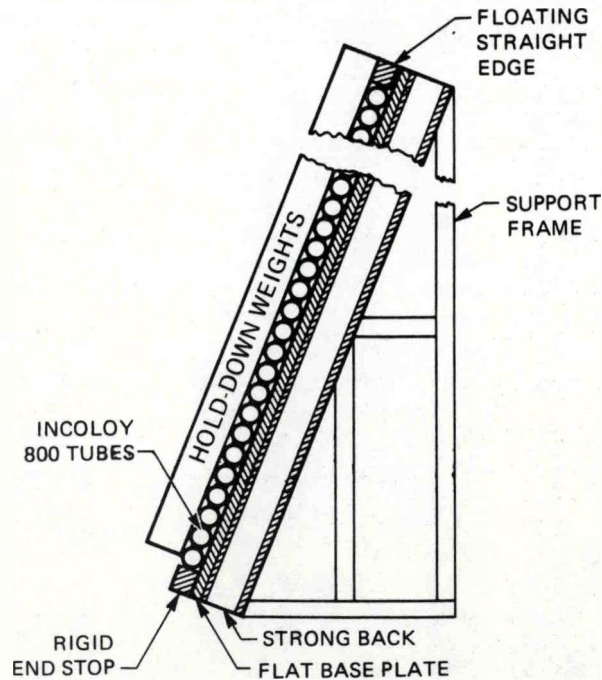
Specimen Number	Brazing ² Temp. °C (°F)	0.2% Offset Yield, MPa (Ksi)	U.T.S. MPa (Ksi)	% Elong. in 25.4 mm (1")
<u>Ambient Test Temperature</u>				
AR 1	None	427 (62.0)	741 (107.5)	39.2
AR 2	None	340 (49.3)	676 (98.0)	38.2
1860-10-2	1015 (1860)	325 (47.2)	689 (100.0)	37.6
1860-10-2	1015 (1860)	306 (44.4)	669 (97.0)	44.9
1900-10-1	1038 (1900)	328 (47.5)	741 (107.5)	35.2
1900-10-2	1038 (1900)	321 (46.6)	707 (102.5)	36.0
<u>621°C (1150°F) Test Temperature</u>				
AR 3	(Failed during set up test in machine)			
AR 4	None	216 (31.3)	503 (72.9)	35.7
1860-10-3	1015 (1860)	216 (31.3)	463 (67.2)	29.9
1860-10-4	1015 (1860)	201 (29.1)	464 (67.3)	28.8
1900-10-3	1038 (1900)	195 (28.3)	493 (70.1)	36.8
1900-10-4	1038 (1900)	185 (26.8)	482 (69.6)	30.4
<u>ASTM B-407 Specified Minimum Values</u>				
No. 1 Temper (Annealed)	--	207 (30.0)	517 (75.0)	30.0 (in 2")
No. 2 Temper (Half-Hard)	--	172 (25.0)	448 (65.0)	13.0 (in 2")

1. Huntington Heat No. 8793, tubing.
 2. Simulated brazing time was 10 minutes for all specimens heat treated.



80-075-11

Figure 2-14. Typical Brazed Joints Made with Filler Metal AD939 Using the Self-Closing Gap Fixture Shown in Figure



80-075-12

Figure 2-15. Solar Panel Brazing Fixture for Self-Closing Joint Gaps

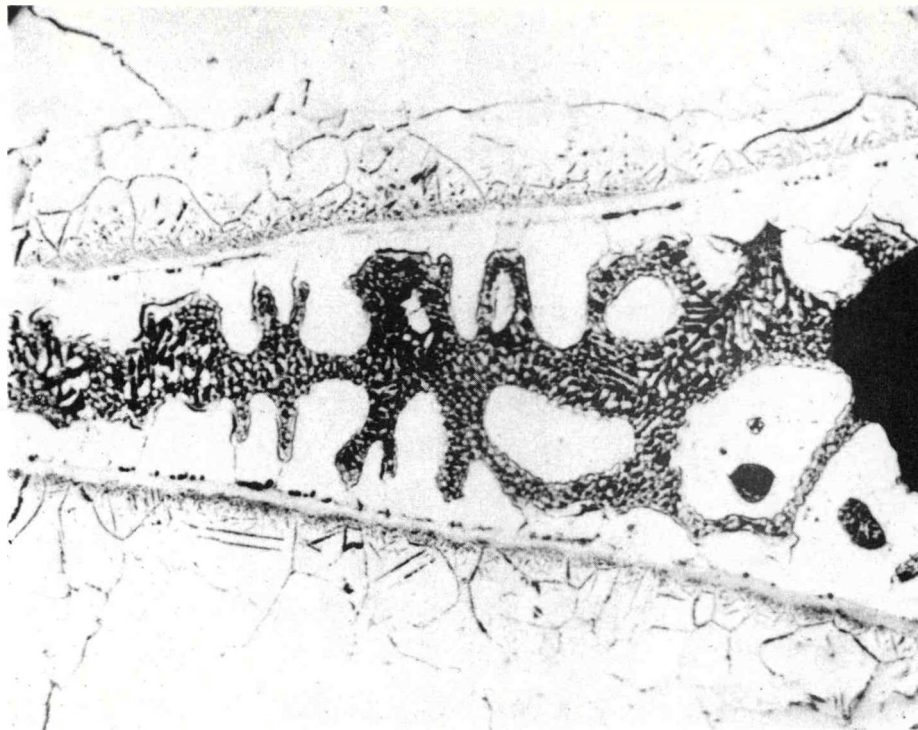
2.3.1.5 Joint Design

The triangular shape filler strips used did not have sides contoured to fit the tubing. As a result, this design was not very effective in providing similar advantages to the hourglass shape strips. A contoured side triangular filler strip was considered for use, since it would provide a narrow joint, approximately 0.03-0.05 mm (1-2 mil) maximum width, similar to the hourglass strip, and would not reduce the effective heat transfer area of the panel front side as a symmetrical hourglass strip does. However, fabrication and handling of a contoured triangular strip proved to be difficult due to the thin knife-edge of this design, and again, there was concern about asymmetry causing distortion. Based on the results of on-going panel heat transfer and thermal stress analyses, however, a non-symmetrical hourglass shape strip may be evaluated as an alternate design.

Despite the excellent brazing success achieved using the hourglass strips, panel brazing development was continued both with and without a filler strip, due to the added costs associated with the strip and the undetermined joint strength requirements. The multiphase microstructures resulting with variable width tube-to-tube joint designs are illustrated in Figures 2-16 and 2-17.

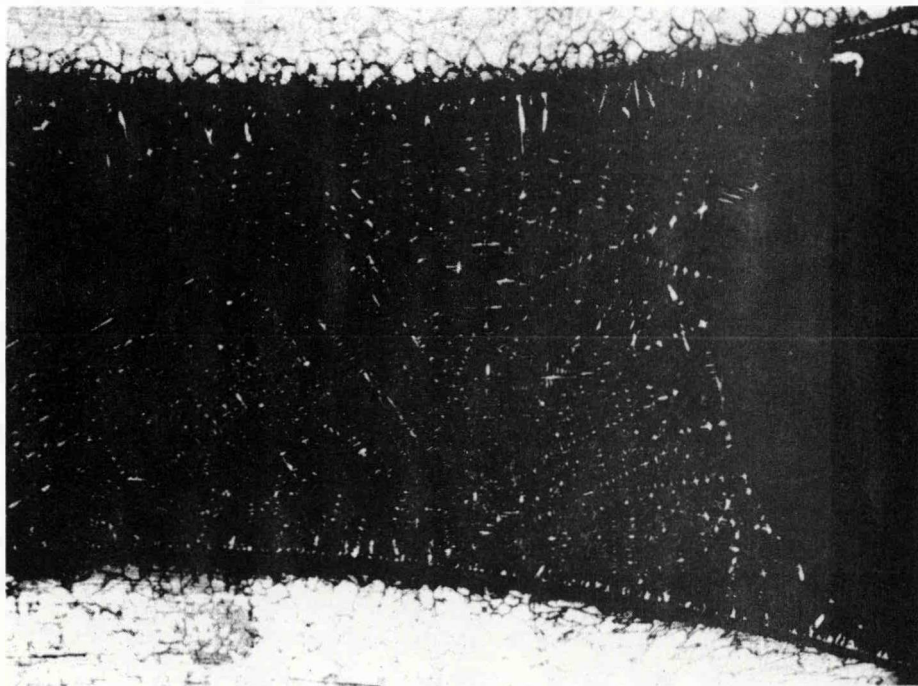
Two alternatives to a joint without a filler strip were evaluated. The first approach was to fill the joint with a 20/80% mixture of brazing filler metal and non-melting powder, and to cover this with a layer of pure filler metal (as described previously). The second approach was to fill the joint with a fillet of non-melting powder, sinter the compact, apply filler metal and allow this to infiltrate the joint during the subsequent brazing operation. The results of these techniques are shown in Figures 2-18, 2-19 and 2-20, which indicate that although large porosity was not observed as often as in conventional joints without filler strips, there are numerous fine pores. The penetrating qualities of different filler metals was demonstrated with these techniques in that the filler metal NB 51, which was observed to have much greater wetting and flow ability than NB LM, also penetrated and filled the non-melting powder structure more easily and, in turn, reduced the porosity. Filler metal flow-through for both these techniques was minimal.

Due to the fact that excess brazing filler metal was necessary to ensure complete joint filling, it became necessary to control tube wetting away from the joint



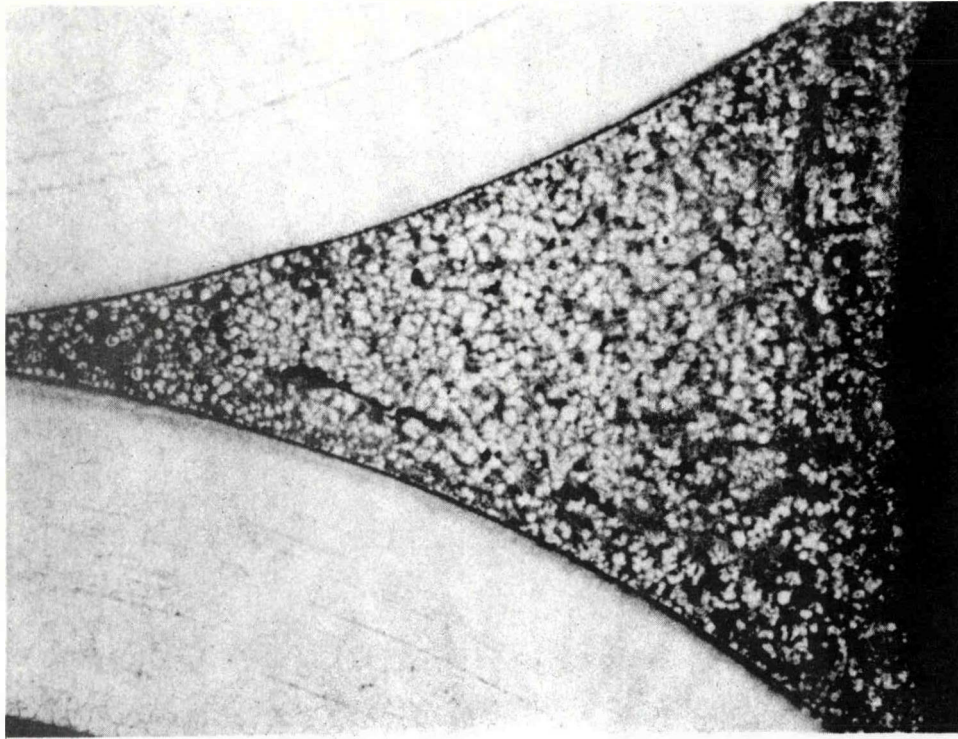
80-075-13

Figure 2-16. Typical Filler Metal Microstructure Occurring in a Tube Contacting Tube Joint Fillet (30X Magnification)



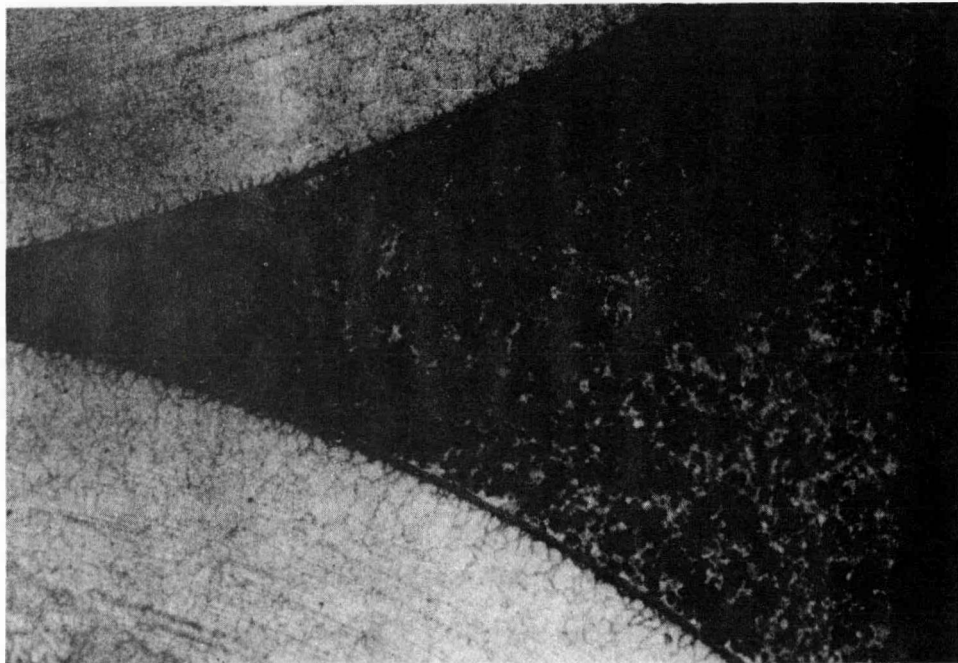
80-075-14

Figure 2-17. Typical Filler Metal Microstructure Occurring in a Tube-Tube Joint Fillet, with Tubes Spaced 0.050" (1.27 mm) Apart (30X Magnification)



80-075-15

Figure 2-18. Results of Combining 20% Filler Metal NB51 and 80% Non-Melting Powder NG108, Overlayed with NB51, Then Brazed (30X Magnification)



80-075-16

Figure 2-19. Results of Combining 20% Filler Metal NB LM and 80% Non-Melting Powder NG108, Overlayed with NB LM, Then Brazed (30X Magnification)



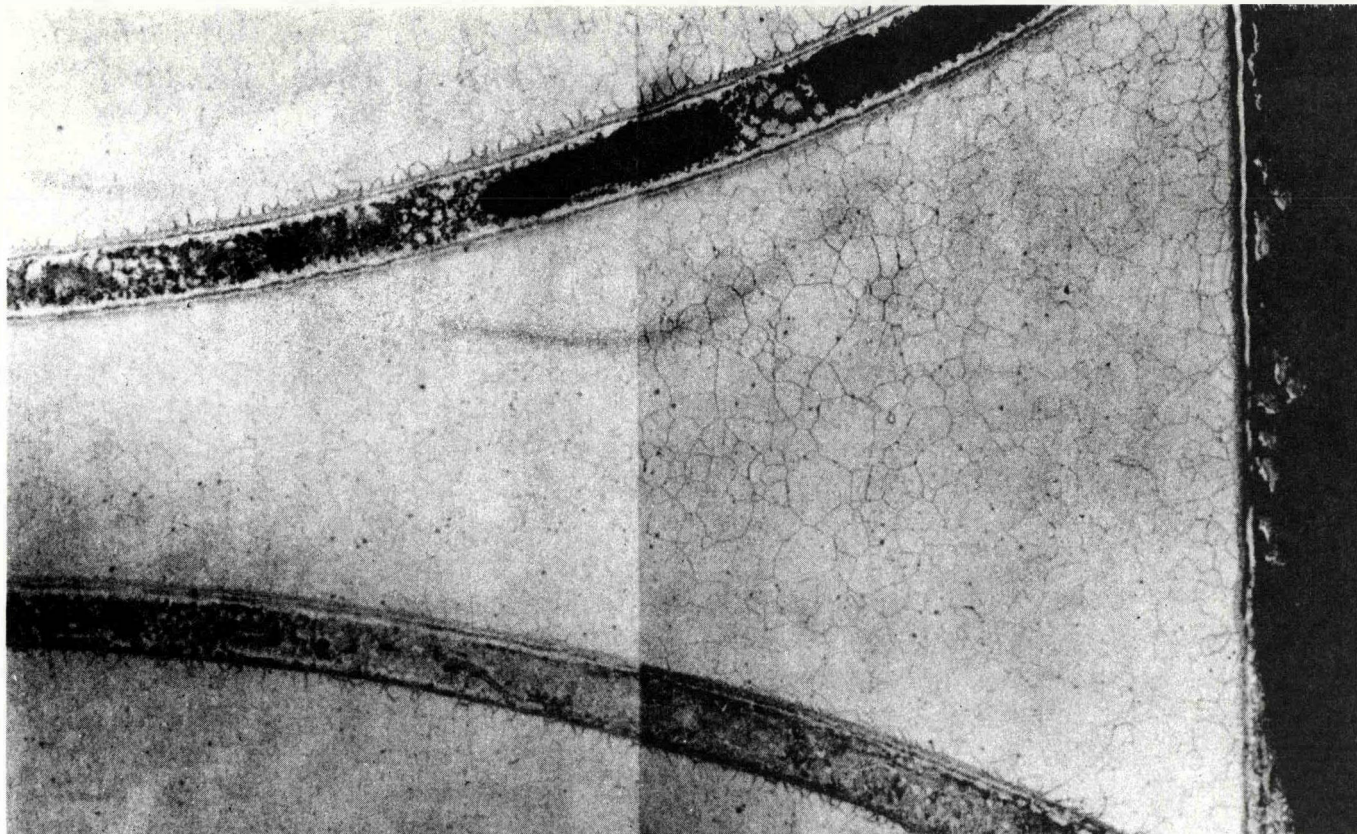
80-075-17

Figure 2-20. Results of Sintering the Non-Melting Powder NG108, Then Infiltrating with Brazing Filler Metal NB LM (30X Magnification)

area by the excess filler metal when applied to the nickel plated tubing. This control was especially necessary for the filler metals NB 50, NB 51, AD 936 and AD 939, which would flow around the complete circumference of the tube unless stop-off compound was applied. Both of the stop-off compounds evaluated, Microbraz Green and boron nitride, gave excellent results when painted on to form a continuous coating between masked braze areas.

2.3.1.6 Braze Filler Metal Forms and Types

The various types and application of brazing filler metal evaluated required approximately the same labor and produced approximately the same brazing results, with the exception of the tape and foil. The adhesive-backed tape filler metal was easier to apply. However, the resulting joint had a high degree of porosity as shown in Figure 2-21, despite the excess width of tape used (beyond the joint edges) to allow for binder burn-out. The tape and foil, both of which were much more expensive than the various powder forms, required use of the contoured filler strip, adding to the overall cost. The foremost reason for discontinuing development of the tape and foil



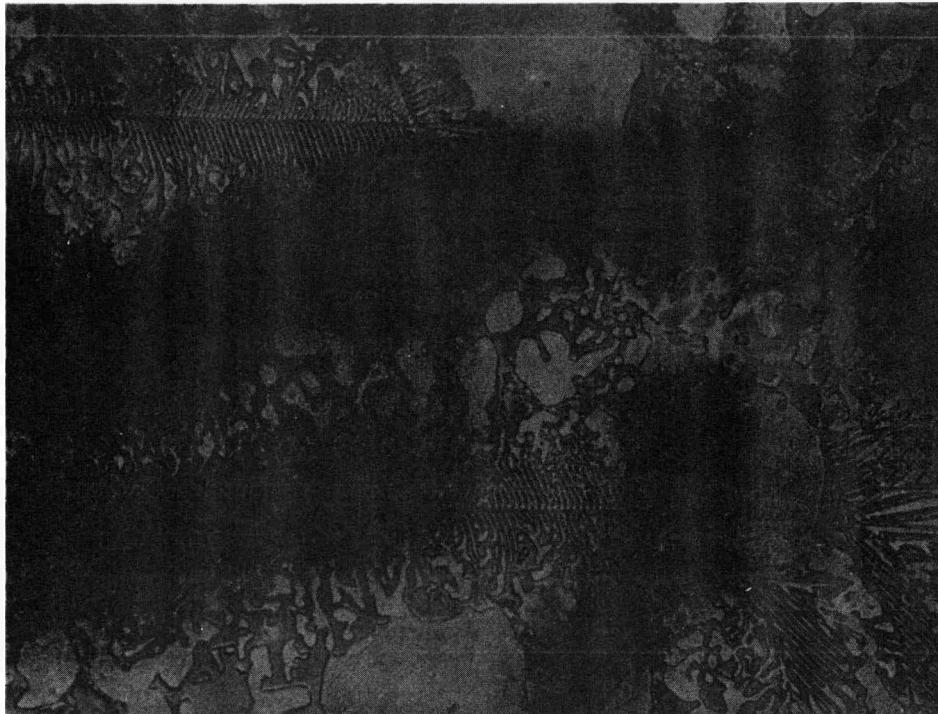
80-075-18

Figure 2-21. Results of Brazing Hourglass Strip Filled Joint with AD770 Adhesive Backed Tape 0.15 mm (0.006") Thick (30X Magnification)

was the fact that both forms of filler metal are relatively intolerant to the increased variation in joint gaps that would probably exist during the brazing of approximately 6.2 meter (20 ft.) long tubes. This condition would lead to incomplete joint filling and/or porosity.

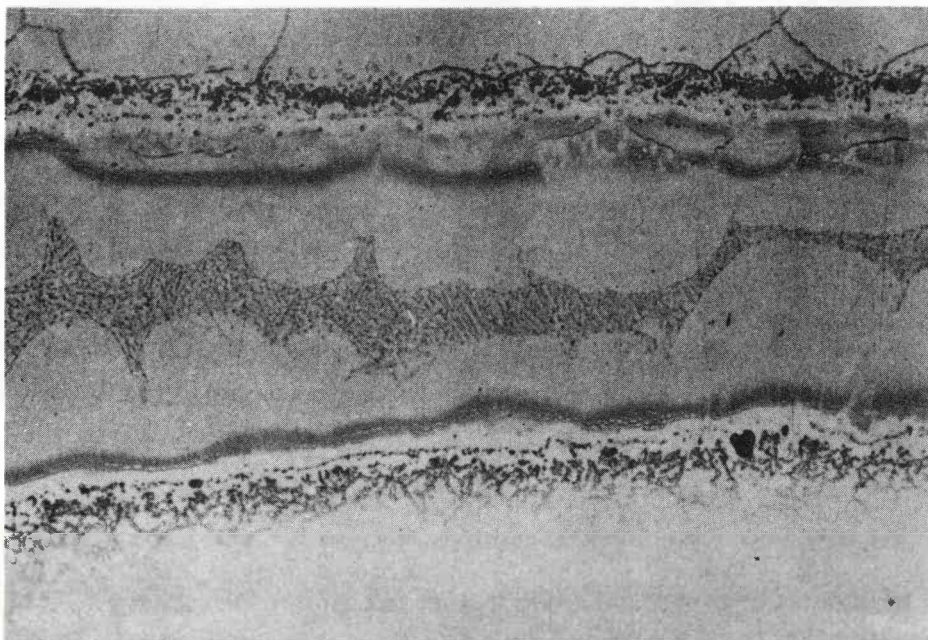
The main advantage of the powder based forms of filler metals -- the ability to easily provide the required excess filler metal -- is also their greatest disadvantage in terms of mechanical properties, in that the excess filler metal becomes a brittle surface fillet. The joint surface will see the highest bending strains, and so the fillets, which have an increased degree of crack susceptibility, may lead to earlier crack initiation and, therefore, a shorter panel life for given service conditions. Use of a contoured filler strip should aid in arresting these potential cracks, since the cracks may initiate in the brittle fillet, but to continue growing would have to propagate through the more ductile narrow-gap joint.

The microstructure of a typical joint with an hourglass shaped filler strip, brazed with AD 939 between 1104 and 1016°C (1840 and 1860°F) for ten minutes after equalizing between 899 and 910°C (1650 and 1670°F), is shown in Figures 2-22 through 2-25. Figure 2-22a shows the fillet on the side of the joint to which the filler



80-075-19

Figure 2-22(a). Typical Fillet Microstructure of AD939 on Side of Joint Filler Metal was Applied, Braze 1840-1860°F (1004-1016°C) (500X Magnification)

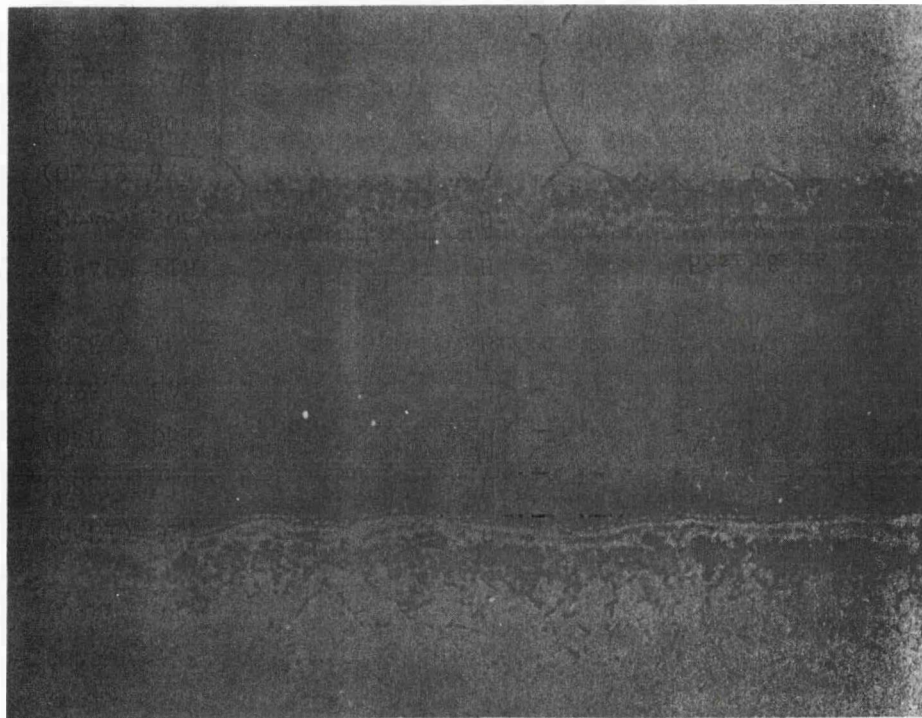


80-075-20

Figure 2-22(b). Single Phase/Multiphase Portion of Same Joint as in 2-22(a), Near Fillet Region Shown in 2-22(a) (500 X Magnification)

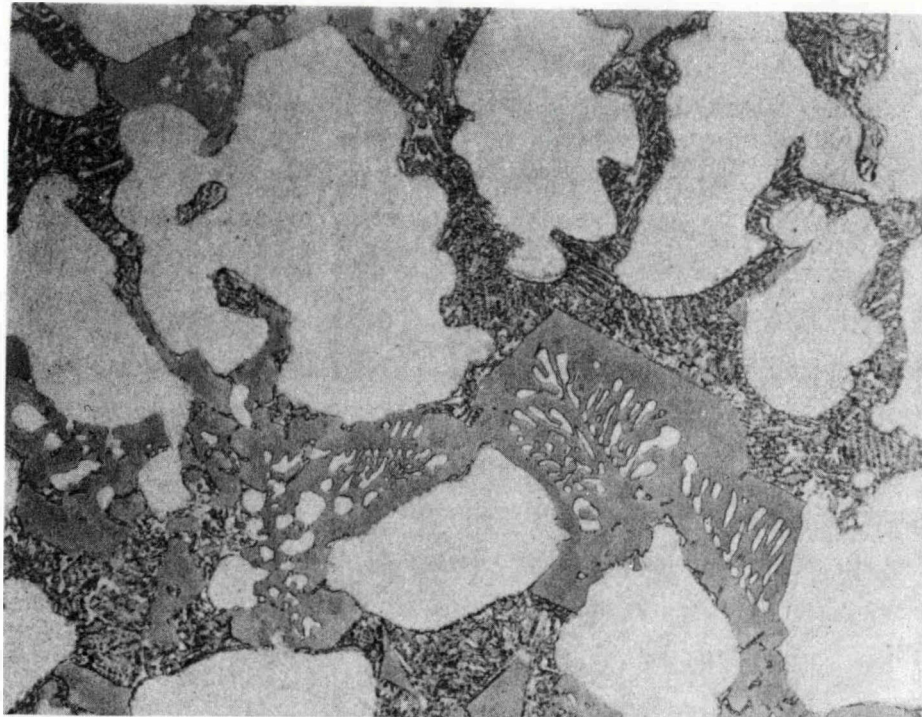
metal was applied, and shows a fine eutectic type feather structure interspersed with islands of a solid solution phase. Figure 2-22(b) shows the joint gap near the fillet as a single phase with a very fine multi-phase structure, which is now visible along the center of the joint gap.

Figure 2-25 shows the adjacent region. In this region the gap is slightly narrower, allowing the multi-phase constituents to diffuse completely into the base metal, which results in the preferred ductile filler metal. The fillet on the back (or opposite) side of the joint to which the filler metal was applied, is shown in Figure 2-24. This region is similar in morphology to that shown in Figures 2-22, except that the aligned eutectic feather structure is absent. The gap width in the single phase region of this joint is approximately 0.061 mm (0.0024 inch), which appears to be the widest gap in which an all single phase structure will form under these brazing conditions. Without a filler strip, the large fillets which were required for the same joint area often produced large dendrites and porosity, as shown in Figure 2-25.



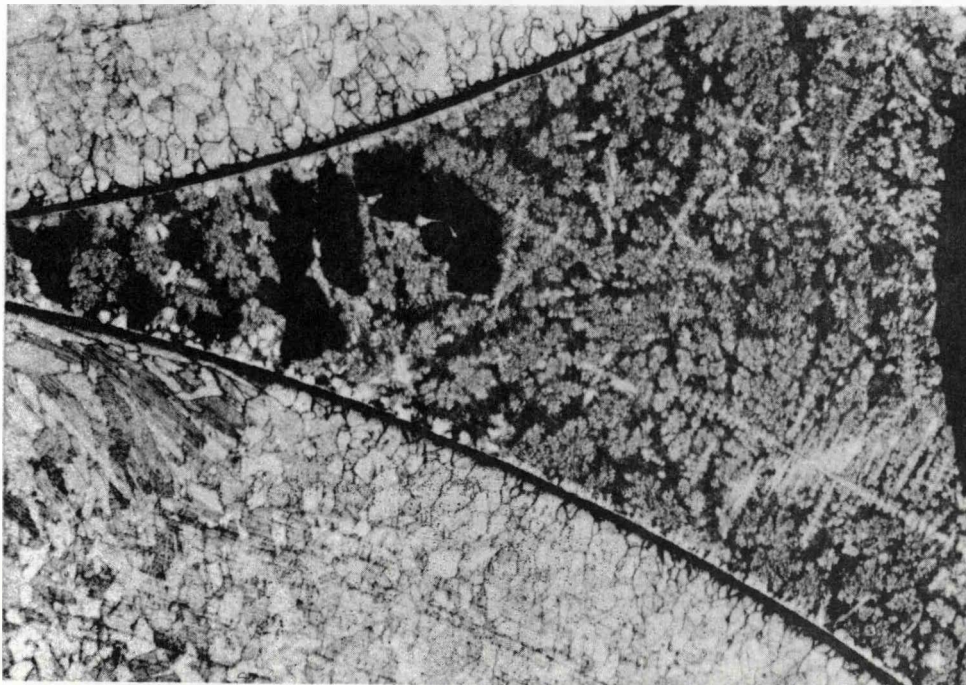
80-075-21

Figure 2-23. Single Phase Microstructural Region of Same Joint as in 2-22 Typical with Narrow Joint Clearance (500 X Magnification)



80-075-22

Figure 2-24. Typical Fillet Microstructure of AD939 on Opposite Side of Joint Filler Metal was Applied (500 X Magnification)

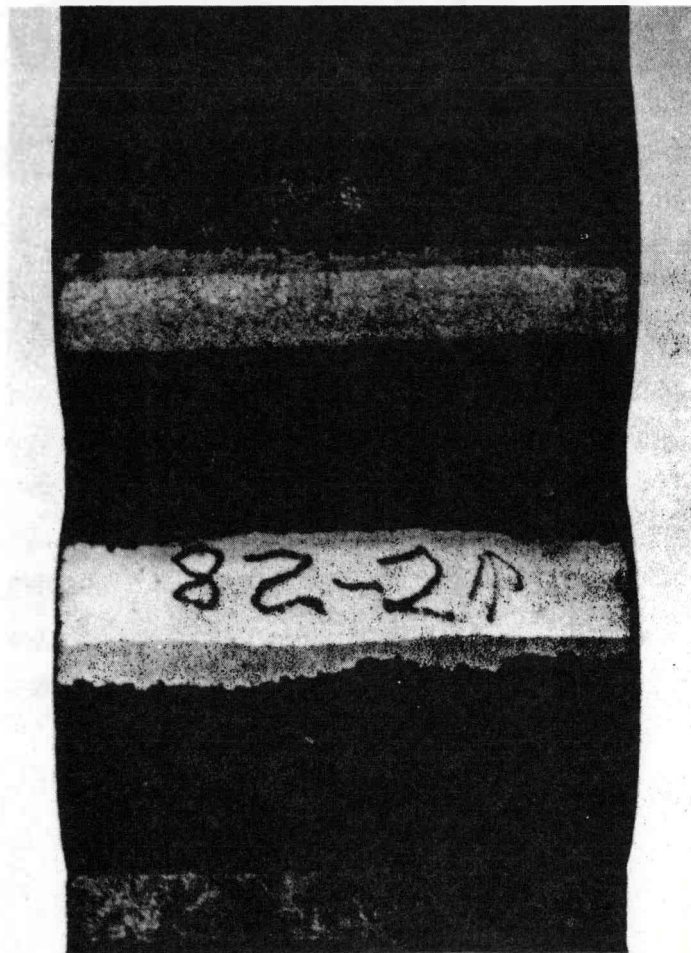


80-075-23

Figure 2-25 Typical Microstructure of Large Fillet on Joint Without a Filler Strip, Showing Dendritic Grains and Porosity (30 X Magnification)

3.1.7 EFFECT OF THERMAL AGING

The results of aging joints made with fillet metals NB LM, NB 51 and AD 939 at 566°C (1050°F) for 1,000 hours in air are encouraging in that the oxides of all three filler metals and the base metal were tightly adherent. Previous work has shown that after aging at a similar temperature of 500°C (932°F) for 500 hours), a filler metal similar to NB LM, but with less chromium, showed flaking oxide which exposed metallic areas. This was suspected to have occurred during cooling.⁴ The three filler metals are shown in Figure 2-26 from top to bottom, respectively (the light-colored areas on each side of the NB 51 joint were coated with stop-off compound). After aging joints made with filler metals NB lm and AD 939 at 677°C (1250°F) for 1,000 hours, which is roughly equivalent to a solar plant lifetime at a maximum temperature of 620°C (1150°F), the oxide of both filler metals was tightly adherent and dark grey in color.



80-075-24

Figure 2-26. Appearance of Joints Made With Filler Metals NB LM, NB51, and AD939 (Top to Bottom) After Aging in Air at 566°C (1050°F) for 1,000 Hours

2.3.2 BRAZING PROCEDURES

Based on the braze development discussed, a typical braze procedure has been recommended, details of which are contained in Appendix A. A summary of the materials and fixtures for brazing an I800 thin walled tubular solar panel is contained in Table 2-5. The brazing sequence is presented in Table 2-6. Some details may change as ongoing test results are evaluated.

Table 2-5
BRAZING MATERIALS AND FIXTURES

- Cleaning Agent - Acetone and Nylon Cloth
- *Tube - I800 19-05 mm OD (.75 in) ASTM B407 HC Cold Drawn Annealed
- *Filler Strip - I800 Hourglass (Rolled or Machined)
- *Plating - Nickel Electroplate, Dull Finish, 0.0107 - 0.0152 mm
- Braze Alloy - Nickel-Manganese-Silicon Metal Powder, - 140 mesh AD 939
- Stop-off-Compound - Microbraz Green
- Platers Tape
- Purge Gas - Argon or Nitrogen, then Hydrogen
- Furnace - Horizontal with Hydrogen DP of $< -62^{\circ}\text{C}$ (-80°F)
- Fixtures - Flat Metal Hearth, Clamps and Weights

*Tubes and filler strips will be nickel-plated prior to brazing.

2.3.3 RESULTS OF MECHANICAL TESTING

2.3.3.1 Hardness Testing

The variation in hardness between the multiphase fillet and the single phase narrow gap portion of the contoured filler strip type joint was considerable. Typical average hardness values for the multiphase fillet were Rockwell C Scale 57, 51, 49 and 28 for brazing filler metals NB 135, AD 914, NB LM and AD 939, respectively. The average hardness values for the single phase structure in the narrow gap part of the joints fabricated with contoured filler strips were, for the same filler metals, Rockwell B Scale 74, 75, 91 and 77.

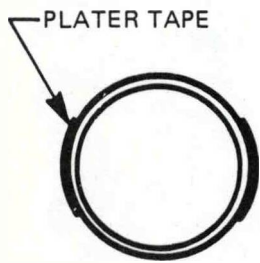
The braze joint hardness measurements were taken with a Knoop indenter using a 500 gram weight, and were converted to Rockwell values using the standard Wilson Chart 60. The hardness data shows that the single phase structure of these filler

Table 2-6
BRAZING PROCEDURE SUMMARY

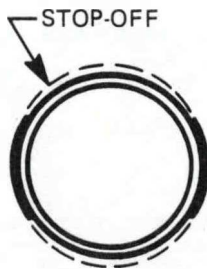
Step

1. Clean tubing exterior with acetone/nylon cloth
2. Apply plater's tape on alloyed portion of tube
3. Apply stop-off compound to tubes and fixtures
4. Remove plater's tape
5. Assemble tubes and filler strips on fixture
6. Apply braze alloy in continuous fillet
7. Apply cement spray to braze alloy (if not mixed with alloy)
8. Install tubes and fixture on 45°-60° angled hearth in furnace
9. Prepare fixture clamps, guides and weights
10. Seal and purge furnace muffle
11. Heat panel segment to approximately 900°C (1650°F) - Hold
12. Heat rapidly to approximately 1015°C (1860°F) - Hold 10 min.
13. Cool rapidly to below 904°C (1660°F)
14. Cool to room temperature
15. Remove and inspect panel
16. Clean and reinspect as necessary

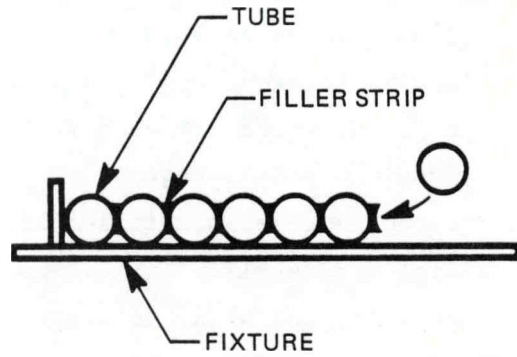
NOTE: Steps are illustrated in Figure 2-27



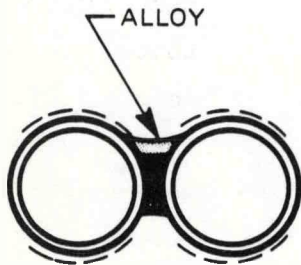
STEP 1 AND 2



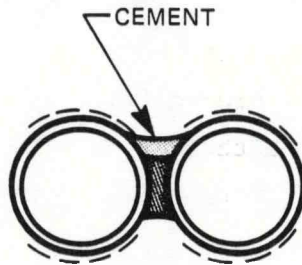
STEP 3 AND 4



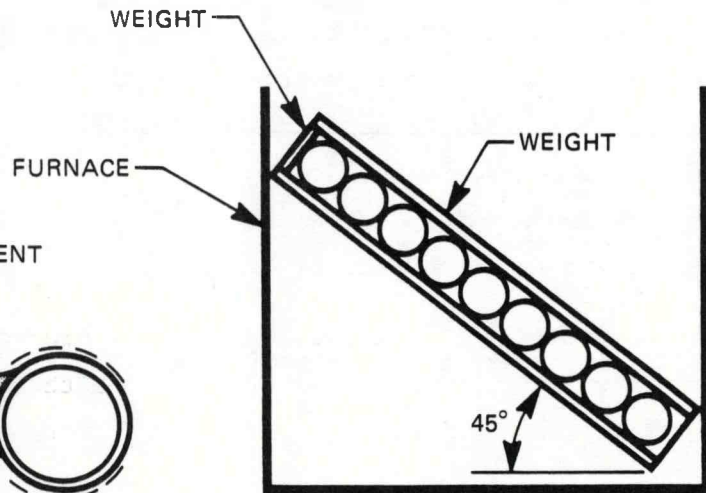
STEP 4 AND 5



STEP 6



STEP 7



STEPS 8 AND 9

NOTE: STEPS 9 THROUGH 13 ARE CONDUCTED IN THE FURNACE

Figure 2-27. Braze Procedure Illustrated

metals is relatively soft, compared to the fillet hardness, and about the same hardness as the tube base metal after brazing. The notable exception is the lower fillet hardness of AD 939, which is the only braze metal type evaluated containing copper (4%), manganese (19%), and rare earth (0.03%). (Filler metal AD 936 is a lower brazing temperature variant of AD 939 with the same copper, manganese and rare earth contents.) In the as-received condition (cold-drawn, annealed), the tubing had an average Rockwell B hardness of 79, which dropped to 75 after brazing at 1016°C (1860°F) for ten minutes. After brazing at 1049°C (1920°F), the tubing hardness had decreased to Rockwell B 73. After brazing and rebrazing (to simulate furnace repair), the tubing hardness decreased to an average of Rockwell B72. Both brazing and rebrazing operations had a duration of ten minutes each. The tubing hardness values were measured using a Rockwell tester on 3.18 mm (1/8 inch) wall thickness Incoloy 800 tubing procured for eventual drawing down to the prototype test panel tubing thickness of 1.27 mm (0.050 inch).

2.3.3.2 Tensile Testing

Room temperature tensile tests were performed on brazed tube/tube joints made with the braze metals which showed acceptable brazability. These joints included specimens without any filler strip, specimens with an hourglass shaped filler strip, and specimens with a triangular shaped filler strip. Based on the results of these tensile tests, additional specimens were brazed for tensile tests performed at 621°C (1150°F), as listed in Table 2-7. Due to the fact that none of the commonly employed specimen configurations for the mechanical testing of brazed joints⁵ were representative of the loading modes and joint designs evaluated for the panel, the tensile tests were performed with the special fixture shown in Figure 2-28. Due to the tube-to-tube joint geometry which produced a cylindrical surface between the brazing filler metal and the tube, the filler metal was not in uniaxial stress when the tubes were pulled apart in tension via a load applied to the tight-fitting pins running through the tubes. As the specimens were loaded, the tubes began to plastically deform before the joint failed, increasing the degree of non-uniformity of the stress distribution across the joint above that inherently present due to the geometrical stress concentration at the intersection of the fillet edge and tube surface. Therefore, the values reported are joint design dependent tensile strengths, rather than filler metal unit stresses at maximum load capacity.

Table 2-7
BRAZE JOINT TENSILE TEST RESULTS

Results of Standard Temperature Brazing without Aging Treatments

<u>Alloy Code</u>	<u>Sample Type¹</u>	<u>Joint Type²</u>	<u>Fracture Load/Joint Length³</u>	
			<u>N/mm (lb./in.)</u>	
Room Temp. 621°C (1150°F)				
NB LM	As Brazed	H	535 (3058)	447 (2550)
"	"	H	630 (3600)	412 (2350)
"	"	H	1130 (6460)	-
"	"	H	382 (2180)	-
"	"	H	777 (4440)	-
"	"	H	786 (4490)	-
NB 51	As Brazed	H	320 (1830)	179 (1020)
"	"	H	417 (2380)	180 (1030)
"	"	H	180 (1030)	-
"	"	H	243 (1390)	-
"	"	H	231 (1320)	-
AD 939	As Brazed	H	611 (3490)	455 (2600)
"	"	H	602 (3440)	383 (2190)
"	"	H	476 (2720)	-
"	"	H	406 (2320)	-
"	"	H	1440 (8250)	-
"	"	H	415 (2370)	-
"	"	H	364 (2080)	-
"	"	H	350 (2000)	-
"	"	H	622 (3550)	-
"	"	H	296 (1690)	-
"	"	H	689 (3820)	-
"	"	H	807 (4610)	-
AD 770A	"	H	425 (2430)	-

Table 2-7
(Continued)

Results of Standard Temperature Brazing without Aging Treatments

<u>Alloy Code</u>	<u>Sample Type¹</u>	<u>Joint Type²</u>	<u>Fracture Load/Joint Length³</u> <u>N/mm (lb./in.)</u>
Room Temperature			
NB LM	As Brazed	T	379 (2162)
"	"	T	287 (1640)
"	"	T	305 (1740)
NB 51	As Brazed	T	282 (1610)
NB 125	"	T	835 (4770)
NB 150	"	T	450 (2570)
NB 210	"	T	528 (3020)
AD 914	"	T	235 (1340)
NB LM	As Brazed	X	308 (1760)
"	"	X	208 (1190)
"	"	X	310 (1770)
"	"	X	371 (2120)
"	"	X	284 (1620)
"	"	X	173 (990)
NB 51	As Brazed	X	200 (1140)
NB 125	"	X	464 (2650)
NB 150	"	X	459 (2620)
NB 210	"	X	578 (3300)
AD 914	"	X	299 (1710)
AD 939	As Brazed	X	310 (1770)
"	"	X	254 (1450)
"	"	X	264 (1505)
"	"	X	417 (2380)
"	"	X	256 (1460)

Table 2-7
(Continued)

Results of Various Brazing/Aging Treatments

<u>Alloy Code</u>	<u>Sample Type</u> ¹	<u>Joint Type</u> ²	<u>Fracture Load/Joint Length</u> ³	
			<u>N/mm (lb./in.)</u>	
			<u>Room Temp.</u>	<u>621°C (1150°F)</u>
NB LM	Brazed at 1065°C(1950°F)	H	877 (5010)	-
"	"	H	385 (2200)	-
AD 939	"	H	441 (2520)	-
"	"	H	249 (1420)	233 (1330)
AD 939	Rebrazed	H	-	552 (3150)
"	"	H	-	672 (3840)
AD 939	Rebrazed 677°C(1250°F)/1000 hr.	H	471 (2690)	-
"	"	H	570 (3260)	-
NB LM	Aged 565°C(1050°F)/1000 hr.	H	709 (4050)	397 (2270)
"	"	H	562 (3210)	397 (2270)
NB 51	"	H	408 (2330)	420 (2400)
"	"	H	326 (1860)	420 (2400)
AD 939	"	H	1467 (8380)	541 (3090)
"	Brush Plated	H	485 (2770)	310 (1770)
"	"	H	282 (1610)	313 (1790)
"	"	H	403 (2300)	-
"	"	H	415 (2370)	-
AD 770	Tape Brazing Filler Metal	H	203 (1160)	461 (2630)

Table 2-7
(Continued)

Results of Various Brazing/Aging Treatments

<u>Alloy Code</u>	<u>Sample Type</u> ¹	<u>Joint Type</u> ²	<u>Fracture Load/Joint Length</u> ³	
			<u>N/mm (lb./in.)</u>	
			Room Temp. 621°C (1150°F)	
AD 939	Rebrazed	X	425 (2430)	522 (3150)
"	"	X	317 (1810)	466 (2660)
AD 939	Brush Plated	X	-	310 (1770)
"	"	X	-	313 (1790)

1. All base metal was electroplated with nickel (bath process) except as noted; all filler metal was applied as -140 mesh powder except as noted.
2. Joint Type: H = Hourglass filler strip; T = Triangular filler strip; X = No filler strip.
3. Tensile load was applied perpendicular to tube axis; however, stress in braze joint was not uniaxial due to the curvature of the joint around the tubing.

The tensile (pull-apart) test results show that generally only a small strength increase resulted from using the triangular shaped filler strip compared to similar joints without any filler strip. However, an appreciable strength increase resulted from using the contoured hourglass shaped filler strip. This increased strength was attributed to the maintenance of a narrow joint gap with a fine structured ductile filler metal. No benefit was achieved using either filler strip for filler metal NB 51, which retained a brittle multiphase dendritic microstructure even in joint gaps less than 0.025 mm (0.001 inch).

The strongest panel joint tested at room temperature was obtained with AD 939, using an hourglass strip, and aged in air (after brazing) for 1,000 hours at 566°C (1050°F); the panel maximum service temperature. Failure occurred in the filler metal in all tests performed, except for one specimen brazed with AD 939 using an hourglass strip. This specimen is shown in Figure 2-29, which indicates that the tubing underwent extensive deformation before failure. (The small holes in the specimen were used for extensometer attachment.) The failure mode was intergranular fracture through all multiphase fillets, both on joints with and without filler strips. In the predominantly single phase regions, the failure mode was transgranular shear.

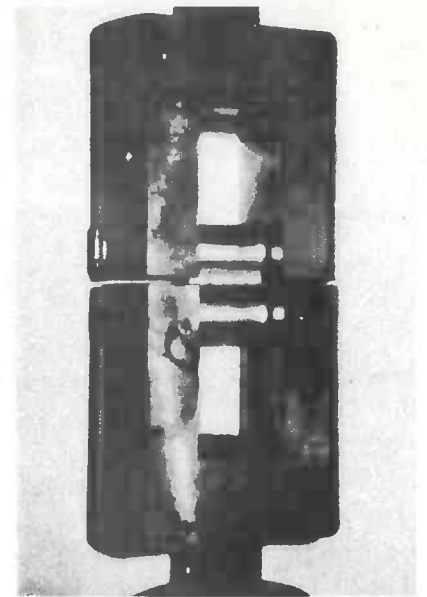
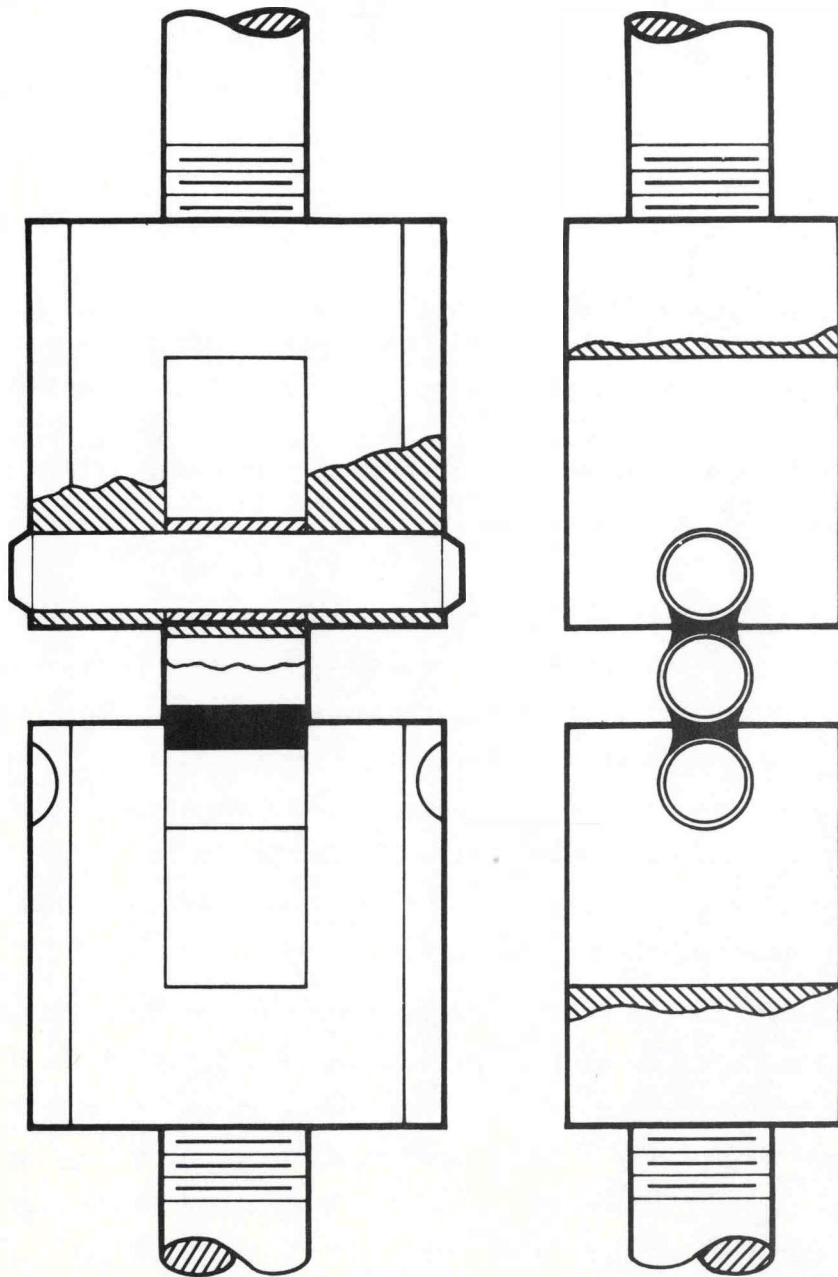


Figure 2-28. Absorber Panel Tensile Test Fixture

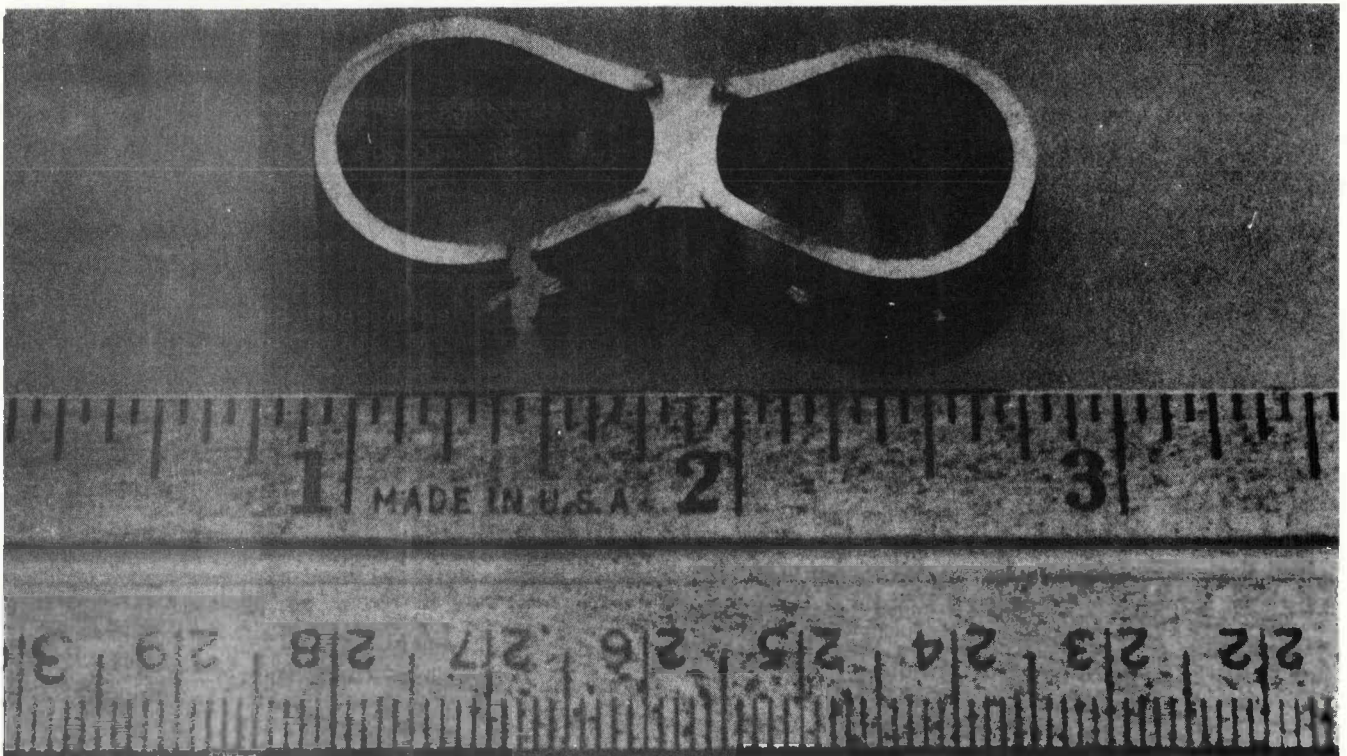


Figure 2-29. Brazed Joint with Hourglass Shaped Filler Strip After Tensile Test, Showing Tube Deformation Before Failure

Other brazed specimens made with the same filler metals and joint designs as those reported here, are being aged at 649°C (1200°F) for 100 and 1,000 hours to determine the effects of post-braze heat treatment and accelerated lifetime aging on strength, and oxidation resistance. Post-braze treatments have been shown to enhance joint strength, particularly thermal fatigue strength.⁶

2.3.3.3 Shear Testing

The initial scoping tensile tests were performed to select the higher strength filler metals of all those that showed acceptable brazability. After the completion of this phase, room temperature and 621°C (1150°F) shear tests were performed. The fixture used to load specimens in shear, applied the load along the longitudinal axis to two brazed joints simultaneously, as shown in Figure 2-30. Shear strengths at ultimate load were calculated by dividing the maximum load carried by both joints by the joint length of the joints. The load required to fail the second joint after the first had cracked was not representative, due to the loading eccentricity and resulting bending moment present in the joint, and was therefore not reported.

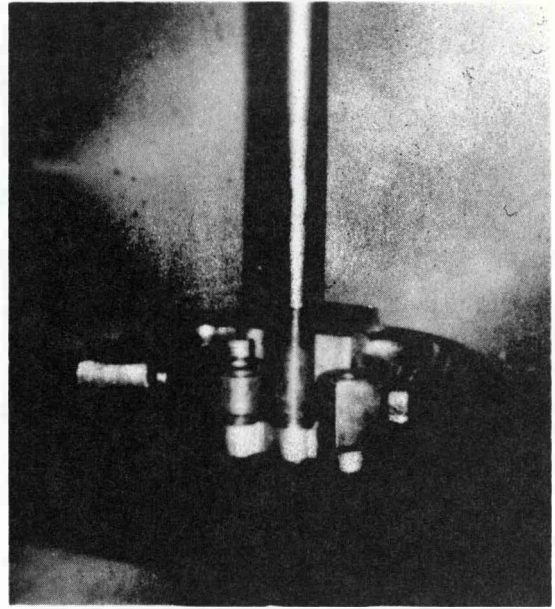
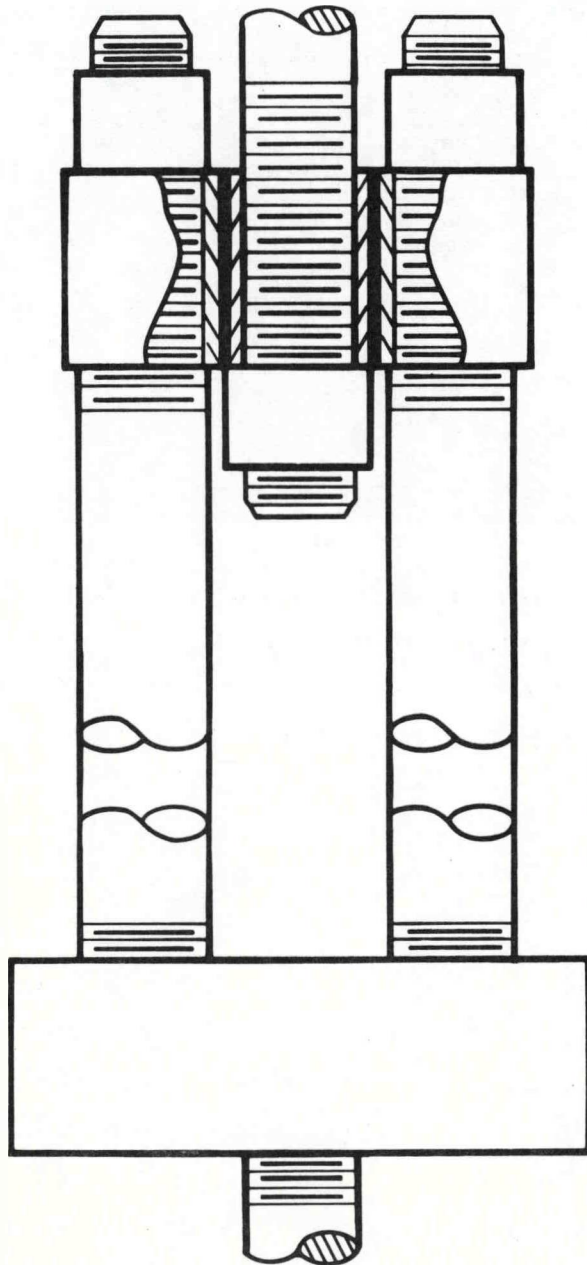


Figure 2-30. Absorber Panel Shear Test Fixture

The results of the shear tests (which are incomplete to date) are given in Table 2-8. Since in both the shear and tensile tests the joint strengths are sensitive to sample size, all specimens were made approximately 12.7 mm (0.500 inch) long. In contrast with the tensile test results, the shear strengths for joints without a filler strip were as high as those for joints with the hourglass shaped filler strip.

2.3.3.4 Bend Testing

Longitudinally loaded three-point bend tests have been performed on three-tube panel segments made with the four combinations of preferred and alternate brazing filler metal and joint designs. These combinations were AD 939 and NB LM, both with and without the hourglass shaped filler strip. A 25 cm (10 inch) span of tubing was loaded incrementally and visually inspected between load increments for surface cracking or delamination on either side. All joints tested with this fixture, shown schematically in Figure 2-31 deflected by approximately 19 mm (0.75 inch) before cracking was observed on the tensile side of the joint. This deflection was measured during load application and, therefore, was the result of elastic and plastic strain. Bending of the joints was subsequently continued through a 90 degree angle, during which delamination began at about a 45 degree bend. Due to the three-point loading used, the deformation was concentrated in the central half of each specimen.

Transverse three-point bend tests are in progress on the same filler metals and joint designs as evaluated in longitudinal bending. These tests are being performed with the fixture shown in Figure 2-32 at both room temperature and 621°C (1150°F). Preliminary results are presented in Table 2-9, and show the increased load carrying capacity of the joints with the hourglass filler strip over those without a filler strip.

Table 2-8
BRAZE JOINT SHEAR TEST RESULTS¹

Results of Various Brazing/Aging Treatments

<u>Alloy Code</u>	<u>Sample Type</u>	<u>Joint Type</u> ²	<u>Fracture Load/Joint Length</u> ³	
			<u>N/mm (lb./in.)</u>	
			Room Temp. 621°C (1150°F)	
AD939	Brush Plated	H	1110 (6330)	823 (4700)
AD 939	Rebrazed ⁴	X	958 (5740)	837 (4780)
"	"	X	942 (5380)	-
AD 939	Rebrazed at at 1950°F ⁵	H	865 (4940)	-
AD 939	As Brazed	X	982 (5610)	-
NB LM	"	X	1010 (5760)	-
AD 939	"	H	1030 (5870)	-
NB LM	"	H	1130 (6460)	-
AD 939	"	X	1560 (8920) ⁶	-
NB LM	"	H	>2560 (>14630) ^{6,7}	-

1. Loading was applied along the tubing axis. Results are incomplete; additional shear tests are in progress.
2. Joint Type: H = Hourglass filler strip; X = No filler strip.
3. All base metal was electroplated with nickel (bath process) except as noted; all filler metal was applied as - 140 mesh powder with cement.
4. Additional brazing filler metal applied before rebrazing.
5. Samples brazed at 1065°C (1950°F); all other samples were brazed at 1015-1038°C (1860-1900°F), except as noted.
6. Specimen outer two tubes were restrained from moving apart radially during test with a rigid clamp.
7. Fracture load exceeded test machine capacity.

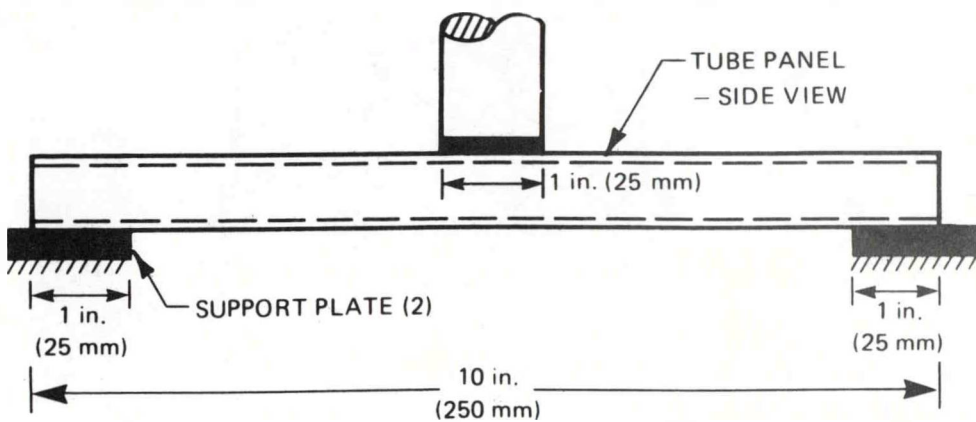
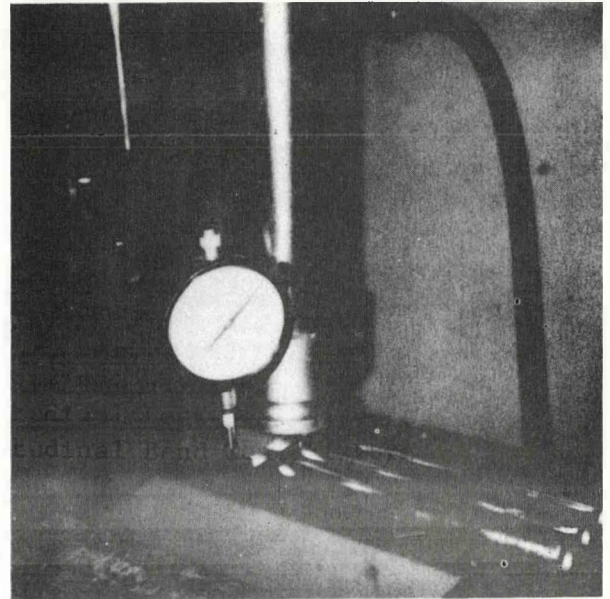


Figure 2-31. Absorber Panel Longitudinal Bend Test Fixture

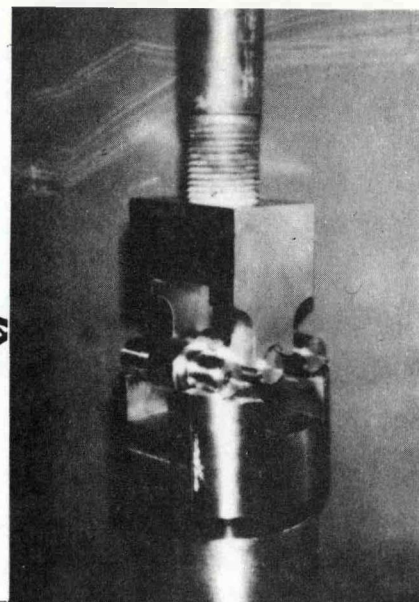
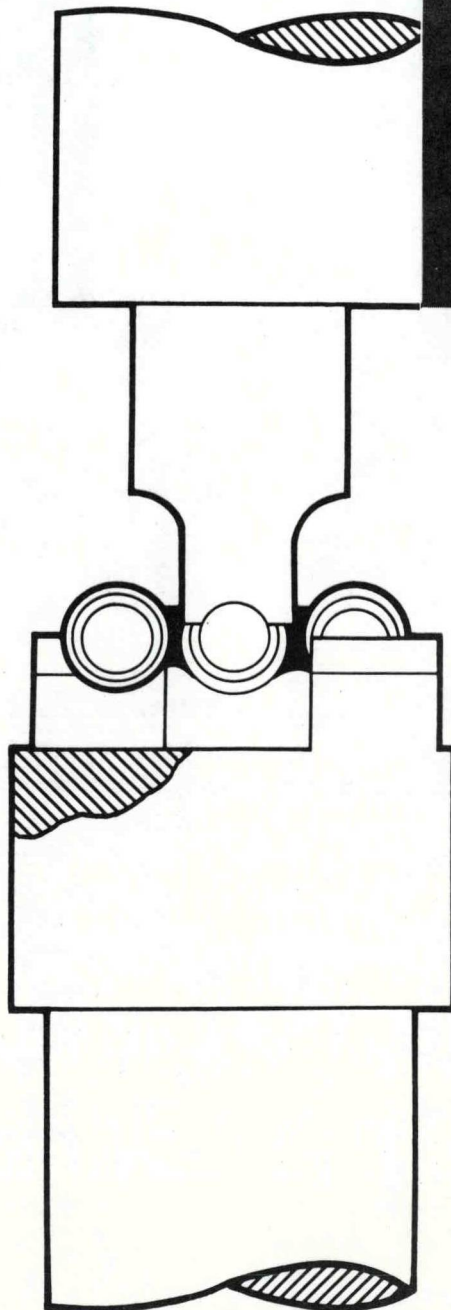
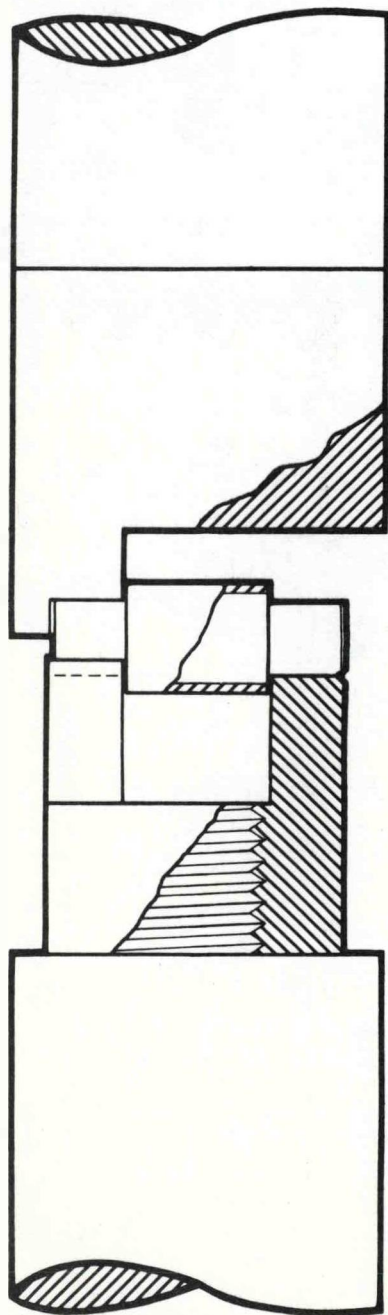


Figure 2-32. Absorber Panel Transverse Bend Test Fixture

Table 2-9
BRAZE JOINT THREE-POINT TRANSVERSE BEND TEST RESULTS

Alloy Code	Joint Type ¹	Tensile Side of Joint ²	Fracture Load/Joint Length ³	
			N/mm (lb./in.)	
AD 939	H	Front	383 (2190)	488 (2790)
"	H	Back	376 (2150)	313 (1790)
"	X	Front	224 (1280)	-
"	X	Back	198 (1130)	-
NB LM	H	Front	401 (2290)	583 (3330)
"	H	Back	382 (2180)	282 (1610)
"	X	Front	189 (1080)	-
"	X	Back	219 (1250)	-

1. Joint Types: H = Hourglass filler strip between tubes; X = No filler strip.
2. The front side of the joint was the side to which the brazing metal flowed through; whereas the back side was area to which the filler metal was applied.
3. Loading was applied to a section of panel three tubes wide and 12 mm (1/2 inch) long, by displacing hardened mandrels to the tubes perpendicular to both the tube axis and panel plane.

2.4 CONCLUSIONS

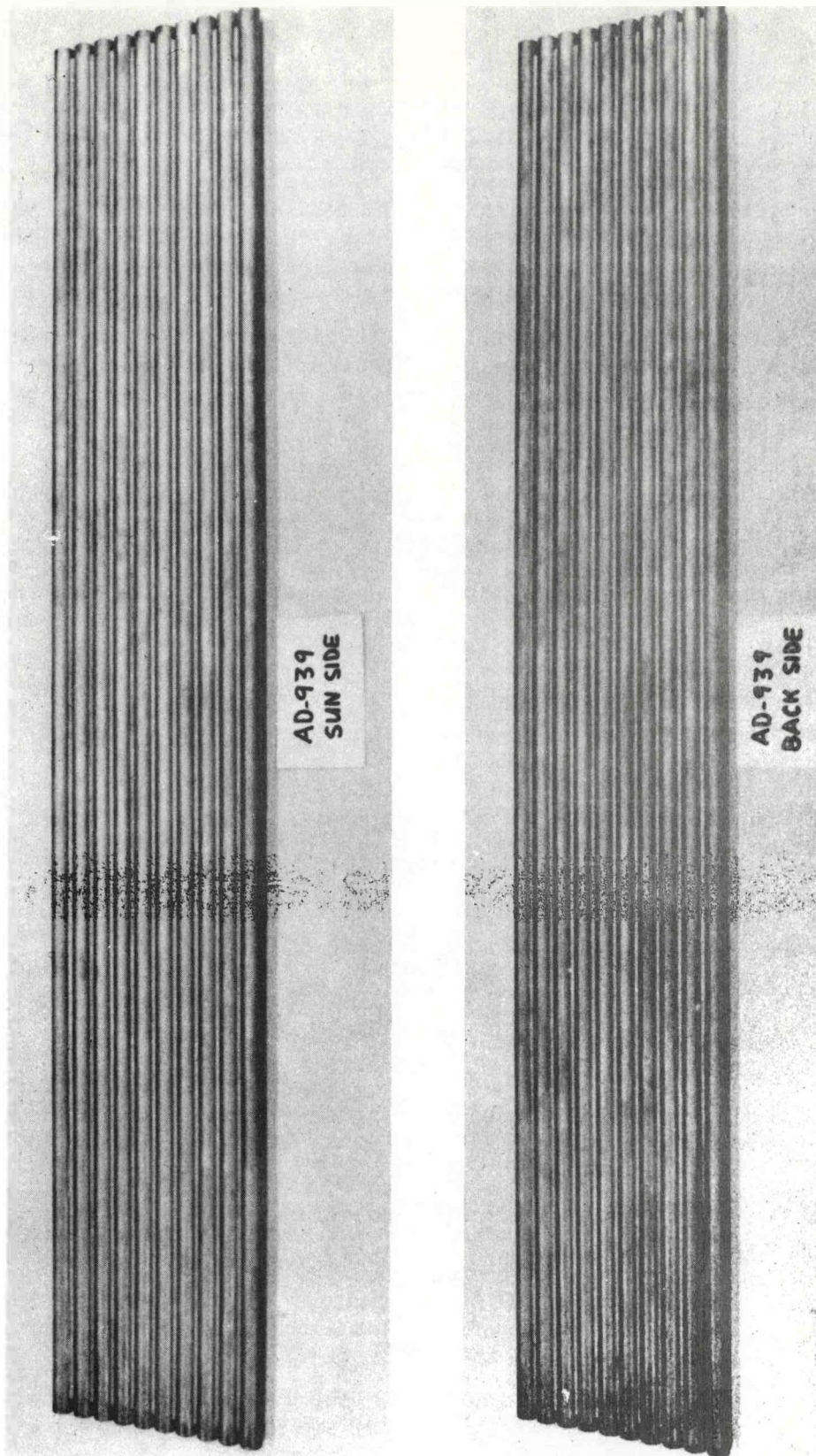
The development project was performed to provide a technique/procedure for the furnace brazing of thin walled tubular Incoloy 800 absorber panels for sodium cooled solar central receiver plants, with an immediate objective of providing a technique for the brazing/fabrication of a test panel scheduled for solar testing at the Central Receiver Test Facility in Albuquerque, N.M. in late 1980 and early 1981. This development included selection of a brazing filler metal type, form and application procedure; determination of a joint design that would result in good brazability and mechanical properties; and the selection of an appropriate panel brazing procedure, atmosphere control, orientation and fixturing method in a brazing furnace.

Over 100 test specimens were fabricated and brazed. The results of this work were demonstrated by the successful brazing of 122 cm (4 foot long), ten tube wide absorber panel segments as shown in Figure 2-33. Some of the major conclusions resulting from the project are listed below.

2.4.1 SPECIFIC CONCLUSIONS AND RECOMMENDATIONS

The following specific conclusions relating directly to the brazing of the test panel can be made:

- Tube Material - Incoloy 800, ASME SB407, High Carbon Cold Drawn Annealed.
- Braze Material - Nickel-manganese-silicon brazing filler metal AD 939 was preferred due to the relatively high ductility of the braze fillets, the ease of brazing, and the good tensile and shear strength. Nickel-chromium-silicon filler metal NB-LM was selected as an alternative.
- Non-Melting Filler Material - Incoloy 800 filler material was selected. The filler strip material should be of similar composition to the tube to minimize differential thermal expansion.
- Braze Filler Metal - The form of filler metal chosen was - 140 mesh powder, applied to the joint as a dry fillet and subsequently bonded with an aerosol spray acrylic base cement. Alternative methods were to apply the fillet as a slurry of powder, acetone and cement, or a paste of powder and cement.
- Tube Plating - Approximately 0.0107 to 0.0152 mm nickel electrolytic plating in a sulfamate bath per specification AMS 2424 was selected. Specification AMS 2403 could be used as an alternative.
- Tube Cleaning - Tubes wiped with acetone on a nylon cloth before applying filler metal and stop-off compound is recommended as a minimum.



80-075-30

Figure 2-33 Absorber Panel Segment Brazed to Demonstrate on a Large Scale the Preferred Set of Parameters Evaluated

- Stop-off Compound - The use of stop-off compounds on tubing was found to be necessary to control braze metal flow. Microbraz Green or boron nitrate brushed on were found acceptable.
- Joint Configuration - An hourglass (double concave) shaped I800 filler strip between adjacent tubes was selected as the preferred joint design to provide maximum joint area, strength and ductility. An alternative joint design used simple tube-to-tube line contact with large brazing filler metal fillets on each side of the joint.
- Panel Orientation - A horizontal orientation of the tube axis resulted in the least filler metal runoff and minimum tube erosion. The panel was inclined 45 to 60 degrees to facilitate fixturing and weighting. The alternative orientation, although less satisfactory for fixturing maintained the panel horizontal in all planes.
- Panel Fixturing - Weights were selected as the fixturing method. This method provided both a self-closing joint gap during brazing, and kept the panel flat along its length and width.
- Furnace Design - Use of a pure dry hydrogen controlled atmosphere furnace with an all metallic furnace hearth is recommended (hydrogen inlet purity 99.999%).
- Braze Conditions - Braze conditions selected were:
 - Equalize temperature - 900°C (1650°F)
 - Braze temperature - 1004°-1016°C (1840-1860°F)
 - Hydrogen dewpoint - \leq -62.2°C (-80°F)
 - Hydrogen exchange - 35 to 40 volumes
 - Braze time - 10 to 12 minutes

2.4.2 GENERAL CONCLUSIONS

The following general conclusions are presented:

- Longitudinal bend tests, both with and without concave filler strips, showed good ductility with AD 939 and NB LM filler metals where gap widths were reasonable.
- The tubing grain boundary penetration was low when braze temperatures were held below approximately 1065°C (1950°F) --- 1 - 2 grains deep. Excessive grain growth occurred at 1121°C (2150°F) with high carbon I800.
- Braze filler metal run-off was found to be controllable with contoured filler strip and horizontal braze orientation.
- The use of stop-off compounds on tubing was found to be necessary to control braze metal flow.
- Excess filler metal was found to be essential for complete joint fill. (Gaps of approximately > 1 mm are difficult to fill.)
- The use of a filler strip controls braze run-off, microstructure quality, joint width and surface area, and can aid in tube fixturing.

Wide gaps can be readily bridged with some alloys, but large fillets and wide braze metal joint gaps are susceptible to excessive porosity and low ductility.

Results of tensile tests show both strong and ductile braze joints when contoured joints with selected filler material are used.

Results of test panel tubing tensile tests show < 1038°C (1900°F) braze cycle does not degrade cold drawn annealed I800 yield and ultimate strengths below ASTM minimums.

Braze foil is relatively expensive and difficult to work with in contoured joints.

Braze tape results in excessive porosity or incomplete filling of variable gap joints.

Erosion of tubing and filler strips was found to be high when brazing at 1121°C (2150°F), and negligible at 1065°C (1950°F). Tubing erosion can be essentially eliminated by controlling the amount of alloy, minimizing the braze temperature, or by braze composition control.

Nickel plating of I800 tubing and filler strip is necessary to produce uniform, consistent wetting and flow-through.

SECTION 3

STRESS CORROSION

The objective of this Subsystem Research Experiment (SRE) is to determine the effect of high temperature water and steam environments on the stress corrosion cracking (SCC) behavior of the reference superheater and reheater alloy, Incoloy 800, and a backup candidate material, Inconel 617. The need is to determine the SCC susceptibility of the alloys in environments that are directly related to conditions of operation from startup to normal operation. The environments are expected to vary from superheated steam containing dissolved impurities to wet-dry cyclic conditions. The impurities, primarily caustic, are expected due to off-chemistry conditions likely to occur during plant operation.

The SRE included two efforts. First, a literature survey to identify industry experience with SCC of Incoloy 800, then a series of experiments to define the metallurgical and heat treatment states of the alloys which establish an SCC-immunity regime or minimize the onset of SCC.

3.1 LITERATURE SURVEY

A literature survey has been conducted concerning the SCC of Incoloy 800 in contaminated superheated steam. In 482°C (900°F) superheat studies, with caustic dissolved in the steam, Incoloy 800 was extremely resistant to SCC, while Inconel 600 and 300 series austenitic stainless steels were very susceptible. In other superheat studies, Incoloy 800 was far more resistant to chloride and caustic SCC than the austenitics. Oxygen (>2 ppm) was needed for the chloride cracking of Incoloy 800, but was not required for caustic cracking. The worst environment for SCC appeared to occur under conditions of dryout. In superheat regions, the chloride concentration is limited to that dissolved in the steam. However, above the critical temperature, water droplets containing caustic can form a liquid film on superheater surfaces. These films have a very high caustic concentration and a very low vapor pressure. It has been estimated that caustic solutions can exist above 500°C (932°F), but chloride solutions would not exist above 40°C (104°F) superheat.

The full survey is provided as Appendix B.

3.2 EXPERIMENT DESCRIPTION

The experimental approach during the Phase II testing involves both short and long term experiments. Short-term straining electrode (SET) and constant extension rate (CERT) tests in 5 and 10 percent NaOH solutions at 316°C (600°F) will screen the test matrix parameters to establish the metallurgical conditions for improved SCC resistance. CERT refers to a tensile type test with a predetermined constant strain rate conducted at high temperature in a caustic environment. The objective is determination of the stress corrosion potential susceptibility of the material. SET experiments are similar to CERT but utilize an applied potential as determined from the polarization curve. In preparation for the SET, polarization curves will be determined in electrolytic solutions containing 5 and 10 percent NaOH at 316°C (600°F). The test matrix for the SET and CERT experiments is shown in Table 3-1. Long-term tests will be performed on Incoloy 800 under prototypic temperature (538 to 593°C (1000 to 1100°F)) and pressure conditions, which determine the level of stress for tubing and tubesheets in the superheaters. These tests will determine the SCC behavior of Incoloy 800 under superheat conditions in steam containing NaOH.

Table 3-1

TEST MATRIX FOR THE ELECTROCHEMICAL STRESS CORROSION STUDIES AT 316°C (600°F)

<u>Experiment</u>	<u>Environment</u>	<u>Material</u>	<u>Variables</u>
SET	5% NaOH	Incoloy 800	Potentials(2)* Metallurgical(4)**
		Inconel 617	Potentials(2) Metallurgical(2)***
	10% NaOH	Incoloy 800	Potentials(2) Metallurgical(4)
		Inconel 617	Potentials(2) Metallurgical(2)
CERT	5% NaOH	Incoloy 800	Metallurgical(4)
		Inconel 617	Metallurgical(2)
	10% NaOH	Incoloy 800	Metallurgical(4)
		Inconel 617	Metallurgical(2)

* Based on the polarization curves.

** As-received, sensitized, and cold worked Incoloy 800H and mill annealed Incoloy 800.

*** Annealed and cold worked.

Previous SCC studies⁽¹⁾ at 316°C (600°F) indicate that the susceptibility of

(1) M.E. Indig, "Constant Extension Rate Testing for LMFBR Steam Generator Applications," NEDO-12668, May 1977.

Incoloy 800 in caustic is a function of metallurgical conditions and oxidizing potential. The most susceptible condition was mill annealed followed by sensitized, Grade 2 annealed, Grade 1 annealed and cold worked. At specific oxidizing potentials, even highly resistant conditions could be failed.

3.3 SRE STATUS

Tensile specimens of as-received and cold worked Incoloy 800H and annealed and cold worked Inconel 617 have been fabricated for the SET and CERT experiments. To complete the material test matrix, specimens of as-received Incoloy 800H will be sensitized, and mill annealed Incoloy 800 material is needed for specimen fabrication. The mill annealed material is presently in shipment.

Incoloy 800H tubing material is being cold drawn to size for the long-term prototypical tests. From the cold drawn material, solution annealed, mill annealed, and sensitized conditions will be produced by heat treating. There has been no success in locating Inconel 617 tubing.

The polarization curves in 5% NaOH for the material conditions listed in Table 3-1 being determined in a test facility at the GE-Vallecitos Atomic Laboratory (VAL). The SET experiments in 5% NaOH are also being performed. These efforts will be completed in late Spring 1980.

The General Electric Company provided components needed to duplicate the VAL test facility within GE-ARSD (SET and CERT use the same facility) are presently on order. The expected delivery date for the autoclave, Instron, potentiostat, etc. is July 1980. The required structural components will be machined during the interim, with an expected completion date for the test facility of September 1980. The polarization curve determination and SET experiments in 10% NaOH will be initiated in October 1980. The CERT experiments in 5 and 10% NaOH will follow and will be completed in February 1981.

The Incoloy 800H tubing material being cold drawn from the long-term tests is expected during May 1980. In the interim, the type of experiment that will be performed to simulate the prototypical superheat conditions will be determined.

SECTION 4

FATIGUE CRACK GROWTH AND FRACTURE TOUGHNESS

Since the ASME Code is used for the design of solar components, specifically the steam generators and the absorber panels, the code requirement for fatigue crack growth data in prototypical environments is a basic need, since only limited crack growth data in air are available for Incoloy 800. The objective of this material SRE is to provide preliminary scoping data for the design of the solar power plant. These data will be necessary to complete the structural analyses of the solar panel header regions, heavy section of the superheater, and transition joint regions. The matrix for testing that will be performed during Phase II was developed from anticipated service conditions envisioned for the solar power plant. Sodium environments have been chosen to be scoped in Phase II. The tests in air will primarily be used for control so that an assessment of the sodium property enhancements can be made.

4.1 FATIGUE CRACK GROWTH

These data will be used to quantify absorber header tube resistance to the thermal striping phenomenon. Thermal striping may result from cyclic impingement of side by side sodium streams in the discharge region of the absorber panel which may be at different temperatures. The near threshold crack growth data will be required for this analysis. Fatigue crack growth data are also needed to complete the piping integrity analysis for the sodium piping system.

4.1.1 BACKGROUND DATA

Environmental effects strongly influence the fatigue crack growth behavior of materials. Therefore, the elevated temperature fatigue crack growth behavior will be characterized under prototypical conditions. Defects may arise from fabrication processes, fatigue loadings (mechanical or thermal), and creep processes, such as triple point wedge crack formed by grain boundary sliding. The use of linear elastic fracture mechanics has been shown to be quite useful in characterizing the crack growth behavior. For many years, a large effort has been devoted towards understanding the crack growth behavior in the power law regime, typically the crack growth rates ranged from 10^{-5} to 10^{-2} mm/cycle ($\sim 10^{-6}$ to 10^{-3} inch/cycle), indicated as regime B in Figure 4-1. The fatigue crack propagation behavior could be

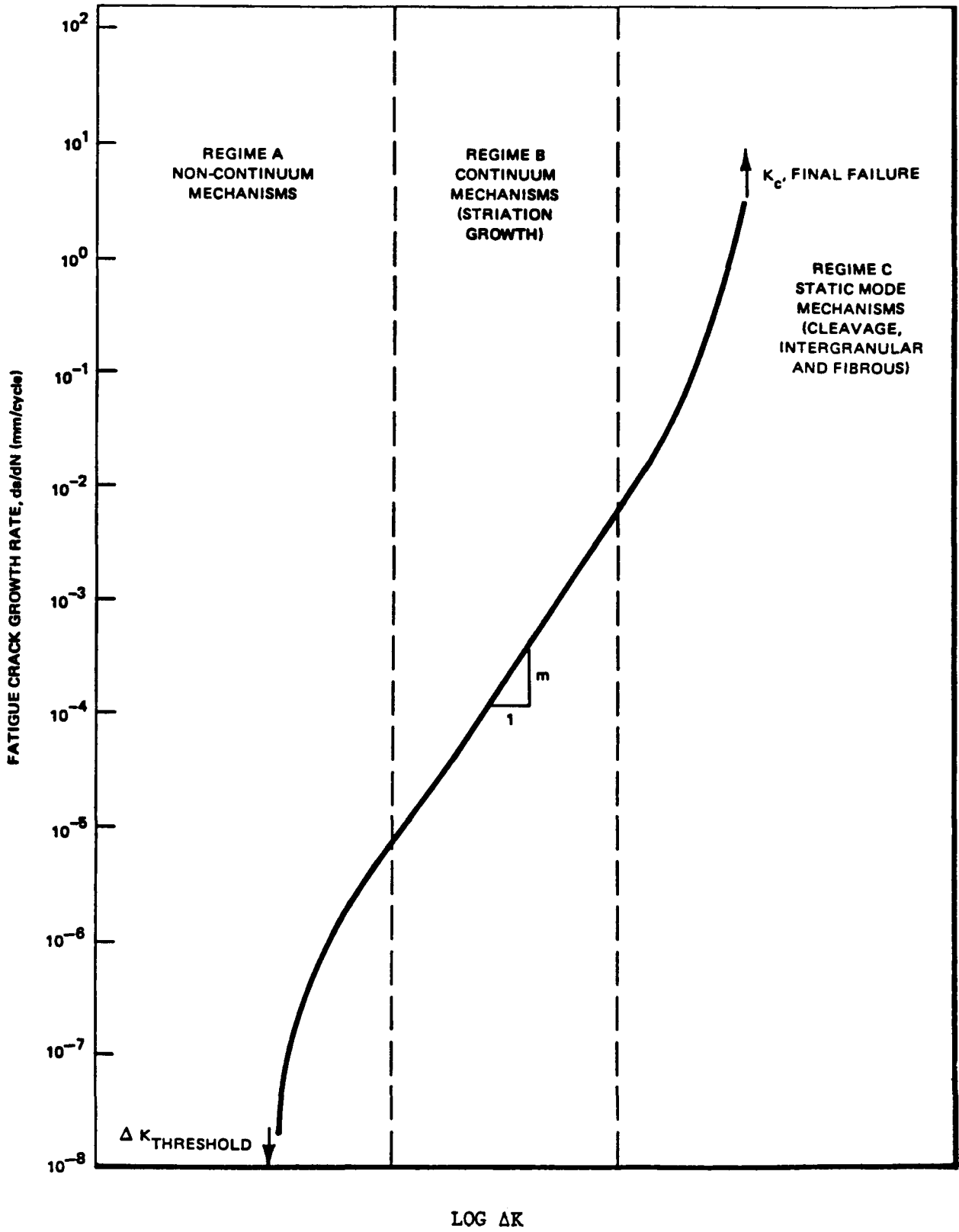


Figure 4-1. Three Stages of Fatigue Crack Growth Behavior

described with the well-known Paris equation:

$$\frac{da}{dN} = C_0 (\Delta K)^m$$

where da/dN is the crack extension per cycle, ΔK is the applied stress intensity range, and C_0 and m are constants that include material and test or service condition effects. Fatigue crack growth tests by James⁽¹⁾ and Carden⁽²⁾ have been performed to characterize the fatigue crack growth behavior of Incoloy 800 and 800H in air. James found in his air test at 0.67 Hz that da/dN increased by a factor of 4.1 between room temperature and 649°C (1200°F). In contrast, Type 316 stainless steel increased by a factor of 11.8 over the same range. James suggested the difference in crack growth rate was attributed to the superior oxidation resistance of Incoloy 800 at this temperature. No differences in crack growth behavior of Incoloy 800 and 800H were detected. The effect of thermal aging at 538°C (1000°F) for 5000 hours resulted in a lower da/dN than the unaged specimens. Carden's work on Incoloy 800 included the effects of frequency, wave form and temperature. At crack growth rates above $\sim 10^{-2}$ mm/cycle ($\sim 10^{-3}$ in/cycle), the crack growth behavior departs from the power law behavior and increases rapidly with increasing ΔK , regime C in Figure 4-1.

Ultimately, the fracture toughness of the material is reached, K_c . The growth rate is strongly affected by both microstructural features and mean stress. Failures occur by modes that are characteristic of static fractures, including cleavage, intergranular and fibrous fractures. At very low crack growth rates below approximately 10^{-5} mm/cycle (10^{-6} in/cycle), the crack growth rate again departs from the power law behavior but in the opposite direction. As ΔK is reduced, the crack growth decreases rapidly and ultimately a threshold stress intensity is reached below which the crack ceases to propagate, regime A in Figure 4-1. At these low growth rates, the measured growth rates are less than a lattice spacing per cycle, suggesting that the crack does not grow uniformly across the entire crack front.

4.1.2 EXPERIMENT DESCRIPTION

The test material used will be representative of the material to be used in the header, absorber panel tubes, and heat exchanger materials. Evaluation of the fatigue crack growth behavior will be done through the use of linear elastic fracture mechanics methods. A 0.5 inch thick 3-point bend specimen geometry will be used to measure the fatigue crack growth rates in the linear growth regime. The test temperature of 1000°F will be used in all the tests, at a cyclic frequency of 0.5 cycles per minute. Test environments will include air and sodium. The sodium environments will be prototypical of the chemistry in the solar power plant

components. The crack growth tests in sodium are intended to scope the environmental influence. The test data will establish the design data for the solar power plant components.

Near threshold fatigue crack growth tests will be performed with 1/2-inch thick compact tension specimens. To accumulate the required cycles for near threshold growth rates, a test frequency of approximately 100 Hz is required. Tests will be performed at 100 Hz in inert environments at elevated temperatures.

4.1.2 TEST PROGRAM

A plate of Incoloy 800H, 40" x 23" x 2" thick, has been procured as test material for the test program. The chemical composition of this material is given in Table 4-1. The exact heat treatment of this material could not be ascertained from the manufacturer (Huntington Alloys). A mill run of this material could have been either air cooled or water quenched from the solutionizing temperature of 1177°C (2150°F). To eliminate this uncertainty, the test specimens were heat treated prior to testing. The test specimens were solutionized at 1177°C (2150°F) for one hour per inch of thickness and water quenched to room temperature.

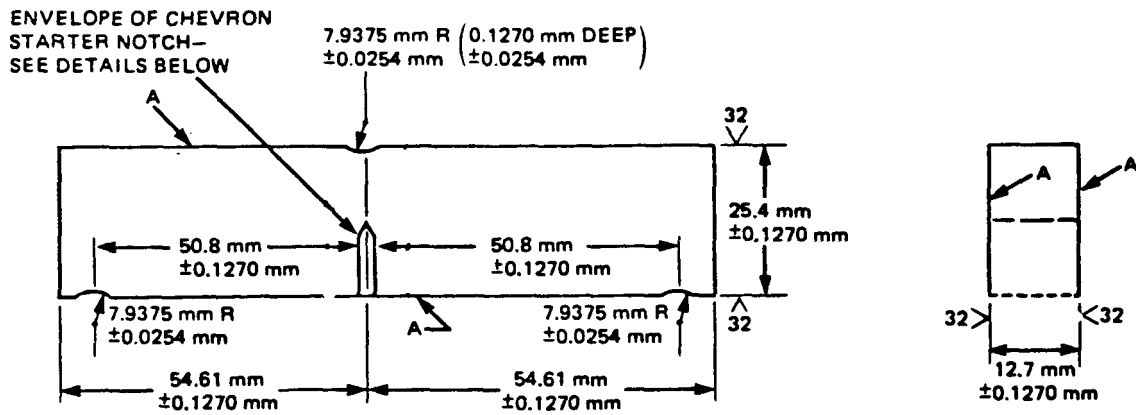
Table 4-1

CHEMICAL COMPOSITION OF TEST MATERIAL, WT % ALLOY 800H

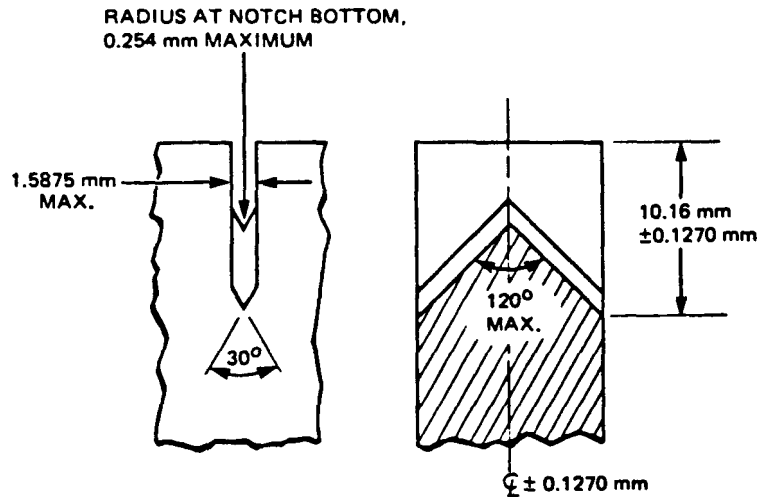
	Heat* HH 8703A
C	0.06
Mn	0.82
Fe	45.35
S	0.002
Si	0.16
Ni	32.53
Cr	19.82
Cu	0.35
Al	0.49
Ti	0.42

* Vendor Test Report

Two types of test specimens have been fabricated for use: 1/2-inch thick bend specimens (Figure 4-2), and 1/2-inch thick 1-T compact tension specimens (Figure 4-3). The bend specimens will be used in low frequency tests at elevated temperatures in air and sodium. These tests will be performed with two existing facilities, one for air and the other for sodium tests. The sodium crack growth test facility is shown schematically in Figure 4-4. Each facility can concurrently test three bend

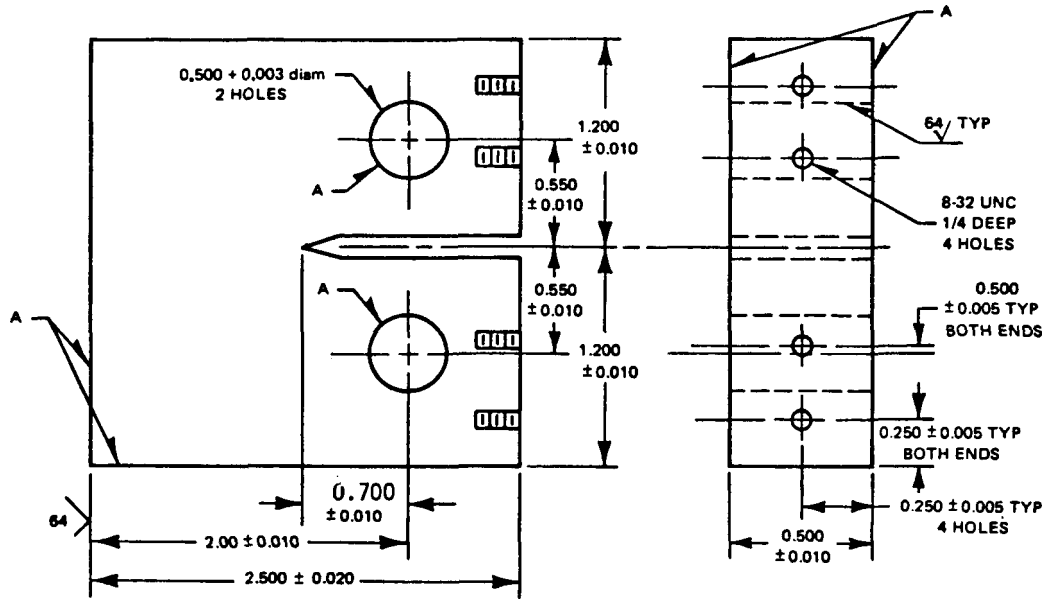


NOTE 1: A SURFACES SHALL BE PERPENDICULAR AND PARALLEL WITHIN 0.0254 mm TIR
 NOTE 2: CRACK STARTER PEPENDICULAR TO SPECIMEN LENGTH AND THICKNESS TO WITHIN ±2 DEGREES



CHEVRON NOTCH CRACK STARTER DETAILS

Figure 4-2. Fatigue Crack Growth, 3-Point Bend Specimen, Showing Weld Metal Location for Weld Tests



NOTES:

1. "A" SURFACES SHALL BE PERPENDICULAR AND PARALLEL WITHIN 0.002 TIR
2. CRACK STARTER SHALL BE PERPENDICULAR TO SPECIMEN LENGTH AND THICKNESS TO WITHIN ± 2 deg

Figure 4-3. 0.5-in.-Thick 1T Geometry Compact Tension Specimen, All Dimension in Inches

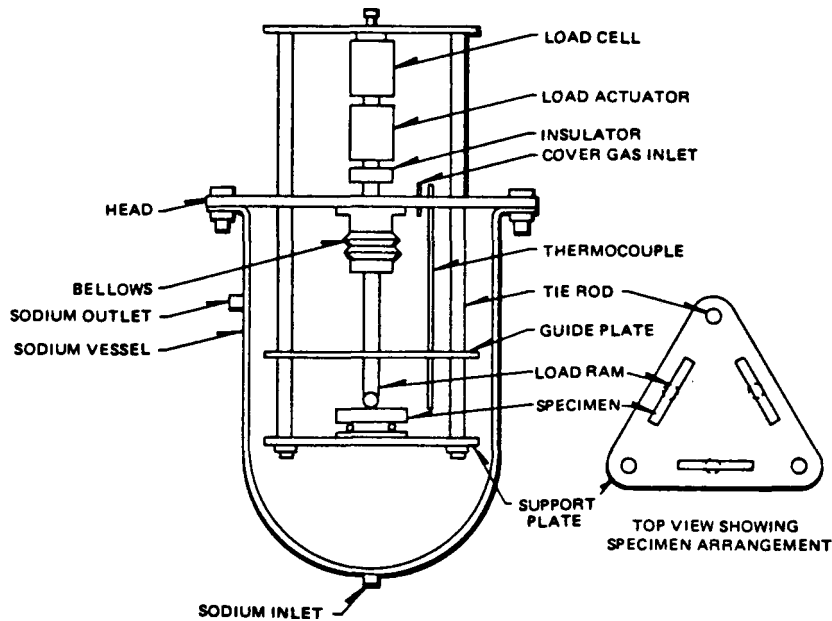


Figure 4-4. Schematic Illustration of Fatigue Test Sodium Pot

specimens under the same or different loading conditions. The facilities are designed for test temperatures up to 538°C (1000°F). The crack growth tests in the near threshold crack growth regime will be performed with the compact tension specimen. These tests are to be performed in a new test facility provided by the General Electric Company. The environmental chamber for the system is currently being fabricated. Preliminary tests in air at room temperature are in progress. An example of the early data is shown in Figure 4-5. The scatter band of the data compares well with that of James.⁽¹⁾

Fatigue crack growth tests in air and sodium in the linear crack growth regime will be continued in the current facilities. The high temperature load frame and environmental chamber for performing the elevated temperature inert atmosphere tests are currently being fabricated. These will be incorporated into the test system as time permits when they are received from the manufacturer.

4.2 FRACTURE TOUGHNESS

In the heavy sections of the central receiver system, there is a possibility that the flaws will reach a critical flaw size where catastrophic failures may result. Justification for leak before break must be made as part of the system integrity analysis.

Objective

Plane strain fracture toughness values, K_{IC} , are needed to justify resistance to nonductile fracture. Because of the low yield strength of the material at elevated temperatures of the reference alloy, large crack tip plasticity occurs. Elastic-plastic test procedures, J-integral methods, must be used to determine the K_{IC} at elevated temperatures.

Work Planned for Phase II

The test material used will be representative of the material to be used in the heavy sections of the central receiver system. Two-inch thick compact tension specimens will be used to evaluate the fracture toughness of the material at room temperature and at elevated temperatures. The test environment will be air.

Status

Eight 2-inch thick compact tension specimens have been fabricated from the 2-inch thick plate. The tests will be performed in air at room temperature and 538°C (1000°F). Because of the low yield strength of Incoloy 800 at elevated temperatures, large crack tip plasticity occurs. Elastic-plastic test procedures and J-Integral methods must be used to determine the K_{IC} at elevated temperatures. These data will

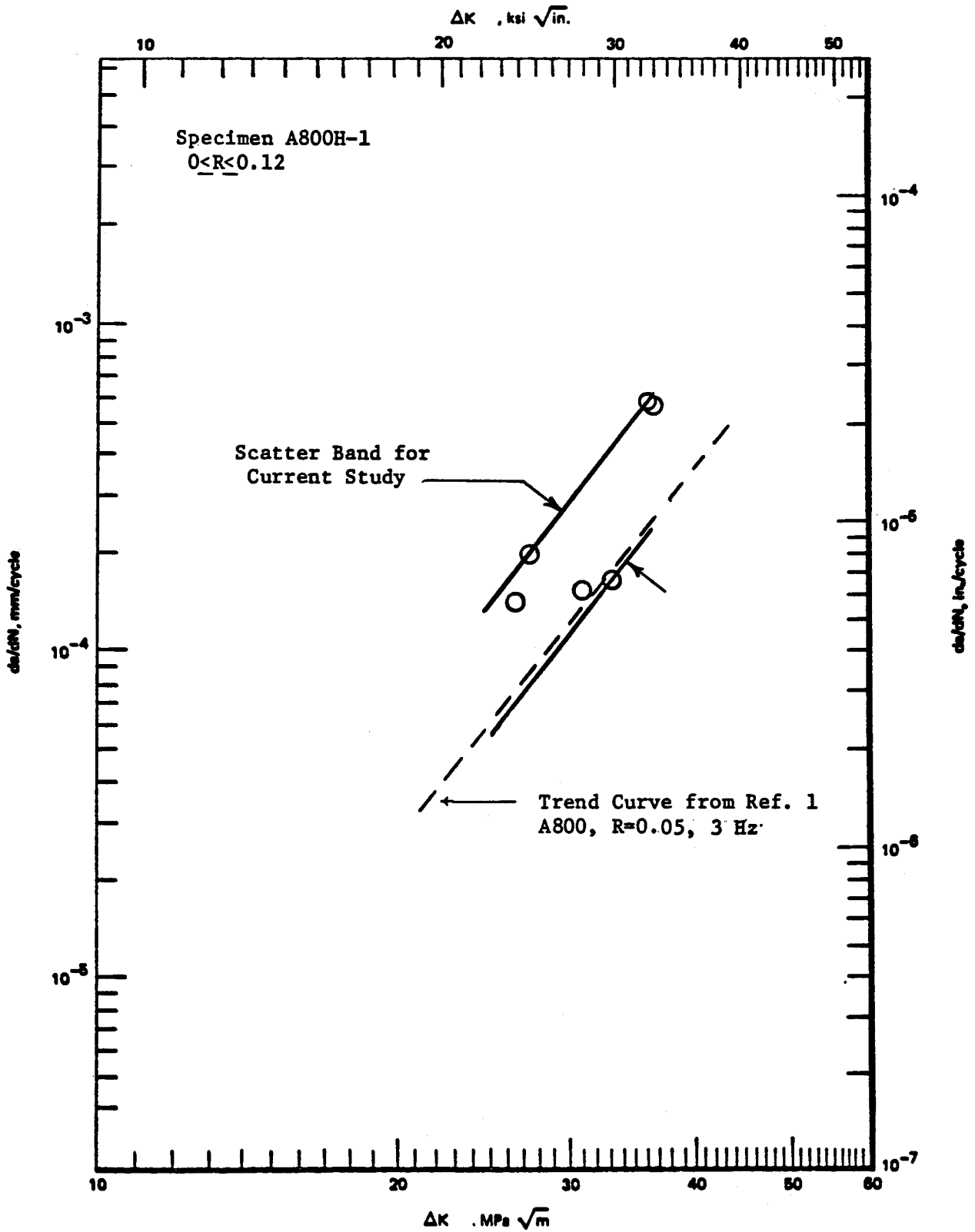


Figure 4-5. Fatigue Crack Growth Behavior of Incoloy 800H in Air at 24°C (75°F) at 100 Hz.

serve as a basis of comparison for tests characterizing the effects of thermal aging and welding, variables not addressed in this Phase II program. Lai and Kimball⁽³⁾ found from their aging studies of Incoloy 800H that the room temperature Charpy impact toughness decreased substantially after initial short-time aging and then leveled off with long-time aging. The aging temperatures ranged from 538°C to 816°C (1000 to 1500°F). Specimens aged for 8000 hours at 593 and 649°C (1100 and 1200°F) resulted in the highest hardness and the lowest toughness values (75J)(55 ft-lbs). The fracture was predominantly intergranular.

Fracture toughness tests for the material will be performed as soon as the specimens are precracked.

SECTION 5

REFERENCES

Section 1

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Section 2

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2. A.E. Carden, "A Phenomenological Analysis of the Interaction of Creep and Fatigue," Ph.D. Dissertation, Dept. of Metallurgy, University of Connecticut, 1972.
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APPENDIX A

PRELIMINARY PANEL BRAZING SPECIFICATION

Rev. 0, 9/26/79

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FURNACE BRAZING LONGITUDINAL JOINTS BETWEEN 1800 TUBES

1. SCOPE

1.1 This specification establishes the procedures for hydrogen furnace brazing of Incoloy 800 tubing to form flat solar absorber panel segments. These segments are to be assembled into a test panel 51 tubes wide with a brazed joint length of 4.57 m (15 ft).

1.2 This specification is to be used for fabricating the General Electric absorber test panel for the Advanced Central Receiver Solar Power System.

2. APPLICABLE DOCUMENTS

2.1 The following documents form part of this specification to the extent specified herein. Unless otherwise indicated, the issue in effect on the submittal date of this specification shall apply.

2.2 Federal Specification QQ-N-290A, Class 1, Grade F to E, Electrodeposited Nickel Plating.

2.3 ASTM Standard B407, Specification for Nickel-Iron-Chromium Alloy Seamless Pipe and Tube.

2.4 AMI Amdry Standard Brazing Alloys Data Sheet, Copyright 1979.

2.5 GE-ARSD Drawing No. HPO-6-28-79, Rev. 2, "Tubing Joint Filler Strip."

3. REQUIREMENTS

3.1 General

3.1.1 This specification was developed specifically for brazing of longitudinal joints between Incoloy 800 tubing with 19.05 mm (3/4 in) outside diameter. The number of tubes in a panel segment may be from 3 to 17, as specified by Design Engineering.

3.2 Materials

3.2.1 Braze Alloy - The Braze Alloy will be Amdry 939, manufactured by Alloy Metals, Inc., Troy, Michigan.

3.2.2 Alternate Braze Alloy - AWS Classification BNi-2 (Microbraz LM, produced by Wall Colmonoy Corp., Detroit, Michigan; Filler Metal 4777, produced by Fusion, Inc. Willoughby, Ohio and Amdry 770 or 770A are all adequate).

3.2.3 The braze alloy form will be metal powder of approximately-140 mesh, applied as a continuous fillet of dry powder to one side of the joint, and then bonded in place with acrylic enamel aerosol sprayed at a distance of 45 to 60 cm (18 to 24 in) for 25 to 30 seconds per 30.5 cm (1 ft) of joint length. The aerosol will be Sprayon #2000 Clear Color Code and Refinishing Enamel, supplied by Industrial Supply, Sprayon Products, Sherwin-Williams Corp., Anaheim, California 92806.

3.2.4 An alternate lacquerized enamel aerosol is Osborn #1800 Clear, supplied by Sherwin-Williams Corp.

3.2.5 An alternate alloy form will be either metal powder bonded with either acrylic-base cement to form a slurry, or organic-base premixed paste.

3.2.6 Cement will have a viscosity of 500 centipoise, and when used to form a slurry will be mixed with metal powder in the volume ratio of approximately 50% powder/50% cement. Cement will be Wall Colmonoy Microbraz Cement Grade 500 or equivalent type and viscosity.

3.2.7 Tubing will be Incoloy 800 with 19.05 mm (3/4 in) outside diameter, procured and certified to meet ASTM B407. Tubing will be handled during all operations so as to prevent any scratches, denting or bending. Tubing will be cleaned with acetone only and lint-free nylon cloth on the outside before assembly. Tubing will be handled with clean white gloves after cleaning, and will be stored under dust-proof cover.

3.2.8 Filler strip will be concave lens shaped, and made of Incoloy 800. The filler strip will be free of bends and burrs, and will have a camber concavity (depth of chord) not to exceed 0.8 mm/m (0.01 in/ft), and will meet the dimensional and finish requirements of GE-ARSD Drawing #HPO-6-28-79, Rev. 2. Filler strip will be cleaned, handled and stored according to the same requirements as the tubing, as listed under Section 3.2.5.

3.2.9 Tubing and concave lens shaped filler strip are to be electroplated with nickel per Federal Specification QQ-N-290A. A dull finish produced using no brighteners, to be used for brazing purposes, is to be specified to plating vendor. Plating thickness will be 0.0102 to 0.0152 mm (0.0004 to 0.0006 in). Any bubbles, pits or after hydrogen annealing, are unacceptable. A continuous metallurgical bond between the plating and the base alloy is required. Archive samples of tube and filler strip, each 31 cm (1 ft) long will be plated with each batch of material plated.

3.3 Brazing Procedure

3.3.1 Tubes and filler strips are to be assembled on a surface plate which is flat across the width and across the length within 1.8 mm (1/32 in). Braze alloy will be applied per Section 3.2.3 of this specification such that the width of the alloy is 6.35 to 7.62 mm (0.250 to 0.300 in) wide, as measured across its top surface. The braze alloy will extend continuously along the 3.81 m (15 ft) length of joint to be brazed.

3.3.2 Stop-off compound type Microbraz Green or equivalent is to be applied around the full circumference of the tubes for a 25 mm (1 in) length, located 6.4 to 12.7 mm (1/4 to 1/2 in) from each end of the braze joint area. Stop-off is to be applied to all fixture surfaces which are in contact with the tubes and to all fixture surfaces in the proximity of the tubes which may be exposed to inadvertent braze runoff.

3.3.3 Stop-off compound is to be applied to completely protect the tube surfaces between alloyed areas. Extreme care must be taken in order to prevent any stop-off from coming in contact with the braze alloy. This is to be accomplished by protecting the cemented braze alloy with plater's tape before stop-off application. Any areas of braze alloy contaminated with stop-off must be mechanically removed and alloyed again per Section 3.2.3 before brazing.

3.3.4 Brazing flux is not to be used, including any vaporizing types.

3.3.5 Brazing is to be performed in a pure dry hydrogen atmosphere with a furnace inlet dew point of -62°C (-80°F) or lower. During brazing the tubes are to be oriented with their axis horizontal, and leveled with a bubble-tube level. The panel segment and fixture reference surface is to be oriented 45° to 60° from the horizontal. Fixturing will include a rigidly fixed straight edge along the full length of the panel segment against the lowermost tube, and a free-floating straight edge against the uppermost tube. The upper straight edge will be composed of approximately 15 independently floating 30.5 cm (1 ft) long pieces. Floating straight edges will also be placed transversely across the tubes at a minimum of one foot intervals along the tube length. Each upper tube straight-edge piece and each transverse straight-edge piece will have approximately 4.5 kg (10 lb) of weight attached to it. Corresponding pairs of upper and transverse straight-edge pieces may be mechanically linked, if desired. All fixture materials are to be made of metal only, including muffle internals.

3.3.6 The brazing sequence will be as follows:

- a. Purge furnace muffle with inert gas as required for safety considerations;
- b. Heat panel segment in hydrogen atmosphere to 899 to 904°C (1650 to 1660°F), and hold until all areas are equalized within this temperature range;
- c. Heat as rapidly as possible to within 1016 to 1027°C (1860 to 1880°F) and hold for 10 to 12 minutes;
- d. Cool rapidly to below 904°C in hydrogen atmosphere;
- e. Cool so as to minimize thermal distortion, to room temperature;
- f. Control flow rate of hydrogen during the brazing cycle so as to provide 40 or more furnace volume changes.

3.4 Rebrazing and Rework

3.4.1 Furnace brazing of the panel segments is allowable only after GE representative inspection of the brazed panel and approval for rebrazing. Approval for rebrazing will be applicable only for rebrazing once, and subsequent rebrazing will require additional GE representative inspection and approval. Torch rebrazing (either electric or arc or exothermic gas) is not allowed.

3.4.2 The braze alloy for rebrazing will be Amdry 939 only, as specified under Section 3.2, Materials. The rebrazing temperature range will be 1000 to 1010°C (1855 to 1875°F), which is 3°C (5°F) lower than the initial brazing temperature range. All other brazing materials will be the same as specified under Section 3.2, Materials. All other rebrazing procedures will be the same as specified under Section 3.3, Brazing Procedure, except that the braze alloy may be applied from either side of the panel segment. All technical reporting requirements under Section 3.8, Reporting, will be the same as for initial brazing, with the additional requirement that an accurately dimensioned map of the panel segment which shows the rebrazed area will be provided.

3.4.3 No tube grinding or metal removal of any kind, or tube or panel straightening after brazing is allowed without GE representative prior inspection and approval to do so.

3.5 Post Braze Cleaning

3.5.1 After brazing, and after inspection and approval by GE representative, the test panel segments may be cleaned at GE option on all surfaces except the unbrazed length at the tube ends, by grit blasting with Ballentina Impact Beads, Size AE,

supplied by Clementina Co., Belmont, California or approved equivalent material. Grit blasting will be followed by acetone washing and wiping with clean nylon cloths.

3.5.2 All cleaning performed should be such that the nickel plating is left intact on all plated surfaces. Tight fitting tube end caps are to be used on both ends of all tubes during cleaning and handling to prevent internal tube contamination. The end caps will be removed during brazing. No water-containing solutions are to be used.

3.5.3 After cleaning, the panel segments are to be handled with clean white gloves only in order to preserve cleanliness for future surface treatment. End caps are to be left in the tubes after cleaning.

3.6 Inspection

3.6.1 Supplier of brazing services will perform all inspections and quality checks as required by this specification, including those listed under Section 3.8, Reporting. A record of the results of these inspections will be included in the report.

3.6.2 Customer will be notified three working days in advance of panel segment assembly and brazing. Customer representatives may be present at customer option, to witness and inspect panel segments during all operations required by this specification.

3.7 Post Braze Packaging

3.7.1 Immediately after brazing the panel segments are to be sealed in polyethylene sheet or equivalent to keep them dry and dust free during storage and shipment. Panel segments will be sealed with adequate desiccant.

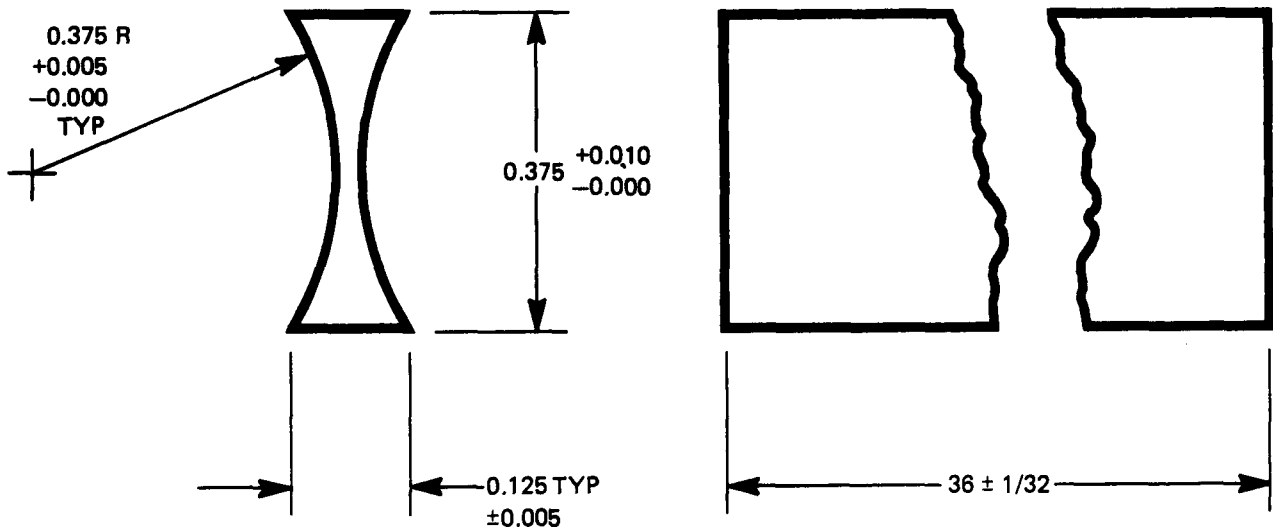
3.7.2 Panel segments will be weatherproof crated for truck shipments so as to prevent bending, scratching or other damage. Crates will be identified with customer name and address, purchase order number and list of contents.

3.8 Reporting

3.8.1 A technical evaluation report (3 copies) will be submitted to the customer on the supplier's letterhead within 10 working days after completion of each panel segment. The report will include the following minimum information:

1. Panel segment identification number, length, number of tubes and fixturing used.
2. Braze alloy type, form and amount used, and manufacturer's name and lot number.

3. Hourglass filler strip length, drawing number and revision number.
4. Orientation of panel segments during brazing (angle to horizontal).
5. Type of nickel plate used on tubes and/or filler strips.
6. Specify hydrogen dew point attained, volume changes and flow rates.
7. Panel segment center and edge heating and cooling rates and equalizing time.
8. Panel segment time in brazing temperature range.
9. Brazing temperatures at panel segment center and edge.
10. Extent of filler metal wetting and fillet size (note any surface voids).
11. Amount of panel segment warping from flat shape (both longitudinal and transverse).
12. Amount and location of excess alloy localized build-up, if any.
13. Amount and location of braze metal run-off, if any.
14. Minimum clearance of joint after jiggling, average longitudinal clearance.
15. Any abnormal reaction in furnace or materials to be explained.
16. Close-up photographs representative of the braze joint area, taken on both sides of each panel segment.



DO NOT BREAK ANY EDGES

MATERIAL - INCOLOY 800

SURFACE FINISH - 64

80-273-02

Figure A. Tubing Joint Filler Strip

APPENDIX B

LITERATURE SURVEY, STRESS CORROSION CRACKING OF
INCOLOY 800 IN CONTAMINATED SUPERHEATED STEAMSummary

Few in depth stress corrosion studies have been conducted on 300 series austenitic stainless steels, and even fewer on Incoloy 800 in contaminated steam. However, it appears that Incoloy 800 is far more resistant to stress corrosion than 300 series austenitic stainless steels in steam contaminated with either caustic or chloride. In many of the tests cited, 300 series austenitic stainless steels stress cracked readily, while Incoloy 800 did not. However, chloride stress corrosion cracking of Incoloy 800 did occur in the rather severe cyclic tests by Hammond et al.⁽⁹⁾ Also, a single case of cracking in caustic superheat was reported by Indig.⁽²⁾

There is a general agreement that the most damaging environmental condition occurs as a result of dry-out of dissolved impurities from the moisture carried over in the steam. This particular combination can result in the formation of liquid films that contain high concentrations of dissolved salts. With enough superheat, Ham et al.⁽⁵⁾ indicated that at 715°C (1320°F) stress corrosion would not occur. However, if the presence of a liquid film on the surface is required, Hearn et al.⁽⁴⁾ point out that NaOH solutions may exist at much higher temperatures than NaCl solutions. They postulated that NaCl solutions would not exist above 40°C (104°F) superheat, while NaOH solutions may exist at 125°C (257°F) superheat. Thus, NaOH appears more dangerous than NaCl as a contaminant in steam. Snowden⁽⁶⁾ showed that caustic cracking of austenitic stainless steel was far more severe than chloride cracking in steam with about 16°C (61°F) superheat. In addition, chloride stress cracking in steam apparently requires the presence of oxygen in substantial concentrations. For example, Ham et al.⁽⁵⁾ found that 0.5 ppm O₂ would not result in chloride cracking but >2 ppm would. Hammond et al.⁽⁹⁾ used 8 or 20 ppm O₂ in their chloride-steam cyclic test to obtain cracking in stainless steels and Incoloy 800. These high oxygen levels are not realistic in superheaters under operating conditions, but may occur as a transient resulting from poor wet lay-up or start-up procedures.

Introduction

The presence of specific salts on the steam side of the reheater and the superheater sections of the Solar Plant steam generator may give rise to stress corrosion cracking. The present survey attempts to determine the susceptibility of Incoloy 800 to stress corrosion in the reheater and superheater of the Solar Plant when the steam is contaminated with OH^- or Cl^- . The contamination will be considered with and without moisture carry-over.

Dissolved salts present in the evaporator of a steam generator can be introduced into the superheater by two mechanisms. First, salts can be carried physically with entrained moisture from the steam drum to the superheater. Second, with increasing pressure and temperature, the steam phase exhibits greater solvent characteristics, and salts which are present in the water phase will be partitioned in an equilibrium between the steam and the water phases. The solubilities of sodium chloride, sodium hydroxide and sodium sulphate in superheated steam at 299°C (570°F), as a function of pressure, are given by Hicks et al.⁽¹⁾ They report that the solubilities of NaCl and NaOH are about 100 ppb in superheated steam at 1000 psi. Extrapolating their data to evaporator temperature and pressure conditions gives a solubility for these two salts of about one ppm. Unless the salts are also introduced by moisture carry-over or some other mechanism, the one ppm level would be the maximum concentration that could occur in the superheater. Of course, if the ionic species are physically carried over in the steam phase, the concentration could be much higher in the higher temperature steam. If carry-over occurs, there may be dryout regions where the local concentration on a metal surface can be extremely high. While there have been no definitive studies on the solubilities of NaCl or NaOH in superheated steam, measurements obtained by Indig⁽²⁾ indicate that the solubility of NaOH at 482°C (900°F) and 1750 psi is about 10 ppm, and as a first approximation the solubility of NaCl can be considered the same. Finally, NaOH can be introduced into the superheater by a breach in the tubing that separates the liquid sodium from the steam followed by a sodium-water reaction. In the case of a leak, the concentration of NaOH in the steam phase will not be >10 ppm, but there can be local regions where the mole fraction of NaOH will be almost one. In the steam phase the NaOH can be present on the metal surface as a slurry with some water dissolved in it. However, reactions with the metal

substrate will occur and calculations of the local concentrations become difficult. In summary, the maximum concentration of NaOH or NaCl appears to be about 10 ppm in the steam phase with the possibility of locally high concentrations due to moisture entrainment and dry-out. In addition, the possibility of high caustic concentrations exists at the site of a leak.

Stress Corrosion Cracking of Superheater Tubes in Power Plants

Austenitic stainless steels have been used for superheater tubes in high temperature, high pressure (>2200 psi) sections of modern fossil fired power plants. Operational failures of types 316, 347, 321 and 304 stainless steel have been reported by Board.⁽³⁾ Many superheater failures have not been fully documented because they have occurred in fossil-fuel fired plants where failure analysis is often not conducted or reported. However, from limited information it appears that the stress corrosion cracking failures in the field have usually occurred in the areas of bends or welds (stressed regions), and have been associated with chloride or caustic contamination. No information appears to be available on the use of Incoloy 800 for similar applications.

Based on the information from the failed superheater tubes, guidelines were set for the United Kingdom nuclear programs concerning the use of austenitic stainless steel superheater tubes. The guidelines were aimed at minimizing impurity transport, specifically chloride and caustic, into the superheater section and reducing residual stresses.

Environmental Conditions for the Stress Corrosion of Superheaters

A study by Hearn et al.⁽⁴⁾ discussed mechanisms by which chlorides and hydroxides could form in evaporators, superheaters and turbines of power plants. For 300 series austenitic stainless steels, the authors presumed that one of these aggressive anions must be present in the steam for stress corrosion to occur. On the other hand, they pointed out that high nickel alloys may stress corrode in pure steam. Although the stress corrosion behavior of Incoloy 800 is unknown, the nickel content is probably below the amount required for stress corrosion in pure steam. The authors further indicated that water droplets may penetrate the superheaters at temperatures above the critical temperature,

>374°C (705°F), but will not wet the tube walls. Water droplets containing NaCl or NaOH behave differently than droplets of pure water. The droplets containing salts will survive longer and penetrate further into the superheater. In addition, the critical temperature of the droplets increases with increasing concentration so that they can wet the tube walls above 374°C (705°F). It is postulated that concentrated NaOH solutions may exist as films on the tube walls at 500°C (932°F) or above. Liquid films of NaCl probably do not exist above 40°C (104°F) superheat, and thus NaCl contamination is not as dangerous as NaOH. A final reason for less concern with NaCl is that oxygen or some other oxidizing agent is required for chloride cracking, but not for caustic cracking. This latter reasoning is not mentioned by Hearn et al.⁽⁴⁾

Laboratory Studies of Stress Corrosion in Superheated Steam

There have been only a few controlled studies of stress corrosion cracking of austenitic stainless steels in contaminated superheated steam. In general, these studies indicate that the worst environmental condition results from moisture carry-over followed by dry-out and salt deposition.

Stress corrosion studies were performed by Ham et al.⁽⁵⁾ in a once through superheated steam system. These experiments for the Advanced Gas Reactor program in the United Kingdom were designed to offer a substitute for 2-1/4Cr-1Mo steel in superheater tubes. Type 316 stainless steel, 9Cr-1Mo steel and Incoloy 800 were tested. The fewest number of tests were performed on Incoloy 800. The tests were conducted in environments faulted with either NaCl plus O₂ or CaCl₂ plus O₂. The steam temperature was raised from 350°C (662°F) at the inlet to 400-480°C (752-896°F) at the outlet. Test sections were lengths of tube stressed at approximately design level. For the present purpose, just the type 316 stainless steel and Incoloy 800 results will be discussed. Essentially no attack occurred in the dry superheat sections of the tubes. The attack was limited to the dry-out region or where the steam had <15°C (59°F) superheat. Stress corrosion and severe pitting occurred on type 316 stainless steel in the dry-out region when CaCl₂ and >2 ppm O₂ were injected into the system. In a similar environment, pitting rather than stress corrosion of Incoloy 800 occurred. When <2 ppm O₂ was present, or where NaCl plus high oxygen (20 ppm) was injected into the steam, stress corrosion of type 316

stainless steel did not occur. The authors note, however, that the NaCl high O₂ test on type 316 stainless steel was of short duration.

Tests in superheated steam were also conducted at a lower temperature for the study of stress corrosion behavior of materials for the Steam Generating Heavy Water Reactor. In these studies, steam was introduced at 280-290°C (536-554°F) and discharged at 5°C (41°F) superheat. The metal temperatures were of the order of 310 to 380°C (590 to 716°F). Two austenitic, iron rich stainless steel alloys (18Cr-10Ni and 20Cr-25Ni-Nb) and Incoloy 800 were tested. The steam environments with dissolved contaminants were passed through the inside of tubes in one series of tests and on the outside of the tubes in another series of tests. The main contaminants were CaCl₂ and NaCl with various concentrations of dissolved oxygen. Generally, the oxygen was maintained at 0.5 or ~20 ppm. No stress corrosion occurred when NaCl was the contaminant added to the steam, regardless of the oxygen concentration. On the other hand, CaCl₂ resulted in failures of the iron rich austenitic alloys when high oxygen was present. Incoloy 800 did not show any evidence of stress corrosion cracking, but did pit in the CaCl₂ plus high O₂ tests. In general, the authors believe that increased nickel content leads to decreased chloride stress corrosion cracking susceptibility.

Tests on austenitic stainless steels, ranging in composition from 18Cr-8Ni-Fe to 25Cr-20Ni-Fe, were performed in condensing steam at 300°C (572°F) and in superheated steam at 330°C (626°F) by Snowden.⁽⁶⁾ The stressed specimens were coated prior to test with either chloride as NaCl or caustic as KOH or NaOH, and then exposed to steam containing compressed air. In the chloride environments, wet (saturated) steam produced the worst cracking as all the austenitic steels failed completely in a few minutes, except for the 25Cr-20Ni-Fe alloy. In superheated steam [~16°C (61°F) superheat], more time was required for cracks to initiate and, although the austenitic steels showed cracks to various depths, most did not fail completely. The dry superheat tests indicate that stress corrosion can occur with no moisture carry-over. The tests on samples contaminated with NaOH or KOH were conducted only in the superheated steam environment. In comparison with chloride contamination, the caustic contamination resulted in earlier failure and more severe cracking. As in the chloride tests, 25Cr-20Ni-Fe did not crack and Snowden concluded

that the higher Ni content resulted in increased resistance to stress corrosion. The increased severity of cracking in the caustic-superheat environment may have been due to the ease of formation of liquid films on the NaOH contaminated specimens.

Several studies exposed stressed alloys to cyclic wet-dry steam environments with contaminants. The cyclic testing probably is the most severe environmental condition because it exposes materials to different concentrations of aggressive solutions and simulates various dry-out stages in the superheater. Pearl et al. (7) studied internally heated and stressed sheaths (tubes) exposed to flowing steam. During testing, the heater sheaths were exposed to several cycles of saturated steam at 286°C (546°F) and 1050 psi, with its associated moisture carry-over, and to superheated steam with a maximum temperature >500°C (932°F). The feedwater for the tests was contaminated with 1.5 ppm chloride as NaCl in one series of tests and 0.5 ppm chloride as FeCl₃ in a second series. In addition to the chloride, 20 ppm O₂ and 2.5 ppm H₂ were added to the steam. Transgranular failure of type 304 stainless steel occurred in less than two weeks in the NaCl-cycle test. Type 347 stainless steel also failed, while Incoloy 800 and Inconel 600 and type 310 stainless steel did not. In the FeCl₃ tests intergranular stress corrosion cracking failures of sensitized types 304 and 316 stainless steel occurred in short times. Again, Incoloy 800, Inconel 600 and type 310 stainless steel showed no indication of cracking. During the general corrosion and stress corrosion test program, various system components failed, including a desuperheater coil fabricated from type 316 stainless steel, a type 304 stainless steel cover plate and superheated steam samples. Some of these components had operated at >500°C (932°F) and were completely sensitized. The cracks in the sensitized regions were intergranular. Other regions cracked transgranularly. In all of the failures, the authors concluded that the presence of moisture at one time or another was a prerequisite for stress corrosion.

This same conclusion concerning chloride stress corrosion cracking of austenitic stainless steels appeared early in studies by Edeleanu and Snowden. (8) Their work indicated that stress corrosion cracking of austenitic stainless steel occurred only at pressures and temperatures consistent with a water film on the surface of the steel. Cracking was rapid in saturated steam and decreased

with increasing superheat. On the other hand, Snowden's⁽⁶⁾ later studies indicated that moisture carry-over may not be necessary for liquid films or stress corrosion.

The most recent cyclic tests in chloride-oxygen-steam environments were reported by Hammond et al.⁽⁹⁾ U-bend and C-ring specimens of many austenitic and ferritic steels were cycled in either saturated steam or water injected with varying amounts of NaCl and O₂ from 280°C (536°F) to the superheat temperature of 430°C (806°F). The pH of the feedwater was controlled to 10.5 by phosphate additions. Types 304 and 316 stainless steel were considered to have a high susceptibility to stress cracking under the cyclic environmental conditions. Incoloy 800 was ranked as slightly to moderately susceptible to cracking.

The caustic stress corrosion cracking of high nickel alloys in deaerated superheated steam was studied by Indig with internally pressurized tube samples.^(2,10) The samples were exposed to steam at 482°C (900°F) and 1750 psi that contained up to 10 ppm dissolved NaOH. Incoloy 800 was found to be extremely resistant to this environment, while Inconel 600 failed quickly by intergranular stress corrosion when stressed near the yield strength. The cracking rate for Inconel 600 was calculated to be about 0.04 cm/hr. A single case of intergranular stress corrosion of Incoloy 800 was reported. The sample was sensitized, highly stressed, and exposed to an environment of steam and impinging molten caustic. In some of the first tests by Indig,⁽²⁾ the pressure vessel used to contain the pressurized tubes and the boiler used to generate the superheated steam were constructed from type 316 stainless steel. These vessels generally failed in hours by transgranular stress corrosion after being contaminated by the caustic-superheated steam.

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