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DEVELOPMENT OF DIFFUSION-TYPE
BONDING TECHNIQUES FOR BERYLCO-25 ALLOY

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UNIVERSITY OF CALIFORNIA

Lawrence Radiation Laboratory

Livermore, California

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DEVELOPMENT OF DIFFUSION-TYPE BONDING TECHNIQUES

FOR BERYLCO-25 ALLOY

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September 8, 1961

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DEVELOPMENT OF DIFFUSION-TYPE BONDING TECHNIQUES FOR BERYLCO-25 ALLOY*

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September 8, 1961

ABSTRACT

Bond tensile strengths in excess of 90,000 psi in joining precipitation-hardened beryllium-copper have been obtained by diffusion techniques. Ordinary brazing techniques for beryllium-copper have resulted in an average of 40,000 psi tensile strength in the bond.

Bonds with tensile strengths averaging 108,400 psi have been developed using high-vacuum, diffusion-type bonding techniques. The hardness of the base metals in these bonded specimens (after subsequent heat treatments) ranged from 38 to 42 Rockwell "C." The microstructures exhibited complete diffusion of the silver-copper-indium filler alloy into the base metals, leaving a base-metal-to-base-metal interface. Also, dry hydrogen techniques (dewpoint less than -80 °F) were used to develop bonds that have an average of 96,000 psi tensile strength, with a base metal hardness range (after heat treating) of from 36 to 37 Rockwell "C." The microstructures of these bonds showed almost complete diffusion of the silver-copper-indium filler alloy into the base metal. The diffusion-type bonds in beryllium-copper were effected at 1550 and at 1475 °F. Data have been obtained on time-temperature parameters for bonding, solution heat treating, quenching, and precipitation hardening. These data were correlated with grain growth effects, tensile strengths

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of the base metal and the bond, hardness data in various conditions of heat treatment, percentage of elongation, percentage of reduction in area, and modulus of elasticity.

Hardnesses averaging 38 Rockwell "C" can be obtained by air-blast cooling of the beryllium-copper after solution heat treatment (followed by precipitation hardening). The differences in tensile strengths and final hardnesses of the base metal were very slight between beryllium-copper with extremely large grain size and that with smaller grain size after bonding at 1550 and 1475°F (followed by solution heat treatment, air-blast cooling, and precipitation hardening), and only minor variations in elongation resulted.

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I. INTRODUCTION

This program was initiated to find a way to produce stronger bonds in Berylco-25 than could be obtained by ordinary brazing.

To meet bond strength requirements of 90,000 psi or better in tension, it was originally decided that the most promising approach was to select filler metals which melt higher than the melting point of the Berylco-25, and attempt to create a solid-state diffusion reaction which would result in a low-melting eutectic, causing complete fusion at the bond interface. This bond might be further diffused into the Berylco-25 to obtain a hardenable base-metal-to-base-metal joint. Gold, with a melting point of 1945°F, was selected for the initial diffusion bonding studies.

Another proposed method involved selecting filler alloys which melted lower than the melting range of Berylco-25 (which is 1587 to 1750°F), and, through high bonding temperatures and lengthy holding times at temperature, effecting another type of diffusion or semidiffusion bond.

Inasmuch as 1475°F is the recommended upper limit for brazing Berylco-25 by ordinary methods, and rapid grain growth is said to occur at temperatures higher than 1475°F, it was deemed necessary to conduct a limited number of mechanical tests (within the scope of the available funds) on Berylco-25 after it had been subjected to temperatures up to 1550°F to determine the effects of such unorthodox heat treatments on the tensile strength, hardness, elongation and other properties of the material.

In the course of the program it was found that some filler metals showed relatively rapid alloying and diffusion into Berylco-25 at lower temperatures than 1550°F. The program was then expanded to determine whether diffusion-type bonds of the strength required could be produced by using the same filler metals and increasing the holding times at lower temperatures; such as 1475°F.

II. PREVIOUS RESEARCH

This program is a continuance of P. O. 37012-07 (Final Report submitted May 15, 1959), and P. O. 42738-07 (Final Report submitted July 15, 1959). We here briefly review the conclusions drawn from these two reports.

P. O. 37012-07

1. The four filler metals which have shown optimum results in joining Berylco-25 to itself in dry hydrogen atmospheres are pure gold powder, Incusil 10 (63Ag, 27Cu, 10In) foil, silver-copper eutectic (72Ag, 28Cu) foil, and vacuum vapor-plated gold.
2. A minimum of 6 psi pressure is required on the joint during bonding to obtain satisfactory joint strength and bond continuity by diffusion bonding methods.
3. In sealed-retort brazing (necessary to provide low dewpoint reducing and/or vacuum atmospheres), the cooling cycle is not sufficiently rapid to put the Berylco-25 into a satisfactory solution-heat-treated condition preparatory to precipitation hardening. Therefore, a subsequent solution heat treatment is required, followed by a precipitation hardening treatment.
4. Titanium oxide and aluminum oxide stopoff compounds are effective in controlling and containing the flow of molten filler metals on the surface of Berylco-25 during bonding at temperatures as high as 1550°F.
5. Aging of Berylco-25 at ambient temperatures over extended periods in the solution heat-treated condition (prior to the precipitation-hardening heat treatment) does not appreciably harden the alloy. Therefore materials which have been stored for extended periods in the solution heat-treated condition should be formable after the storage period.

P. O. 42738-07

1. Nickel plating of Berylco-25 prior to bonding appears to discourage the formation of diffusion-type bonds using gold as a filler metal. It appears to assist in effecting conventional brazed bonds using the 72% silver-28% copper eutectic alloy as a filler metal, without the use of added pressures, by reducing the tendencies of the filler metal to alloy and diffuse away from the bond into the Berylco-25.

2. A copper-phosphorous filler alloy, "Phoson O," proved unsatisfactory for joining Berylco-25 to itself.

3. Vacuum vapor-plated gold has exhibited considerably more promise in effecting diffusion-type bonds in Berylco-25 than have either electroplated gold or gold powder in an acrylic binder, in preliminary tests. The quality of the bond, however, depends heavily on the quality of the vacuum gold plating, which has proven to be an important variable over which little control could be developed in this program.

4. Preliminary work indicates that the solution-heat-treatment cooling-rate parameters are not critical in producing hardnesses in the range of 36 to 40 Rockwell "C" upon subsequent precipitation hardening treatments. It appears possible that air-blast cooling may prove satisfactory for solution heat treating Berylco-25 after bonding.

5. Pretinning of Berylco-25 with "Easy Flo" silver filler alloys prior to brazing resulted in generally sound bonds without the use of added pressure. Upon subsequent heat treatment, however, the specimens bonded without pressure failed through the joints. Specimens bonded using 2-4 psi pressure did not fail.

III. OBJECTIVES

1. To develop a joining process for Berylco-25 which will give 90,000 psi minimum tensile strength in the joint and 32-40 Rockwell "C" hardness (142,000 to 181,000 psi tensile strength) in the base metal after solution heat treatment and precipitation hardening.

2. To develop a lower-temperature sealing technique for possible use in plug seals.

3. To prepare detailed specifications on the techniques for effecting the above bonds.

4. To develop heat treating parameters for Berylco-25 base metal to be used as guides to allowable compromises required in obtaining and preserving optimum bond strength and bond continuity in joining and heat treating this alloy.

5. To develop the optimum holding time at 1475°F in solution heat treating Berylco-25 to obtain the lowest hardness in the solution-annealed condition. This is to assist in spinning the alloy to difficult shapes.

6. To develop data on heat treatments vs tensile strengths vs grain growth in Berylco-25 base metal.

7. To study the characteristics of the various types of bonds developed in this program, including metallographic and photomicrographic work.

IV. DISCUSSION AND CONCLUSIONS

1. High Strength Joining

Diffusion-type joining techniques capable of showing more than 90,000 psi tensile strength in the bond and 32-40 Rockwell "C" hardness in the base metal after heat treating were developed in this program. These techniques are described below.

a. Vacuum furnace bonding

Filler alloy: Incusil 10 foil, 0.002 in. thick

Diffusion bonding cycle: 30 minutes at 1475°F, reheat-cool

Solution heat treating cycle: 60 minutes at 1475°F, air-blast-cool to R. T.

Precipitation hardening cycle: 2 hours at 600°F, air-cool

Average tensile strength of joint: 108,400 psi

Hardness of Berylco-25: 38-42 R_C (converted from KHN)

Metallurgical studies of the bond structure indicate that this vacuum bond is a completely diffused joint, Berylco-25-to-Berylco-25, with no filler alloy remaining. The grain structure of the Berylco-25 base metal is duplex, consisting of large grains in a finer grain matrix.

b. Dry hydrogen furnace bonding

Filler alloy: Incusil 10 foil, 0.002 in. thick

Diffusion bonding cycle: 15-30 minutes at 1475°F, retort-cool

Solution heat treating cycle: 60 minutes at 1475°F, air-blast-cool to R. T.

Precipitation hardening cycle: 2 hours at 600°F, air-cool

Average tensile strength of joint: 96,000 psi

Hardness of Berylco-25: 36-37 R_C (converted from KHN)

Studies of the metallurgical structures of the bonds indicate that the dry hydrogen bond (b) is a partially diffused joint, with some Incusil 10 still existing at the bond interfaces. The grain structure is duplex, with some large grains in a matrix of finer grains.

2. Low-Temperature Sealing Techniques

a. 50Pb-50Sn (melting range: 362°F to 413°F). This alloy was applied to Berylco-25 by the oxyacetylene torch method, using standard solder flux. Experimental soldering indicated satisfactory wetting and flow on Berylco-25. Soldered specimens showed no visible intergranular penetration of Berylco-25 after holding 3 hours at 600°F. At higher temperatures in the diffusion bonding range, the solder penetrated the Berylco-25 very deeply, intergranularly.

50Pb-50Sn solder appears satisfactory for low-temperature sealing of plugs, etc., in previously bonded and/or heat-treated Berylco-25 parts below the precipitation hardening temperature.

3. Heat Treating Parameters

The parameters within which a minimum hardness of 32 Rockwell "C" (142,000 T. S.) can be obtained in Berylco-25, as determined in this study, are:

- a. Holding time at solution heat
treating temperature (1475°F): 5-60 minutes
- b. Variation in solution heat
treating temperature: 1400-1475°F
- c. Holding time at precipitation
hardening temperature (600°F): 15-120 minutes

- d. Variation in precipitation
hardening temperature: 500-600°F
- e. Variation in rate of quench in
solution heat treating: Oil, air-blast, water, or
brine satisfactory
- f. Softening of Berylco-25 for
spinning by increasing S.H. T. Maximum softening
holding time (no precipitation reached at 150 minutes
hardening done):

4. Determination of Hardness Range Through Bond Zone with Various Filler Alloys

<u>Filler Alloy</u>	<u>Bond Hardness Range</u>
Gold powder	90-104 R _B
Incusil 10	88-104 R _B
BT + 0.1% Li	90-101 R _B
Vacuum-plated gold	30-26 R _C

No extremely hard, brittle layers were found in the bonds effected with any of these filler alloys.

5. Effects of Heat Treatment Variations on Tensile Strengths of Berylco-25 Base Alloy (0.505-in. Tensile Specimens)

The figures shown are for specimens subjected to the simulated bonding cycles shown, then given a solution heat treatment of 60 minutes at 1475°F, air-cooled to ambient temperature, then precipitation-hardened 2 hours at 600°F.

These data indicate that excessive grain growth in Berylco-25 has a relatively negligible effect on tensile strength, yield strength, and final hardness in the range studied. Since in the hardened state the percent elongation and percent area reduction are low, the specimens bonded at 1475°F show only a slight increase over those bonded at 1550°F. This same situation exists for the modulus of elasticity.

Simulated Bonding Treatment	30 min @ 1550 °F	45 min @ 1550 °F	90 min @ 1550 °F	30 min @ 1475 °F	45 min @ 1475 °F	90 min @ 1475 °F
Average Tensile Strength (psi)	160,000	160,000	164,000	167,000	166,000	164,000
Average Yield Strength (psi)	143,000	142,000	—	147,000	146,000	—
Average Hardness (R _C)	35	35	36	37	37	37
Average Percent Elongation in 2 inches	3.0	3.1	2.5	3.9	4.3	4.0
Average Percent Reduction in Area	4.8	5.0	3.5	6.2	6.2	6.1
Average Modulus of Elasticity (×10 ⁶)	17.9	17.9	17.3	19.0	18.0	17.6

a. Effects of bonding temperature on grain growth. Data indicate that grain growth occurs in bonding beryllium-copper at 1475 °F as well as at 1550 °F. At 1475 °F, grain growth is slower than at 1550 °F when holding times up to 30 minutes are used. The rate of grain growth at 1475 °F increases upon increasing the holding times above 30 minutes, until at 120 minutes the grain size obtained by bonding at 1475 °F and 1550 °F appears almost identical.

6. Plug Tensile Tests

Berylco-25 press-fit plugs, plated with vacuum vapor-deposited gold, exhibited 24,400 psi tensile strength when held 45 minutes at 1550 °F in dry hydrogen atmosphere, then solution- and precipitation-heat-treated.

The studies were limited to the filler metal, bonding cycle, and furnace atmosphere described above, and were done early in the program before optimum bonding cycles had been developed. We believe additional work on this phase will improve the results appreciably.

7. Butt-Joint Bonding for Welding Experiments

These five butt-bonded specimens showed excellent joints with Incusil 10 filler alloy, with the Berylco-25 interfaces either copper-plated or unplated. With special preparation techniques, sound-appearing joints were effected without alloy filleting or flow on those surfaces designated as the "inside surfaces."

No data have been received as yet on the results of Heliarc weld tests on these diffusion-bonded specimens.

8. Bonding and Heat Treating Specifications for Berylco-25

Detailed specifications for bonding Berylco-25 in high vacuum and in dry hydrogen atmospheres are attached to this report. These specifications cover the procedures used to obtain the optimum tensile strength and hardness levels developed in this program. In accordance with the heat treating parameter studies developed, variations in heat treating times and temperatures within the limits shown in this report can be used, depending upon the mechanical properties desired for a particular application.

V. RECOMMENDATIONS

This program was designed to develop certain data about diffusion bonding and heat treating of Berylco-25 to meet specific mechanical property requirements desired in a particular phase of LRL work. It was not intended as a complete and all-inclusive investigation of the bonding, heat treating, and mechanical property characteristics of the alloy.

Additional work in widening the scope of all phases of the program would be beneficial, in view of the interesting results obtained thus far.

It would be especially useful to conduct a study of the effects of bonding temperature, holding time, and grain size variations on the resistance of the material to impact loading.

VI. MATERIALS

The materials used in this program, with the exception of 0.505-in. tensile specimens and some vacuum vapor-plated gold specimens, were furnished by Pyromet Company, and consisted of the following:

1/4-in. -thick Berylco-25 plate (solution-annealed condition) — capable of being hardened to 39-40 Rockwell "C" per test reports

Gold powder (high purity, -150 mesh)

Electroplated gold

Vacuum vapor-plated gold

Incusil 10 (63Ag, 27Cu, 10In) — 0.002-in. foil

Silver-copper eutectic (72Ag, 28Cu)

Silver-copper eutectic plus 0.1% lithium — 0.002-in. foil

Easy Flo (50Ag, 15.5Cu, 16.5Zn, 18Cd)*

Easy Flo 35 (35Ag, 26Cu, 21Zn, 18Cd)*

Easy Flo 45 (45Ag, 15Cu, 16Zn, 24Cd)*

Hydrogen-fired steel weights and supports were also furnished by Pyromet Company for this program.

VII. METHOD OF APPROACH

A. Bonding and Heat Treating Techniques

Before the application of various filler metals to specimen interfaces, contamination and oxides were carefully removed from the Berylco-25 by sanding (#1 grit emery) and acetone-cleaning the mating surfaces. The vacuum-plated gold specimens were not sanded, but were acetone-cleaned.

Specimens were diffusion-bonded in a weld-sealed retort furnace with high-purity dry hydrogen atmosphere (dewpoint -80°F or lower), or in a high-vacuum furnace (pressure 10^{-4} mm or lower). Bonding temperatures were controlled by thermocouples placed directly on the specimens and recorded on L & N eight-point recorder charts. All specimens were retort-cooled to ambient temperatures after bonding.

* These alloys were "tinned" on the surfaces of the Berylco-25; thickness varied from 0.002 to 0.010 in.

B. Solution Heat Treating and Precipitation Hardening

After bonding, specimens were examined to determine whether complete melting and filleting of the filler metals had been accomplished. The general appearance of the bonded area and other pertinent data were recorded. Bonded specimens were then solution heat-treated in a thermocouple-controlled box-type electric furnace. An endothermic atmosphere was provided in the furnace to minimize oxidation on the surface areas of the specimens. Upon removal from the furnace, specimens were water-quenched, oil-quenched, salt-bath-quenched, and rapid-air-blast-quenched by electrically controlled circulatory fans.

Precipitation hardening was done in a thermocouple-controlled box-type electric furnace, and specimens were air-cooled to ambient temperatures.

C. Testing

After bonding and heat treating, sections were cut from each bonded specimen, mounted, and prepared for metallographic studies. The balance of the specimens were sent to LRL for physical testing.

The microscopic examinations included studies of bond continuity, diffusion of the bonding alloy into the Berylco-25 base metal, and grain growth of the Berylco-25 as a function of holding time and bonding temperature. These studies also included microhardness traverses across the bond zones and into the base metal.

VIII. EXPERIMENTAL DATA

A. Diffusion-Type Bonding of Berylco-25 at 1550°F in Dry Hydrogen

Table I presents tabulated data on joining work done at the 1550°F bonding temperature in dry hydrogen. The table also includes heat treating and mechanical testing data for these specimens.

These data include work done with the four filler alloys selected for optimum bonding properties from the filler materials tested in previous programs:

- a. Gold powder in acrylic binder
- b. Incusil 10 foil
- c. Silver-copper eutectic (+0.1% lithium) foil
- d. Vacuum vapor-plated gold

Table I. Data for diffusion-type bonding of Berylco-25 in dry hydrogen at 1550°F.

Specimen Number	Filler Metal	Thickness of Filler Metal (in.)	Holding Time at 1550°F (min)	Heat Treatment After Bonding	Pressure on Interface (psi)	Average Hardness of Berylco-25 (R_C)	Tensile Strength of Bond (psi)	Tensile Strength of Base Metal (psi)
				60 min @1475°F, air-blast-cooled; 2 hr @600°F, air-cooled				
I-221	Gold powder (-150 mesh)	0.002-0.003	30	"	6	37	34,800	165,800
I-224	"	"	45	"	"	38	34,700	165,500
I-227	"	"	60	"	"	36	39,700	147,900
I-230	"	"	90	"	"	39	46,700	169,600
I-233	"	"	120	"	"	36	37,800	175,900
I-222	Incusil 10	0.002	30	"	"	37	46,700	142,700
I-225	"	"	45	"	"	36	Failed during cutting	150,000
I-228	"	"	60	"	"	38	"	155,400
I-231	"	"	90	"	"	38	"	161,500
I-234	"	"	120	"	"	37	40,000	180,800
I-223	BT + 0.1% Li	0.002	30	"	"	39	Failed during cutting	178,300
I-226	"	"	45	"	"	37	10,500	159,600
I-229	"	"	60	"	"	35	Failed during cutting	163,300
I-232	"	"	90	"	"	37	28,700	148,500
I-235	"	"	120	"	"	36	Failed during cutting	154,600
I-239	Vacuum-plated gold	0.002	45	"	"	42	10,000	—
I-240	"	"	90	"	"	42	18,500	—

1. Metallographic Studies of Bond Interfaces – Diffusion-Type Bonds at 1550°F

a. Gold powder bond. Figures 1 through 5 illustrate the microstructures of Berylco-25 bonded with high purity gold powder in an acrylic binder at 1550°F. A constant pressure of 6 psi was maintained on the interface of each specimen during the bonding cycle. Holding times at the bonding temperature varied from 30 minutes up through 120 minutes.

These photomicrographs show extensive alloying and diffusion of the gold powder into the Berylco-25 base metal on either side of the interfaces. Since pure gold melts at 1945°F, and these bonds were made at 1550°F, the mechanism of this type of diffusion bonding (which is not a true diffusion bond in the full meaning of the word) must be, first, a solid-state diffusion reaction between the gold and the beryllium-copper, resulting in an interfacial alloy which melts and flows at some temperature below 1550°F; second, a liquid or semiliquid flow of this alloy to fill the joint and effect sound and complete bonding.

Figure 1

Filler Metal: Gold Powder in Acrylic Binder
Bond Cycle: 30 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 2

Filler Metal: Gold Powder in Acrylic Binder

Bond Cycle: 45 min @1550°F

100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 3

Filler Metal: Gold Powder in Acrylic Binder

Bond Cycle: 60 min @1550°F

100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 4

Filler Metal: Gold Powder in Acrylic Binder
Bond Cycle: 90 min @ 1550°F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 5

Filler Metal: Gold Powder in Acrylic Binder
Bond Cycle: 120 min @ 1550°F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

To obtain as rapid a rate of solid-state diffusion as possible, the bonding temperature should be as high as possible without entering the melting range of the Berylco-25. This is the reason 1550°F was selected as the initial temperature to be used in this program for diffusion bonding.

It will be observed that as the holding time at 1550°F is increased, the distance of diffusion into the base metals increases, until at 60 minutes holding time and above, diffusion passes beyond the bounds of the photomicrographs.

In Fig. 2, some stringer-type voids can be seen along the original interface between the base metals. These are attributed to lack of sufficiently intimate contact between the two Berylco-25 plates. Intimate contact, heat, and pressure are important requirements for effecting diffusion-type bonds.

The light-colored central matrix of the bond consists of an incompletely diffused alloy of high gold content which is attempting to follow the grain boundaries up into the Berylco-25.

b. Incusil 10 Foil Bond. Figures 6 through 10 illustrate the microstructures of Berylco-25 bonded with Incusil 10 foil at 1550°F. A constant pressure of 6 psi was maintained on the interface of each specimen during the bonding cycle. Holding times at the bonding temperature varied from 30 to 120 minutes.

Using Incusil 10 filler alloy, which melts at 1265°F and flows at 1319°F, a different bonding sequence occurs than with the gold, since the filler alloy first melts and then diffusion occurs as the temperature increases to 1550°F and the holding times increase.

In this case, the diffusion zone does not etch out as plainly as it does in the gold bond; however, it is interesting to note that good interfacial contact has been obtained in all of these specimens. A considerable amount of filler alloy, or its constituents, has diffused into the Berylco-25, yet all discontinuities at the interfaces remain filled with alloy. Careful scrutiny of Figs. 6 through 10 reveals a slight shading on either side of the bond, where changes in composition of the Berylco-25 have been brought out faintly by the etchant. It will be noticed that as the holding time at 1550°F is increased, this shaded area extends farther away from the interface on either side of the joint, indicating deeper diffusion of filler metal constituents into

Figure 6

Filler Metal: Incusil 10
Bond Cycle: 30 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 7

Filler Metal: Incusil 10
Bond Cycle: 45 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 8

Filler Metal: Incusil 10
Bond Cycle: 60 min @ 1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 9

Filler Metal: Incusil 10
Bond Cycle: 90 min @ 1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 10

Filler Metal: Incusil 10
Bond Cycle: 120 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

the Berylco-25. All of these specimens appear soundly and continuously bonded; the mechanical properties developed on tensile testing will determine which of these holding times is best for the purpose of the program.

c. Silver-copper (BT) plus 0.1% lithium bond. Figures 11 through 15 illustrate the microstructures of Berylco-25 bonded with silver-copper-lithium foil at 1550°F. A constant pressure of 6 psi was maintained on the interface of each specimen during the bonding cycle. Holding times at bonding temperature ranged from 30 to 120 minutes.

This alloy, like Incusil 10, melts below the 1550°F brazing temperature. It is a eutectic alloy with a melting point of 1435°F and an ordinary brazing temperature of 1475-1500°F. Both its constituents appear to alloy well with the base metal. One would therefore expect that as the bonding temperature is raised from 1435 to 1550°F, and as the holding time is increased, diffusion of filler alloy constituents into the Berylco-25 will increase. Such is in fact the case (Figs. 11-15).

Figure 11

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 30 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 12

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 45 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 13

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 60 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 14

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 90 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 15

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 120 min @ 1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Even at 30 minutes holding time (Fig. 11), complete diffusion appears to have occurred into the Berylco-25. As the holding times are increased up to 120 minutes, the distance of diffusion into the base metals will be even greater. All of these photomicrographs indicate that essentially complete diffusion of the filler metal into the base metal has occurred, leaving a base-metal-to-base-metal bonded interface in each instance.

d. Vacuum-plated gold bond. The vacuum-plated gold bond work at 1550°F was done with 45-minute and with 90-minute holding times, using an applied pressure of 6 psi. The bond effected at 45 minutes failed during sectioning of the specimen; therefore only the 90-minute bond is shown in a photomicrograph (Fig. 16).

It was found in this work that while electroplated gold showed poor diffusion bonding characteristics, the vacuum vapor-plated gold showed excellent results in some instances, and poor results in other instances.

Figure 16

Filler Metal: Vacuum Vapor-plated Gold
Bond Cycle: 90 min @1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Indications are that the quality of the vacuum vapor-plated gold bond is primarily dependent upon the quality of the plating. In some instances during this program, good, adherent plating was obtained; in other instances, poor plating. Therefore results are not too dependable. Because the plating was done by an outside source, close control of plating techniques was difficult.

2. Metallographic Structures of Berylco-25 Base Metal After Diffusion

Bonding at 1550°F

Figure 17 illustrates the microstructure of Berylco-25 in the solution-annealed condition as received from the supplier. Notice the fine-grained structure complete with annealing twins. Duplex grain size is prominent in the material, however, even in this condition.

After a 30-minute holding time at 1550°F (Fig. 18), some small grain size is still evident, but noticeable grain growth has occurred generally. Annealing twins are still definitely delineated.

Figure 17

Berylco-25: As Received
Bond Cycle: None
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 18

Bond Cycle: 30 min @ 1550°F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 19

Bond Cycle: 45 min @ 1550°F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 20

Bond Cycle: 60 min @ 1550°F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 21

Bond Cycle: 90 min @ 1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 22

Bond Cycle: 120 min @ 1550°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Fig. 23. Bar graph, holding time vs grain growth at 1550°F.

After a 45-minute holding time at 1550°F (Fig. 19), the twinning is not as definitely delineated as at lower holding times; however, the duplex grain structure has become considerably more evident, with very large grains and areas of relatively small grains interspersed in the structure.

After a 60-minute holding time at 1550°F (Fig. 20) the grain structure is not too different from that at 45 minutes. Some twinning is still visible on etching.

After a 90-minute holding time at 1550°F (Fig. 21) a dark etching material, believed to be a high-beryllium compound with copper, has precipitated at some of the grain boundaries. This precipitate could possibly indicate a depletion of beryllium content in the grains themselves, which might be expected to have an effect on the response of the Berylco-25 to hardening upon heat treatment.

After 120-minute holding time at 1550°F (Fig. 22), the beryllium compound precipitated in the grain boundaries is still visible. The grain structure is still duplex in character and even the smaller grains have shown appreciable growth.

It is apparent that holding times of 30 minutes and longer at 1550°F have an appreciable effect on grain growth in Berylco-25. How this and other structural changes affect the properties of the material for the intended service is indicated in mechanical tests covered in Section VIII-F of this report.

Figure 23 compares the grain sizes in the duplex grain structure of Berylco-25 that result upon varying the holding time at 1550°F.

B. Bonding of Berylco-25 at 1475°F in Dry Hydrogen

This group of experiments was conducted to compare the bond strengths and base-metal characteristics of Berylco-25 as joined to itself by the higher temperature diffusion-type technique, and as bonded by the technique of using extended holding times in the recommended brazing range. The aim of the experiments was to develop a stronger joint by the diffusion technique than could be obtained by standard brazing procedures, but without any marked drop in the properties of Berylco-25.

It should be mentioned here again that because of grain growth tendencies, and concern over possible incipient melting, the producers of Berylco-25 do not recommend brazing this material at temperatures above 1475°F.

Table II. Data for diffusion-type bonding of Berylco-25 in dry hydrogen at 1475°F (1-1/4 × 1/2 × 1-1/4-in. specimens).

Specimen Number	Filler Metal	Thickness of Filler Metal (in.)	Holding Time at 1475°F (min)	Heat Treatment After Bonding	Pressure on Interface (psi)	Hardness of Berylco-25 (R _C)	Tensile Strength Joint (psi)
				60 min @1475°F air-blast-cooled; 2 hr @600°F, air-cooled			
I-258	Gold powder (-150 mesh)	0.002-0.003	5		6	36.5	57,000
I-259	"	"	5	"	"	36	69,500
I-264	"	"	15	"	"	36	86,500
I-265	"	"	15	"	"	37	71,000
I-270	"	"	30	"	"	36	78,000
I-271	"	"	30	"	"	36	68,500
I-254	Incusil 10	0.002	5	"	"	36.5	87,000
I-255	"	"	5	"	"	36.5	84,000
I-260	"	"	15	"	"	36	94,500
I-261	"	"	15	"	"	36.5	99,000
I-266	"	"	30	"	"	36.5	99,000
I-267	"	"	30	"	"	37	95,000
I-256	BT	0.002	5	"	"	36	55,000
I-257	"	"	5	"	"	36	Failed during cutting
I-262	"	"	15	"	"	36	"
I-263	"	"	15	"	"	36	59,000
I-268	"	"	30	"	"	36	85,000
I-269	"	"	30	"	"	36	80,000
I-236	Vacuum-plated gold	0.002	30	"	"	41	30,000
I-237	"	"	60	"	"	38	Failed during testing
I-238	"	"	120	"	"	38	6,230

Table II presents tabulated data on joining work done in dry hydrogen at 1475 °F, and also includes heat treating and testing data on bonds effected at this temperature.

The data comprise work done using the following filler alloys:

- a. Gold powder in acrylic binder
- b. Incusil 10 foil
- c. Silver-copper eutectic (+0.1% lithium) foil
- d. Vacuum vapor-plated gold

1. Metallographic Studies of Bond Interfaces — Diffusion-Type Bonds at 1475 °F

The holding times at bonding temperatures in these experimental bonding heats varied from 5 minutes to 30 minutes. A pressure of 6 psi was exerted on the interfaces during bonding.

a. Gold powder bond. Because gold powder melts at 1945 °F and these Berylco-25 bonds were effected at 1475 °F in this phase of the program, solid-state diffusion of the gold with the Berylco-25 first had to occur to initiate the bonding. Figures 24 through 26 illustrate the microstructure of the bond layer resulting from the use of gold powder plus an acrylic binder, with holding times of 5, 15, and 30 minutes, respectively.

These photomicrographs indicate that solid state diffusion of gold into Berylco-25 is surprisingly rapid even at 1475 °F.

Figure 24, where a holding time of only 5 minutes at 1475 °F was used, shows a remarkable degree of diffusion and complete bonding. The diffusion zone in this specimen, however, is noticeably reduced in extent when compared with Fig. 1, where a 30-minute holding time at 1550 °F was used.

This comparative reduction is also true of Fig. 25, where a 15-minute holding time was used. With a 30-minute holding time at 1475 °F (Fig. 26), diffusion appears to have progressed considerably further, and the bond structure more closely resembles that of Fig. 1. All of these bonds appear to be continuous and relatively void-free.

b. Incusil 10 foil bond. Figures 27 through 29 illustrate the bonds effected with Incusil 10 foil as a filler metal and with 5 to 30 minutes holding time at 1475 °F.

In comparing these bonds with those effected at 1550 °F (Figs. 6 through 10), the restricted amount of filler metal diffusion into the base metal is again apparent.

Figure 24

Filler Metal: Gold Powder + Acrylic
Bond Cycle: 5 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 25

Filler Metal: Gold Powder + Acrylic
Bond Cycle: 15 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 26

Filler Metal: Gold Powder + Acrylic
Bond Cycle: 30 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 27

Filler Metal: Incusil 10 Foil
Bond Cycle: 5 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 28

Filler Metal: Incusil 10 Foil
Bond Cycle: 15 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 29

Filler Metal: Incusil 10 Foil
Bond Cycle: 30 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

c. Silver-copper eutectic (BT) plus 0.1% lithium bond. Figures 30 through 32 illustrate the bonds effected with silver-copper eutectic foil as a filler metal, using 5 to 30 minutes holding time at 1475 °F.

All three of these bonds show almost complete diffusion of the filler alloy into the Berylco-25. In the first two photomicrographs the diffusion zone is restricted to a localized dark-etching band on either side of the bond interface. In Fig. 32, however, as far as the etchant can be used to indicate the limits of discernible diffusion, the filler alloy appears to have completely diffused away from the bond interface, leaving a base-metal-to-base-metal joint.

d. Vacuum-plated gold. Because gold melts at 1945 °F, and because the brazing temperature used for these specimens was 1475 °F, the 5-, 15-, and 30-minute holding times were extended to 30, 60, and 120 minutes, respectively.

Figure 30

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 5 min @ 1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 31

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 15 min @ 1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 32

Filler Metal: BT + 0.1% Lithium
Bond Cycle: 30 min @ 1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 33

Filler Metal: Vacuum-Plated Gold
Bond Cycle: 30 min @ 1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 34

Filler Metal: Vacuum-Plated Gold
Bond Cycle: 60 min @ 1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 35

Filler Metal: Vacuum-Plated Gold
Bond Cycle: 120 min @1475°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 36

Bond Cycle: 30 min @1475°F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

All of these bonds (Figs. 33, 34, and 35) are characterized by a narrow zone of diffusion, although the gold appears to have effected sound and complete bonds.

2. Metallographic Studies of Berylco-25 Base Metal After Bonding at 1475 °F

As a basis of comparison between the microstructure of Berylco-25 base metal in the "as-received" solution-annealed condition, and the microstructures of Berylco-25 after various holding times at the bonding temperature of 1550 °F and again at 1475 °F, the following photomicrographs (Figs. 36, 37, and 38) should be compared with Figs. 18, 20, and 22, respectively.

Notice that after an exposure of 30 minutes at 1475 °F (Fig. 36), large grains are already beginning to form in the structure, although it remains predominantly fine grained. The twinning shown so prominently in Fig. 17 (as-received specimen) is not at all prominent in Fig. 36.

After a heat treatment of 60 minutes at 1475 °F (Fig. 37), approximately one-third of the area of the photomicrograph shows grains that have grown extremely large. The balance of the structure also shows some grain growth when compared with Fig. 17.

After a heat treatment of 120 minutes at 1475 °F (Fig. 38) the grain size appears approximately the same as shown in Fig. 22 (120 minutes at 1550 °F).

It is apparent that grain growth occurs at 1475 °F as well as at 1550 °F, except that at 1475 °F the growth is at a slower rate for holding times below 30 minutes. This rate increases for holding times longer than 30 minutes until, at 120 minutes, the grain sizes obtained by heating to 1475 °F and to 1550 °F are approximately identical.

Figure 39 shows a graphical comparison between fine and coarse grain sizes as holding times at a bonding temperature of 1475 °F are increased from 30 to 60 minutes. It is apparent that duplex grain structures with some very large grains are prevalent at the 1475 °F brazing temperature, as well as at the 1550 °F temperature.

These data enabled a determination of the proper cycle to select for highest tensile strength of the bonded Berylco-25. Other mechanical properties (such as impact strength), though of great interest, were not investigated due to limited funding of the program.

Figure 37

Bond Cycle: 60 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 38

Bond Cycle: 120 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Fig. 39. Bar graph, holding time vs grain growth at 1475°F in hydrogen

C. Bonding of Berylco-25 at 1475 °F in High Vacuum

This phase involved the experimental brazing of Berylco-25 in high vacuums varying between 10^{-6} and 10^{-4} mm Hg.

The most promising filler metal (Incusil 10 foil) selected from the results of Phases A and B was used in this work.

The main advantage of vacuum furnace bonding over dry hydrogen bonding is that impurities in the filler metals, and on the surfaces of the base metals, that volatilize within the bonding temperature-vacuum range used can be removed. In addition, occluded gases in the filler metal and on the surface of the metals are released at various temperatures during heating and removed from the bond interface. It has been found in the course of previous vacuum joining research that deleterious entrapped gases, not readily driven out by a reducing or inert gas atmosphere under positive pressure, are readily removed from a tight interface in high vacuum.

Table III presents tabulated data on joining of Berylco-25 under high-vacuum conditions at 1475 °F.

It will be observed that the average tensile strength of the bond obtained with a 15-minute holding time at bonding temperature is quite high. A holding time of 30 minutes has produced the highest tensile strength of bond yet obtained consistently in this program.

1. Metallographic Studies of Bond Interfaces — Vacuum-Bonded Joints at 1475 °F

Figure 41, where a 30-minute holding time was used at brazing temperature, shows an average tensile strength of 108,400 psi in the bond. The structure exhibits essentially complete diffusion of the filler metal into the Berylco-25 base metal.

Figure 42 graphically illustrates the hardness variations through the bond zone in vacuum-bonded Berylco-25. Although the average visible diffusion zone on either side of the bond (after etching) extends only approximately 0.0025 in. into the base metals, microhardness figures have picked up diffusion of filler metal into the base metal as far as 0.006 in. distance from the original interface.

Figure 40

Filler Metal: Incusil 10
Bond Cycle: 15 min @1475 °F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 41

Filler Metal: Incusil 10
Bond Cycle: 30 min @1475 °F
100×. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Fig. 42. Graph — Hardness Data Across Bond — Vacuum

Table III. Data for diffusion-type braze-bonding of Berylco-25 in high vacuum at 1475°F.

Specimen Number	Filler Metal	Thickness of Filler Metal (in.)	Holding Time at 1475°F (min)	Heat Treatment After Brazing	Pressure on Interface (psi)	Hardness of Berylco-25	Tensile Strength of Joint (psi)	Average Tensile Strength of Joint (psi)
I-243 - a	Incusil 10	0.002	15	60 min @ 1475°F, air-blast-cooled; then 2 hr @ 600°F	6	See graph (Fig. 42)	89,200	52,500
- b	"	"	"	"	"	"	96,200	
- c	"	"	"	"	"	"	99,400	
- d	"	"	"	"	"	"	95,400	
I-244 - a	Incusil 10	0.002	15	"	"	"	49,000	52,500
- b	"	"	"	"	"	"	30,000	
- c	"	"	"	"	"	"	34,600	
I-245 - a	Incusil 10	0.002	15	"	"	"	38,500	52,500
- b	"	"	"	"	"	"	11,500	
- c	"	"	"	"	"	"	17,300	
- d	"	"	"	"	"	"	14,400	
I-246 - a	Incusil 10	0.002	30	"	"	"	112,500	108,400
- b	"	"	"	"	"	"	93,300	
- c	"	"	"	"	"	"	76,900	
- d	"	"	"	"	"	"	70,400	
I-247 - a	Incusil 10	0.002	30	"	"	"	130,000	108,400
- b	"	"	"	"	"	"	127,300	
- c	"	"	"	"	"	"	123,800	
- d	"	"	"	"	"	"	112,000	
I-248 - a	Incusil 10	0.002	30	"	"	"	105,600	108,400
- b	"	"	"	"	"	"	90,000	
- c	"	"	"	"	"	"	128,000	
- d	"	"	"	"	"	"	131,200	

2. Metallographic Studies of Berylco-25 Base Metal After Vacuum Bonding Treatment

Although Fig. 36 (dry-hydrogen-bonded 30 minutes at 1475°F) shows only a slight tendency toward the growth of excessively large grains, Fig. 44 (vacuum-bonded 30 minutes at 1475°F) shows marked tendencies in that direction. The explanation for this difference is quite interesting.

It should first be pointed out that a considerable amount of duplexing and precipitation were found in almost all specimens examined in this program. Areas of almost any grain size desired could be photomicrographed in any of the bonded specimens, regardless of whether the bonding temperature was 1475°F or 1550°F, or whether the holding time was 15 minutes or more. Some isolated fine-grained areas could be found even in specimens bonded with long holding times at 1550°F. The contrast between the upper and lower Berylco-25 plates in Fig. 34 (brazed 60 minutes at 1475°F) attests to the wide variation found in grain size; both pieces were cut from the same plate before bonding.

In view of such variations, particular care was exercised to insure that each photomicrograph in this program was taken of an area most representative of the overall structure of the specimen.

But as to the difference in grain size of Figs. 36 and 44, both brazed 30 minutes at 1475°F, a new factor appears. The materials in these specimens came from two different mill heats of Berylco-25.

The material used for Figs. 43 and 44 and for the entire program with the exception of Phase B (dry hydrogen bonding at 1475°F) was purchased almost two years ago at the start of the Berylco-25 bonding studies. The material for all of Phase B, to which Fig. 36 belongs, was purchased well over a year later, and consistently shows a greater tendency to retain a fine grain structure at elevated temperatures. This difference was observed in many mounted, polished and etched specimens examined during the latter part of the program.

Figure 45 is a graphic comparison between the sizes of grains found in the duplex structures existing in Berylco-25 from the older mill heat which was used in this portion of the program.

Figure 43

Bond Cycle: 15 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Figure 44

Bond Cycle: 30 min @1475 °F
100X. Etchant: $\text{NH}_4\text{OH}-\text{H}_2\text{O}_2-\text{KOH}$

Fig. 45. Bar graph, holding time vs grain growth at 1475°F in vacuum

D. Studies of Heat Treating Parameters vs Tensile Strength in Berylco-25 Base Metal

During the bonding phases of this program, it was found that stresses set up at the interfaces on water-quenching bonded Berylco-25 plates caused failures along the bonded joint in some instances. It was therefore thought desirable to determine the ranges of holding time, temperature, and quenching rate within which the final minimum hardness of 32 R_C (142,000 psi tensile) could be maintained or exceeded after the final precipitation hardening treatment.

All specimens tested in this phase, with the exception of the ones involving the rate of quench studies, were given the standard water quench following solution heat treatment.

1. Effects of Varying Holding Time at Solution Heat-Treating Temperatures

Figure 46 graphically illustrates the effects on tensile strength of varying the holding time at solution heat-treating temperature from 60 minutes down to 5 minutes.

The solution heat-treating temperature was held constant at 1475 °F, and was followed in each case by a 3-hour precipitation hardening treatment at 600 °F.

The normal solution heat-treating and precipitation hardening cycle recommended for Berylco-25 (60 minutes at 1475 °F, water-quenched, and precipitation-hardened 2-3 hours at 600 °F) results in a tensile strength of approximately 200,000 psi.

Upon decreasing the holding time at 1475 °F to 45 minutes, a tensile strength of 199,000 psi resulted.

Upon decreasing the holding time to 30 minutes, the tensile strength rose from approximately 198,000 psi to 218,000 psi. Upon further reducing the holding time to 15 minutes, the tensile strength dropped off sharply to around 192,000 psi. Further reduction of the holding time to 5 minutes did not seem to have an appreciable effect on reducing the tensile strength below that shown by the 15-minute holding time.

2. Effects of Varying Solution Heat-Treating Temperature

Figure 47 illustrates the effects on the tensile strength of varying the solution heat-treating temperature from 1475 to 1400 °F.

Fig. 46. Graph — Solution Heat Treatment — Variations of Holding Time vs Tensile Strength

Fig. 47. Graph — Solution Heat Treatment — Variation of Temperature vs Tensile Strength

Fig. 48. Graph — Precipitation Hardening Treatment — Variation of Holding Time vs Tensile Strength

Fig. 49. Graph — Precipitation Hardening Treatment — Variation of Temperature vs Tensile Strength

In this work, the time at solution heat-treating temperature (60 minutes) was held constant. In each case the specimen was given a 3-hour precipitation hardening treatment at 600°F subsequent to solution heat treating.

Upon decreasing the solution heat-treating temperature from 1475 to 1460°F, and then to 1450°F, only a slight drop in tensile strength resulted. On decreasing to 1430°F, a sharper drop in tensile strength resulted (185,000 psi). In further decreasing the solution heat-treating temperature to 1400°F, a slight increase in final tensile strength to 189,000 psi resulted.

3. Effects of Varying Holding Times at Precipitation Hardening Temperatures

Figure 48 illustrates the effects on the tensile strength of Berylco-25 of varying the holding time at the precipitation hardening temperature from 120 minutes to 15 minutes. The precipitation hardening temperature was held constant at 600°F.

In decreasing the holding time from 180 minutes (3 hours) to 120 minutes (2 hours) the tensile strength decreased from approximately 200,000 psi to 187,000 psi, which is still well above the 142,000 (32 R_C) minimum established for this program. Upon decreasing the holding time further to 60 and then to 30 minutes, the tensile strengths still remained well above the 142,000 psi minimum. Upon further decrease in holding time to 15 minutes, a sharp drop in tensile strength resulted, but the strength was still higher than the minimum.

As a result of these data, a conservative decrease in precipitation-hardening holding time to 2 hours instead of the recommended 3 hours for Berylco-25 was instituted for the 1550°F and 1475°F bonding work in the balance of the program.

4. Effects of Varying Precipitation Hardening Temperatures

Figure 49 illustrates the effects on tensile strengths of Berylco-25 of varying the precipitation hardening temperature from 600°F to 500°F.

Very little drop in tensile strength resulted when the temperature was reduced to 585 and then to 575°F (192,000 psi). As the temperature was decreased further, the drop in tensile strength appeared steady and almost linear until, at a temperature of 500°F, the tensile strength had dropped to 151,000 psi, which is still above the allowable minimum (for this project) of 142,000 psi.

Fig. 50. Graph — Effect of Quenching Rate on Tensile Strength

Fig. 51. Graph — Increased Holding Time vs Tensile Strength after Solution Heat Treating (for Spinning)

5. Effects of Varying Rate of Quenching in Solution Heat Treating

Figure 50 illustrates the effects of varying the rate of quenching of Berylco-25 upon the effectiveness of solution heat treating. This was done by varying the quenching media.

The holding time, solution heat-treating temperature, and precipitation hardening cycle were held constant (60 minutes at 1475 °F, and 2 hours at 600 °F, respectively).

The quenching media used were oil at ambient temperature, oil at 200 °F, air-blast, water, and brine.

The thickness of Berylco-25 quenched was 1/2 in. in plate form.

The results of this study were interesting in that oil quenching both at ambient temperatures and at 200 °F gave a final tensile strength, after precipitation hardening, of above 190,000 psi.

Air-blast, water, and brine quenching all resulted in approximately the same tensile strengths of around 176,000 psi.

Water quenching in this program did show some detrimental effects on the joint strengths of the bonded specimens of Berylco-25. No failures were experienced using the air-blast quenching technique.

To further substantiate these findings, a separate program was conducted at a later date to determine the effects of air-blast quenching of Berylco-25 joined specimens (1/2 in. total thickness of plate) enclosed in a 0.035-in. -thick carbon-steel protective envelope. After precipitation hardening, these envelope-air-blast-quenched specimens showed a hardness of 35 R_C, equivalent to 155,000 psi tensile strength; this is approximately the same hardness as obtained by air-blast quenching the unprotected Berylco-25 bonded specimens in previous work.

As a result of data obtained in these quenching studies during solution heat treatment, all bonded specimens covered in this report were air-blast-quenched rather than water-quenched as recommended.

6. Effects of Increasing Holding Times at Solution Heat-Treating Temperatures on Softening Berylco-25 for Spinning

Difficulties have been encountered by LRL engineers in spinning Berylco-25 in the solution heat-treated condition as received from the mill.

This phase of the program was conducted to determine whether increased holding times at the solution heat-treating temperature of 1475 °F, followed by air-blast quenching, could further increase the ductility of Berylco-25 to improve spinning characteristics.

Figure 51 illustrates the results of these studies. It was found that a tensile strength of 80,000 psi (equivalent to a hardness of 84 R_B on conversion) was obtained by using the normal 60-minute holding time.

Data obtained on increasing the holding time at solution heat-treating temperatures are as follows:

Holding Time at 1475 °F (air-blast-quenched) (min)	Resulting Tensile Strength (psi)
60 (normal)	80,000
70	75,800
90	75,000
105	75,000
120	74,700
150	72,800
240	72,800

It appears from these data and from the curve shown in Fig. 51 that a maximum softening effect is reached on solution heat treating at 1475 °F by holding 150 minutes at temperature.

E. Studies of Microhardness Variations Across Bond Zones

In order to develop data on the hardening effects resulting from diffusion-type bonds in Berylco-25, microhardness traverses were made through the bond zones in specimens bonded with each of the four filler alloys selected from previous work. The range of hardness shown in the bond zones includes data taken from specimens held from 30 minutes through 120 minutes at 1550 °F, then solution heat-treated at 1475 °F, air-blast-cooled, and precipitation-hardened at 600 °F.

Filler Alloy Used	Hardness Range Through Bond Layer (Knoop converted to R_B)
Gold powder	90-104
Incusil 10	88-104
BT + 0.1% Li	90-101
Vacuum-plated gold	30-36 (R_C)

Table IV. Data for tensile testing of Berylco-25, 0.505-in. specimens after simulated bonding, solution heat-treating, and precipitation hardening.

Specimen Number	Simulated Bonding Treatment*	Tensile Strength* (psi)	Yield Strength* (psi)	Percent Elongation in 2 in.	Hardness Data		
					After Simulated Bonding	After Solution Heat Treat	After Precipitation Hardening
A-1	30 min @1550°F	158,000	139,000	3.5	88 R _B	80 R _B	35 R _C
A-2		157,000	140,000	2.5	88	80	34
B-1		162,000	144,000	3.0	88	79	35
B-2		162,000	147,000	3.0	89	79	35
A-3	45 min @1550°F	158,000	139,000	3.5	88	80	34
A-4		160,000	142,000	3.5	88	80	34
B-3		161,000	145,000	2.5	88	79	35
B-4		160,000	143,000	3.0	88	80	35
A-5	90 min @1550°F	164,000	—	3.0	89	77	34
A-6		165,000	—	3.0	87	80	37
B-5		162,000	—	2.0	88	77	35
B-6		165,000	—	2.0	88	78	36
A-7	30 min @1475°F	168,000	146,000	4.0	88	78	37
A-8		169,000	148,000	3.5	87	77	37
B-7		166,000	147,000	4.0	88	77	36
B-8		165,000	145,000	4.0	89	78	36
A-9	45 min @1475°F	168,000	146,000	4.5	88	76	37
A-10		166,000	144,000	4.5	89	77	37
B-9		165,000	146,000	4.0	90	76	37
B-10		167,000	146,000	4.0	90	77	36
A-11	90 min @1475°F	164,000	—	3.5	89	77	37
A-12		165,000	—	4.0	89	77	37
B-11		162,000	—	4.5	89	77	37
B-12		166,000	—	4.0	89	78	37

*After the simulated bonding treatments shown above, and prior to tensile testing, each specimen was solution heat-treated 60 minutes at 1475°F and immediately air-blast-cooled, then precipitation-hardened to holding 2 hours at 600°F, then air-cooled.

All of the bond layers are quite ductile with the exception of the vacuum-plated gold bond zone, which is in the middle Rockwell "C" range. This increase in hardness may be a result of the thinner, more controlled thickness of gold applied by vacuum plating as compared with gold powder applied in an acrylic binder. The thinner gold would be likely to diffuse into the Berylco-25 more completely, resulting in higher copper and beryllium content in the bond zone, and possibly some response in this zone to the precipitation hardening treatment.

None of these hardnesses are sufficiently high to give indications of possible zones of brittleness in the bond layers.

F. Studies of Effects of Various Heat Treatments on Tensile Strength of Berylco-25 Base Metal (0.505-in. Standard Tensile Tests)

In prior diffusion bonding work, metallographic examinations have indicated some wide variations in microstructures and in grain sizes after bonding of Berylco-25 at varying times and temperatures, followed by the solution heat-treating cycle developed in Phase D (60 minutes at 1475°F, air-blast-cooled) and the precipitation hardening treatment (2 hours at 600°F, air-cooled).

In an effort to study the effects of these bonding time-temperature variations on the tensile strength of Berylco-25 in the heat-treated condition, 30 0.505-in. standard tensile specimens were given various simulated bonding heat treatments, followed by the solution heat treatment (air-blast-cooled), and the precipitation hardening treatment. These specimens were then subjected to tensile testing.

Specimens marked "A" were cut with their longitudinal axes transverse to the direction of rolling of the Berylco-25, and specimens marked "B" were cut with their longitudinal axes parallel to the direction of rolling.

Figures 52 and 53 graphically illustrate the differences in tensile strengths in the longitudinal and transverse directions as the holding times at simulated bonding temperatures of 1550°F and 1475°F, respectively, are varied.

Figures 54 and 55 illustrate the differences in yield strengths in the longitudinal and transverse directions as the holding times at 1550°F and 1475°F, respectively, are varied.

Fig. 52. Graph — Holding Time at 1550°F vs Tensile Strength (0.505-in. Tensile Tests)

Fig. 53. Graph — Holding Time at 1475°F vs Tensile Strength (0.505-in. Tensile Tests)

Fig. 54. Graph — Holding Time at 1550°F vs Yield Strength (0.505-in. Tensile Tests)

Fig. 55. Graph — Holding Time at 1475°F vs Yield Strength (0.505-in. Tensile Tests)

Fig. 56. Graph — Holding Time at 1550°F vs Elongation in 2 in. (0.505-in. Tensile Tests)

Fig. 57. Graph — Holding Time at 1475°F vs Elongation in 2 in. (0.505-in. Tensile Tests)

Figures 56 and 57 illustrate the differences in percent elongation (in 2 in.) in the longitudinal and transverse directions as the holding times at 1550°F and 1475°F, respectively, are varied.

Figures 58 and 59 illustrate the differences in percent reduction in area in the longitudinal and transverse direction, as the holding times at 1550°F and 1475°F, respectively, are varied.

Figures 60 and 61 illustrate the differences in modulus of elasticity in the longitudinal and transverse directions as holding times at 1550°F and 1475°F, respectively, are varied.

A comparison of the differences in properties resulting from bonding at 1550°F and at 1475°F can be made from these graphical representations and from Table III. They are summarized at the beginning of this study under "Discussion and Conclusions," p. 4.

G. Diffusion-Bonded Plug Tensile Tests

This phase of the program involved the bonding of 3/4-in. -diameter Berylco-25 plugs into a press-fitted female Berylco-25 joint. Both the male and female mating surfaces were vacuum gold plated.

These plugs were bonded at various holding times at 1550°F, then solution heat-treated at 1475°F, air-blast-cooled to ambient temperatures, and precipitation-hardened for 120 minutes at 600°F.

Tensile strength tests of the bonds showed results as follows:

<u>Specimen Number</u>	<u>Holding Time at Bonding Temperature (min)</u>	<u>Tensile Strength (psi)</u>
I-125	45	24,400
I-126	60	Failed on machining
I-127	90	Failed on machining
I-128	120	16,300

It appears from these tests that the highest tensile strength in the bond layer was obtained with a 45-minute holding time at bonding temperature.

H. Butt-Joint Bonding for Subsequent Welding Experiments

Five vacuum-plated 1/4-in. -thick butt-joint specimens were brazed with vacuum-plated gold. They were bonded in dry hydrogen using specially designed fixtures so that a pressure of 6 psi could be applied to the joint exactly perpendicular to the bond interfaces.

Fig. 58. Graph — Holding Time vs Per Cent Reduction in Area (0.505-in. Tensile Tests)

Fig. 59. Graph — Holding Time at 1475°F vs Per Cent Reduction in Area (0.505-in. Tensile Tests)

Fig. 60. Graph — Holding Time at 1550°F vs Modulus of Elasticity (0.505-in. Tensile Tests)

Fig. 61. Graph — Holding Time at 1475°F vs Modulus of Elasticity (0.505-in. Tensile Tests)

Three specimens were bonded using a 90-minute holding time at 1550°F in dry hydrogen atmosphere, and two specimens were bonded using a 30-minute holding time at 1475°F.

All five of the specimens failed through the bond on very slight impact loading. Examination of the specimens showed a dark oxidation at the junction of the plating with the Berylco-25, which inhibited solid-state diffusion of the gold into the Berylco-25. It appeared that the vacuum plating was defective.

In an effort to avoid this vacuum plating variable, the specimens were remachined and rebrazed as follows:

<u>Specimen Number</u>	<u>Preparation of Berylco-25</u>	<u>Filler Alloy</u>	<u>Bonding Cycle</u>
I-249	—	Gold powder	90 min @1550°F
I-250	—	Gold powder	90 min @1550°F
I-251	—	Incusil 10 foil	30 min @1475°F
I-252	—	Incusil 10 foil	30 min @1475°F
I-253	Electroplated with 0.0005 in. of copper	Incusil 10 foil	30 min @1475°F

Each of these specimens was machined with a very small lip on the inside surface to eliminate any excess filler alloy from building up on the inside surface of the part. All specimens were bonded in dry hydrogen (dewpoint -80 to -100°F) with 6 psi pressure applied perpendicular to the joints.

All five of the specimens showed good alloy flow around the joint upon visual examination. Two of the specimens, bonded with the inside lip pointing downward, showed some filler-metal flow past the lip on the inside surface of the parts. The other three specimens, bonded with the lip pointing upward, showed no filleting or alloy flow on the inside surfaces of the parts.

All five specimens appeared soundly bonded and were resistant to the low-level impact tests that were applied.

These specimens have been submitted by Pyromet to LRL for welding tests. The results of the tests are not yet available.

APPENDIX

SPECIFICATION 24-I - VACUUM FURNACE TECHNIQUE
DIFFUSION-TYPE BONDING OF BERYLCO-25Pyromet Company
Research and Development Laboratory

-
1. Finish: Parts should have a #1 grit emery finish, or equivalent.
 2. Cleaning: After preparing the mating surfaces, wash entire part in acetone to remove dirt and grit. Dry in warm air blast.
 3. Filler Alloy: 0.002-in foil - 63% Ag, 27% Cu, 10% In (Incusil 10).
Clean filler alloy in accordance with (1) and (2), or acid-clean if preferred.
 4. Placement of Filler Alloy: Preplace filler alloy in the joint so that all of the area to be joined is covered.
 5. Pressure: Six psi minimum pressure should be exerted on the interface either by weighting, jigging, or some other method when the part is set up in the vacuum furnace.
 6. Vacuum: Maintain vacuum below 1×10^{-4} mm Hg throughout entire cycle.
 7. Bonding Cycle (Vacuum):
 - a. Raise from ambient temperature to 1475 °F in 40 minutes.
 - b. Hold at 1470-1480 °F for 30 minutes.
 - c. Cool to below 1250 °F in 30 minutes.
 - d. Retort-cool to ambient temperature.
 8. Heat Treating Cycle (Endothermic Atmosphere):
 - a. Heat to 1475 °F.
 - b. Hold 60 minutes at 1475 °F.
 - c. Air-blast-cool immediately to ambient temperature.
 9. Precipitation Hardening Cycle (Air or Reducing Atmosphere):
 - a. Heat to 600 °F.
 - b. Hold 2 hours at 600 °F.
 - c. Air-cool.

This procedure should result in above 100,000 psi average bond strength, and above 38 R_C average hardness in the Berylco-25 base alloy.

SPECIFICATION 24-II - ATMOSPHERE FURNACE TECHNIQUE
DIFFUSION-TYPE BONDING OF BERYLCO-25

Pyromet Company
Research and Development Laboratory

-
1. Finish: Parts should have a #1 grit emery finish, or equivalent.
 2. Cleaning: After preparing the mating surfaces, wash entire part with acetone to remove dirt and grit. Dry in warm air blast.
 3. Filler Alloy: 0.002-in. foil - 63% Ag, 27% Cu, 10% In (Incusil-10). Clean filler alloy in accordance with (1) and (2), or acid-clean if preferred.
 4. Placement of Filler Alloy: Preplace filler alloy in the joint so that all of the area to be joined is covered.
 5. Pressure: Six psi minimum pressure should be exerted on the interface by weighting, jiggling, or by some other method when the part is set up in the furnace.
 6. Atmosphere: Dry hydrogen (dewpoint -80 °F or lower).
 7. Bonding Cycle (Dry Hydrogen):
 - a. Raise from ambient temperature to 1475 °F in 170 minutes.
 - b. Hold at 1470-1480 °F for 15 to 30 minutes.
 - c. Cool to below 1000 °F in 30 minutes.
 - d. Retort-cool to ambient temperature.
 8. Heat Treating Cycle (Endothermic Atmosphere):
 - a. Heat to 1475 °F.
 - b. Hold 60 minutes at 1475 °F.
 - c. Air-blast-cool immediately to ambient temperatures.
 9. Precipitation Hardening Cycle (Air or Reducing Atmosphere):
 - a. Heat to 600 °F.
 - b. Hold 2 hours at 600 °F.
 - c. Air-cool.

This procedure should result in above 90,000 psi average bond strength, and above 36 R_C average hardness in the Berylco-25 base alloy.

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