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# EVALUATION OF REFRACTORY/AUSTENITIC BIMETAL COMBINATIONS

by

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and

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First Quarterly Progress Report

Covering the Period

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# FOREWORD

This report was prepared by personnel of the Astronuclear Laboratory of the Westinghouse Electric Corporation under Contract NAS 3-7634. This work is administered under the direction of the Nuclear Power Technology Branch of the National Aeronautics and Space Administration with Mr. P. L. Stone acting as Technical Manager.

This work is being administered at the Astronuclear Laboratory by R. T. Begley with J. L. Godshall and R. W. Buckman, Jr. serving as principal investigators. This report covers the work performed during the period June 22, 1965 to September 22, 1965.

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# EVALUATION OF REFRACTORY/AUSTENITIC BIMETAL COMBINATIONS

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## ABSTRACT

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Sixteen refractory/austenitic bimetal combinations produced by explosive bonding were evaluated for bond integrity by ultrasonic resonance and dye penetrant techniques, metallographic examination, and reverse bend and tensile tests. Nine combinations were selected for complete evaluation of thermal stability. Equipment for diffusion annealing, high temperature thermal cycling and creep rupture determinations was procured and is being assembled.

Author

## SUMMARY

The compatibility of 16 refractory/austenitic bimetal combinations is being evaluated by determining properties before and after long time isothermal and cyclic thermal exposure. The bimetal combinations were formed by explosively bonding refractory metals and alloys to austenitic material. The various thermal treatments include thermal exposures at 1400-1600°F for times up to 2700 hours, low temperature (room to liquid nitrogen),and high temperature (600-1350°F) cycling. The effects of these thermal treatments on the properties of the composites will be determined using room temperature bend and tensile tests and 1400°F creep-rupture tests. Metallographic examination, microhardness traverses, and electron beam microprobe analysis will be used to determine the interdiffusion between the austenitic and refractory metals during the elevated temperature exposures.

The as-bonded sheets were received and non-destructively inspected using ultrasonic resonance and dye penetrant techniques. In general, all of the various combinations exhibited sound metallurgical bonds with the exception of the FS-85/austenitic combinations. Metallographic examination of the refractory/austenitic bimetal interface was initiated to verify the ultrasonic resonance indications.

Room temperature bend tests were conducted on the as-bonded material and on material given the low temperature thermal cycle. The data show no differences in the fracture characteristics as a result of the thermal cycle.

Various items of equipment to be used for creep rupture testing, thermal exposure, and high temperature thermal cycling are in the fabrication and/or assembly stage.

From the original 16 composite sheets, nine were selected for complete evaluation after the initial screening tests.

# I. INTRODUCTION

This is the first quarterly report under Contract NAS 3-7634 "Evaluation of Refractory/ Austenitic Bimetal Combinations". The primary objective of this program is to evaluate the compatibility of various refractory/austenitic bimetal combinations after isothermal and cyclic thermal exposure. Refractory metal alloy sheets of Cb, Ta, Cb-1Zr, FS-85 (Cb-27Ta-10W-1Zr) and T-222 (Ta-10W-2.5Hf-0.01C) bonded to Inconel 600, and AISI 347 and AISI 321 stainless steel by explosive cladding techniques are being evaluated. Selection of the austenitic grades was made on the recommendations of C. A. Barrett and P. L. Stone of NASA-Lewis who have made detailed analyses on the expected compatibility behavior[1]. The bimetal sheet combinations are made of 0.03 inch thick refractory metal bonded to 0.06 inch thick austenitic material, 12 inches wide and 20 inches long. The Explosives Department of E. I. duPont deNemours and Company supplied the explosively-bonded bimetal composites.

A compatibility problem exists between Fe and Ni base materials and Group V-A (Ta, Cb) refractory metal alloys as a result of the strong tendency for brittle intermetallic compound formation between these two classes of materials[2]. However, the good liquid metal corrosion resistance and strength characteristics of the refractory alloys, combined with the good oxidation resistance of the austenitic alloys make this combination particularly attractive for use in ground test installations of space power systems which use liquid metals as working and coolant fluids. The explosive cladding process permits sound metallurgical bonding to occur between the two classes of material without the formation of a continuous brittle interface in the as-bonded condition[3]. Thus the rate of growth of the brittle interface which will occur during service will be a function of the use temperature and time.

The interdiffusion of the refractory/austenitic material will be studied for times up to 2700 hours at temperatures up to 1600°F under ultra high vacuum conditions. Electron beam microprobe analyses will be used to determine compositional changes across the interface. The effects of time and temperature through isothermal and cyclic thermal exposures will be evaluated by determination of the room temperature tensile and bend properties. Elevated temperature strength properties will be determined by means of creep rupture tests. The bond integrity of the as-bonded material will be evaluated by destructive and non-destructive test techniques. Diffusion annealing and creep rupture testing will be done at pressures below $1 \times 10^{-8}$ torr in bakeable, all metal sealed ion pumped vacuum systems.

During this quarter, the explosive bonded bimetal composites were subjected to detailed inspection and specimens required for testing were prepared. The equipment required for the cyclic thermal exposure, long time diffusion studies and creep rupture testing has been procured. The components for the diffusion annealing furnaces were delivered and are being assembled and checked out. The thermal cycling apparatus is being assembled and will be checked out during the next period. The ultra high vacuum creep rupture units are scheduled for delivery during the next report period.

## II. EXPERIMENTAL PROGRAM

The refractory/austenitic bimetal composites are being evaluated according to the program outlined in Figure 1. Nine of the 16 combinations listed in Table I will be selected for complete evaluation. Incoming inspection by dye penetrant, ultrasonic resonance, and reverse bend testing of as-received and liquid nitrogen thermally cycled material, is being used to identify the least promising combinations. All of the bimetal combinations were formed by explosive bonding by the DuPont Explosives Department. Material in each stage of preparation is being subjected to dye penetrant testing to determine if any delamination has occurred.

The program has been divided into two tasks:

### Task I - Diffusion Study

Diffusion annealing treatments for 1600 and 2700 hours at 1400, 1500, and 1600°F will be

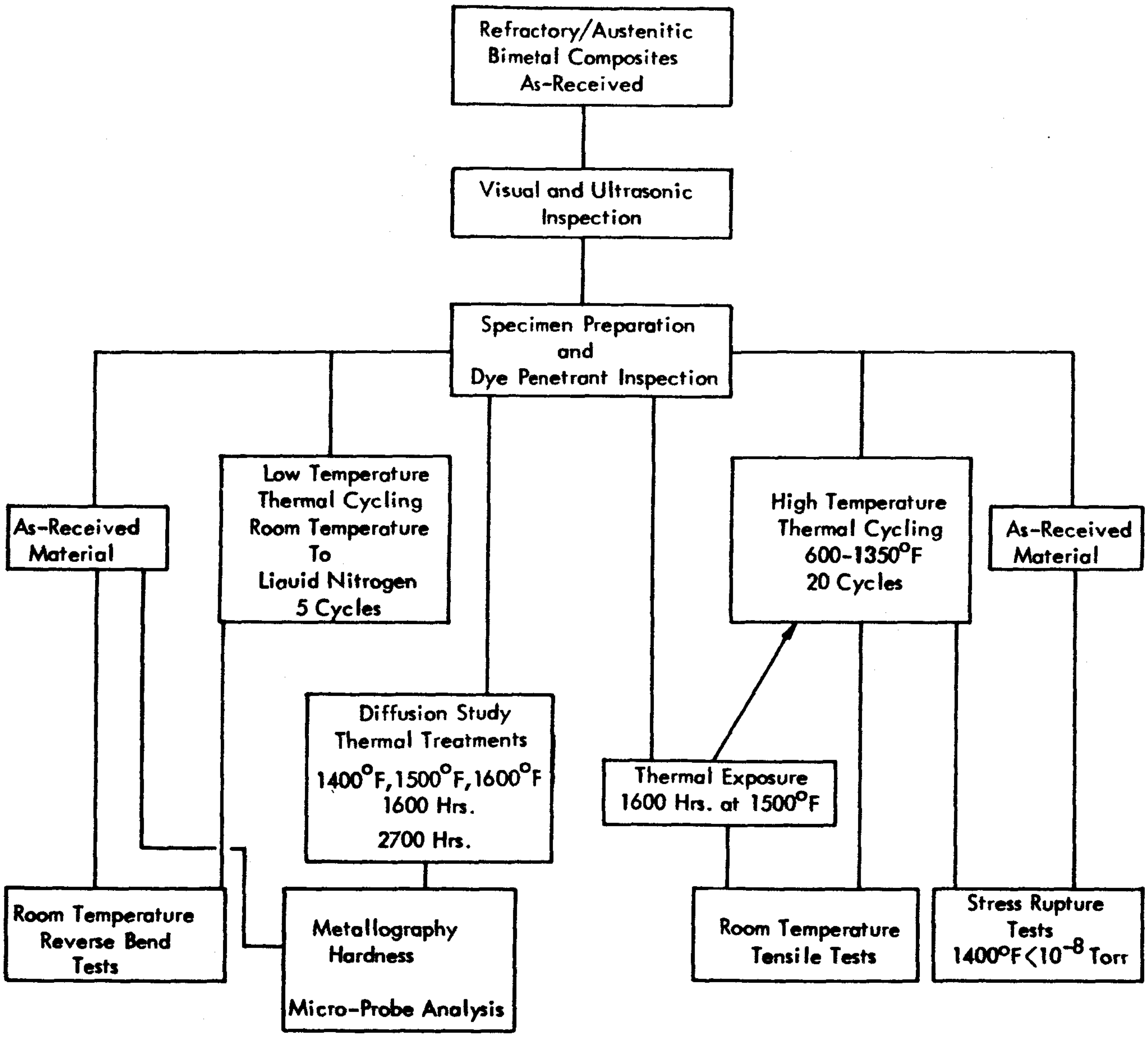


FIGURE 1. Testing and Inspection Schedule for Refractory/Austenitic Bimetal Combinations

TABLE 1. Refractory/Austenitic Explosively Bonded Bimetal Composites

| Composition (Refractory Metal/Austenitic Metal) |
|---|
| Cb/321 |
| Cb/347 |
| Cb/Inconel 600 |
| Cb-1Zr/321 |
| Cb-1Zr/347 |
| Ta/321 |
| Ta/347 |
| Ta/Inconel 600 |
| Ta/Hastelloy N |
| FS-85/321 |
| FS-85/347 |
| FS-85/Inconel 600 |
| FS-85/Hastelloy N |
| T-222/321 |
| T-222/347 |
| T-222/Inconel 600 |

Remarks (Nominal Composition)

AISI 321 (Fe-18Cr-10Ni-0.08C Max - Ti stabilized)

AISI 347 (Fe-18Cr-11Ni-0.08C Max - Cb stabilized)

Inconel 600 (Ni-16Cr-7Fe-2Cb)

Hastelloy N (Ni-17Mo-7Cr-5Fe-0.06C-0.1B-0.5 Al)

FS-85 (Cb-27Ta-10W-1Zr)

T-222 (Ta-10W-2.4Hf-0.01C)

used to study the rate of inter-diffusion between the austenitic and refractory metal alloy. Metallographic examination, micro-hardness transverses, and electron beam microprobe analysis will be the techniques used to study the specimen interface before and after exposure.

Task 2 - Mechanical Property Evaluation

The effects of isothermal and cyclic thermal exposure on the bond integrity will be determined by room temperature tensile tests and elevated temperature creep rupture testing at 1400°F. The isothermal exposure consists of 1600 hours at 1500°F and the cyclic thermal exposure consists of the following:

1. heat to 1350°F and soak for 15 minutes
2. cool to 600°F in 10-30 seconds
3. heat to 1350°F in 5 minutes.

A total of 20 such cycles constitutes a complete test. Temperature is measured at the austenitic-refractory metal interface and helium gas directed at the refractory metal will be used to accelerate cooling.

All elevated temperature creep rupture testing and diffusion annealing runs are being conducted in all metal sealed, sputter ion pumped bakeable systems operating at pressures of $1 \times 10^{-8}$ torr or less.

## III. PROGRAM STATUS

### A. STARTING MATERIAL

The technique, explosive bonding, by which the bimetal combinations were formed is a unique method for effecting a metallurgical bond between dissimilar metals. The physics of the process are involved(3) and hence will not be discussed in detail. However, certain features of the process are summarized in the following discussion taken from the paper of Holtzmann and Cowan(4).

Bonding is accomplished by the collision at high velocity of explosively propelled sheet. Two sheets of material, one of which has a layer of explosive affixed to it, are positioned with an included angle between them and one edge of each in contact at the apex. Detonation of the explosive charge is initiated at this apex, and the explosively propelled sheet collides at high velocity with the stationary sheet. A sufficiently large pressure is generated ahead of the point of collision which causes the free surfaces of the metal just ahead of the collision point to flow into the space between the plates. It is the characteristics of this jet flow which determine the characteristics of the bond. When the jet flow becomes trapped, sufficient portions of the kinetic energy is converted into heat as frictional forces bring part of the jet to rest, and a layer of molten material is formed. The exact mechanisms are, however, uncertain. When the collision region becomes unstable, oscillations occur and the result is the formation of an interface which has a sinusoidal wave form. Most of the energy is expended in the formation of the waves and melting, but a continuous layer of molten material is not formed. This type of interface is considered optimum. However, the wave propagation is very directional and this sinusoidal wave form will be observed only when viewing the interface transverse to the direction of wave propagation. A planar interface would be observed when viewing the interface parallel to the direction of wave propagation.

The bimetal compositions prepared for this investigation were made by bonding 12 inch x 20 inch x .03 inch refractory metal sheet to the same size sheet of austenitic material. The direction of wave propagation was along the 20-inch dimension. Each composite was sheared to provide two sheets 6 inches x 20 inches which were then stretcher straightened.

During the current report period, sheets of each of the refractory/austenitic bimetal composites listed in Table 1 were formed by explosive bonding, stretcher straightened, trimmed and forwarded to the Westinghouse Astronuclear Laboratory for inspection and processing. Chemical analyses of the starting materials furnished by the vendors are shown in Tables 2A and 2B. All of the material was fully recrystallized with the exception of the tantalum which had been stress-relief annealed prior to bonding. During the initial stages of the material

TABLE 2A - Vendor* Analysis of Refractory Starting Materials

| Alloying Additions (w/o) | Cb-1Zr Ingot | Sheet (1) | Cb-1Zr Ingot | Sheet (2) | T-222 Ingot | Sheet | Ta Ingot | Sheet | Cb Ingot | Sheet | FS-85 Ingot | Sheet |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Cb | 98.9 | | 98.9 | | | | | | | | Bal | |
| Zr | 1.04 | | 1.0 | | | | | | | | .93 | .92 |
| Ta | | | | | Bal | | | | | | 27.8 | 27.8 |
| W | | | | | 9.2 | | | | | | 10.6 | 10.7 |
| Hf | | | | | 2.6 | | | | | | | |
| Impurities ppm | | | | | | | | | | | | |
| Al | | | | | | | <10 | | <20 | | <20 | |
| C | <30 | 60 | 35 | 40 | 115 | 170 | 35 | 70 | 35 | <30 | 45 | 80 |
| H | 4.4 | 2.5 | 4.2 | 27 | 3.1 | 2 | 2.7 | 1.4 | 4.2 | 2.7 | 3.7 | 1.7 |
| N | 65 | 65 | 87 | 55 | 20 | 16 | 23 | 70 | 37 | 35 | 35 | 25 |
| O | 145 | 190 | 180 | 240 | <50 | 220 | 75 | 80 | 50 | 140 | 75 | 120 |
| B | <1 | | <1 | | | | <1 | | <1 | | <5 | |
| Cb | | | | | 485 | | <50 | | | | | |
| Cd | <5 | | <5 | | | | <1 | | <5 | | <5 | |
| Co | <20 | | <20 | | | | <5 | | <10 | | <10 | |
| Hf | <80 | | <80 | | | | | | <80 | | 80 | |
| Fc | <100 | | <100 | | 40 | | <15 | | <50 | | <50 | |
| Pb | <20 | | <20 | | | | <5 | | <20 | | <20 | |
| Mn | <20 | | <20 | | | | <10 | | <20 | | <20 | |
| Mo | <20 | | <20 | | 30 | | <10 | | 27 | | < 10 | |
| Ni | <20 | | <20 | | <20 | | <10 | | <20 | | <20 | |
| Si | <100 | | <100 | | | | <10 | | <50 | | <50 | |
| Ta | 520 | | <500 | | | | | | 705 | | | |
| Ti | <150 | | <150 | | | | <10 | | <40 | | <40 | |
| V | <20 | | <20 | | | | <10 | | <20 | | <20 | |
| W | <300 | | <300 | | | | <10 | | 37 | | | |
| Zr | | | | | | | <50 | | <100 | | | |
| Sn | | | | | | | <10 | | <10 | | <10 | |

*All refractory metals purchased from Wah Chang Corp. (1) Heat 912-1211-Cb-1Zr. (2) Heat 912-1189-Cb-1Zr

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TABLE 2B - Vendor* Analysis of Austenitic Starting Materials

| Element (w/o) | Inconel 600 | 321 (1) | 321 (2) | 347 | Hastelloy N** |
|---|---|---|---|---|---|
| C | 0.026 | 0.061 | 0.055 | 0.046 | 0.06 |
| Mn | 0.10 | 1.56 | 1.67 | 1.36 | 0.51 |
| P | 0.014 | 0.023 | 0.025 | 0.020 | 0.001 |
| S | 0.006 | 0.016 | 0.010 | 0.011 | 0.007 |
| Si | 0.39 | 0.56 | 0.72 | 0.58 | 0.39 |
| Cr | 15.74 | 17.10 | 17.94 | 18.47 | 6.44 |
| Ni | 76.00 | 10.57 | 10.44 | 10.64 | Bal |
| Cu | 0.01 | 0.15 | 0.22 | 0.17 | 0.01 |
| Ti | 0.22 | 0.46 | 0.58 | | 0.01 |
| Co | 0.06 | 0.03 | | 0.04 | 0.17 |
| Al | 0.13 | | | | 0.01 |
| Fe | 6.80 | Bal. | Bal. | Bal. | 4.05 |
| Mo | | 0.29 | 0.27 | 0.17 | 16.42 |
| Cb | | | | 0.68 | -- |
| Ta | | | | 0.01 | -- |
| V | | | | | 0.24 |
| B | | | | | 0.004 |

* Austenitic Metals purchased from Industrial Standard Steel Inc.
** Vendor - Stellite Division, Union Carbide
(1) Heat 31094
(2) Heat 60171

procurement effort, duPont indicated that they would not be able to stretcher straighten all of the material unless the delivery schedule could be extended. A check of the adjacent industrial concerns showed a limited capacity to perform this operation. Therefore, the composites shown in Table 3 were straightened by roller leveling. This treatment was not as efficient as stretcher straightening in producing flat plates.

## B. INCOMING INSPECTION

### 1. Visual and Ultrasonic

The results of the visual and ultrasonic resonance inspection are recorded in Table 4. Two types of surface defects were observed visually, a "bubble" or "blister" and a "ripple" with

TABLE 3 - Refractory/Austenitic Bimetal Composites Straightened by Roller Levelling

| COMPOSITION (Refractory Metal/Austenitic Metal) |
|---|
| Ta/Hastelloy N |
| T-222/347 |
| T-222/Inconel 600 |
| T-222/321 |
| Cb-1Zr/321 |
| FS-85/Hastelloy N |

the bubble or blister-type defect predominating. The bubble or blister defects which were observed on the refractory metal side were attributed to air entrapment between the sheets during the bonding process[7]. Severe localized buckling of the refractory metal which resulted in a surface defect with a rippled appearance was also observed but the cause for this type of defect is not understood. An example of this type of defect is shown in Figure 2. Prior to ultrasonic inspection all visual surface defects were marked and ultrasonic measurements were not made at these locations.

A grid of one inch line spacings was drawn on the austenitic side of the composite and ultrasonic determinations were made at the intersection of the grid lines using a "Vidigage" thickness measuring device. The quartz crystal transducer was 3/8" diameter and the induced frequency was 9 mcs. A 0.090" thick piece of austenitic stainless steel was used as the reference standard, thus unbounded areas on the composite would indicate a thickness of 0.060" and areas of intimate metallurgical bond would give the same indication as the reference standard.

TABLE 4. Summary of Visual and Ultrasonic Resonance Inspection Results

| Composition | Ultrasonic Indications | Visual Indications |
|---|---|---|
| Cb/321 | None | None |
| Cb/347 | None | None |
| Cb/Inc 600 | Two defects, ~1" square | None |
| Cb-1Zr/321 | None | None |
| Cb-1Zr/347 | None | Two defects, 1" x 5", 2-1/2" x 1" |
| Ta/321 | None | None |
| Ta/347 | One defect 3" x 10" | 1/2" x 2" within area of ultrasonic indication |
| Ta/Inc 600 | Two defects, 3-1/2" x 2", 1" x 3" | Two defects, 1" x 3-1/2", 2" x 4" |
| Ta/Hastelloy N | One defect 2" x 2" | One defect ~2" x 2" |
| FS-85/321 | Two defects, 1" x 6", 1" x 12" | None |
| FS-85/347 | 1/4 the area of the sheet unbonded | None |
| FS-85/Inc 600 | Two defects, 1" x 6", 2" x 16" | One area 2" x 17" |
| FS-85/Hastelloy N | Two defects, 1" x 9", 1" x 6" | One area 2" x 2" |
| T-222/321 | None | None |
| T-222/347 | None | None |
| T-222/Inc 600 | None | None |

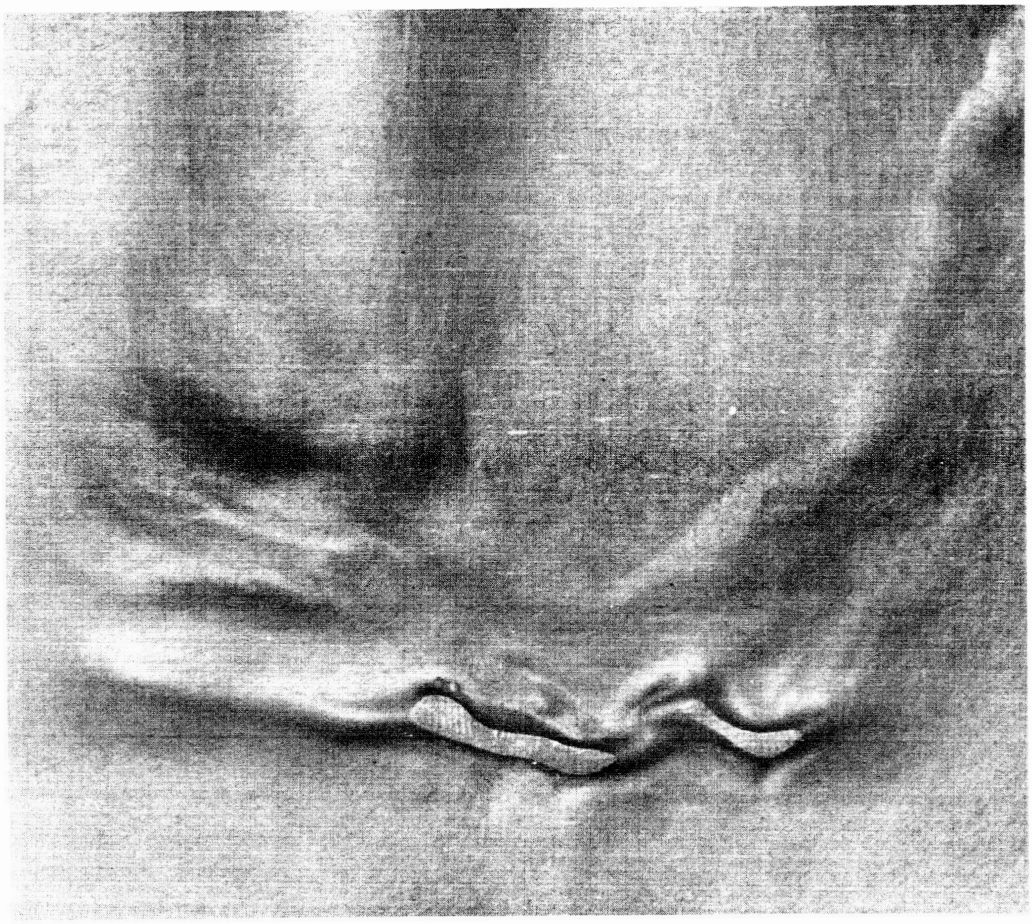


FIGURE 2 - "Rippled" Surface Defect in Ta/Inconel 600. 2X
Defect in Ta Side of Composite

Ultrasonic testing revealed the presence of unbonded areas which were not apparent on visual inspection. Generally, the results of the ultrasonic inspection indicate excellent bonding between the refractory and austenitic material with the exception of the FS-85/ austenitic combinations. A typical defect area determined on the FS-85/347 bimetal sheet by ultrasonic inspection is shown in Figure 3. The preliminary results would indicate that the explosive cladding technique reqults in excellent metallurgical bonding between the austenitic and refractory materials but there are apparently some aspects of the process which bear further investigation to improve reproducibility.

## 2. Reverse Bend Testing

Two specimens 1" x 4" x 0.09" of each of the bimetal combinations were subjected to reverse bend testing. The purpose of this test was to evaluate bond integrity by observing any tendency for delamination at the interface. One specimen of each composite prior to reverse bend testing was quenched to liquid $N_2$ temperature and warmed to room temperature, the cycle being repeated five times. Reverse bend testing was performed by clamping the specimen vertically in a vise. The free half of the specimen was bent, by impacting, $90^{\circ}$ from the original portion, and then returned to original position and then $90^{\circ}$ in the opposite direction. This process was repeated until the composite fractured. Visual inspection of the bend area was made after each $90^{\circ}$ bend. The results are summarized in Tables 5A and 5B.

Only the FS-85 combinations showed any delamination tendency and then separation of each base material extended approximately 3/16" from the point of fracture. Generally the composites fractured in a ductile manner and the fracture or delamination characteristics were not influenced by the prior low temperature thermal cycle. The total number of reversals each composite withstood before fracture appeared to be a function of the properties of the refractory metal portion only. Composites with the high strength FS-85 and T-222 fractured in the least number of cycles and the weak pure Cb withstood the greatest number of reversals before failure.

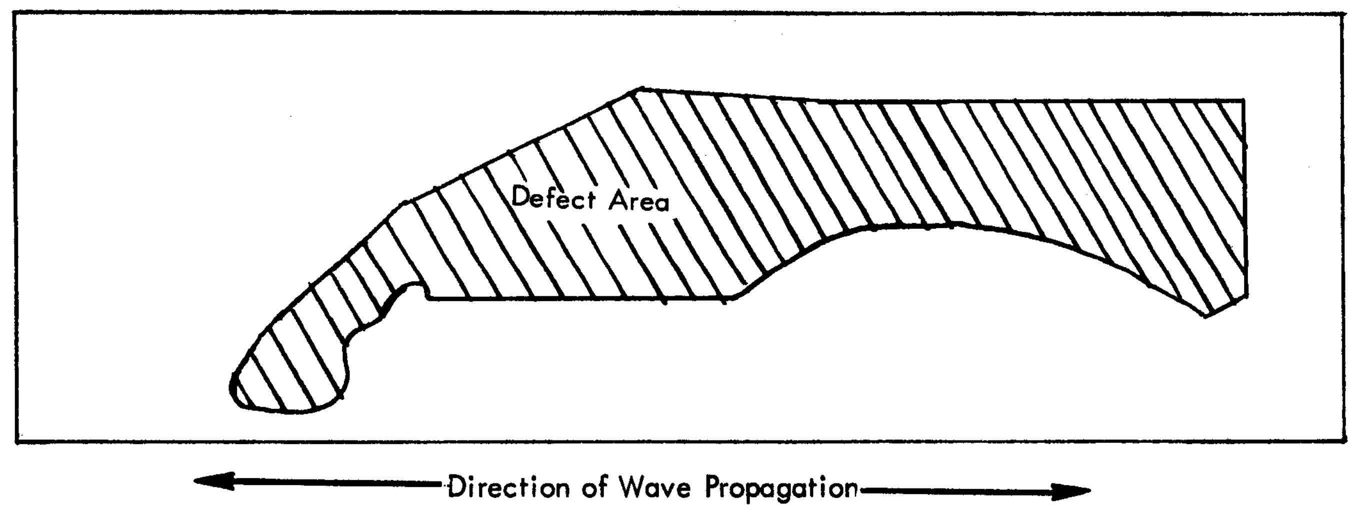



FIGURE 3 - Typical Defect Area in FS-85/347 Revealed by Ultrasonic Inspection - Scale 3/8" = 1"

TABLE 5A – Reverse Bending Test Data for As-Bonded Refractory/Austenitic Bimetal Composites

| Number of 90° Bends* | Cb/321 | FS-85/ 347 | Cb/ In 600 | Cb-1Zr/ 347 | Cb/347 | FS-85/ 321 | Ta/ In 600 | Ta/347 | Ta/321 | FS-85/ In 600 | Cb-1Zr/ 321 | T-222/ In 600 | T-222/ 321 | Ta/ Hast N | T-222/ 347 | FS-85 Hast N |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | N | N | N | N | N | N | N | N | N | N | N | N | N | N | N | N |
| 2 | N | N | N | N | N | N | N | N | N | N | N | S | N | N | N | N |
| 3 | N | N | N | N | N | N | N | N | N | N | S | S | N | N | M | N |
| 4 | N | S | N | N | N | S | N | N | N | S | S | S | L | S | L | S |
| 5 | S | S | N | N | S | S | S | N | N | S | S | L | L | S | F | S |
| 6 | S | S | S | N | S | F | S | N | S | S | M | F | F | S | | L |
| 7 | L | F | S | N | S | | S | S | S | L | L | | | M | | F |
| 8 | L | | S | S | S | | L | S | S | F | F | | | L | | |
| 9 | L | | L | S | S | | L | L | S | | | | | F | | |
| 10 | F | | L | L | S | | F | F | L | | | | | | | |
| 11 | | | F | L | L | | | | F | | | | | | | |
| 12 | | | | F | F | | | | | | | | | | | |

TABLE 5B – Reverse Bending Test Data for Thermally Cycled (Liq Ni to RT) Refractory/Austenitic Bimetal Composites

| Number of 90° Bends | Cb/321 | FS-85/ 347 | Cb/ IN 600 | Cb-1Zr/ 347 | Cb/347 | FS-85/ 321 | Ta/ In 600 | Ta/347 | Ta/321 | FS-85/ In 600 | Cb-1Zr/ 321 | T-222/ In 600 | T-222/ 321 | Ta/ Hast N | T-222/ 347 | FS-85/ Hast N |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | N | N | N | N | N | N | N | N | N | N | N | N | N | N | N | N |
| 2 | N | N | N | N | N | N | N | N | N | N | N | N | S | N | N | N |
| 3 | N | N | N | N | N | N | N | N | N | S | N | N | S | N | L | N |
| 4 | N | S | N | N | N | N | N | N | N | S | S | S | L | S | F | S |
| 5 | S | S | S | N | S | N | N | N | N | L | S | L | F | S | | S |
| 6 | S | F | S | N | S | S | S | S | S | F | S | F | | M | | L |
| 7 | S | | M | S | S | M | S | S | S | | L | | | L | | F |
| 8 | L | | M | S | S | F | L | L | S | | L | | | L | | |
| 9 | L | | L | S | S | | L | L | M | | F | | | F | | |
| 10 | L | | F | L | L | | F | F | M | | | | | | | |
| 11 | F | | | F | F | | | | F | | | | | | | |

N = No Effect; S = Slight crack; M = Medium Crack; L = Large crack; F = Failed.

* 90° refers to the angle from the original position of the sample in the vise.

### 3. Metallography

Metallographic samples were taken from each bimetal composite and mounted to view the thickness of the sheet transverse to the direction of wave propagation. Microstructural examinations revealed a wide variation in the interfacial area for all the as-received material, particularly in regard to the amplitude of the wave. Typical areas of the bonded interface are shown in Figures 4 and 5. While there is a significant variation in the appearance of the bond interface, there is little correlation of the microstructural features and the ultrasonic or bend tests. Some of the composites which exhibited essentially a planar interface were shown to have as high a bond integrity as those composites which exhibited the wave form which could be considered to provide additional bond strength as a result of the mechanical locking effect. Voids and what appeared to be foreign particles were observed in all the interfaces examined.

Additional samples, taken from the center and either end of each composite, are being prepared for examination to try to establish the point of explosive detonation which was not recorded when the composites were formed.

## C. SPECIMEN PREPARATION

After visual and ultrasonic inspection, the specimens required for the diffusion annealing study, thermal exposure and mechanical property tests were sheared from the defect-free portions of the bimetal sheet as shown in Figure 6. All specimens were cut such that the long axis of the mechanical property test specimens was parallel to the direction of wave propagation. No delamination at the interface was observed as a result of the shearing operation. The sheared edges of each specimen was belt-sanded and dye penetrant inspected to verify the absence of any delamination.

# IV. EQUIPMENT DESIGN AND ASSEMBLY

## A. THERMAL CYCLE APPARATUS

During the current report period, the design of the thermal cycling furnace has been completed and most of the components fabricated. A schematic representation of the thermal

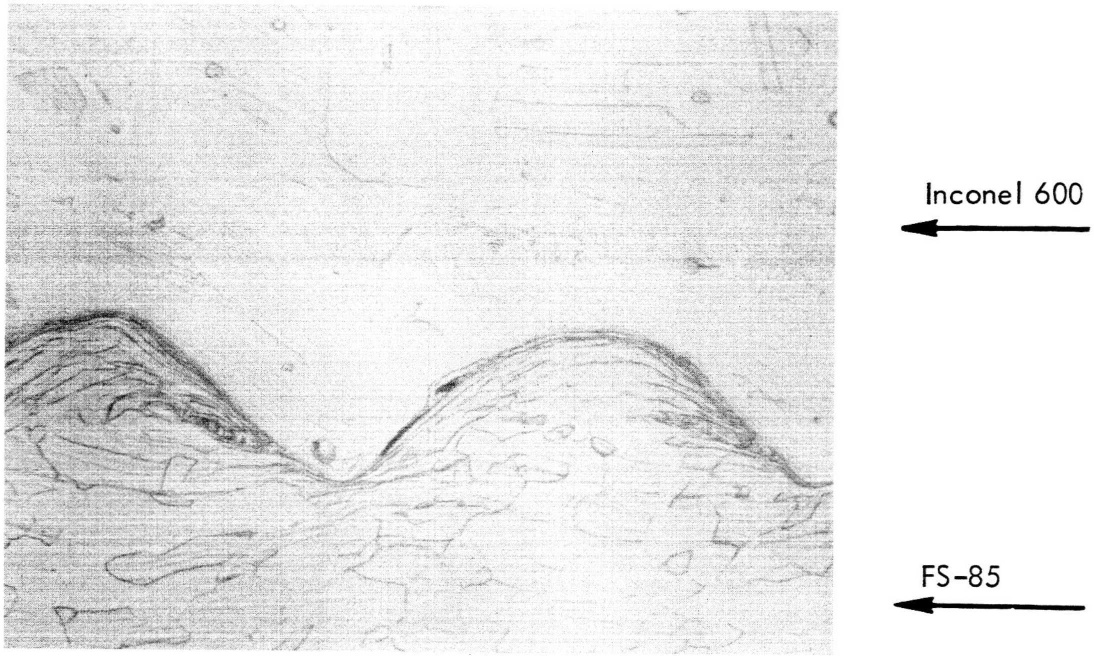



FIGURE 4. Interfacial Area of FS-85/Inconel 600 (400X)

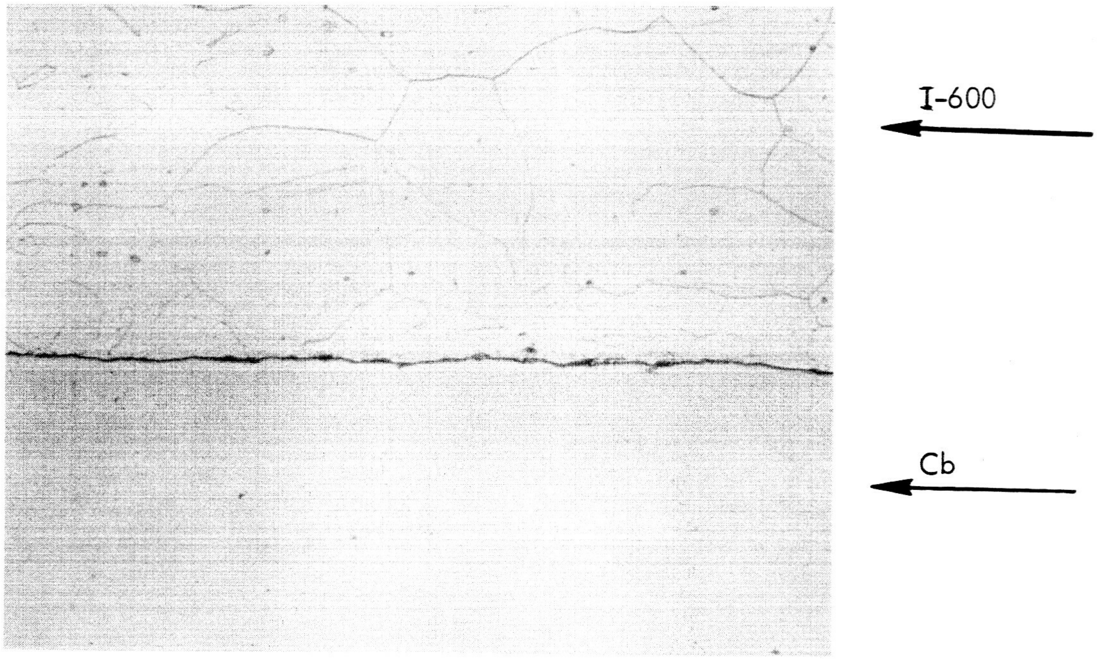
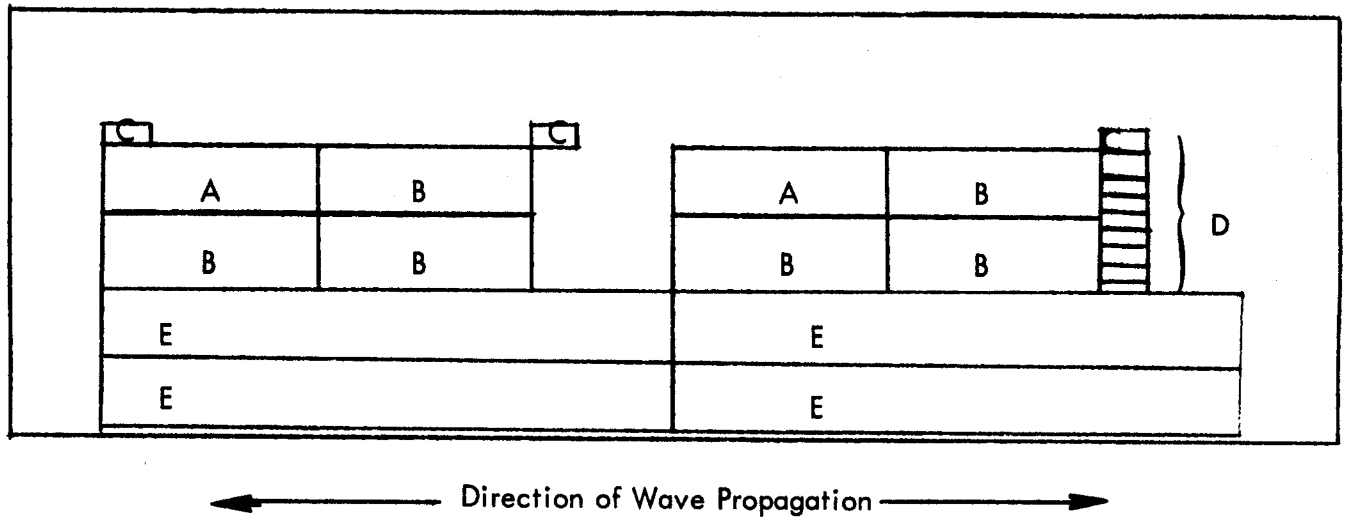



FIGURE 5. Interfacial Area of Cb/Inconel 600 (400X)




A) Bend specimens
B) Tensile and creep rupture specimens
C) Metallographic specimens
D) Diffusion annealing specimen
E) Specimens for thermal exposure and thermal cycling tests

FIGURE 6 - Specimen Orientation of Refractory/Austenitic Bimetal Composite with 100% Integral Bonding
Scale 3/8" = 1"

cycling apparatus is shown in Figure 7. This furnace will incorporate a RF induction coil using 450 kcs at a power level of 10 Kw. The design calls for heating to 1350°F ±25°F within 15 minutes, holding for 15 minutes, cool to 600 ±25°F at the bimetal interface in 1-30 seconds, heat to 1350°F within 5 minutes. This cycle is to be performed a total of 20 times. The cooling gas will be directed against the refractory metal to produce the greatest thermal stress upon the samples which are held securely in a fixture during the treatment to prevent the specimens from bending. The present design calls for running three samples simultaneously in a partial pressure of helium gas which contains less than 10 ppm total active impurities.

## B. DIFFUSION ANNEALING FURNACE

Diffusion annealing studies will be conducted using the ultra high vacuum annealing system shown in Figure 8. This system incorporates an all metal sealed unit which is bakeable, and uses a 15 liter/sec sputter ion pump and a titanium sublimation pump with a rated pumping speed of 45 liters/sec for obtaining very low pressures. Initial evacuation of the system is accomplished with cryogenic sorption pumps. Roughing is done through a 3/8-inch diameter copper tubulation which is subsequently pinched off after the system pressure is less than 5 microns. The diffusion annealing samples will be contained within the 1-1/2-inch diameter vycor glass tube which is attached to the metal flange through a graded glass seal. The glass tube will be inserted into a resistance heated furnace and heated to the desired operating temperature. A Pt-Pt-13 Rh thermocouple attached to the specimen load will be used to monitor the test temperature. Preliminary tests at 1600°F on the empty and unbaked system resulted in an operating pressure of $1.5 \times 10^{-8}$ torr after 24 hours of operation.

The use of a hydrocarbon-free pumping system precludes any carbon contamination of the test specimens. Tests at WANL[4] have shown that the use of liquid nitrogen cooled traps does not prevent oil backstreaming, leading to carbon contamination of test specimens at elevated temperatures in oil diffusion pumped systems.

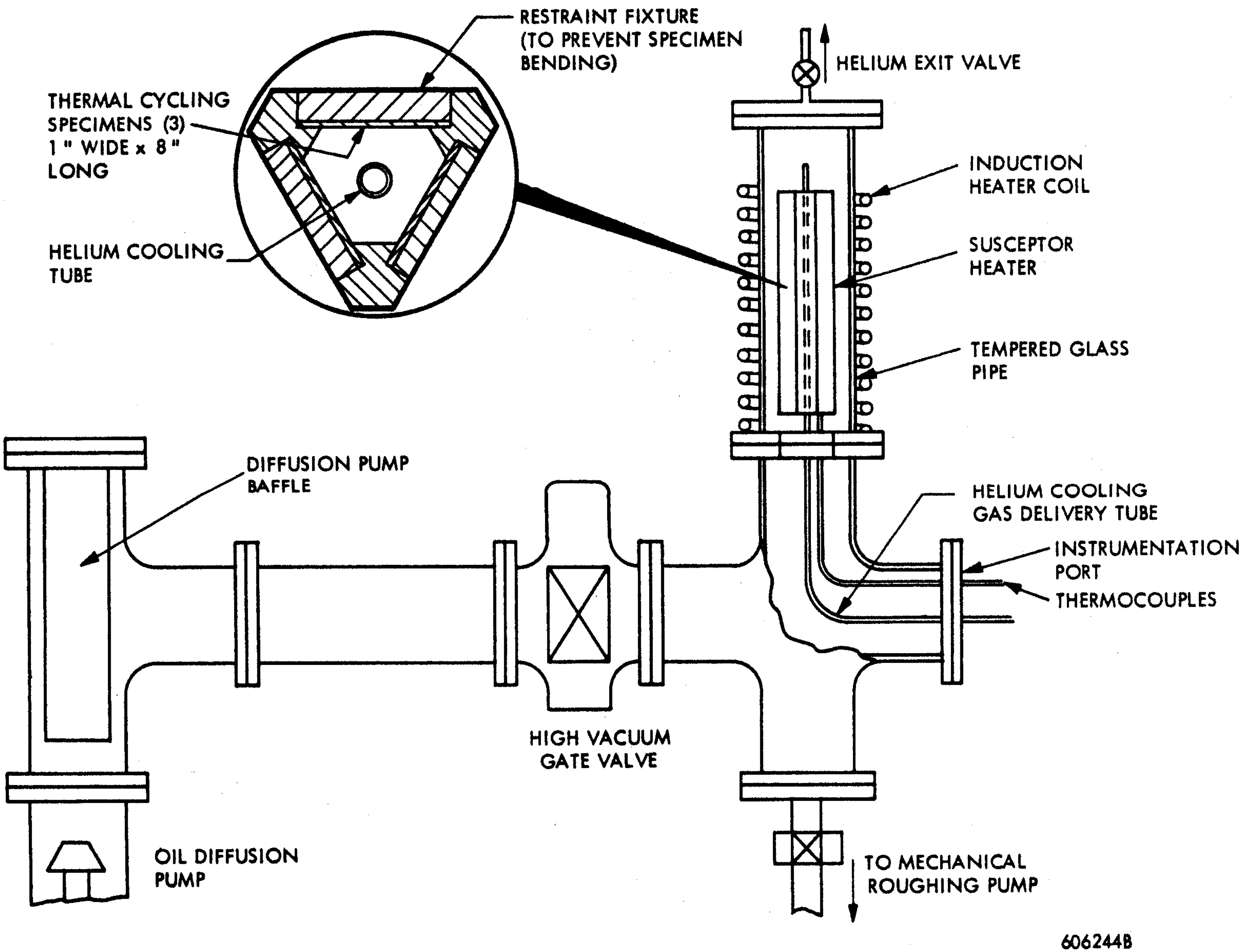



FIGURE 7. Schematic of Thermal Cycling Apparatus

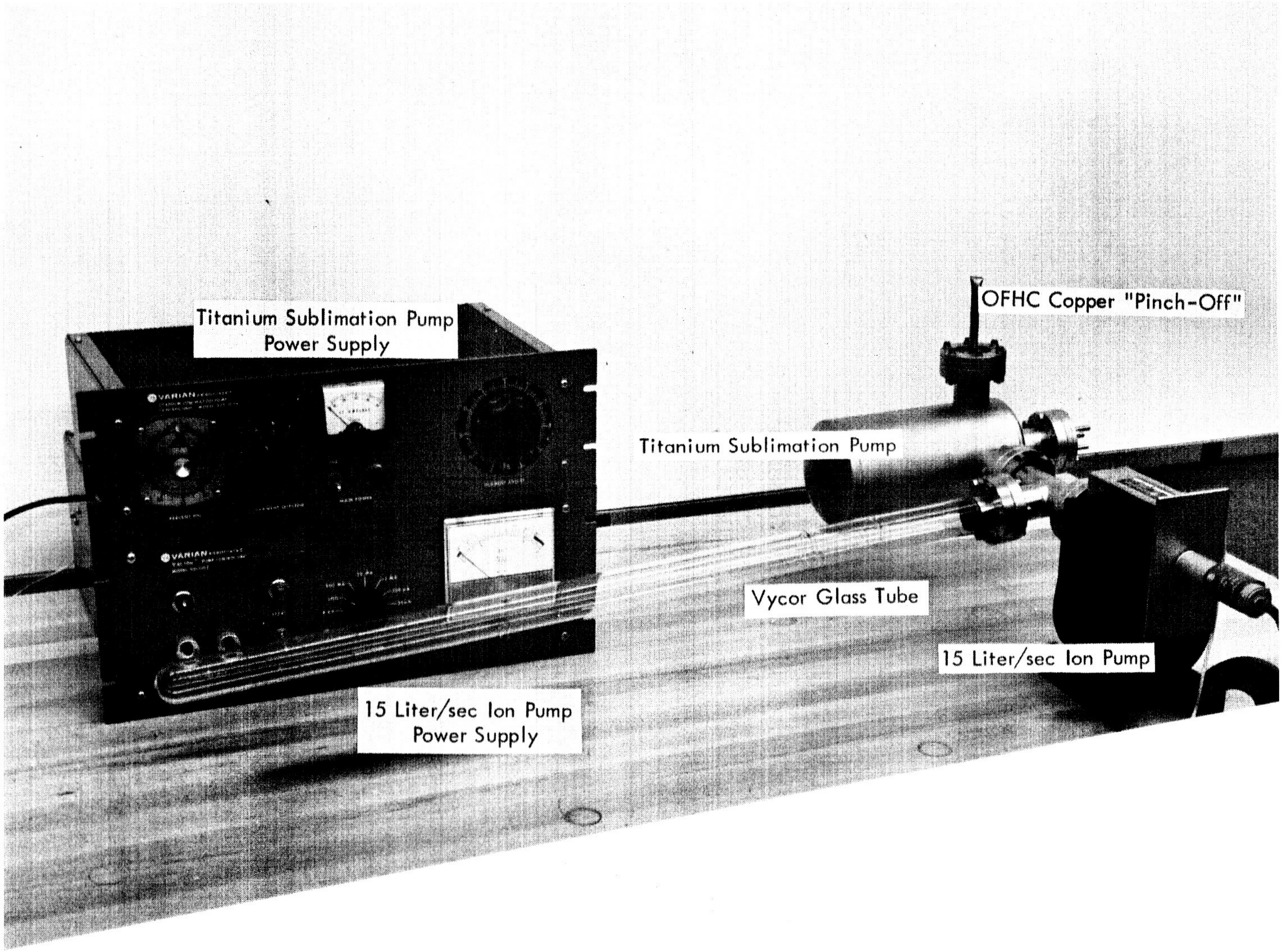



FIGURE 8 - Vacuum System for Diffusion Annealing Study

Temperature uniformity of the hot wall furnaces to be used for the diffusion annealing and thermal exposure testing is within $\pm$ 13°F over a 8-inch heated length and within $\pm$ 2°F over a 3-inch heated length. The total heated length of the furnace for the thermal exposure test is approximately 18 inches and for the diffusion annealing furnace approximately 7 inches. The total length of the diffusion annealing and thermal exposure furnace loads are 1-1/2 and 8 inches, respectively.

## C. CREEP RUPTURE TESTING

Creep rupture testing will be done in sputter ion pumped, internally loaded, ultra high vacuum units that have been described by Buckman and Hetherington[(4)]. The system shown in Figure 9 consists of a 14-inch diameter bell jar, a feedthrough spool and weight chamber which is pumped by a 500 liter/sec internally baked sputter ion pump. Roughing is accomplished by using molecular sieve cryogenic sorption pumps. The test specimen, 0.250 inch wide by 1 inch gage length, is heated by radiation from a resistance heated split tantalum corrugated element.

Power is provided to the hot zone by a 7.5 Kva low voltage, high current power supply that employs a magnetic amplifier driven saturable reactor in series with the primary winding of a step-down transformer. Temperature is controlled by means of a thermal watt converter. This device measures the true ac power delivered to the hot zone and provides a dc millivolt signal to a three mode precision controller which maintains the power input to the heating element at a constant value.

The weight is contained internally within the weight chamber and is applied by retracting a support platform which is activated by means of a screw jack connected to the bellows assembly.

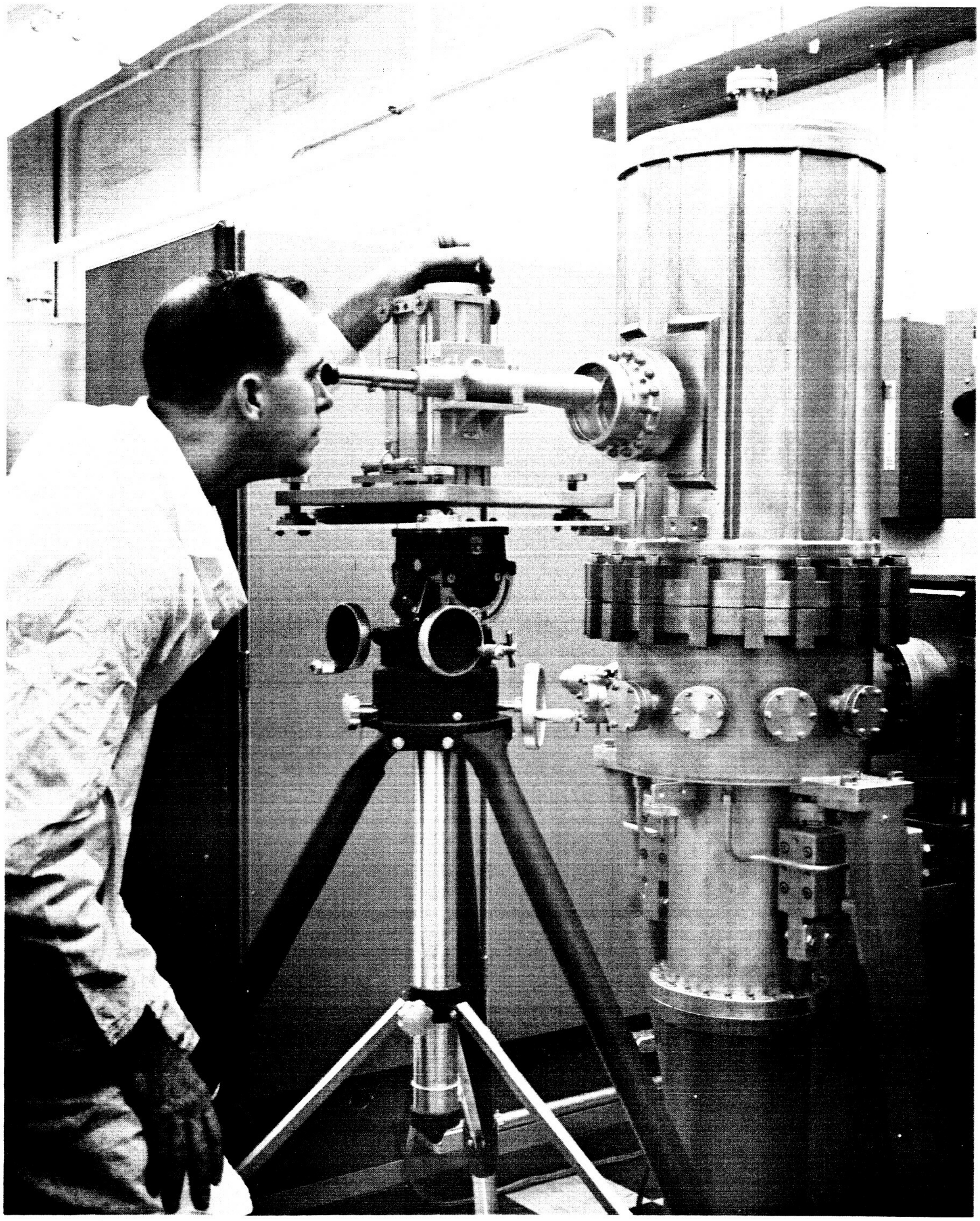


FIGURE 9 -- Creep Rupture Furnace

Creep strain is determined optically by direct measurement of the separation of fiducial scratches applied to the extremes of the uniform gage section of the test specimen. Readings are made with a vertical scale and a 100 part drum equipped with a 10 part vernier. The instrument can be read to 0.00005 inch.

Two vacuum stress rupture furnaces are scheduled for delivery during the next report period.

## V. FUTURE WORK

During the next quarterly period, experimental work will proceed as follows:

1. Room temperature tensile testing of the as-received material will be completed. The test will be continuously monitored through the use of a movie camera to record the failure and tendency to delaminate through the yield point.

2. All of the furnaces will be assembled, calibrated, and the thermal exposure studies initiated.

3. Metallographic examination will be completed and microhardness traverses taken on all of the as-received materials.

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