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**DEVELOPMENT OF
A BRAZING PROCESS
FOR SEAL BLOCKS**

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INTRODUCTION

In order to prevent hydrogen leakage from within the NRX-A1 reactor pressure vessel, each of the 343 instrument leads must be bonded into one of ten cylindrical plugs. These plugs (or seal blocks) are then sealed by mechanical seals into circular openings in the pressure vessel.

This report covers the development and qualification of a brazing process capable of forming the bond between the instrument leads and the seal block. The process developed is unusual in that it permits the formation of a bond between austenitic stainless steel surfaces at temperatures below 1350°F without a flux. The requirements for any brazing process to be used in joining the instrumentation tubes to the seal block were:

1. The brazing process must not exceed a temperature of 1375°F. This temperature was considered to be the maximum that could be encountered without affecting the calibration of the instrumentation leads. This temperature is too low to permit the use of a reducing atmosphere, such as hydrogen.
2. The use of corrosive brazing fluxes was not permitted. The fluxes normally used in brazing operations contained fluoride compounds which might become trapped in the joint and which are difficult to remove after brazing even with hot water. The use of large amounts of water during reactor assembly would not be an acceptable practice.
3. The finish of the surface of that portion of the seal block where the mechanical seal seats must not be changed by the brazing process.

4. The sealing operation had to be accomplished on the reactor itself after the instrumentation was installed so that it was not possible to use a conventional brazing furnace.

The requirements, 1 and 2 above, narrowed the choice of brazing filler materials to one alloy (ASTM B 260 or B Cu P-S), namely 80% copper, 15% silver and 5% phosphorous (trade name Sil-Fos). This alloy is designed to braze copper to copper (and to some noble metals) without the addition of fluxes. The brazing flow temperature is 1300°F, 75° below the permissible maximum (requirement 1). However, the Sil-Fos braze alloy will not effectively wet the ferrous alloys and particularly the chrome bearing stainless steels. It was, therefore, necessary to develop a surface on the stainless components that would be compatible with the braze alloy. This requirement was met by the addition of a copper inlay to the seal block and by gold plating the surface of the instrumentation leads.

Requirements 3 and 4 were met by the use of an argon-gas-filled quartz chamber. Requirement 4 also required the application of high frequency induction heating (450KC). The seal block assembly was not heated directly by the induction coil. A graphite susceptor surrounding the seal block was heated inductively which in turn heated the seal block. This minimized wide temperature gradients in the seal block caused by skin effect of the high frequency current. To further protect the seal block from oxidation, a titanium "getter" tube surrounded the seal block. Figure 1 is a cutaway view of the induction brazing furnace. Figure 2 is a photograph of an "as-brazed" test sample. (909E375 GR04).

In the prosecution of the development effort, the several separate processes that are required to produce the sealing bond were evaluated. The processes, which are described in subsequent portions of this report are:

1. Copper inlay brazing
2. Plating of the surfaces of the leads
3. Assembly of the seal block and placement of the 80% Cu-15% Ag-5% P
Braze Alloy

4. Establishment of the gap between surfaces being joined
5. Cleaning
6. Brazing process qualification and pre brazing trials

EVALUATION OF COPPER INLAYING PROCEDURES

Since plating the internal surface of each of the holes in the seal block was not practical, a wettable surface was developed on the seal block by inlaying a OFHC*type copper disc into the upper surface prior to drilling the holes.

Originally the copper inlay was brazed to the seal block with a low temperature alloy using flux. However, the quality of braze necessary for this application was not obtained by this procedure due to voids in the braze. During subsequent drilling of the instrumentation lead holes, the drill would break through a void producing a trap for contamination from drilling fluids. Even ultrasonic cleaning would not remove the contamination. During brazing the contaminating material would be driven out of the void and would degrade the braze joint of the instrument tube.

Copper inlay braze feasibility samples were then run in a vacuum furnace using an 82% gold - 18% nickel alloy (Premabraz 130). These samples when subsequently drilled and brazed to the instrument tubes. Elimination of the flux from the joint reduced contamination problems. WANL process specification 294543-8 was written to cover this operation. Subsequently a fluxless, commercial brazing operation was qualified to carry out the inlay operation on the seal blocks used for qualification and for assembly of the first reactor.

*"Oxygen Free-High Conductivity"

EVALUATION OF PLATING SYSTEMS FOR INSTRUMENTATION LEADS

Copper, silver and gold plating respectively were evaluated in a series of brazing tests as a means for producing a wettable surface on the instrument leads.

Copper Plating:

Copper plated instrumentation tubes were used in initial trials because of the low material cost and availability. A plating thickness of .002" proved most effective with a clearance of .004". However, bond integrity between copper and the tube was not reliable. Further, there was a tendency for the copper to outgas and produce voids. Finally there was metallographic evidence that the braze alloy was penetrating the relatively thin copper plate and destroying the bond between the copper and the stainless. This action produced a considerable number of voids in the joint (Figure 3). Tests on the copper plated samples, however, did prove the feasibility of plating as a means for producing a wettable surface on the instrumentation leads.

Silver Plating:

In the effort to improve the integrity of the brazed joint, a series of tests was run with silver plated instrumentation tubes. These tests were encouraging but there remained inconsistencies in the process. Joints exhibited voids and the fillets formed were small and irregular in size. The voids (Figure 4) can be attributed in a large degree to oxidation between the stainless steel base metal and the silver plating during the heating cycle. Penetration of the silver by the braze alloy was not as severe as that observed with copper or gold.

Gold Plating:

A third series of tests was run on gold plated instrumentation tubes. The tests indicated a bond integrity of 95% or better. Filleting of the braze was smooth and traveled up the gold plated instrumentation tube 1/32" higher than either the copper or silver plated tubes. The

brazing alloy showed greater solubility in the gold plate than the copper or silver. There is no evidence that this penetration affected the bond as it did with silver and copper. The ability of the gold to resist oxidation during assembly and heating is believed responsible for the superior results obtained. The as-brazed appearance of the joint was brighter and cleaner than either the copper or silver plated samples (Figures 5 and 6). A test sample containing 73 leads was brazed using leads that were gold plated (.002" thick) and clearances of .004" (.008" diametrically). This sample was pressure tested with helium at 1000 psi at room temperature and also at -325°F. No leaks were detected using a helium leak detector set at 2×10^{-9} cc/sec. WANL specification 294543-4 was prepared to document the process established by the above tests.

EVALUATION OF BRAZE ALLOY PLACEMENT PROCEDURES

Work done prior to and for purposes of developing WANL-294543-4 included an evaluation of brazing alloy quantity and placement as well as desired joint clearances.

At the outset, wire (.032" diameter) was used which was cut into a ring-shaped preform having an I.D. equal to the O.D. of the instrumentation lead being brazed. These rings were placed at each joint area prior to brazing. Experimentation indicated two rings at each joint area was the most desirable number regardless of instrumentation diameter as long as the gap between the instrument tube and the copper remained .004". Later experiences with gaps up to .010" indicated the need for at least one extra ring. Another means of preplacing the brazing alloy involved .040" brazing alloy sheet. This sheet was machined to the proper diameter and holes were drilled in it to accommodate the instrumentation leads.

The preplaced rings produced good fillets and ample joint fill. It was felt that the drilled sheet method of alloy placement might exhibit greater potential as an assembly method during production brazing. However, the shim did not consistently braze the outer tubes because the edge of the shim melted before the tubes were up to brazing temperature, due to a high frequency skin effect from the induction coil.

ESTABLISHMENT OF CLEARANCES

In the original development of the process (i.e., when the various methods for inlaying, plating and alloy placement were under investigation) the clearance between the plated surface and the I.D. of the hole in the copper was evaluated in the range of .002" to .009" for an .042" diameter lead. A gap of .004" appeared to be the most desirable and was used during subsequent development, proof testing and qualification.

Recognizing that the plugs had to be slipped over 30' of leads, supplementary tests were run to determine that a metallographically sound joint would be produced with a gap of .008", and drawing clearances were established between .004" and .008" using standard drill sizes.

When seal blocks were produced for production brazing and pre-production tests, the complex hole patterns were produced by the vendor using a drilling fixture. The fixture was such that the holes were drilled through the copper inlay first then the stainless steel. Because it was known the stainless steel chips passing out through the copper inlay area would scar the softer copper surface, a reaming operation was added. Reaming was confined to the copper portion of the hole which resulted in gap sizes in excess of the drawing dimensions as follows:

BRAZING GAP

Diameter of Lead (In Inches)*	Gap as Specified on the Drawing (In Inches)	Gap in Production Parts After Reaming (In Inches)
.040-.045	.008	.011*
.060-.065	.004	.007*
.085-.090	.007	.011*
.125-.130	.005	.009*

* Average values based on actual measurement of parts.

Because joint widths as great as .011 inches were to be encountered in production brazing, several tests were run on actual seal blocks with these larger gaps. The following test assemblies were produced and evaluated as noted below:

Drawing Number	Number of Brazes Per Unit by Diameter				Pressure Test*		Metallographic
	.055	.073	.1015	.140	Room Temp. (psi)	Liq. N ₂ (psi)	
909E375 Gr 02 (TP-6)		24	9			--	Yes
909E375 Gr 03 (TP-7)	4	19	2		100+1000 OK	--	No
909E375 Gr 05 (TP-8)		2	9	6	600 OK	--	No
909E375 Gr 09 (TP-9)	3		24		100+1000 OK	--	No
909E375 Gr 08 (TP-10)	3		21		600 OK	--	No
909E375 Gr 06 (TP-11)		1	6	9	100+1000 OK	1000 OK	No
909E375 Gr 04 (TP-12)	5	26	3		100+1000 OK	--	No
909E375 Gr 06 (TP-13)		1	6	9	100+1000 OK	--	No
909E375 Gr 07 (TP-17)**	6	32	1		700 OK	--	No

* Leak testing was accomplished using a CEC Leak Detector set at 2×10^{-9} cc/sec. Sensitivity.

** TP-17 contained six (6) copper plugs inserted into three .073", ten .055" and one .1015" diameter hole for purposes of evaluating the feasibility of sealing off seal block holes which would not require instrumentation.

The joints with the larger clearances were strong enough and also appeared to exhibit better penetration of the braze through the copper than had the qualification tests. This latter observation suggests that regardless of the method of drilling, the copper hole surfaces in all previous tests had been subjected to some damage from stainless steel chips. Apparently, the drilling practices encountered on the production parts simply increased the degree of damage until it became obvious on visual inspection (usually in the form of a smeared blue film on the I.D. of the copper portion of the hole).

OBSERVATIONS REGARDING CLEANING PRACTICES

Because of the complex array of small holes, ultrasonic cleaning in methyl-ethyl-ketone or in acetone was used on all seal plugs. The braze alloy rings were lightly etched by immersion in a proprietary dilute phosphoric acid solution with one of the higher alcohols as an inhibitor ("Metal Conditioner" which in turn is diluted to form "Metal Preparation Number 10"). This etching treatment removed tarnish and appeared to improve flow during the early stages of melting. The copper surface of each hole was wiped (using a pipe cleaner) with the same solution to remove tarnish. Immediately prior to assembly all surfaces were rinsed with alcohol to remove phosphate and chloride residues. Final rinsing with acetone was not effective. Parts were handled with clean cotton gloves and all tools were cleaned in alcohol. It is important to realize that all cleaning must be accomplished prior to assembly since no effective washing or rinsing is possible once the plug is in place on the reactor assembly. (The leads, however, were frequently wiped with alcohol soaked rags during assembly.)

TEST RESULTS

Once a clearance of .004", a gold plated surface, and a fluxless inlay process had been established through the brazing and macro sectioning of test plugs (with a simplified hole pattern), two plugs were produced for hydrostatic testing by the Mechanics Testing Laboratory*. These plugs represented an earlier design**, one contained 30 holes for .125 diameter pressure tubes while the other contained 73 holes for .062 diameter thermocouples.

The block containing 30 holes was assembled using gold plated tubes plated by a proprietary process involving a manually manipulated brush***. This part was a failure as far as braze joint is concerned. The gold plate - stainless interface became unbonded when brazing heat was applied, permitting numerous leaks along this surface. The plate also was severely spalled. As a result this process was dropped.

* Work Request 41:41:06:3W

** Item 15 of 936J429

***This process and its application is described in TME-628

The second block (73 holes) was brazed using leads gold plated by the gold cyanide process* carried out in tanks where plating conditions could be controlled. To assure adherence, this process involved both a nickel and gold strike. Visually this block, after brazing, was sound and passed a 1000 lbs pressure (helium) test at both room temperature and at -325°F . A mass spectrometer failed to detect any leaks. Figure 7 shows the testing apparatus.

QUALIFICATION TESTS

Using the furnace shown in Figure 1, a series of trials on simplified plugs suggested the following detailed procedures for each of the two major styles of seal block:

Date	Qualification Number	Type	<u>Machine Settings</u>		Argon (psi - Gage)
			Current	Time	
10/12/63	1	978D108 Gr 03	135A	10 min.	6 lbs.
10/13/63	2	978D108 Gr 03	135A	10 min.	6 lbs.
10/13/63	3	909E375 Gr 03	135A	12 min.	10 lbs.
10/13/63	4	909E375 Gr 03	135A	14 min.	10 lbs.
10/16/63	5	909E375 Gr 03	135A	14 min.	10 lbs.
10/16/63	6	909E375 Gr 03	135A	14 min.	10 lbs.
10/16/63	7	978D108 Gr 03	135A	10 min.	10 lbs.
10/16/63	8	978D108 Gr 03	135A	12 min.	10 lbs.

*This process and its application is described in TME-628

ND 101 Production Brazing Parameters

Date	Detail	Type	Machine Settings		Argon (psi - Gage)
			Current	Time	
10/20/63	--	909E375 Gr 02	135A	11 min.	10 lbs.
11/ 8/63	H	909E375 Gr 04	140A	14 min.	10 lbs.
11/ 8/63	K	909E375 Gr 04	130A	*12 min.	10 lbs.
11/ 8/63	L	909E375 Gr 04	135A	*18 min.	10 lbs.
11/ 9/63	--	909E375 (copper shim and plug)	--	16 min.	10 lbs.
11/ 9/63	AD	978E375	130A	11 min.	10 lbs.
11/ 9/63	B	909E375	120A	16 min.	10 lbs.
11/10/63	D	909E375	--	11 min.	10 lbs.
11/10/63	E	909E375	130A	*28 min.	10 lbs.
11/10/63	AE	978D108	130A	17 min.	10 lbs.

* Rebraze

Both styles were heated by the same induction coil. The 978D108 style required a 16% increase in heating efficiency because of its greater mass and, consequently, improved R. F. loading.

The Gr 03 configuration was selected for qualification because it represented the greatest mix of lead diameters in one seal block (.042, .063, and .098). Figure 8 illustrates the two configurations and the hole pattern. Two crews were qualified. Each crew consisted of a brazing operator and a helper. The qualification required that each crew successfully braze two specimens of each of the two styles (four pieces altogether) without interruption. Evaluation consisted of a pressure test, fillet examination and microscopic examination of several representative joints. The results of these examinations are listed in the following table.

Plug Number & Identification	Pressure Test Conditions with Helium (psi)	Remarks
1 - 978D108 Gr 03	700 @ Room Temperature	O.K. 10/14/63
2 - 978D108 Gr 03	700 @ Room Temperature	O.K. 10/14/63
	700 @ Cryogenic (-196°C)	
3 - 909E375 Gr 03	700 @ Room Temperature	O.K. 10/14/63
4 - 909E375 Gr 03	700 @ Room Temperature	O.K. 10/14/63
5 - 909E375 Gr 03	100 @ Room Temperature	O.K. 10/24/63
6 - 909E375 Gr 03	100 @ Room Temperature	O.K. 10/24/63
7 - 978D108 Gr 03	100 @ Room Temperature	*O.K. 10/18/63
8 - 978D108 Gr 03	100 @ Room Temperature	O.K. 10/18/63

* The leak in this plug was not due to the brazing technique. It was caused by a thermocouple piece which did not contain plating for the full length, the part apparently slipping prior to brazing and in doing so the joint area was exposed to the non-plated section. The same condition could not occur in a production braze.

The procedures used to braze the above are documented in Test Book K 17124, pages 1712400 through 1712443 inclusive.

PREPRODUCTION BRAZING TRIALS

Using the qualified procedures, equipment and personnel, a series of trials was run on other hole patterns. These trials were initiated prior to the start of production brazing but those tests marked below were run during production just prior to the brazing of the corresponding style and configuration on the reactor.

Plug Number and Identification	<u>Pressure Test Conditions-Results</u>		Metallographic Examination
	Room Temp. (psi)	Cryogenic(N ₂) (psi)	
TP- 1 909E375 Gr 02	100 OK	Not tested	No
TP- 2 909E375 Gr 02	100 OK	Not tested	No
TP- 3 909E375 Gr 02	100 OK	Not tested	No
TP- 4 909E375 Gr 04	100 OK	Not tested	No
TP- 5 909E375 Gr 04	100 OK	Not tested	No
TP- 6 909E375 Gr 02	Not tested	Not tested	Microscopic exami- nation of reamed holes to verify large clearance OK
TP- 7 909E375 Gr 03	100+1000 OK	Not tested	No
TP- 8 909E375 Gr 05	600 OK	Not tested	No
TP- 9 909E375 Gr 09	100+1000 OK	Not tested	No
TP-10 909E375 Gr 08	600 OK	Not tested	No
TP-11 909E375 Gr 06	100+1000 OK	1000	No
TP-12 909E375 Gr 04	100+1000 OK	Not tested	No
TP-13 909E375 Gr 06	100+1000 OK	Not tested	No
TP-17 909E375 Gr 07	700 OK	Not tested	No

CONCLUSIONS

1. A method of brazing instrumentation leads to seal blocks meeting all the engineering requirements has been developed and qualified for application to NRX-A1.
2. 1076 brazed joints, which include qualification, pre-production and production pieces were made with only 5 failing to meet leak test requirements.
3. A gold plate is superior to a silver or copper plate in that the gold-stainless bond can better withstand the temperature of brazing and because the braze alloys will flow or wet better against a gold plate surface.
4. It is essential to use fluxless brazing methods capable of producing a uniform bond with minimum porosity when brazing the copper inlay into the stainless steel seal blocks.
5. Handling during assembly must be such that no dirt, grease or chips are introduced into the joint since very little cleaning can be accomplished after threading the seal block over the instrumentation tubes.

ACKNOWLEDGMENT

The assistance and cooperation given by Messrs. J. Kehoe, A. Dixon, H. E. Ricks, and their people of the R & D Center has been most helpful in completing this program.

FIGURE 1

Induction Furnace for Brazing Gold Plated Instrument
Leads to Seal Plugs

Approx. 1 X

FIGURE 2
AS-BRAZED TEST SAMPLE

This specimen shows the bright surface that existed on the braze fillets and copper inlay after brazing. Note freedom from discoloration on the cylindrical surfaces of the seal block.

Unetched

10X

FIGURE 3

80 Cu-15 Ag-5 P BRAZED COPPER PLATED LEADS

Filleting appears to be better than on silver plated leads. Note severe void in inlay braze.

Unetched

10X

FIGURE 4

80Cu-15Ag-5P BRAZED SILVER PLATED LEADS

Poor filleting can be observed.

Unetched

100X

FIGURE 5

80Cu-15Ag-5P BRAZED GOLD PLATED LEAD

The .004 inch gold layer has been penetrated until only about .001 inch remains.

Unetched

100 X

FIGURE 6

80 Cu-15 Ag-5 P FILLET ON GOLD PLATED LEAD

Inlet from source of
pressurized (2000 psi
maximum) helium

Fixture with specimen
extending through cover

FIGURE 7

PRESSURE TEST FIXTURE

Gauge shown was used only for 100 psi tests. Fixture (less gauge) was immersed in container of liquid N_2 for low temperature tests. Insert shows assembly of test specimen in container. Mass spectrometer probe inserted into bundle of leads.

GR-03 HOLE PATTERN

909 E 375

GR-03 HOLE PATTERN

978 D 108

FIGURE 8

SEAL BLOCK AND QUALIFICATION HOLE PATTERN

596491A