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Westinghouse Astronuclear Laboratory

THE RELATIONSHIP BETWEEN BRAZING PROCESS
VARIABLES AND GEM-MOLYBDENUM JOINT
QUALITY FOR A CHROMIUM-NICKEL-GOLD

BRAZE ALLOY (u)

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THE RELATIONSHIP BETWEEN BRAZING PROCESS
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QUALITY FOR A CHROMIUM-NICKEL-GOLD
BRAZE ALLOY

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SPECIAL REVIEW	REVIEWER	Class.	Date
DETERMINATION	JRP	u	4-28-82
Class: u			

Classification cancelled (or changed to _____)

by authority of _____

by H. F. C. TIC, date SEP 12 1973

~~GROUP 1~~

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~~INFORMATION CATEGORY~~

~~CLASSIFIED (CRD)~~

E. L. Layland 3/17/69
Authorized Classifier Date

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ABSTRACT

A series of NbC (GEM)-Molybdenum-AuCr Ni braze alloy assemblies was produced to simulate a proposed brazed orifice configuration. The effect of brazing temperature, brazing time and the gap (between the coated channel and the orifice) on braze quality was observed. Gap had a major effect. Temperature had less effect if it was controlled within a reasonable range. Time had no effect except when coupled with marginal temperatures. Secondary variables such as the composition of the Molybdenum braze alloy form, and orifice shape were also evaluated qualitatively.

Threaded orifices were not included in this study. Specimens were produced from unfueled graphite only.

TABLE OF CONTENTS

	<u>Page No.</u>
I. INTRODUCTION	1
II. MATERIALS	2
III. ESTABLISHING BRAZING CLEARANCE (GAP)	3
IV. PREPARATION OF SPECIMENS	3
V. EQUIPMENT	5
VI. TEMPERATURE MEASUREMENT	5
VII. TABLE	6
VIII. ANALYTICAL PROCEDURES	7
IX. BRAZING PROCEDURES	8
X. EXPERIMENTAL RESULTS	9
XI. DISCUSSION OF RESULTS	11
XII. CONCLUSIONS	13
XIII. FUTURE ACTIVITY	14

ILLUSTRATIONS

- FIGURE 1   ORIFICE AS ORIGINALLY DESIGNED
- FIGURE 2   ORIFICE AS RE-DESIGNED FOR SPIRAL PREFORM
- FIGURE 3   BRAZING APPARATUS
- FIGURE 4   LEAK DETECTION APPARATUS
- FIGURE 5   TYPICAL BRAZED JOINTS (2100°F-1 MINUTE-0.001" TO 0.004" DIAMETRAL GAP)
- FIGURE 6   UNMELTED JOINTS AT 1850°F (30 SECONDS)
- FIGURE 7   BRAZE WITH OPEN CHANNEL
- FIGURE 8   BRAZE WITH MARGINAL FILL
- FIGURE 9   RESPONSE SURFACE FOR INDUCTION HEATED GRAPHITE-CRONIRO-MO
BRAZED JOINTS

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-1-

INTRODUCTION

(CRD) Present NERVA reactor design requires the brazing of the metal orifices into graphite fuel elements. The problem is one of mismatched coefficients of expansion. Graphite expansion is typically $2.5 - 3.0 \times 10^{-6}$ in/in/° F. Metals, such as the austenitic stainless steel presently used in orifices, commonly exhibit expansivities of 12×10^{-6} in/in/° F. At low brazing temperatures, martensitic stainless steel matches graphite fairly well, and because of the low temperature, the total interference is not severe. Thus, an initial process was successfully developed using a low temperature silver-copper braze alloy and type 416 stainless steel. This process had to be abandoned when the GEM type of fuel element coating process was substituted for the GED process. The silver braze would not wet the GEM coating. An earlier candidate braze alloy, (chromium, nickel, gold) was reconsidered. It has a brazing temperature 300° - 400° F higher than the silver-copper brazing alloy. Under these conditions, the expansion dissimilarity between 416 stainless steel and graphite became too great. Therefore, molybdenum had to be substituted as the orifice material.

(CRD) In this study, a series of NbC (GEM)-Molybdenum Orifice-AuCrNi braze alloy assemblies was produced, leak tested, and then sectioned for metallographic evaluation in order to determine the interaction between the independent process variables of brazing temperature, time at brazing temperature, and gap between the wall of the coated channel and the cylindrical surfaces of the orifice. The interaction was established in terms of defective brazes - a dependent variable. The results of the interaction were displayed graphically as a response surface. A response surface is useful in determining, visually in this instance, the sensitivity of the dependent variable to changes in the independent variables.

(CRD) The study did not cover threaded orifices. Threaded orifices involve a direct braze to uranium-graphite and might respond differently than either a GED-to-metal or GEM-to-metal systems such as those described in this report.

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Materials

(U) The brazing was accomplished using a gold-nickel brazing alloy with 6 % chromium added. The chromium forms carbides with any free graphite that might be present on either surface of a joint. The pertinent metallurgical data is:

Composition - 72% gold (Au)
22% nickel (Ni)
6% chromium (Cr)

Brazing temp. 1938 - 2050° F

(CRD) The channels in these tests were coated by the GEM process, which appears to produce some form of free carbon as an integral part of the coating. At least, braze alloys, which do not contain carbide forming elements, will not wet the GEM coating, according to preliminary tests. The Au-Ni-Cr braze alloy was selected because of the possible presence of free carbon in the joint and because of its affinity for molybdenum. The graphite substrate was PDS 30022 material with an expansion coefficient of 2.65×10^{-6} in/in/° F.

(U) Molybdenum was selected as the orifice material because the carbide - forming braze alloy dictated a high brazing temperature. At such temperatures only molybdenum, with a coefficient of expansion of 3.0×10^{-6} in/in/° F, is sufficiently matched to the expansivity of graphites to avoid joint breakage upon cooling. Early tests had indicated that type 416 martensitic stainless steel (6.3×10^{-6} in/in/° F) was sufficiently matched if the brazing temperature did not exceed about 1600° F. Carbide formers are not very active at these lower temperatures, however. Thus, the selection of molybdenum was made to accommodate the high-temperature, carbide-forming brazing process.

(U) In certain test specimens, commercially pure (99.98%) molybdenum and a recrystallized molybdenum alloy (0.25% Zr and 0.25% Ti-TZM) were used in alternating orifices to evaluate the brazeability of both forms. This test was conducted because molybdenum is a difficult material from which to machine orifices and it was felt that one or the other might prove more machinable in the manufacture of the small orifices. Most of the tests involved recrystallized TZM alloy material.

(U) The braze alloy was available as 0.005" thick sheet and 150 mesh powder. In most of the tests the sheet form was used. In order to form the sheet into preforms for assembly with the orifice, it was cut into strips approximately 0.100" wide.

Establishing Brazing Clearance (GAP)

CRD) Irregularities in channel coatings prevented the establishment of the brazing clearance over a range of discrete measurements, (i.e in increments of 0.001 inch.) Instead, the specimens were grouped in a semi-quantitative manner as follows:

0.000 - 0.001 inch clearance - Very tight, required an unusually smooth, round channel.

0.001 - 0.004 inch clearance - typical tight fit condition.

0.004 - 0.008 inch clearance - Loose fit, usually achieved by etching stock from the orifice.

A few specimens were found (after brazing) to exhibit clearances above 0.008 inch so that another category (0.008" and over) had to be established.

(U) Etching (0.008 and over) caused the O.D. of the orifice to become tapered. The reported clearance is the dimension at the mid-length of the tapered section.

(U) All reported dimensions refer to the diameter.

Preparation of Specimens

(U) Orifices and braze strip (0.005" x 0.100") were cleaned in acetone.

Orifices were also chemically milled in a solution of

Milling: Lactic acid 20 parts

HNO₃ 20 parts

HF 10 parts

Post-Milling Bright Dip: KFeCn 10 gm

KOH 10 gm

H₂O 100 ml

for differing lengths of time to obtain varying degrees of diametral clearance between the orifice and the channel. All orifices were given a light etch in the above

solution to assure that the braze alloy would be exposed to uniform surface conditions during the wetting process.

(U) Assembly of the orifice and preformed braze alloy into the small diameter channel proved difficult. At first the alloy was formed in a "C" ring, and expanded mechanically and allowed to snap into a groove midway up the brazing shank of the orifice (Figure 1). Forming to the tolerances required to fill the groove and yet not overhang the diameter of the tight fitting orifice created severe problems. Two commercial producers of a preformed alloy recommended against such an approach. A cement-powder braze application technique was substituted on a tentative basis in an attempt to retain the center groove (Figure 1) configuration. The Figure 1 configuration appeared to assure better centerline alignment between the channel and the orifice.

(U) A novel approach to preplacement was then developed. First the center groove was extended to the inner end of the brazing shank as shown in Figure 2. Then the braze alloy strip was wound in a spiral around a mandrel. The mandrel was approximately 0.005" larger than the brazing shank. The brazing shank of the orifice was easily inserted in the spiral and the spiral cut to match the length of the brazing shank. Typically this left 2-1/2 turns (about 0.0124 ounce) of braze alloy on the shank. The cutting operation caused the cut end to twist outward. This cut end was then carefully introduced into the channel. The assembly was then turned into the channel much as a screw is turned into a thread but in the opposite direction. By counter rotating the spiral preplacement (and keeping the upper end of the spiral from rotating), the spiral pulled tight on the shank of orifice, and because it then had a smaller diameter, slipped readily into the channel. Once the turning action stops, the spiral tries to return to its original diameter. This brings it into close contact with the channel wall. This spring-out effect prevents the spiral of braze alloy from slipping further into the channel. In fact, it makes it virtually impossible to move inside the rough channel - a special tool is required if it must be

moved. Spring-out also enhances heat transfer from the graphite to the braze alloy which in turn melts and bridges the gap to the orifice to complete the heating cycle efficiently.

(U) Using the spiral method, orifices were assembled into the element in a pattern that involved two intersecting diagonal rows of orifices. The common center orifice was then completely surrounded with orifices to represent an unthreaded orifice under maximum restraint during cooling. The as-assembled clearance between the orifice and the coated wall of the channel was measured and recorded for each orifice in the intersecting rows of orifices.

Equipment

(U) After assembly, the brazement was placed in a Vycor retort inside the coil of a 10 KW induction heater (Figure 3). The system was evacuated to approximately 40 microns and refilled with welding grade argon gas to a positive pressure of 1-3 psig. This purge cycle was repeated three times.

Temperature Measurement

(U) The primary temperature measuring instrumentation was a total radiation pyrometer with a variable emissivity correction. For all brazing cycles, the emissivity was set at 0.91. A second instrument, of the disappearing filament type, was used to scan the workpiece for gradients and observe the actual melting. The recorded process data is actually the brightness temperature of the specimen taken by the primary instrument focused on a $1/8" \times 1/8"$ area of the flat surfaces of the element $1/4"$ from the end of the element. Temperatures observed in the brazing area with the disappearing filament instrument were recorded and can be correlated later with a two-color instrument to establish spectral emissivity at 650 μ . With this information, the disappearing filament data could then be translated from the brightness temperature to the true temperature. True temperatures are very important in transferring the process from one organization to another.

(CRD) During the program, some temperature variance was observed. Potential sources for variance include variation in emissivity and the presence of the Vycor (99.5% quartz) tube (with or without films). The effect of emissivity can be considerable as shown in the following table:

<u>Brightness Temperature</u>	<u>Emissivity</u>	<u>True Temperature</u>
2100° F	0.9	2127° F
	0.8	2158
	0.7	2194
	0.6	2237
	0.5	2291
2200° F	0.9	2229° F
	0.8	2263
	0.7	2302
	0.6	2305
	0.5	2408

Surface emissivity of an element can vary within the above range so that some control of the surface may be required. Reported variations were about 50-70° F. Additionally, the emissivity of a machined and leached element containing fuel is somewhat higher than a machined unfueled element because of differences in surface roughness.

(U) The transmission characteristics of the Vycor tube because of variations in optical quality may also introduce a temperature variable. Even when optical quality is controlled (as in quartz windows) each 1/8" thickness introduces a 1° C negative error for each 100° C rise in the true temperature of the body. Additionally, films on the surface of any tube may selectively absorb a particular wave length of light. This could be particularly disturbing when using a two-color pyrometer.

(U) Thus, it is important to realize that temperatures in this report are not true temperatures but are within about $\pm 100^\circ \text{F}$. Because temperature variance is hard to eliminate, the selected process should be relatively insensitive to temperature. Also, care should be taken in transferring process data.

Analytical Procedures

(U) After brazing, open channels were plugged with plastic and the channels of the sample were pressurized (Figure 4). A leak detecting fluid was poured over the joint area around each orifice. The presence of leaks was detected by bubbling of the solution. By careful application of the fluid, the leak can be traced to the defective area. The maximum pressure for leak testing was 20 psig.

(U) During metallographic examination, one full diagonal row of each specimen was sectioned to reveal the axial profile of each of the five orifices in that row. Additionally, the end orifices of the intersecting row were also sectioned axially. The gap between the wall and the braze surface was measured using a filar micrometer at 100X. A sketch was made revealing the extent of flow and the presence of voids. Figure 5 shows a typical, acceptable joint.

(U) In other joints one of three categories of defect were observed. These categories were:

1. Failure of the preplaced alloy to melt or evidence that melting was incomplete. (Figure 6)
2. An open void axially thru the entire joint in the plane of the metallographic specimen. (This included specimens which leaked on pressure test even though the metallographic section failed to reveal a leak path.) (Figure 7)
3. The 0.043" long braze surface was not completely bonded to the adjacent channel for its full length. It was felt that such joints could be marginal even if they did not leak. (Figure 8)

(U) The uniformity of the location of the orifice with reference to the face of the element was established by measuring the gap between the base and the shoulder of the orifice after brazing and sectioning. No attempt was made to measure the uniformity of the location of the outer end of the orifice with respect to another orifice.

Brazing Procedure

(U) After the assembly had been purged and the temperature measuring equipment sighted on the target area, the power was introduced into the workpiece from the coil. The power input rate was controlled manually to provide a temperature rise of approximately 50° F per second until 1500° F was reached. At 1500° F an automatic control was allowed to maintain the temperature $\pm 25^\circ$ F. This closed loop control operated from a third radiation pyrometer which viewed the entire workpiece. The changeover was accomplished when the direct reading pyrometer discussed in the previous section registered 1500° F.

(U) The powder braze alloy specimens were first heated to 1000° F in a vacuum (40 microns) in order to burn out the binder. The 1000° F temperature was held for 5 minutes. Then the inert gas was permitted to enter the chamber and the assembly was brought to 1500° F.

(U) The 1500° F hold point was adopted to permit the part to come to a uniform temperature. The orifices heat by radiation at 1500° F and the gap between the channel wall and the orifice does not have such a detrimental effect as it does at temperatures below about 1500° F. At the same time the temperature is sufficiently below the 1938° F melting point of the alloy, to prevent premature metallurgical reactions. The 1500° F temperature and time at temperature are probably not critical.

(U) Once the workpiece has been held one minute at 1500° F, the power is again applied to the workpiece. The exact power level was not controlled and may have been a factor in causing the observed 50 - 100° F gradients. After using the automatic control to maintain the selected brazing temperature for a predetermined time period, the power was removed and the specimen cooled.

Experimental Results

(U) Incomplete melting was observed only when attempting to braze at 1850° F for only 30 seconds. Specimens held for 60 and 180 seconds did not exhibit the effect. Understandably, the condition was worst at the joint nearest the center. It was evident to a lesser extent on adjacent orifices. Evidence of complete melting in the outer ring of orifices in these same samples suggests that the melting point for this brazing alloy must be between 1800 and about 1850° F since such a temperature gradient probably existed between the center and the outer surface of the specimen. (See the section on "Temperature Measurement.") This defective condition was not included in the response curve because it indicated that the process was running below some minimum threshold. The absence of these incomplete brazes in the 60 second specimens suggests that, even under marginal temperature conditions, 60 seconds is sufficient to avoid a severe gradient at the center orifice.

(U) In the remainder of the 139 specimens brazed at temperatures, averaging from 1850° F (60 seconds or more) to 2100° F, the following defects were observed:

Open Joints	8	
Marginal Joints	<u>12</u>	
Total Defects	20	(14.4% of experimental sample)

This indicates that the experimental plan had been successful in identifying some areas of interaction between the several procedure variables and the rate of defect occurrence.

(U) The response surface which identifies this interaction was generated (Figure 9) by plotting the percentage of defects that occur in any given sample against the independent variables presented by that sample. A sample in this case represents a specific combination of gap and temperature. Time did not seem to affect quality, except, as noted, at marginal temperatures. Predictably large diametral clearance gaps accounted for most of the voids and open brazes. Also predictably, this situation appeared to be most serious at low temperatures where the gap caused not only flow problems but also heating problems.

(U) The process appears to be very reliable over a temperature range extending from a nominal 1900° F to 2200° F if the diametral clearance between the channel and orifice is kept in the 0.000 - 0.004 range. This is to say that no defects were observed in this range.

(U) Beyond the above boundaries, the trend is to more defects and a less reliable process. The most significant departure from reliability occurs as the diametral gap exceeded the .001-.004 inch category. A detailed review of the specimens in the next higher diametral gaps category (.004 - .009 inches) suggests that the marked increase in failure rate occurs at about 0.0065 inch. Available brazes below this value (specifically in the range of 0.0054-0.0059) appeared to be sound.

(U) An observed tendency for high brazing temperature to starve the joint appears to place an upper limit on the allowable temperature variations. At the high temperatures within the 0.005-0.009 inch range of gaps there was some evidence (Figure 9) that the frequency of defects increased. Supplementary specimens were run at temperatures of 2200° F, 2300° F, and 2400° F. (3 minutes each.) These specimens revealed no change in the failure pattern up to 2200° F if the 0.000-0.004 inch gap was maintained. Above 2200° F, a slight increase in marginal brazes was noted in specimens with a gap of 0.001-0.004 inch. These marginal brazes appear to be the result of excessive capillary flow out toward the face of the element. In such cases, the 0.043 inch long braze zone would become starved of alloy. This is a situation which could be remedied by adding more braze alloy if the overtemperature condition had to be tolerated.

(U) Metallographic analysis of the specimens involving two grades of molybdenum (pure and TZM) showed no difference in wettability between the two materials. This observation is limited to tests at only one temperature (1900° F). However, at this low temperature variances in wettability would have been expected to occur if they were significant.

(U) Supplementary brazed joints using powdered braze material with an acrylic cement as a binder were leak tested and evaluated metallographically. The center-groove orifice configuration with a 0.001-0.004 inch diametral gap was used. Generally, the brazes appeared more porous and spongy than brazes made with spiral strip. Two brazes out of eight were defective. With the same 0.001-0.004 inch diametral gap no defects were observed in over 40 spiral-strip specimens brazed in the same temperature range. The centergroove (figure 1) configuration was more effective in preventing tilting of the orifice during brazing than is the figure 2 configuration. This may reflect the tapering action of the chemical milling process on figure 2 configuration.

(U) A difference in the metallographic appearance of the braze was observed as temperature increased from 1900 to 2400° F. No process or structural significance was attached to this in terms of this program.

(U) The shoulder of the orifices that were not tapered was always either in contact with the element face or within 0.0017" of it. Where orifices were tapered they tended to lift out of the channel and tilt causing the distance to the shoulder to increase to 0.004 inches in some cases.

Discussion of Results

(U) The most significant finding of the program is the delineation of the relationship between time, temperature, diametral gap and braze quality. Figure 9 suggests that the process specification should control temperature between 1950° F and 2200° F. The penalty for under-running this range is an increase in defect potential unless gap is inordinately close. The penalty for over-running appears to relate to some loss of control over the location of the alloy as a result of excessive flow and wetting in non-essential areas. Adjunct tests aimed at evaluating temperature control suggest that the nominal process temperature should be at least 150° F from either extreme. No detrimental metallurgical effects such as erosion were observed.

(U) According to the findings of this program (Figure 9) the diametral gap should ideally be restricted to 0.001 inch maximum because this clearance level tolerated the broadest temperature range. However, the diameter of the channel varies about 0.003 inch and allowance must be made for a 0.002 inch machining variance. Thus the process may be forced to tolerate up to 0.005 inch. Figure 9 indicates that this is near maximum tolerable gap. In fact it is slightly over the upper limit of the arbitrary 0.001 - 0.004" category upon which response surface is based. However, a careful review of all defective specimens with clearances above .004 inches suggested that defects existed in brazes with gaps as low as 0.0065 inch, but brazes with diametral gaps of 0.0054 inch to 0.0059 inch were sound. Thus, establishing a maximum clearance limit of 0.005 appears to be justified.

(U) It is desirable to have the largest acceptable diametral gap in order to permit rapid assembly. Occasionally, when working with diametral gaps of 0.001 and 0.002 inch, the braze material broached the channel and disturbed the coating in such a manner that brazing alloy flow was blocked. Additionally, the assembly of an eleven-orifice specimen with gaps of 0.001 - 0.002 inch requires about one hour. Gaps of 0.005" or greater permit the same assembly to be produced in about 30 minutes. These time values are not necessarily representative of production rates but the relationship between them suggests that clearance will affect manufacturing time.

(U) The results also suggest that either unalloyed molybdenum or TZM (Ti-Zr) molybdenum alloy can be brazed. However, it does not appear, at this time, that the powder works as well as the wrought (sheet) braze alloy. There may be two reasons for this. First, it appeared in the metallographic examination that a carbon residue-molybdenum reaction had taken place. This may have inhibited flow. Second, there is a possibility that the tendency for the wrought braze alloy spiral to spring out into good contact with the element makes it easier to reach molten condition during the initial heatup. Once the braze alloy becomes liquid, it can bridge the gap to the

orifice and act as a relatively efficient heat transfer medium. Efficient heating is always desirable in a brazing process to minimize the chance for side reactions with the base materials, brazing atmosphere or (in the case of powder alloy) with carbon residue from the binder cement.

(CRD) The relationship between the shoulder of the orifice and the face of the element can be considered an indication of the magnitude of the problem associated with maintaining a uniform orifice height above the element face. A uniform height is required if the orifices are to butt against the cluster plates. If the O.D. of the orifice is not tapered, the problem is not severe since the orifice rests within 0.0017 inch of the same axial location regardless of brazing condition. Possibly even some of this variation relates to assembly. Tapering the O.D. (or probably encountering excessive clearance) permits tilting and should be avoided. Additionally tapering may result in a wedging action during the braze cycle which ejects the orifice from the channel.

CONCLUSIONS

(U) This program revealed several quantitative characteristics relating to a brazing procedure using:

Molybdenum orifices (regular - not threaded)

Nickel Chromium Gold Brazing Alloy

GEM Coated Channels

Low Expansion Graphite

(U) First, the total diametral gap between the orifice and the channel affects the quality of the brazed joint.

(U) A gap of 0.006 inch appears to be the maximum that can be tolerated if the amount of preplaced braze alloy weighs 0.0124 ounce. Above the 0.006" width gap there is evidence of insufficient braze alloy which may result in leaks thru the joint.

(U) Process temperatures above 2300° F evidenced a slight tendency to cause joint starvation through excessive flow out of the joint.

(U) If a time of one minute at brazing temperature is maintained, process temperatures as low as 1850° F can be tolerated. 2100° F appears to be an acceptable nominal brazing temperature which would tolerate observed variances in temperature in the form of gradient in the specimen, or as a result of pyrometric variables.

However, temperatures below 1950° F occasionally produce defective brazes unless impractically small gaps are maintained.

(U) Time (30 sec to 180 sec) is of little consequence above 1850° F.

(U) Rate of heating can result in non-uniform temperature distribution. Surface coatings on the exterior of the specimen can influence temperature measurements.

(U) Whether the molybdenum is alloyed with Ti (0.25%) or Zr (0.25%) or unalloyed does not influence brazing characteristics.

(U) Preplacement of braze alloy in the form of a spiral strip placed around the inner end of the orifice was effective. Speed of assembly varies inversely with gap.

(U) Preplacement of strip in a notch proved very difficult. Powdered braze alloy, bonded with acrylic cement could be placed in the notch. The brazes produced from powdered alloy appeared somewhat more porous but were acceptable in a large percentage of cases (the powder-braze heating sequence was not optimized).

(CRD) Orifices with a uniform O.D. are more likely to maintain a uniform height or projection beyond the element face.

Future Activity

(U) While the present effort has provided a quantitative picture of such basic procedure variables as time, gap and temperature and does permit the design of hardware, the information is not sufficient to prepare a process specification.

(CRD) Future activity must establish the ability of a threaded center orifice to be brazed by the same process that seals the orifices described herein. This capability must be established over a realistic range of procedure variances (time, temperature and gap).

(CRD) The problem of precisely assuring the location of the outer end of the regular (non-threaded) orifices must be considered. This is necessary to assure that each orifice butts against the cluster plate. This may introduce tool and/or position constraints.

(U) Only after the above tasks are completed can the process (including tentative tooling, position, preplacement method, clearance, time and temperature be applied to produce complete test pieces which use hardware produced to the configuration and tolerances shown on available reactor design drawings. (This hardware would not, however, involve traceability.) These pieces should be leak tested using the apparatus shown in this report. A transverse metallographic section should then be made to verify the leak test results. When three sequential acceptable specimens have been produced, a process specification may be prepared. The process and at the same time the reference process may then be used to produce hardware for reactor design tests (thermal shock, leak test, and mechanical tests).

(U) A method of placing fresh braze alloy in a leaking joint and rebrazing must be developed.

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Figure 1. Orifice as Originally Designed

Figure 2. Orifice as Redesigned for Spiral Preform

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Figure 3. Brazing Apparatus

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Figure 4. Leak Detection Apparatus

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Figure 5. Typical Brazed Joints 2100°F - 1 Minute - .001" - .004" Diametral Gap

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Figure 6. Unmelted Joints at 1850°F (30 seconds)

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Figure 7. Braze With an Open Channel 1950°F - 1 min. $-.007''$ Diametral Gap

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Astronuclear
Laboratory

Figure 8. Braze with Marginal Fill - 2100°F - 3 Minutes - .007" Diametral Gap

CONFIDENTIAL
RESTRICTED DATA
Atomic Energy Act - 1954

Figure 9. Response Surface for Induction Heated Graphite - Croniro - Mo Brazed Joints

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