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BDX-613-1683 (Rev.)

CERAMIC-TO-METAL SEALING

PDO 6989258, Final Report

W. G. Gates, Project Leader

Project Team:
G. E. Parkhurst

Published March 1977

Prepared for the United States Energy
Research and Development Administration
Under Contract Number EY-76-C-04-0613
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Printed in the United States of America

Available From the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161.

Price: Microfiche \$2.25
Paper Copy \$3.50

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BDX-613-1683 (Rev.), UNCLASSIFIED Final Report, Published
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Prepared by W. G. Gates, D/814, under PDO 6989258

Ceramic-to-metal sealing was accomplished by brazing a metallized alumina insulator to a Kovar (Carpenter Technology Corporation) pin and header. A braze alloy wetting evaluation and a braze joint tensile strength evaluation were completed by brazing the specimens in a hydrogen atmosphere. From these evaluations the best braze alloy and brazing cycle were selected. Twelve braze alloys were evaluated and Nicusil 3 (Western Gold and Platinum Company) braze alloy was found to have good wetting characteristics and optimum strength. Assemblies brazed with this alloy were vacuum leak tight and metallurgically sound.

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States Energy Research and
Development Administration
Contract Number EY-76-C-04-0613
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SUMMARY

Ceramic-to-metal joints were brazed and evaluated to compare various ceramic metallizations, braze alloys, and brazing processes. Twelve braze alloys were selected and evaluated in a braze alloy wetting comparison. Six of the alloys, heated in a hydrogen atmosphere furnace, had acceptable wettability on nickel plated stainless steel coupons. These alloys were evaluated on standard ceramic tensile braze specimens. Resulting data indicated that Nicusil 3 from the Western Gold and Platinum Company (Wesco) was the better braze alloy for ceramic sealing.

Wesco versus Diamonite Type II ceramic (United States Ceramic Tile Company), Sandia Laboratory versus EIMAC (Division of Varian Associates) metallizations, and electroless nickel-boron versus electroplated nickel (nickel-chloride solution) were evaluated. No difference in tensile strength could be attributed to the ceramic vendor, metallization, or type of Nicusil plating. The electroless nickel-boron plating was more uniform than the electroplated nickel and in some cases exhibited slightly better wettability. A plating fixturing problem, resulting in nonuniform current density, caused this nonuniform plating condition and can be corrected.

A metallurgical evaluation of the brazed ceramic tensile specimens indicated that brazing time and temperature were critical. Brazing at 55°C above the braze alloy liquidus for 10 minutes caused severe braze alloy penetration of the ceramic metallization. This was most critical for the higher temperature braze alloys. It resulted in a marked reduction in tensile strength and an increase in the frequency of vacuum leaks. An adequate cycle for Nicusil 3 braze alloy was obtained by slowing the heating rate and reducing the time at temperature. This resulted in higher braze strength and prevented braze alloy penetration of the metallization.

The observed large deviation in tensile strength may be due to tensile fixture misalignment or to the large statistical variation expected with brittle ceramic material. Additional evaluations are required to better characterize the tensile strength of this acceptable ceramic-to-metal seal and to further evaluate the higher temperature braze alloys at each alloy's optimum braze cycle.

DISCUSSION

SCOPE AND PURPOSE

This study was initiated to develop technical knowledge about ceramic-to-metal sealing. It will provide a basis for advising commercial vendors in correcting production problems encountered at Bendix with various ceramic-to-metal seals. Specifically, attention is focused on a sealing problem encountered with a switch circuit manufactured by the EIMAC Division of Varian Associates.

ACTIVITY

Definition of Process

A thorough literature search was conducted to establish a fundamental knowledge of the sealing process and to lay the groundwork for a meaningful development plan. A large number of ceramics and sealing processes are reported to produce satisfactory ceramic-to-metal seals. However, in order to scope the work for a short term process development order, only the ceramic metallizing and sealing processes used for the switch were selected.

There are eight different sealing techniques listed in the literature for joining a metal to a ceramic.¹ Of these eight techniques, only the sintered metal powder seals have gained widespread acceptance for sealing the alumina ceramics used in most Bendix Kansas City applications. This technique is used by EIMAC and with most other supplied alumina-to-metal seals used at Bendix. A brief description of this sealing process is presented to give insight into the terminology and processing details involved in sealing alumina to Kovar.

A typical ceramic-to-metal seal is represented (Figure 1). The ceramic compositions for most Bendix applications are 94 and 99 percent alumina. The percentage refers to the ratio of aluminum oxide (Al_2O_3) to the glassy phase ($\text{Al}_2\text{O}_3 + \text{SiO}_2$). To join ceramic to metal it is necessary to attach a bondable surface on the ceramic. This is accomplished by firing a sintered metal powder (metallization) onto the ceramic at 1300° to 1600°C in wet hydrogen. This metallization is then plated with nickel and brazed to Kovar (29Ni-17Co-53Fe) (Carpenter Technology Corporation) using standard plating and brazing techniques. Kovar is the standard metal chosen for alumina-to-metal seals because its coefficient of expansion ($10.4 \mu\text{m/m}/^\circ\text{C}$) is near that of alumina ($8.28 \mu\text{m/m}/^\circ\text{C}$).

Of the many metallization compositions,¹ the most common is a mixture of manganese and molybdenum powders with added trace amounts

Figure 1. Ceramic-to-Metal Seal

of silica, iron, zirconium, or titanium. Most vendors including EIMAC consider their metallization composition proprietary and will not report the exact composition. Sandia Laboratories Albuquerque uses a mixture of 69.56 weight-percent molybdenum, 17.39 weight-percent manganese, and 13.04 weight-percent titanium, which they fire onto the ceramic at 1495°C in wet hydrogen.² The wet hydrogen is required in the metallizing process to supply sufficient oxygen to oxidize the manganese and titanium which will go into solution with the alumina to form a lower melting glassy phase. This glassy phase provides a mechanically-adherent coating of metallic molybdenum on the ceramic surface. Next, this molybdenum-rich outer layer can be coated with a nickel electrodeposit to cover the porous metallization structure and form a smooth brazable surface.

The literature does not explicitly state which braze alloy works best, but does indicate that the copper, silver, and noble base alloys can be used with standard brazing techniques.³

Sandia Albuquerque has reported that good ceramic brazes should fail under tensile stresses at 75.8 to 82.7 MPa (11,000 to 12,000 psi). Also, a strong braze joint normally fails either in the alumina ceramic or the titania-manganese dioxide-alumina glassy phase diffusion zone. Poor braze joints are caused by a number of factors.

Thin nickel plating (less than 5.08 μm or 0.0002 inch) can completely dissolve in the braze alloy and result in loss of adhesion.

Thick nickel plating (greater than 7.62 μm or 0.0003 inch) can reduce the overall braze joint strength.

Thin metallization can result in loss of nickel plating adhesion.

Poor cleaning procedures can cause blisters and lack of either plating or metallization adhesion.

Wetting Evaluation

A wetting evaluation was conducted to select candidate alloys for hydrogen brazing to metallized ceramics. Three types of wetting samples were selected for this evaluation: 304 Stainless Steel coupons and two similar 304 Stainless Steel coupons--each with a different type of 8 μm thick nickel plating. Electroplated nickel from a nickel-chloride solution and electroless nickel-boron from a nickel-boron solution were selected for this evaluation because they represent EIMACs past and present platings, respectively. EIMAC switched to electroless nickel-boron because of past problems with controlling the electroplated nickel deposits. Twelve braze alloys were examined in this evaluation (Table 1).

Two Palcusil 10 wetting coupons were used in this evaluation; an electroless nickel-boron plated sample and an electroplated nickel sample are shown (Figure 2). Some of the nickel electro-deposited surfaces discolored from nonuniform current density on the specimen during plating. This discoloration was observed to affect the wettability results. However, samples that were not discolored had equivalent wetting to the nickel-boron coated samples. This current density problem appears to be a function of the plating fixturing and can be corrected.

Figure 3 illustrates four wetting samples: Nicusil 3 on electroless nickel plated and bare stainless steel, silver on electroless nickel, and Lithobraz 725 on electroless nickel. None of the braze alloys wetted the unplated stainless steel specimens when they were brazed in the dry hydrogen atmosphere because the hydrogen atmosphere was not adequate to keep the stainless steel from oxidizing. The two types of nickel plated samples were similar; however, the electroless nickel-boron coatings seemed to wet slightly better with some of the alloys. This could result from the previously described plating current density problem encountered with the nickel-chloride electroplating solution. Nicusil 3, Lithobraz 725, Palcusil 10, Nicoro 80, and 80-20 were selected from this wettability study, since each have acceptable wettability on both types of nickel plated specimens in a hydrogen atmosphere. However, Nicusil 3 appeared to be the best.

Table 1. Candidate Braze Alloys

Braze Alloy	Composition (Weight-Percent)	Liquidus Temperature (°C)
Cusil*	72Cu-28Ag	780
Lithobraz 725**	71.5Ag-28Cu-0.5Li	780
Lithobraz 925**	92.5Ag-7.3Cu-0.2Li	890
Nicusil 3*	71Ag-28Cu-1.0Ni	795
Palcusil 10*	58Ag-32Cu-10Pd	852
Incuro 60*	60Ag-32Cu-3In	900
80-20*	80Au-20Cu	910
Silcoro 75*	75Au-20Cu-5Ag	895
Nicoro 80*	81.5Au-16.5Cu-2Ni	925
Silver	100Ag	960
Nicoro*	35Au-62Cu-3Ni	1030
Copper	100Cu	1083

*Western Gold and Platinum Company

**Handy & Harman Company

Metallized Ceramic Samples

Bendix does not have the necessary equipment for metallizing ceramics. Therefore, Type II (96 percent alumina) Wesco ceramic, similar to that used in the switch circuit, was purchased from the Western Gold and Platinum Company. These ceramics were obtained as standard ASTM tensile test specimens (ASTM-F-19). For comparison, some of these test specimens were metallized at EIMAC by the same process used for the switch and the rest were metallized at Sandia Laboratories Albuquerque. Type II metallized Diamonite ceramic (United States Ceramic Tile Company) was also supplied by Sandia Laboratories to provide a comparison with the Wesco ceramic. The standard ASTM ceramic test specimens and the tensile test procedure described in ASTM-F-19 were used to compare the ceramics, metallizations, nickel platings, and selected braze alloys. Fracture specimens are shown in Figure 4. Nicusil 3 was used as the braze alloy to compare the strengths of the ceramics, metallizations, and platings. Wesco ceramic test specimens that were

ELECTROPLATED NICKEL

ELECTROLESS NICKEL-BORON

Figure 2. Palcusil 10 Brazed Electroplated Nickel and Electroless Nickel-Boron Coated 304L Stainless Steel Wetting Coupons (2.75X)

metallized at EIMAC and electroless nickel-boron plated at Bendix were chosen as a base to compare the braze strength of the five different braze alloys.

All specimens were ultrasonically degreased in trichloroethylene and isopropyl alcohol, fixtured according to ASTM-F-19, and brazed in a dry hydrogen atmosphere. The brazing cycle selected for each alloy was 10 minutes at 55°C above its liquidus temperature. Since the samples were placed in the preheated furnace, the heat up time of the specimens was included in this 10 minute brazing time. The samples were allowed to cool in a water cooled, hydrogen purged, chamber.

Nicusil 3 tested very good in every case. The 20 specimens metallized at Sandia and the first five metallized at Wesco vacuum leak checked better than 666 μ Pa (5×10^{-6} torr) and tensile tested at an average strength of 75.8 MPa (10,971 psi).

One group of five ceramic test specimens metallized at Wesco electroless nickel-boron plated at Bendix, and brazed with Nicusil 3, experienced three leakers. Discoloration of this group

NICUSIL 3, ELECTROLESS NICKEL-BORON

NICUSIL 3, STAINLESS STEEL

SILVER, ELECTROLESS NICKEL-BORON

LITHOBRAZE 725, ELECTROLESS
NICKEL-BORON

Figure 3. Braze Alloy Wetting Evaluation

A. DIAMONITE, GROUP 1

B. WESCO, GROUP 6

C. WESCO, GROUP 2

D. WESCO, GROUP 6

Figure 4. Fractured ASTM-F-19 Tensile Test Specimens (1.25X)
(Reference Table 2)

occurred during the hydrogen brazing and the samples with the most discoloration leaked (Figure 4D). This discoloration was a carbonaceous residue. It probably resulted from insufficient cleaning and was reduced to carbon by the hydrogen atmosphere during the brazing cycle. The film is considered to be the cause of the braze joint deterioration in this particular group of Nicusil brazed samples.

A graph (Figure 5) and a table (Table 2) represent the high, low, and mean for all the specimens tensile tested per ASTM-F-19. Typical fractures are shown in Figure 4. Each category of braze strengths consisted of a sample size of four tensile test specimens. The braze joints are categorized (Table 2). It can be seen that most of the Nicusil braze joints were in the same tensile strength range. All of the high strength fractures were similar to those shown in Figure 4 (A and B) with either 100 percent ceramic failure (A) or partial ceramic and metallization failure (B). According to Sandia, failure should occur in either the metallized layer or glassy diffusion zone in good ceramic braze joints. The Group 6 braze that failed at 25.9 MPa (3777 psi) was a contaminated specimen shown in Figure 4 (D) that also failed the vacuum leak test. Samples in Groups 2, 3, and 7 that fractured in the 41.3 to 51.6 MPa (6000 to 7500 psi) range failed 100 percent in the ceramic material (A and C). This ceramic failure mode rather than braze joint failure indicates that the wide range of strengths is due either to tensile test fixture misalignment or to the large variation in strength expected with ceramics. Since a statistical approach is required to characterize strengths of brittle materials, such as ceramics and glasses, the validity of the Group 4 higher braze joint strengths (Figure 5) cannot be verified without further fixture modifications and more tests.

The other alloys examined, 80-20, Palcusil 10, and Nicoro 80 (Groups 9, 10, and 11) had gross vacuum leaks as well as lower tensile strength compared to the Nicusil 3 braze alloy. Subsequent examination revealed that these samples had braze alloy penetration into the ceramic metallization layer which caused this braze joint strength degradation. Although Lithobraz 725 was omitted from this comparison due to an insufficient number of ceramic samples available for evaluation, it is a silver-copper eutectic alloy similar to Nicusil 3 with the exception of containing trace lithium rather than nickel and is considered to give similar results.

A metallurgical examination and fracture analysis were made on each group of brazed specimens. Fracture analysis and cross sections of the Nicusil 3 braze joints indicated that fracture occurred most frequently in the titania-manganese dioxide-alumina diffusion layer.

Table 2. Samples Used to Determine Braze Strength

Group Number	Metallization	Plating	Braze Alloy	Furnace	Ceramic Manufacturer
1	SLA	SLA	Nicusil	Lindberg	Diamonite
2	SLA	SLA	Nicusil	Lindberg	Wesco
3	SLA	Ni	Nicusil	Lindberg	Wesco
4	SLA	Ni-B	Nicusil	Lindberg	Wesco
5	EIMAC	Ni-B	Nicusil	Lindberg	Wesco
6	EIMAC	Ni-B	Nicusil	Lindberg	Wesco
7	EIMAC	Ni-B	Nicusil	BTU	Wesco
8	EIMAC	Ni-B	Nicusil	AVS	Wesco
9	EIMAC	Ni-B	Nicoro 80	Lindberg	Wesco
10	EIMAC	Ni-B	Palcusil	Lindberg	Wesco
11	EIMAC	Ni-B	80-20	Lindberg	Wesco

Figure 5. Braze Joint Tensile Strength

The first four groups of Nicusil 3 specimens had no appreciable braze alloy diffusion (Figure 6). However, Groups 5 and 6 did (Figure 7). EDAX (Energy Dispersive Analysis of X-Rays) analysis of the four locations represented in Figure 6 (C) revealed compositions of alumina-manganese dioxide-titania glass, molybdenum-metallization, nickel-boron plating, and alumina ceramic. No braze alloy penetration other than diffusion into the nickel-boron plating was noted. Region 5 (6A) and 6 (6B) were found to be the glassy phase ($\text{Al}_2\text{O}_3+\text{SiO}_2$) of the alumina ceramic. Examination of the Group 5 test samples in Figure 7 (C) shows the braze alloy has penetrated through the metallization to the glassy phase. The braze is the light colored region 1 in (C), while the glassy phase is region 2 in (C and D). Region 4 in (C) is the molybdenum-manganese metallization diffused with nickel and region 5 in (C) is the nickel plating.

The braze alloy penetration in Groups 5 and 6 was indirectly caused by fixturing. Groups 5 and 6 were the first specimens brazed and were fixtured directly onto the metal boat that carried the assemblies into the furnace. It was found that the tensile specimens for the higher temperature braze alloys (Palcusil 10, Nicoro 80 and 80-20) were cracking because of thermal shock when they were fixtured in this manner. To eliminate cracking due to thermal shock it was necessary to insulate the tensile specimens from the metal furnace boat by placing them on an aluminum disc. This disc eliminates the cracking problem by increasing the brazing tensile specimen heat up time. Groups 1, 2, 3, and 4 were also brazed using these insulating alumina discs. Since all the specimens were brazed by placing them into a hot (55°C above liquidus for the specific braze alloy) furnace for 10 minutes, the alumina disc slowed the sample heat up time, reduced the time at brazing temperature, and minimized the braze alloy penetration as metallurgically observed in Groups 1, 2, 3, and 4.

The braze alloy penetration (Figure 7) for Group 5 test samples indicated that their braze time was too long. Consequently, for the higher temperature braze alloys this degradable effect was more pronounced and resulted in the much lower tensile strengths. If 10 minutes is marginal for the lower temperature Nicusil 3 brazes, then it will have to be reduced significantly for the higher temperatures brazing alloys. Metallurgical cross sections of the high temperature braze alloys (Groups 8, 9, and 10) revealed severe braze alloy penetration and account for their lower strengths (Figure 5).

The resultant structures of SLAs and EIMACs metallizations can be observed in Figures 6 and 7, respectively. EIMACs metallization has more glassy phase than the SLA metallization. Compare the relative thickness in Figures 6 and 7 noting the difference of magnification. The intergranular penetration of this glassy phase

1. ALUMINA-MANGANESE DIOXIDE-TITANIA GLASS
2. MOLYBDENUM-MANGANESE METALLIZATION
3. NICKEL-BORON PLATING
4. ALUMINA CERAMIC
5. AND 6. GLASSY PHASE

Figure 6. SEM Photomicrographs of a Group 4 Ceramic Braze

Figure 7. SEM Photomicrographs of a Group 5 Ceramic Braze
(Note Braze Alloy Penetration)

into the alumina ceramic can be seen in both figures. The glassy phase in both cases was, as determined by SEM-EDAX analysis, composed of alumina-manganese and titanium-oxides. No difference in braze strength could be attributed to the two different metallizations.

Figure 8 shows a Kovar header assembly with an EIMAC metallization and electroless nickel-boron plated Wesco Type II ceramic. It was brazed with Nicusil 3 in a hydrogen atmosphere by the process previously described. The braze worked well, forming a vacuum leak tight electrical feedthrough. The graphite fixture used did not adequately maintain dimensional alignment; therefore, future fixturing will be made out of Kovar to assure a matched thermal expansion between the assembly and braze fixture.

ACCOMPLISHMENTS

Technical information about the ceramic metallizing and sealing processes, as related to a switch circuit, was obtained and reviewed. The wetting characteristics of 12 brazing alloys were determined in a hydrogen environment on 304 Stainless Steel test coupons with electrodeposited nickel coatings. Results indicate that the electroless nickel-boron appeared better and provided slightly better wettability than the electroplated nickel. In addition, Nicusil 3, Lithobraz 725, Palcusil 10, Nicoro 80, and 80-20 alloys exhibited acceptable wetting behavior on both the electroless nickel-boron and electroplated nickel samples and were selected for further evaluation.

Standard ASTM ceramic tensile test specimens and the test procedure described in ASTM-F-19 were used to compare the various ceramics, metallizations, nickel platings, and selected braze alloys. Resulting tensile strengths indicate that no differences can be attributed to either the SLA or EIMAC metallizations or the electroless nickel-boron or electroplated nickel coatings. In addition, the Nicusil 3 produced joints with the highest strengths of those selected alloys following similar brazing cycles. However, metallurgical examinations of the fractured Palcusil 10, Nicoro 80, and 80-20 alloy tensile specimens indicate that the 10 minutes at 55°C above the liquidus for these higher temperature alloys enabled the braze alloy to penetrate into the ceramic metallization layer and cause degradation in tensile strengths. Further evaluations involving shorter brazing times and lower temperatures appear necessary in order to improve these initial tensile strengths and permit their usage.

A Kovar header assembly was brazed with Nicusil 3 by the process described and passed the specified vacuum leak test. The dimensional control of the assembly was marginal due to the thermal

TOP

BOTTOM

Figure 8. MC 2935 Header Brazed With Nicusil 3 (3.4X)

expansion mismatch of the graphite fixturing; however, future improvements are expected with Kovar fixturing.

The technical knowledge gained from this process development order is currently being utilized to aid in the solution of vendor related problems with other parts.

FUTURE WORK

More development work is needed to define the time-temperature relationship of braze alloy penetration and quantity and determine its effect on braze joint strength, and vacuum integrity. These relationships should be established using the braze alloys most frequently selected by commercial vendors.

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