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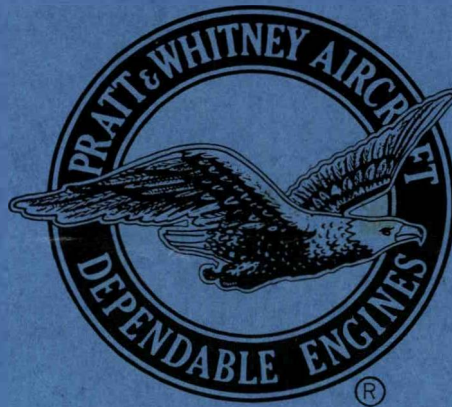
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~~CNLM-6076~~
 SPACE RADIATOR FABRICATION DEVELOPMENT
 FOR
 November and December, 1964
 January and February, 1965

AEC RESEARCH AND DEVELOPMENT REPORT



P R A T T & W H I T N E Y A I R C R A F T
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CNLM-6076
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P R A T T & W H I T N E Y A I R C R A F T
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SPACE RADIATOR FABRICATION DEVELOPMENT
FOR
November and December, 1964
January and February, 1965



UNCLASSIFIED

CLASSIFICATION

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April 8, 1965

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SPACE RADIATOR FABRICATION DEVELOPMENT
FOR
November and December, 1964



V. FABRICATION DEVELOPMENT (V. P. Treciokas, J. R. Stewart)

7701 - Space Radiator Fabrication Development (N.T. Wagenheim, W. F. Hanny)

Objective

This program is intended to 1) evaluate compatibility of space radiator materials under simulated service conditions, 2) develop joining methods for radiator assembly, 3) evaluate strength of joints, and 4) fabricate model radiator panels.

Abstract

All 800-hour compatibility tests were completed. Compatibility couples containing beryllium as one of the materials were examined metallographically; only silver, molybdenum, vanadium and tungsten showed reaction zones less than one mil in thickness after 800 hours at 760C. Beryllium-columbium alloy and beryllium-stainless steel joints brazed with silver separated during aging test at 760C for 800 hours. Compatibility tests were initiated on specimens joined by 1) diffusion bonding with silver foil at 760C, 2) brazing with Ti-42.5 Mn alloy, and 3) brazing with four silver base alloys.

Compatibility Testing

Compatibility couples scheduled for 3000 and 10,000 hours, under test conditions previously described (PWAC-1016, PWAC-1017) have accumulated 1696 hours. All 800-hour compatibility couples were completed. Test results of preliminary evaluations are summarized in Fig. 35. Metallographic examination of the 800-hour beryllium couples were completed: results are summarized in Fig. 36.

All of the beryllium/silver couples tested at 540C, 650C and 760C, for 800 hours were diffusion-bonded. Metallographic examination showed no visible reaction between beryllium and silver tested at 540C and 650C. A 1-mil reaction zone was observed in the 760C couple. Hardness in this zone ranged from 129 to 137 DPH (20 gm load). Qualitative determination of diffusion effects by diffraction analysis on both the 100-hour and 800-hour beryllium/silver couples will be delayed until completion of metallographic examination.

Beryllium/Cb-1 Zr alloy couples all showed visible reactions after 800 hours, with roughening of interfacial surfaces, but no bonding. Metallographic examination revealed only a very slight reaction zone (0.05 mil) at 540C. The zone width increased, however, with increasing test temperature: 0.2 mil at 650C and 1.3 mils at 760C. In addition, the hardness of the zone also increased with temperature: 527 DPH at 650C as compared to 842 DPH at 760C.

Beryllium/316 stainless steel couples all showed surface roughening after 800 hours, but showed no bonding. Metallographic examination revealed an increase in zone width with increasing temperature: 0.15 mil at 540C, 4.0 mils at 650C, and 10.0 mils at 760C. A single reaction layer was seen in the 540C and 650C specimens; three layers were observed in the 760C specimen.

Beryllium/copper couples all bonded after 800 hours with extensive reaction, noted metallographically, at all test temperatures. The 760C specimen had a 100-mil reaction zone with six distinct constituents. Microhardness tests on these samples is in progress.

The only other material which bonded to the beryllium was the 540C/800-hour vanadium specimen. Metallographic examination showed no reaction zone and the bond appeared to be only mechanical. The 650C and 760C couples did not bond. No reaction occurred between beryllium and vanadium at 650C, but a 0.7-mil zone was observed in the 760C specimen.

RADIATOR MATERIAL COMPATIBILITY DATA

(TESTED IN VACUUM $> 5 \times 10^{-5}$ TORR)

	Cb-1 Zr						Cb-20 Ti						316 SS						Cu						Be					
	540C		650C		760C		540C		650C		760C		540C		650C		760C		540C		650C		760C		540C		650C		760C	
	Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours		Hours	
	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800	100	800
Ag	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	D	B	N	B	N	B	B	B	B	B	B	B	
Co	B	B	B	B	B	B	N	N	B	B	B	B	N	N	B	B	B	B	B	B	N	B	N	R	N	R	N	R		
Cr	N	N	B	N	B	N	B	N	B	N	B	B	N	N	N	N	N	N	B	B	B	B	B	N	C	B	C	R	R	
Cu	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	B	B	B	B	R	B		
Fe	N	N	D	B	D	B	N	N	N	N	B	N	N	N	N	N	B	B	B	B	B	B	B	R	C	R	R	D	R	
Mo	N	N	N	B	D	B	N	N	D	B	D	B	N	N	N	B	D	N	D	N	N	B	D	B	R	R	R	C	R	C
Ni	D	N	N	N	N	N	N	N	N	N	N	N	N	B	B	B	B	B	B	B	B	B	B	R	C	R	R	D	R	
Pd	N	B	B	B	B	B	N	B	N	N	N	N	N	B	B	B	B	B	B	B	B	B	B	R	R	B	R	R	R	
Pt	B	B	N	B	N	B	B	N	B	B	B	N	N	N	N	N	N	N	N	B	B	B	B	B	3	R	R	R	R	R
Ta	N	N	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	B	N	R	R	R	R	R	
Ti	D	B	B	B	B	B	B	B	B	B	B	N	N	D	B	B	B	B	B	B	B	B	B	D	N	R	C	D	R	
V	D	B	B	B	D	D	D	B	B	B	D	N	D	N	N	N	N	B	D	N	N	N	B	B	N	B	R	R	D	C
W	N	N	B	N	B	B	N	B	B	B	B	B	N	N	B	B	N	N	N	N	N	N	N	R	C	R	C	D	C	
Zr	D	B	B	B	B	B	D	B	B	B	B	B	B	B	N	B	B	N	N	N	N	N	N	R	R	R	R	D	R	
316 SS	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	N	N	B	B	B	B	R	C	R	R	R	R	
Cb-1 Zr	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	N	N	B	B	B	B	R	C	R	C	R	R	
Cb-20 Ti	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	----	B	B	B	B	B	B	R	C	R	C	R	R	

D = Bond separated during rough Metallographic preparation

R = Visible reaction, No bond

N = No bond, or visible reaction

B = Bonded

I = In test

C = Slight reaction - under .001" - No bond

BERYLLIUM COMPATIBILITY TEST RESULTS TESTED 800 HOURS IN VACUUM
AT 540, 650 AND 760C

Compatibility Couple	Total Width of Reaction Zones in mils			Number of Reaction Zones			Hardness Range in Reaction Zones, DPH						Bond		
	540C	650C	760C	540C	650C	760C	540C		650C		760C		540C	650C	760C
							Max.	Min.	Max.	Min.	Max.	Min.			
Be-Ag	0	0	1	0	0	1	-	-	-	-	137	129	Yes	Yes	Yes
Be-Cu	8	27	100	3	3	6							Yes	Yes	Yes
Be-3i6	0.15	4	10	1	1	3			1314	1314	1314	339	No	No	No
Be-Cb-1Zr	0.05	0.2	1.3	1	1	1	-	-	538	517	885	800	No	No	No
Be-Ni	0.7	8.5	28.0	1	2	2					1400	1235	No	No	No
Be-Pd	2.5	5.8	9 - 12	2	2	2					517	221	No	No	No
Be-Pt	No Spec.	3.8*	4.4*	No	1	1	No Spec						-	No	No
Be-Co	1.2	3.4	3	2	3	2					841	400	No	No	No
Be-Cr	0.6	0.7	2.3	1	1	1					242	201	No	No	No
Be-Fe	0.3	1.5	4	1	1	1							No	No	No
Be-Mo	0	0.1	0.4	0	0	1	-	-	-	-	1235	1235	No	No	No
Be-Ti	0	0.5	2.0	0	1	1	-	-	1400	1314			No	No	No
Be-V	0	0	0.7	0	0	1	-	-	-	-			Yes	No	No
Be-W	0.1	0.1	0.5	1	1	1					661	630	No	No	No
Be-Zr	1.5	3.3	22	1	1	1					801	291	No	No	No
Be-Cb-20 Ti	0.05	0.1	1.5	1	1	1	-	-					No	No	No

*Reacted completely with nominal 4-mil foil

All remaining materials listed in Fig. 35 reacted with the beryllium but did not bond. Metallographic examination showed that the following materials produced a reaction zone less than 1 mil thick after 800 hours at 760C: silver, molybdenum, vanadium and tungsten (Fig. 36). Hardness data on the majority of the 760C-800 hour specimens is given in Fig. 36. Data will be obtained on all specimens tested.

Joining Development

Silver Brazing: Beryllium/Cb-1 Zr alloy and beryllium/316 stainless steel specimens, brazed at 890C using 4-mil silver foil and 0.7-mil electroplated silver, completed 800-hour vacuum (5×10^{-5} torr) compatibility tests at 650C and 760C. Visual examination of the specimens showed all 760C soaked specimens had separated during test. Typical photomicrographs and microhardness data across the joints are given in Figs. 37 and 38. Two distinct diffusion zones are present in the Cb-1 Zr alloy, adjacent to the silver-beryllium eutectic (Fig 37) similar to that previously reported (PWAC-1017) for the 100-hour tests. The type 316 stainless steel/beryllium specimens showed three zones after 800 hours (Fig. 38) similar to those previously seen after 100 hours. Extremely high hardness readings were observed in these zones. For the 650C/800-hour specimens, values reached 1980 DPH in the Cb-1 Zr alloy and 2150 DPH in the 316 stainless steel. Fig. 39 shows clearly that the width of these zones increases with both time and temperature, culminating in failure of the joint. Preliminary metallographic examination of the failed specimens indicate failure occurred either at the interface between the silver-beryllium eutectic and the Zone A and/or in the silver-beryllium eutectic. Identification of the reaction zones by X-ray diffraction techniques is still in progress.

Failure of the 760C soaked specimens may be associated with the formation of the γ phase in the beryllium-silver system (Fig. 40). This phase does not form below approximately 760C. Results of compatibility tests between beryllium and silver show bonding occurs at 760C, 650C, and even 540C. Therefore, joints could be made without forming the γ phase by diffusion bonding at 760C or below. Metallographic examination of the 800-hour/760C compatibility couple showed only a 1-mil reaction zone with a maximum hardness of 137 DPH. Cb-1 Zr alloy/beryllium and 316 stainless steel/beryllium compatibility specimens were diffusion bonded at 760C, using 4-mil silver foil as the interface material. These specimens have accumulated 460 hours of a scheduled 800-hour test at 760C. Due to failure of the compatibility specimen, brazed with silver at 890C, lap-shear tensile strength tests on specimens joined at this temperature were not performed. Performance of strength tests on 760C diffusion bonded specimens will depend upon results of compatibility tests.

Other Braze Alloys: Several titanium, zirconium and nickel base alloys were evaluated for joining beryllium to Cb-1 Zr alloy and 316 stainless steel and results were reported in PWAC-1017. Based on these data, an attempt was made to braze compatibility specimens using these alloys for testing at 760C for 800 hours. The specimens were rectangular in shape, 1-inch by 3/8-inch, with 1/8-inch thick beryllium joined to 1/16-inch thick Cb-1 Zr alloy and 316 stainless steel. Ti-42.5 Mn alloy was the only braze material with which a sound joint could be made. The other alloys cracked on cooling from the braze temperature, possibly due to stresses caused by differences in thermal expansion of the materials being joined. In addition to the Ti-42.5 Mn, compatibility specimens of beryllium/Cb-1 Zr alloy and beryllium/316 stainless steel were also made using the following silver base alloys:

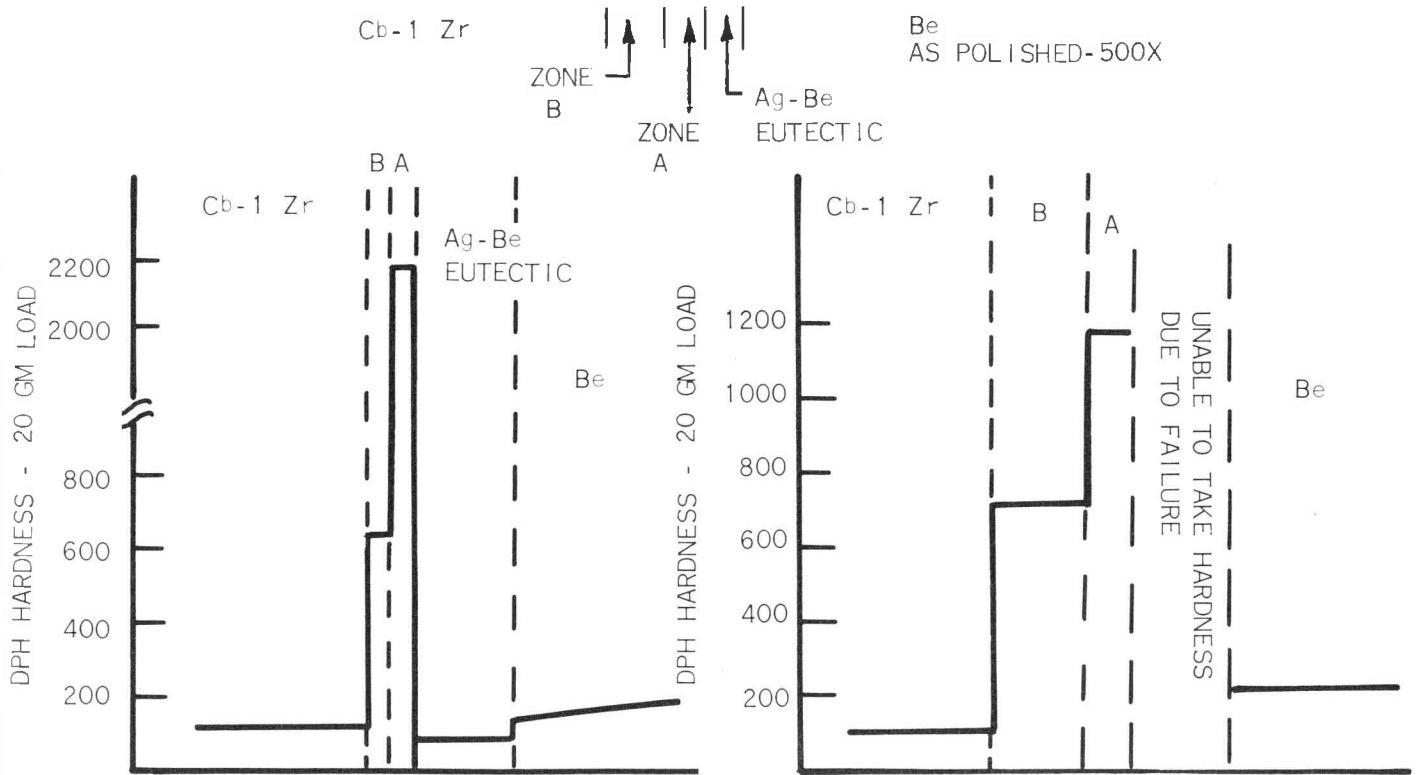
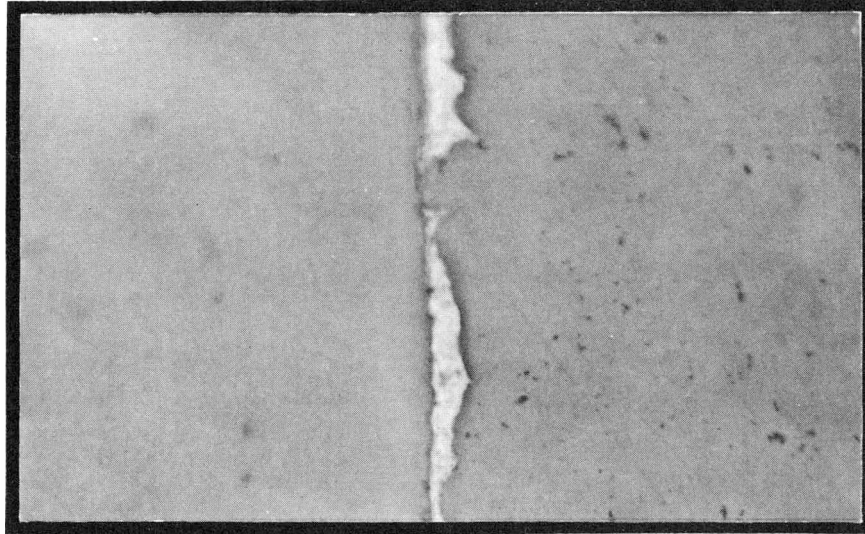
<u>Braze Alloy</u>	<u>Braze Temperature and Time</u>
Ag-28 Cu	830C - 10 min.
Ag-10 Pd	900C - 10 min.
Ag-15 Mn	915C - 10 min.
Ag-9 Cu-0.3 Li	840C - 10 min.

Vacuum (5×10^{-5} torr) compatibility tests at 760C were initiated and have reached 460 hours of a scheduled 800-hour test.

Compatibility testing of silicon-coated beryllium, silver-brazed to Cb-1 Zr alloy (as outlined in PWAC-1017), was not accomplished during this report period. Tests will be initiated during the next report period.

FIG 37

MICRO HARDNESS DATA FOR BERYLLIUM/SILVER/Cb-1 Zr SPECIMEN
 BRAZED IN ARGON-890° C-10 MIN. WITH 4 MIL SILVER FOIL
 AGED FOR 800 HRS AT 650°C



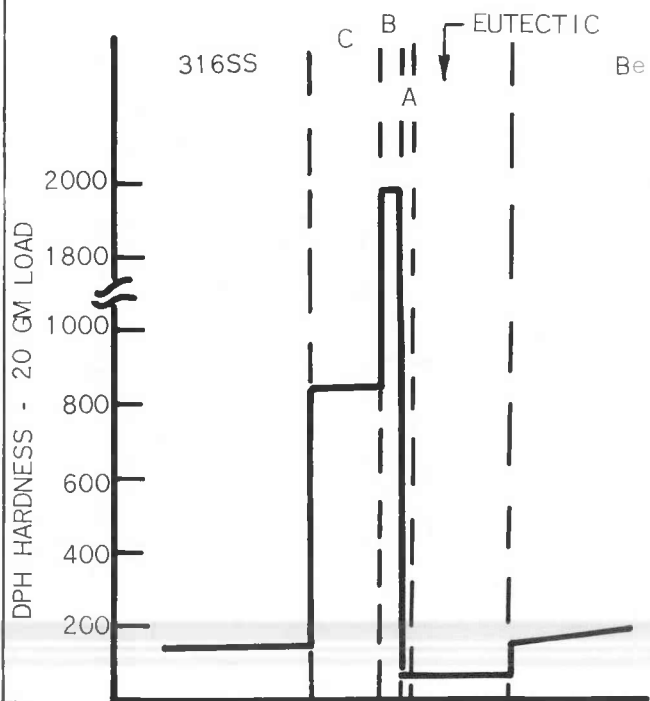
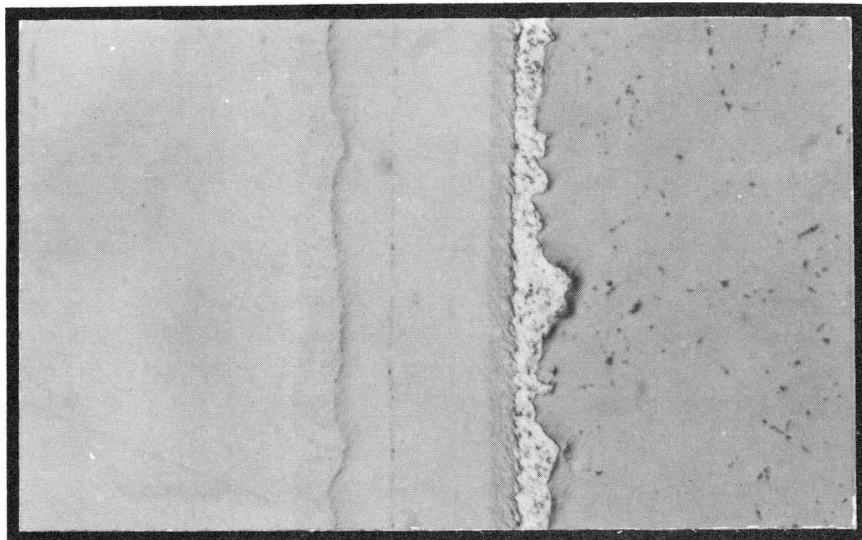
AGED 800 HRS AT 650C

AGED 800 HRS AT 760C

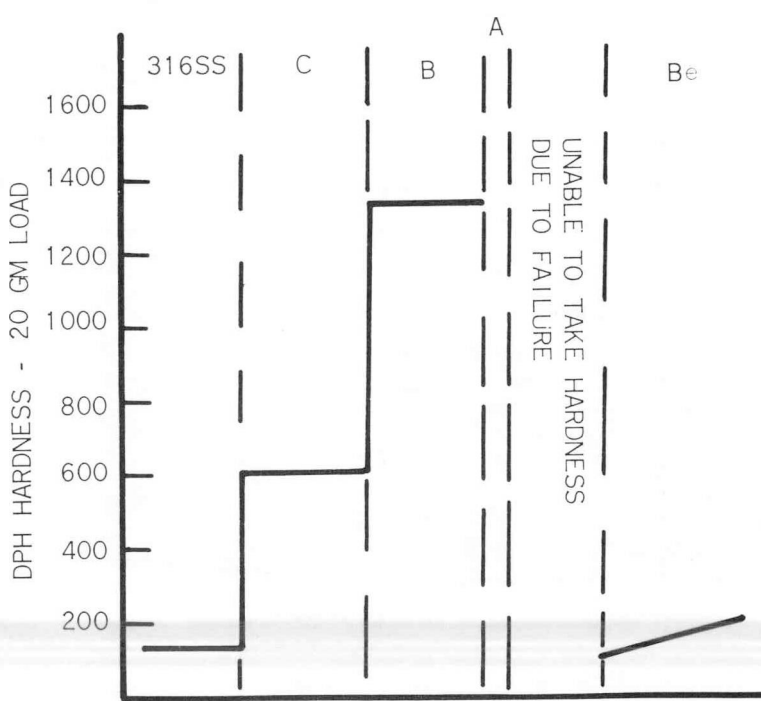
FIG 38

MICRO HARDNESS DATA FOR BERYLLIUM/SILVER/316SS SPECIMEN
BRAZED IN ARGON-890C-10 MIN. WITH 4 MIL SILVER FOIL

AGED FOR 800 HRS AT 650C



AGED - 800 HRS - 650C



AGED - 800 HRS - 760C

EFFECT OF ELEVATED TEMPERATURE AGING ON BERYLLIUM - DISSIMILAR METAL BRAZED JOINTS

BRAZE ALLOY - PURE SILVER

BRAZE CONDITIONS - 890C, 10 MIN, ARGON

AGED - 100 AND 800 HOURS AT TEMPERATURE SHOWN

Total Reaction Zone Thickness - Inches (1)

Aging Temp, C	<u>Be/Cb-1 Zr Joint</u>				<u>Be/Type 316 SS Joint</u>			
	<u>Silver Plate (2)</u>		<u>Silver Foil (2)</u>		<u>Silver Plate (2)</u>		<u>Silver Foil (2)</u>	
	<u>100 Hrs.</u>	<u>800 Hrs.</u>	<u>100 Hrs.</u>	<u>800 Hrs.</u>	<u>100 Hrs.</u>	<u>800 Hrs.</u>	<u>100 Hrs.</u>	<u>800 Hrs.</u>
(As brazed)	0.0004	0.0003	0.0004	0.0003	0.0010	0.0010	0.0010	0.0011
540	0.0004	-----	0.0004	-----	0.0010	-----	0.0010	-----
650	0.0006	0.0005	0.0007	0.0010	0.0015	0.0021	0.0015	0.0022
760	0.0010	0.0030 (3)	0.0012	0.0030 (3)	0.0033	0.0050 (3)	0.006	0.0055 (3)

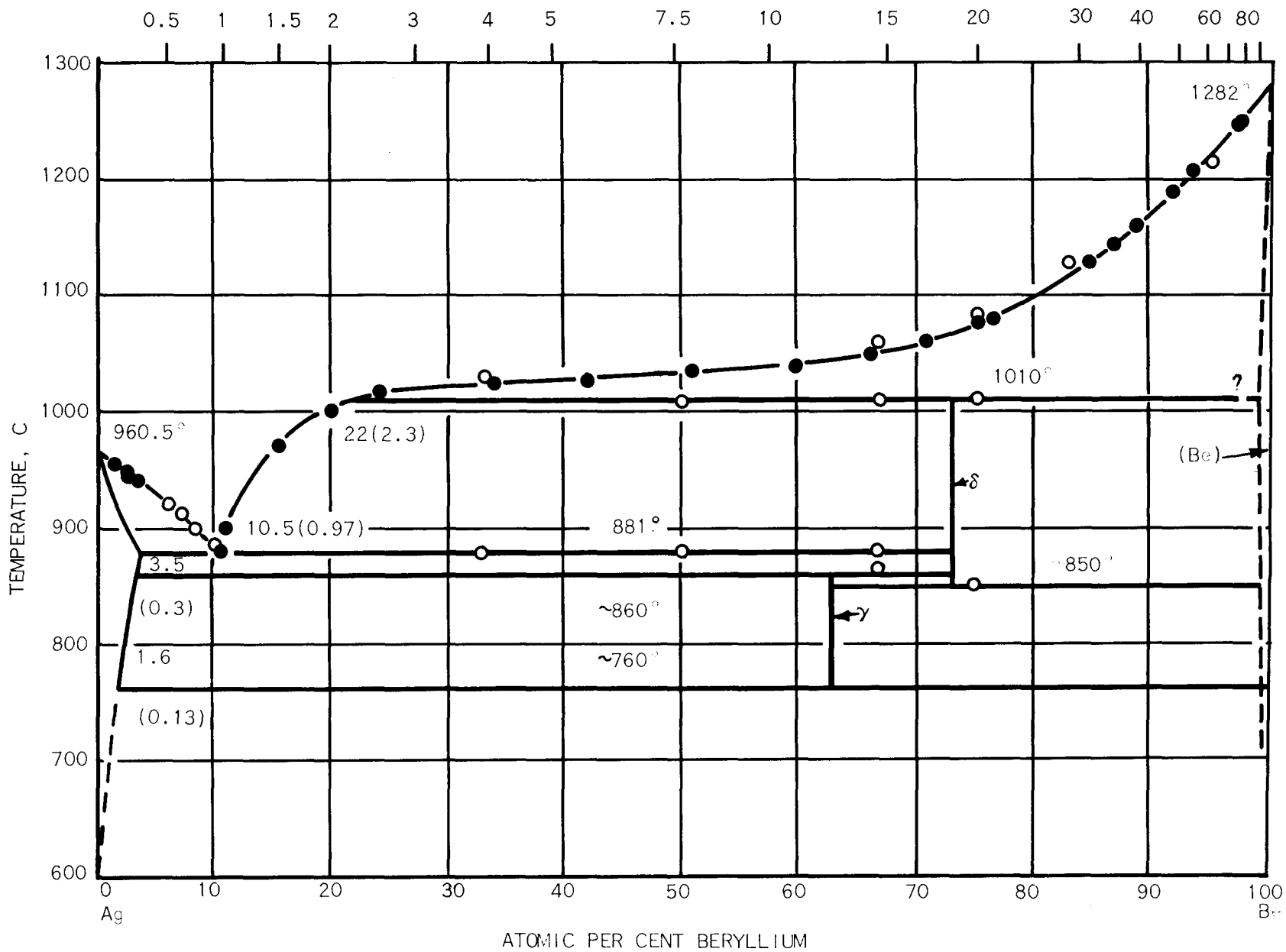
(1) Total reaction depth measured in Cb-1 Zr alloy and type 316 SS side of joint

(2) Refers to form of silver filler: electroplate 0.0007 in. thick or foil, 4 mils thick

(3) Specimens separated during test

SILVER - BERYLLIUM PHASE DIAGRAM

WEIGHT PERCENT BERYLLIUM



16

FIG 40
CNLM - 6076

TAKEN FROM "CONSTITUTION OF BINARY ALLOYS" BY M. HANSEN

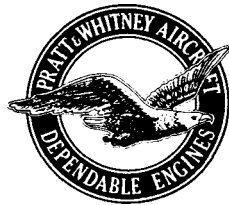
McGRAW-HILL BOOK CO., INC. 1958 SECOND EDITION

Fabrication: Beryllium fin/armor segments and Cb-1 Zr alloy tubes and headers have been machined for use in a three tube-six fin/armor, model radiator panel. Assembly of the unit has been delayed due to difficulties in producing a sound joint for use at 760C. Upon selection of a joining method, assembly will be initiated.

Future Work

1. Continue metallographic analysis of 100-hour and 800-hour compatibility tests.
2. Complete 3000-hour compatibility tests.
3. Complete 800-hour/760C compatibility tests on silver diffusion bonded specimens.
4. Complete 800-hour/760C compatibility tests on titanium and silver base alloys.
5. Initiate compatibility tests on silicon-coated beryllium specimens.

SPACE RADIATOR FABRICATION DEVELOPMENT
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January and February, 1965



V. FABRICATION DEVELOPMENT

(V. P. Treciokas and J. R. Stewart)

7701 - Space Radiator Fabrication Development
(N. T. Wagenheim and W. F. Hanny)

Objective

This program is intended to 1) evaluate compatibility of space radiator materials under simulated service conditions, 2) develop joining methods for radiator assembly, 3) evaluate strength of joints, and 4) fabricate model radiator panels.

Abstract

The 3000-hour compatibility tests were completed. Extrapolation to 10,000 hours showed that of all the beryllium couples tested, only the silver-beryllium combination could be expected to react to thicknesses less than one mil at 760C. At 650C, silver, tungsten, tantalum, molybdenum, Cb-1 Zr, vanadium and possibly chromium reactions with beryllium would probably be less than one to two mils in depth after 10,000 hours. Preliminary X-ray data suggests that the failure of silver-joined specimens of beryllium/Cb-1 Zr and beryllium/316 stainless steel, during heating at 760C for 800 hours, may be associated with the formation of gamma phase (Ag/Be) and brittle columbium or iron beryllide phases. Compatibility tests were initiated at 650C and 705C on brazed specimens of beryllium/Cb-1 Zr and beryllium/316 stainless steel. Good diffusion bonds were formed between silver and beryllium. No other material was found to be satisfactory.

Compatibility Testing

The 3000-hour compatibility tests conducted at 540C, 650C, and 760C were completed. Preliminary evaluation of test results are summarized in Fig. 37. Metallographic examination of the 3000-hour beryllium couples were completed; results are summarized in Fig. 38. The beryllium/silver couples tested at 540C, 650C and 760C for 3000 hours were all diffusion bonded. Metallographic examination revealed no detectable reaction zone in any of the specimens. The 800-hour/760C couple, previously reported as having a one-mil zone (PWAC-1018), showed no detectable reaction on reexamination. Silver was the only material tested which showed no reaction at any of the temperatures or times investigated. Bonding was probably accomplished by a solid solution reaction; there are no silver-beryllium inter-metallic phases which could be expected under the time-temperature conditions used in these tests. Evidence of silver-beryllium solid solution reaction is shown by the fact that when beryllium is joined to Cb-1 Zr with silver, beryllides are formed at the Cb-1 Zr interface.

Beryllium/chromium and beryllium/molybdenum couples were bonded after 3000 hours at 650C (Fig. 38). Similar couples tested at 540C and 760C were not bonded. Metallographic examination of the 650C samples of both couples showed a 0.4-mil reaction layer with no cracking. Bonding of the beryllium/molybdenum couple was less extensive than in the case of the beryllium/chromium couple. Six other materials, copper, 316 stainless steel, Cb-1 Zr, nickel, tungsten, and zirconium, produced bonds with beryllium at various test temperatures (Fig. 38). Metallographic examination, however, showed considerable cracking in the reaction zones of all these couples.

Measurement of reaction zone thicknesses on the 100, 800 and 3000-hour beryllium couples was completed. Plots of the log of the reaction zone thickness versus log time at 540C, 650C and 760C are shown in Figs. 39, 40 and 41, respectively. In most cases the data was consistent and produced straight line relationships. Several couples showed no measurable zone or the results were erratic; these are represented by dashed lines. Based upon these results, an extrapolation of reaction zone thickness at 10,000 hours was attempted for each temperature. This showed that silver was the only material which did not react with beryllium to depths exceeding one mil after 10,000 hours at 760C. At 650C, silver was again the only material with less than one mil reaction, although tungsten, tantalum, molybdenum, Cb-1 Zr, vanadium and possible chromium were between one and two mils. At 540C, several materials produced zones less than one mil (Fig. 39). Completion of the 10,000-hour couples, which have accumulated 3336 hours to date, will be required to check these extrapolated values.

RADIATOR MATERIAL COMPATIBILITY DATA

(TESTED IN VACUUM > 5×10^{-5} TORR)

	Cb-1 Zr												Cb-20 Ti												316 SS					
	540C Hours				650C Hours				760C Hours				540C Hours				650C Hours				760C Hours				540C Hours					
	100	800	3000	10,000	100	800	3000	10,000	100	800	3000	10,000	100	800	3000	10,000	100	800	3000	10,000	100	800	3000	10,000	100	800	3000	10,000		
Ag	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I		
Co	B	B	B	I	B	B	B	I	B	B	B	I	N	N	B	I	B	B	B	I	B	B	B	I	N	N	B	I		
Cr	N	N	N	B	I	B	N	N	I	B	N	N	I	B	N	B	I	B	N	B	I	B	B	B	I	N	N	N	B	I
Cu	N	N	N	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	N	N	N	B	I
Fe	N	N	N	B	I	D	B	B	I	D	B	N	I	N	N	N	I	N	N	N	I	D	B	B	I	N	N	N	B	I
Mo	N	N	N	B	I	N	B	B	I	D	B	B	I	N	N	B	I	D	B	B	I	D	B	B	I	N	N	N	B	I
Ni	D	N	N	N	I	N	N	N	I	N	N	N	I	N	N	N	I	N	N	N	I	N	N	N	I	B	B	B	B	I
Pd	N	B	B	I	B	B	B	I	B	B	B	I	N	N	B	B	I	N	N	B	I	N	N	N	I	B	B	B	B	I
Pt	B	B	B	I	N	B	N	I	N	B	B	I	B	N	B	I	B	B	B	I	B	N	B	I	N	N	B	B	B	I
Ta	N	N	N	N	I	B	B	B	I	B	B	B	I	B	N	N	I	B	B	B	I	B	B	B	I	B	B	B	B	I
Ti	D	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	B	B	B	I	N	N	N	B	B	I
V	D	B	B	I	B	B	B	I	D	D	B	I	D	B	N	I	B	B	B	I	D	N	B	I	D	N	N	X	X	I
W	N	N	N	N	I	B	N	N	I	B	B	N	I	N	B	B	I	B	B	B	I	B	B	B	I	N	N	N	N	I
Zr	D	B	B	I	B	B	B	I	B	B	B	I	D	B	B	I	B	B	B	I	B	B	B	I	B	B	B	B	I	

316
Cb-1Zr
Cb-20Ti

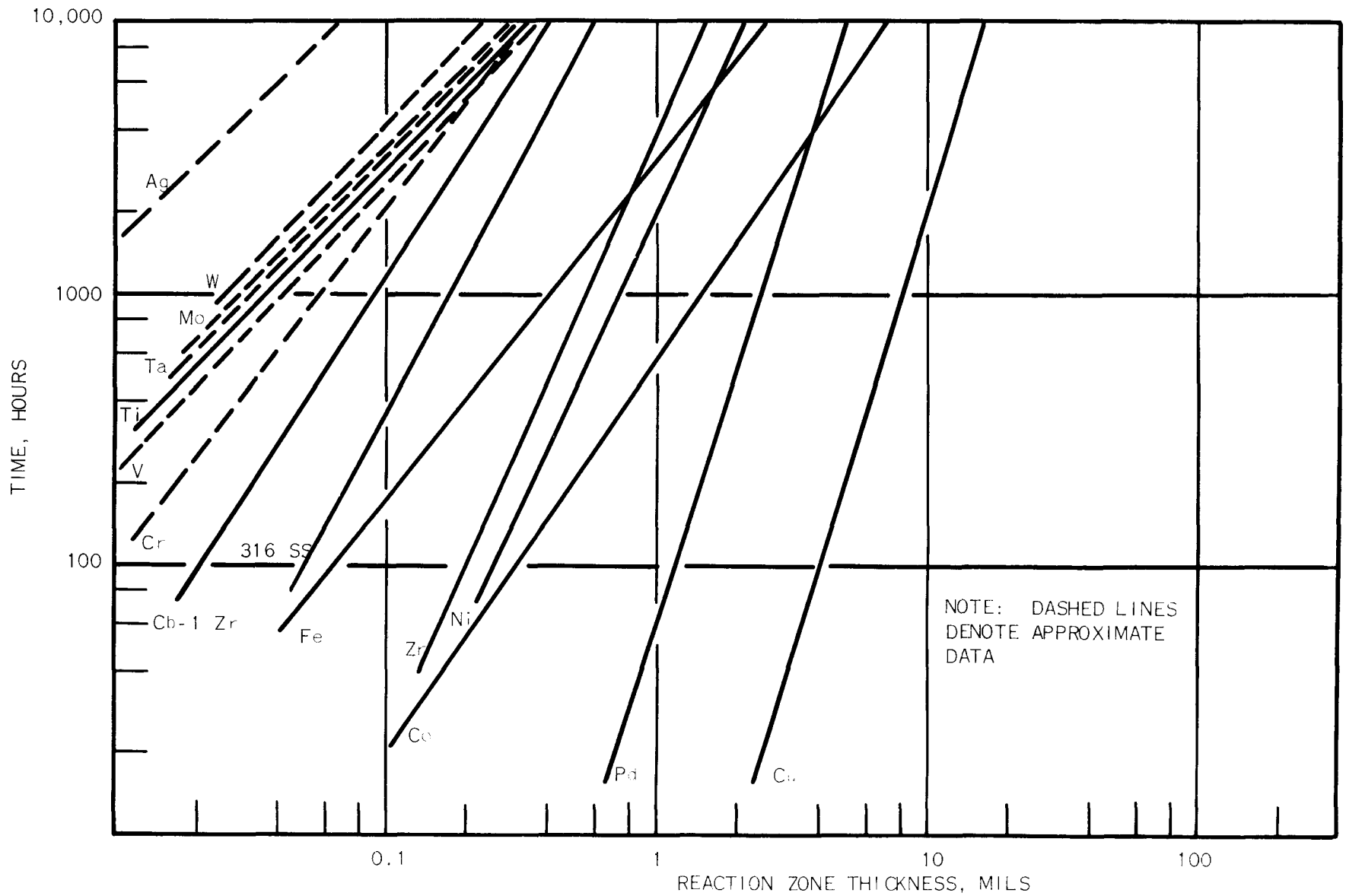
COMPATIBILITY OF BERYLLIUM WITH VARIOUS METALS AT ELEVATED TEMPERATURES

EXPOSURE TIME - 3000 HOURS
ENVIRONMENT - VACUUM $> 5 \times 10^{-5}$ TORR

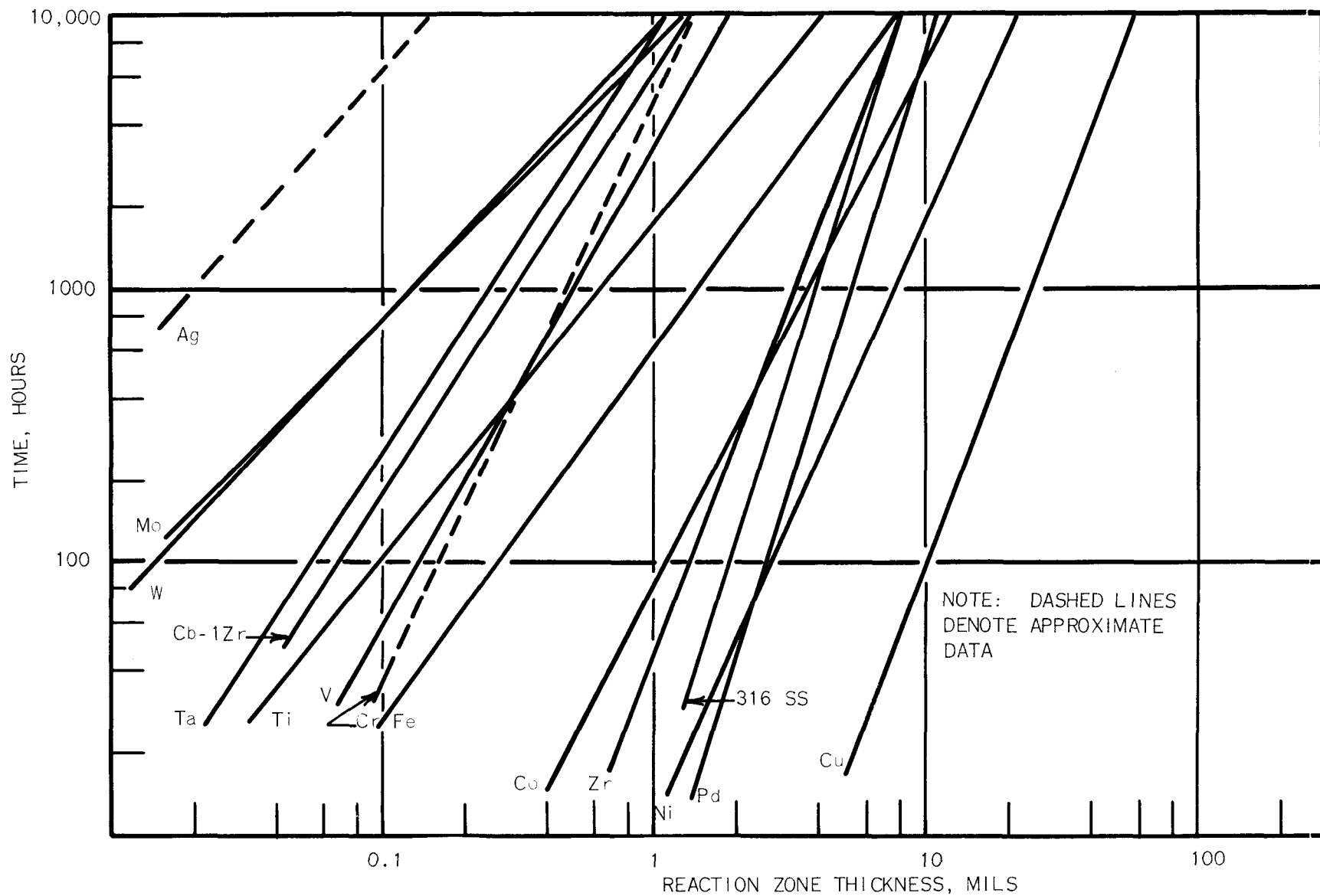
Beryllium Vs.	Total Width of Reaction Zones in Mils			Number of Separate Reaction Zones			Bond		
	540C	650C	760C	540C	650C	760C	540C	650C	760C
Silver	0	0	0	0	0	0	Yes	Yes	Yes
Copper	10	23	125	3	3	6	Yes*	No	Yes*
Type 316 Stain- less Steel	0.3	5	14	1	1	3	Yes*	No	No
Cb-1 Zr	0.2	0.7	3	1	1	1	No	Yes*	No
Nickel	1	10	45	1	2	2	No	Yes*	No
Palladium	3	6	19	2	2	2	No	No	No
Tantalum	0	0.5	1.4	1	1	1	No	No	No
Cobalt	3	-	8.5	2	-	3	No	-	No
Chromium	0	0.4	4.6	0	1	1	No	Yes	No
Iron	1	2.5	8.5	1	1	2	No	No	No
Molybdenum	0	0.4	1.3	0	1	1	No	Yes	No
Titanium	0.1	1.6	4.2	1	1	1	No	No	No
Vanadium	0	0.7	2.0	0	1	1	No	No	No
Tungsten	0	0	1.7	0	0	2	No	No	Yes*
Zirconium	0.5	5	35	1	1	1	No	No	Yes*
Cb-20 Ti	0.1	0.6	1.6	1	1	2	No	No	No

*Specimens bonded; however, metallographic examination showed considerable cracking in the reaction zone.

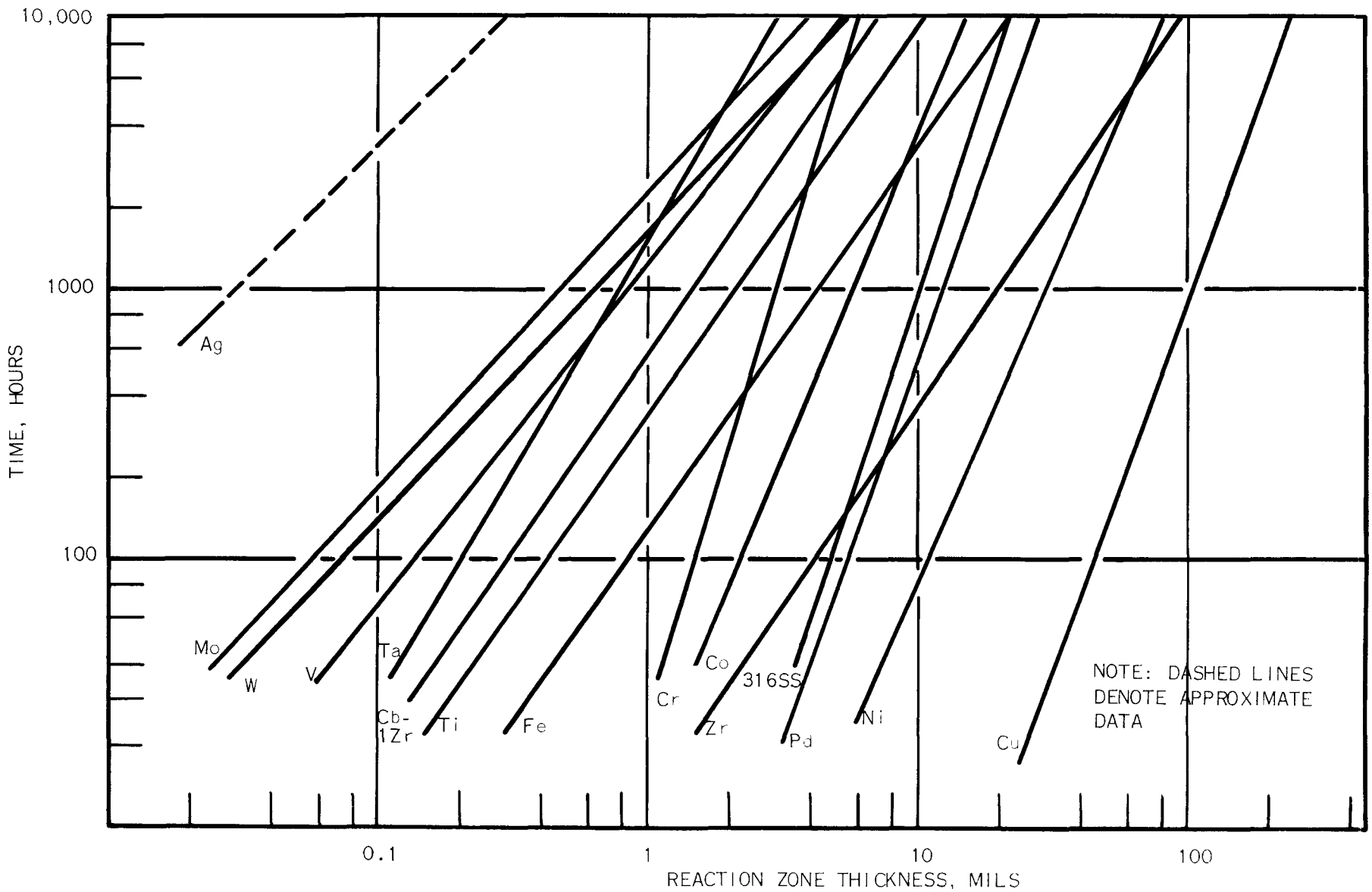
REACTION ZONE THICKNESS VERSUS TIME FOR BERYLLIUM COUPLES TESTED IN VACUUM AT 540C



REACTION ZONE THICKNESS VERSUS TIME FOR BERYLLIUM COUPLES TESTED IN VACUUM AT 650C



REACTION ZONE THICKNESS VERSUS TIME FOR BERYLLIUM COUPLES TESTED IN VACUUM AT 760C



NOTE: DASHED LINES
DENOTE APPROXIMATE
DATA

Joining Development

Silver Alloys: Preliminary X-ray diffraction analysis was completed on beryllium/Cb-1 Zr and beryllium/316 stainless steel brazed specimens. These were brazed at 890C using 4-mil silver foil and subsequently aged 800 hours at 760C. As reported in PWAC-1018, the specimens separated during heat treatment and post-test metallographic examination showed high hardness values on the columbium and stainless sides of the joint. Diffraction patterns showed that the high hardness regions were associated with columbium and iron beryllides. The presence of a γ phase (Ag/Be) near the fracture surfaces is also suspected, although definite identification has not been established. On the basis of these findings, the failure of silver-brazed joints during heating at 760C for 800 hours is presumed to be associated with the formation of γ phase or the formation of columbium or iron beryllides or a combination of both. It is obvious that silver diffusion bonded joints between beryllium and columbium or 316 stainless steel will be useful only for lower temperature service (to avoid brittle phase formation). For higher temperature applications, the brittle phase reactions must be inhibited by alloying or by the use of barriers to prevent reaction of the columbium or stainless with the beryllium by diffusing thru the silver interface.

Specimens of Cb-1 Zr/beryllium and 316 stainless steel/beryllium brazed joints made with various silver base alloys and diffusion bonded joints made with silver foil were heated for 800 hours at 760C in vacuum. None of the specimens was completely satisfactory. The samples brazed with alloys of Ag-28 Cu, Ag-15 Mn and Ti-42.5 Mn all separated with failure at the beryllium/braze alloy interface. Severe voiding was noted in the beryllium in all cases and reaction layers were observed on the Cb-1 Zr and stainless sides of the joints. These reactions were similar to those shown in Figs. 37 and 38 of PWAC-1018 for silver joints subjected to the same heat treat cycle. The specimens brazed with Ag-10 Pd and Ag-7 Cu-0.3 Li did not come apart during the heat treatment cycle, but the beryllium side showed numerous voids and the stainless (or columbium) interface showed considerable second phase. Similar joints made with pure silver, reported in PWAC-1018 and subjected to the same treatments, all failed. The fact that the Ag-10 Pd and Ag-7 Cu-0.3 Li appeared to moderate the rate of reaction and eliminate failure suggests the possibility of further alloying improvement.

Cb-1 Zr/beryllium and 316 stainless steel/beryllium compatibility specimens joined by diffusion bonding with silver at 760C and tested in vacuum 800 hours at 760C were destroyed during test due to a vacuum leak. Replacement specimens were prepared and have accumulated 144 hours at 760C.

Compatibility tests of beryllium/Cb-1 Zr specimens joined with pure silver by brazing at 890C (3 minutes) and by diffusion bonding at 760C (8 hours) are being evaluated at temperatures below 760C. Tests scheduled for 800 hours in vacuum at 650C and 705C have accumulated 144 hours. In addition to these tests, similar specimens brazed with the following silver base alloys are also being evaluated under the same test conditions:

<u>Alloy</u>	<u>Braze Temp., C</u>	<u>Braze Time, min.</u>
Ag-4.5 Si	825	3
Ag-15 Mn	915	5
Ag-10 Pd	900	3
Ag-28 Cu	785	2
Ag-7 Cu-0.3 Li	870	1.5

Barrier Evaluation: An investigation was started to evaluate barriers to prevent reactions between beryllium and silver or other braze alloys. The following materials were blended with magnesium powder and cold-sprayed onto beryllium specimens: vanadium, chromium, tungsten, molybdenum, silicon, and carbon. After spraying, the specimens were heated for 16 hours at 1080C in a flowing argon atmosphere to melt and "boil off" the magnesium and thereby leave a coating of the desired material. The silicon slurry resulted in a visually continuous coating with concomitant weight gain. This coating was file hard and strongly adherent. None of the other metal-magnesium combinations produced a satisfactory coating or significant weight gain.

Silicide and carbide coatings were successfully applied to Cb-1 Zr by a cold spray and gaseous process, respectively. Test specimens of silicon-coated beryllium and silicon and carbon-coated Cb-1 Zr were brazed using silver and silver base alloys. Specimens listed below are in test at 650C, 705C and 760C and should show whether or not these coatings will prevent failure or reduce formation of beryllides in the joints.

<u>Specimen</u>	<u>Braze Alloy</u>	<u>Braze Temp., C</u>	<u>Braze Time, min.</u>
Silicon-coated Be/Cb-1 Zr	Ag-4.5 Si	825	3
Silicon-coated Be/Silicon-coated Cb-1 Zr	Ag-4.5 Si	825	3
Be/Silicon-coated Cb-1 Zr	Ag	837	5
	Ag-4.5 Si	825	3
	Ag-7 Cu-0.3 Li	845	5
Be/Carbon-coated Cb-1 Zr	Ag-4.5 Si	825	2
	Ag-7 Cu-0.3 Li	845	5
	Ag-7 Cu-0.3 Li	870	1.5

Specimens have accumulated 144 hours at 650C, 705C and 760C.

Diffusion Bonding Evaluation: In addition to silver and silver base alloys, other materials were investigated for joining beryllium to stainless steel and columbium alloys. Specimens of beryllium/Cb-1 Zr and beryllium/type 316 brazed with a Ti-42.5 Mn alloy failed during the 800 hours/760C compatibility test. Excepting silver, this was the only material which would braze beryllium to the structural alloys. Diffusion bonding, however, was accomplished with several materials in addition to silver, as shown in Fig. 37. Bonding was generally sporadic and not satisfactory. Therefore, tests were initiated to develop improved materials for diffusion bonding beryllium. The basis for selection of these materials was provided by the compatibility test results; bonding combined with minimum interfacial reactions was considered as a reasonable screening criterion. Materials selected for the initial trials and results are summarized in Fig. 42. All specimens were held in intimate contact for four hours in vacuum with the same holder used in the compatibility tests (Fig. 27, PWAC-1016). As shown in Fig. 42, uranium bonded to beryllium at all temperatures; vanadium bonded at 870C and 980C but not at 760C or 1090C; copper bonded at 760C but not at 870C or 980C. None of the other materials produced bonds with beryllium. Metallographic examination of the beryllium/uranium joint showed a reaction zone at the uranium interface in all specimens. At 760C, this layer was 0.3-mil thick and, in addition, voiding was noted in the beryllium. At 870C, the layer was 0.6 mil and a marked increase of porosity in the beryllium was nearly continuous to a depth of 3 mils. Compatibility tests are scheduled between uranium and beryllium for 800 hours at 540C, 650C, and 760C to determine reaction rates. Metallographic examination of the beryllium/copper and beryllium/vanadium specimens showed ruptures in the reaction zone of all couples. It has thus become increasingly clear that silver is the only metal which bonds satisfactorily with beryllium. Other metals, such as uranium and copper bond with beryllium, but reaction rates are too rapid to allow high service temperatures.

Future Work

1. Continue 10,000-hour compatibility tests.
2. Continue evaluation of silver brazed and diffusion bonded joints for use below 760C.
3. Evaluate coatings on beryllium and Cb-1 Zr for use with silver-brazed specimens.
4. Continue evaluation of copper and uranium as materials for diffusion bonding.

DIFFUSION BONDING TESTS BETWEEN BERYLLIUM AND VARIOUS MATERIALS

(SPECIMENS HELD 4 HOURS IN VACUUM AT TEMPERATURES SHOWN)

<u>Beryllium Vs.</u>	<u>Diffusion Bonding Temperature</u>			
	<u>760C</u>	<u>870C</u>	<u>980C</u>	<u>1090C</u>
Cb-1 Zr	-	No Bond	No Bond	No Bond
Co	-	No Bond	No Bond	No Bond
Cr	-	No Bond	No Bond	No Bond
Cu	Bonded	No Bond	No Bond	-
Fe	-	No Bond	No Bond	No Bond
Mo	-	No Bond	No Bond	No Bond
Ta	-	No Bond	No Bond	No Bond
Ti	-	No Bond	No Bond	No Bond
U	Bonded	Bonded	Bonded	-
V	No Bond	Bonded*	Bonded*	No Bond
W	-	No Bond	No Bond	No Bond

*Considerable cracking observed in reaction zone.