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BDX-613-2414

Failure Analysis of Ceramic-to-Metal Seals

By W. G. Gates

Published March 1980

Topical Report

Prepared for the United States Department of Energy
Under Contract Number DE-AC04-76-DP00613.

**Kansas City
Division**

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Price: Microfiche \$3.00
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Distribution Category UC-38

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Prepared by W. G. Gates

Ceramic-to-metal seal failures that occur at the next assembly level present a critical problem because of the time and expense involved in the assembly of electro-mechanical switches. A typical analysis of a failed assembly and a recommended rework process are presented. In addition, steps are proposed to minimize the occurrence of future ceramic-to-metal seal failures.

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**A prime contractor with the United States
Department of Energy under Contract Number
DE-AC04-76-DP00613**

CONTENTS

Section	Page
SUMMARY.	5
DISCUSSION	6
SCOPE AND PURPOSE.	6
ACTIVITY	6
<u>Sample Preparation</u>	6
<u>Failure Analysis</u>	7
<u>Pin Rework</u>	13
<u>Specification</u>	17
ACCOMPLISHMENTS.	17
FUTURE WORK.	18
REFERENCE.	19
APPENDIX: REQUIREMENTS FOR CERAMIC-TO-METAL BRAZED ASSEMBLIES	20

ILLUSTRATIONS

Figure		Page
1	Crack Observed in Nickel Plating (Polaroids)	8
2	SEM and EDS Analysis of Nickel Plating Contaminant (Polaroids)	9
3	SEM and EDS Analysis of Pin Fracture Contaminant (Polaroids)	10
4	SEM and EDS Analysis of Contaminant on Unbrazed Pins (Polaroids)	11
5	EDS Spectra of Number 3 Pea-Gravel Deburring Medium (Polaroids)	12
6	SEM Photographs of Nonuniformly Etched Pins (Polaroids)	14
7	SEM Comparison of Nickel-Stripped Pins (Polaroids)	15
8	Dark-Field Metallographic Photographs of Dye Penetrant in Cracks (Polaroids)	16

SUMMARY

Ceramic-to-metal seals are used in Bendix-manufactured components because they have higher temperature and strength requirements than can be provided by glass-to-metal sealed headers. Bendix procures ceramic-to-metal seals for electromechanical switches and for an output switch, all of which have multiple applications. Typical requirements for these seals include: high vacuum requirements, typically 1×10^{-8} cm³/s leak rate, high G forces (3500 G for 1 ms), and capability to withstand a 580°C fire for one hour without failure. After receiving these ceramic-to-metal seals from commercial suppliers, they are inspected and laser welded into the 13-8PH stainless steel switch housings. Seal failures at this manufacturing stage present a critical problem because of the time and expense involved in fabrication.

Failure analysis of a switch revealed a vacuum leak caused by poor nickel plating adhesion on the Kovar pin. This plating problem was traced to the vibratory deburring process. An alumina-silica film left on the Kovar pins was not removed by the supplier's cleaning process prior to nickel plating. During the subsequent sintering operation, the nickel plating diffuses past this alumina-silica contaminative layer and leaves it in the center of the nickel plating. This contaminative layer has been the verified source of recently observed pin braze failures.

A rework process has been established for stripping the nickel plating and the accompanying deburring alumina-silica contaminant from the Kovar pins. A Kovar bright dip solution, composed of acetic, nitric, hydrochloric, and hydrofluoric acids, was used. Careful monitoring of the etch time and weight loss per pin and inspection with a scanning electron microscope (SEM) provides a satisfactory rework technique for the pins.

Bendix has prepared a specification to control the recurrence of future ceramic-to-metal sealing problems. This document proposes more vendor process control. The vendor will be required to keep accurate records on lot control, material certification, and the data of subsequent specified tests.

DISCUSSION

SCOPE AND PURPOSE

Recently, ceramic-to-metal seals for a six-pin header have failed vacuum leak specifications. These seals are procured from a commercial supplier, inspected, and laser welded into a 13-8PH stainless steel alloy switch housing. Typically, these seals were passing receiving inspection tests; however, gross vacuum leaks were occurring after the welding operation. An investigation was initiated to determine the cause of this failure, to define the appropriate solution, and to specify appropriate action to minimize future occurrences of similar vacuum leaks.

ACTIVITY

Sample Preparation

A technique has been developed for evaluating ceramic-to-metal seal failures. First, the surface of the seal in question is examined with a stereomicroscope at 50X magnification for braze and ceramic defects. Higher magnification examinations of these seals are done on the scanning electron microscope (SEM), and unknown particles observed with the SEM are analyzed with an energy dispersive spectrometer (EDS) to determine a qualitative elemental analysis.

Before a metallurgical cross section is examined, the exact source of the vacuum leak must be identified. This is accomplished by first vacuum leak testing the assembly and finding the general location of the leak with a helium leak tester. The location of the leak then is pinpointed more exactly by using a bubble test that involves submerging the ceramic-to-metal seal in an alcohol bath and visually examining for bubbles at 50X on a stereomicroscope. This technique will reveal leaks as small as $1 \times 10^{-5} \text{ cm}^3/\text{s}$. The exact location of the leak is scribed on the assembly and is used later to identify the sample's position for cross-sectional examination.

The cracked seal next is given a dye penetrant treatment to highlight any existing cracks. The dye penetrant is a 1 volume percent rhodamine (B,O) solution (a violet 10 biological stain) mixed with 39.5 volume percent alcohol, 39.5 volume percent water, and 20 volume percent wetting agent. The ceramic-to-metal seals are placed in a small chamber and the chamber is evacuated for one hour using a standard vacuum roughing pump. The dye solution is vacuum wicked into the chamber and pressurized to 698 mPa for 30 minutes. The seals then are removed

from the chamber and the dye is completely washed from the seal surface. The trapped dye in any cracks is then dried by baking for one hour at 93°C.

The defective ceramic-to-metal seal is carefully positioned for epoxy mounting so that cross sectioning can be accurately located longitudinally through the marked defect. The sectioned seal is then observed optically using bright and dark field microscopy to examine the seal for cracking, presence of dye, and general seal quality. Further higher magnification analysis of defects is conducted using the SEM with supplemental EDS elemental analysis.

If more than one sample is available, the samples are fractured and the fracture interfaces are analyzed using Auger electron spectroscopy (AES), SEM and EDS analysis, and electron microprobe (EMP) X-ray mapping. The dye penetrant may be used in some cases to highlight the fracture surfaces.

Failure Analysis

The leaks in the defective ceramic-to-metal seals were caused by cracks in the pin braze. Metallurgical cross-sectional analysis (Figure 1) revealed a crack propagating the length of the ceramic-to-metal seal through the plated nickel layer. Examinations of the uncracked areas in Figure 1 and of copper-overplated unbrazed pins in Figure 2 indicate that a continuous line of contaminative material was trapped in the nickel. SEM-EDS analysis indicated the contaminative layer was located midway in the nickel plating and was composed of a high aluminum and silicon bearing material (Figure 2).

These data indicated that the contaminative layer was caused either by poor plating adhesion on the Kovar and the subsequent sintering operation caused the nickel to diffuse past the contaminant, leaving it in the center of the plating, or by a second nickel plating operation on top of the original nickel layer, with subsequent poor plating adhesion.

Fracture analysis of pin seals (Figure 3) revealed that, in every case, fracture occurred at this contaminative layer in the nickel plating. The EDS spectra also revealed that the contaminant again was high in aluminum and silicon and was distinctly different from the high aluminum base ceramic.

Unbrazed pins obtained from the seal supplier were analyzed and were found to have the previously identified contaminative layer. In the areas of the reduced cross sectioning, the alumina-silica containing contaminant was heavy enough to protrude through the nickel plating (Figure 4). According to the supplier, the manufacturer was using a deburring process prior to nickel plating. Historically, uncontrolled deburring media and inadequate cleaning

Figure 1. Crack Observed in Nickel Plating

processes have been a difficulty when subsequent plating operations are required. For example, an earlier study¹ revealed that all deburring media with the exception of dolomite, a calcium magnesium carbonate material soluble in acetic acid and water, will produce poor plating adhesion. However, all the plated and sintered pins used in this study had passed plating adhesion tests. Although the subsequent 800°C, 30-minute sintering operation improved plating adhesion enough to pass standard plating bend and scrape tests, the contaminative layer in the nickel plating provided a continuous cracking path through the ceramic-to-metal seal which failed under typical handling stresses.

Figure 2. SEM and EDS Analysis of Nickel Plating Contaminant

Optical examination of the pin manufacturer's deburring medium revealed four distinct colors of deburring stones. The deburring

Figure 3. SEM and EDS Analysis of Pin Fracture Contaminant

Figure 4. SEM and EDS Analysis of Contaminant on Unbrazed Pins

medium, number three pea-gravel, is apparently a river rock local to the pin manufacturer's area. Two colors of rock were found to be silicon dioxide, the third color was high alumina-silica with a trace of zirconia, and the fourth was a high alumina-silica with traces of zirconia and titania (Figure 5). The high alumina-silica content in the rock matched the high alumina-silica found in the

STONE 1

STONE 2

STONE 3

STONE 4

Figure 5. EDS Spectra of Number 3 Pea-Gravel Deburring Medium

nickel plating and confirmed the contaminant's origin. Sintering tests conducted at Bendix revealed that the trapped deburring contaminant migrated outward as the nickel diffused into the Kovar during the 800°C sinter for 30 minutes. These tests explained the location of the contaminant in the center of the nickel plating shown in Figure 2. In summary, the pea-gravel deburring medium is the source of the current pin plating problem. The resulting contaminative layer in the nickel plating affords a continuous crack path which propagates under stress created during the assembly and welding processes, thus resulting in the observed vacuum leaks.

Pin Rework

The first alternate solution would be to order new pins without the deburring contaminative layer. However, the excessive lead time on ordering new pins was not compatible with current production schedules. Two other proposed solutions were either to chemically strip the nickel and contaminant from the pin or to sinter the pins to further diffuse the nickel and possibly disperse the continuous contaminative layer.

The chemical stripping evaluations were completed at Bendix. An etching solution was found that removed the nickel plating and contaminative layer from the Kovar pins. The solution was a Kovar bright dip etching solution composed of 750 mL of acetic acid, 250 mL of nitric acid, 15 mL of hydrochloric acid, and 5 mL of hydrofluoric acid. This solution was allowed to age for 30 minutes prior to etching the pins. The solution is unstable, and the etch rate on Kovar varies with solution age. The etch rate appears to reach a maximum after approximately two hours. Therefore, to use this bright dip solution, a preliminary batch of pins had to be processed to determine the solution's current etch rate. This etch rate was used to establish the etching time on the first batch of approximately 500 pins. Weight loss of each subsequent batch of pins was monitored, and the etch time was adjusted to maintain a uniform etch and a consistent amount of material removed from each pin. Each batch of 500 pins was etched in 150 mL of solution for approximately 40 seconds using ultrasonic agitation. The ultrasonic agitation is necessary to break off the contaminant from the pin surface as the etch proceeds through the chemically inert alumina-silica contaminative layer, which helps produce a more uniformly etched pin surface. Figure 6 shows contaminant remaining on an etched pin surface and a nonuniform etching condition.

The Kovar bright dip etching technique produces a bright appearing pin surface with the nickel removed and the contaminant reduced to an acceptable level. Weight loss per pin is maintained between 1.1 and 1.8 mg, which produces a clean and dimensionally acceptable

Figure 6. SEM Photographs of Nonuniformly Etched Pins

pin. A weight loss of 0.88 mg/pin shows contaminant remaining on the pin surface, whereas a weight loss of 0.95 mg/pin produced a clean pin (Figure 7). Dimensional measurements on the large and small ends of pins revealed a dimensional spread of 0.742 to 0.757 and 0.340 to 0.358 mm, respectively, when the weight loss was maintained between 1.1 and 1.8 mg/pin.

A high temperature sintering operation was proposed as another pin rework technique. This sintering was expected to diffuse the nickel, break up the continuous line of contaminant, strengthen the seal, and minimize the probability of cracked seals. This rework technique would be less expensive than etching; however, evaluations indicate that sintering does not sufficiently break up the contaminative layer to prevent similar cracking of contaminated seals.

Different brazing alloys were examined by the seal manufacturer in an attempt to diffuse the nickel and to disperse the contaminative layer simultaneously during the brazing operation. A pure silver alloy and a 50 weight percent gold-50 weight percent

Figure 7. SEM Comparison of Nickel-Stripped Pins

copper braze alloy were selected to evaluate this rework technique for salvaging the contaminated pins. The silver braze alloy at lower temperatures did not wet the Kovar pins, and, at higher temperatures, the silver and the gold-copper braze alloys degraded the ceramic seal's metallization adhesion to the ceramic. These brazing defects were readily visible and were highlighted by the rhodamine dye penetrant (Figure 8). The proposed braze temperature or braze alloy changes did not sufficiently disperse the alumina-silica contaminative layer to prevent cracking.

As a result of this rework evaluation, the most reliable solution to this deburring-plating problem is either to rework the existing pins by stripping the nickel plating and contaminant or to machine new pins and eliminate contamination by the pea-gravel deburring media. Although the nickel bright dip stripping technique is difficult to control, it is the only technique evaluated capable of successfully salvaging the pins in time to support current production schedules.

3 MIN AT 980°C

0.02 mm

SILVER

0.02 mm

5 MIN AT 1000°C

3 MIN AT 990°C

0.02 mm

Au-50Cu

0.02 mm

1 MIN AT 990°C

Figure 8. Dark-Field Metallographic Photographs of Dye Penetrant in Cracks

Specification

In an attempt to control the recurrence of future ceramic-to-metal sealing problems, a special specification was prepared. Recognizing that tests available to truly predict ceramic-to-metal seal quality are very difficult to define and require much evaluation to generate reliable data, quality and process controls during seal fabrication seem to provide the most assurance of seal quality. Therefore, more controls are recommended for the ceramic-to-metal seal supplier, such as requiring accurate records on lot control, material certification and the data from subsequent specified tests. Additional requirements are that plating adhesion tests be run prior to the sintering operation, that all deburring processes have prior Bendix approval, that metallization strength tests be conducted on a daily basis, that strength tests be run on each lot of brazed assemblies, and that bend and thermal shock samples be tested for each lot.

After the seals arrive at Bendix, each seal will be visually inspected for defects and will be given a vacuum leak test. Samples will be bent, and other samples will be exposed to the high temperature survival test. A metallurgical evaluation will be conducted on all failures and on at least two samples from every lot. The proposed specification is contained in the Appendix.

ACCOMPLISHMENTS

A failure analysis technique has been established for evaluating ceramic-to-metal seal failures. This technique includes optical and SEM surface examination; dye penetrant impregnation of defects; microsectioning; and SEM-EDS, EMP, and AES analysis. The technique revealed that recent vacuum leaks in a six-pin switch were caused by pin contamination of silica-alumina particles during the vibratory deburring process, resulting in poor nickel plating adhesion. The contamination caused cracking in the ceramic-to-metal seal during subsequent laser welding. A nickel stripping method was developed to salvage nickel plated Kovar pins that had been contaminated with this alumina-silica film. A Kovar bright dip solution, composed of acetic acid, nitric acid, hydrochloric acid, and hydrofluoric acid, was used to strip the nickel plating from the Kovar pins.

By carefully monitoring the etch time and weight loss per pin, as well as examining one pin per stripping batch with the SEM and EDS, the plated nickel and alumina-silica films were removed to an acceptable level without excessive reduction of pin thickness.

A specification has been proposed to control production and inspection of ceramic-to-metal seals in an attempt to minimize the recurrence of similar defects in procured ceramic-to-metal sealed assemblies.

FUTURE WORK

Efforts will be initiated to implement the proposed specification. Approximately 30,000 Kovar pins will be reworked using this stripping process and returned for use in manufacture of six-pin header seals required to meet production schedules. In addition, acoustic emission techniques will be studied to further strengthen inspection and diagnostic capabilities for ceramic-to-metal seal components.

REFERENCE

¹L. K. Gillespie, Effects of Deburring Contaminants on Electroplating Adhesion (Final Report). Bendix Kansas City: BDX-613-1757 (Rev.), February, 1979 (Available from NTIS).

Appendix

REQUIREMENTS FOR CERAMIC-TO-METAL
BRAZED ASSEMBLIES

REQUIREMENTS FOR CERAMIC-TO-METAL BRAZED ASSEMBLIES

1. GENERAL

1.1 Scope

This specification defines requirements and testing procedures to assure process and quality control for metallized and brazed ceramic-to-metal seals. In case of conflict between this specification and drawing requirements, the drawing shall prevail.

1.2 Lot Definition

A lot of sealed assemblies shall be a ship quantity received from the supplier at one time.

1.3 Records

1.3.1 A supplier shall keep material lot numbers and material certification on the materials used in the seal manufacture. This shall include the nickel plating lots and pin, form, metallizing slurry, and ceramic compositions.

1.3.2 The buyer shall keep cross sectioned mounts and photographs of D-tested units and tensile samples for one year minimum after acceptance by the buyer of the associated material.

1.3.3 A report of the quantity of rejected headers as a result of supplier's thermal shock test shall be supplied with each lot.

1.3.4 The supplier shall certify that the nickel plating complies to the requirements listed in Section 3.1.

1.3.5 The supplier shall certify that the parts were deburred per Section 3.2.

1.3.6 The supplier shall include notification of any production process changes and the current process control data for each lot of assemblies. The first lot of assemblies to incorporate any change shall be so identified.

2. DOCUMENTS

The following documents form a part of this specification.

ASTM F-44 -- Metallized Surfaces on Ceramic

ASTM F 134-- Determining Hermiticity of Electron
Devices With A Helium Mass Spectrometer
Lead Detector

MIL STD 202E Method 107D, Condition C -- Test Methods
For Electronic and Electrical Component
Parts

3. REQUIREMENTS

3.1 Nickel Plating

The nickel plating shall meet the adhesion and general quality provisions defined.

3.2 Burrs

The metal piece parts shall be free of burrs and sharp edges and shall be deburred in a manner approved by BKC that has been proven compatible with subsequent nickel plating processes.

3.2.1 If loose abrasive deburring processes (such as barrel tumbling, centrifugal barrel tumbling, vibratory finishing, or spindle finishing) are used, then white dolomite shall be used as a tumbling media. No other abrasive media or abrasive compounds are allowable. The tub, barrel, or container used for loose abrasive deburring must be used with only dolomite media. If other media is used in this container, it will cause unallowable contamination of parts. After loose abrasive deburring in dolomite, the parts must be ultrasonically cleaned in a 8 to 10 percent solution of glacial acidic acid at $115 \pm 20^{\circ}\text{F}$ for 5 minutes. Ultrasonic rinse in distilled or deionized water for at least 5 minutes followed by a slosh rinse in isopropyl alcohol. Air dry or blow off moisture and particulate with filtered air. If parts cannot be cleaned immediately after loose abrasive deburring they may be stored in water or alcohol for 24 hours maximum.

3.2.2 Electrodeburning surface preparation shall be acceptable. The base metal shall be subjected to such cleaning and pickling as necessary to prepare it for uniform polishing.

The electropolished parts shall have a uniform bright surface finish when examined at 7 to 10X on a microscope.

3.3 Process Control

Three brazes each of the pin-to-ceramic and form-to-ceramic braze shall be tested each week for braze strength. Production pins and ceramics shall be used for these supplier process control tests. A Kovar washer may be used for the ceramic to form tests. The washer must have the same thickness dimension and the same processing as the form. All test specimens shall have a minimum strength of 20 pounds when tested in the direction most likely to cause failure (per 4.6).

3.4 Final Assembly. The assembly shall meet all specified dimensional requirements, shall have a maximum leak rate of $1 \times 10^{-8} \text{ cm}^3/\text{s}$ after thermal shock, shall be capable of surviving 30 minutes at 1000°F and have metallurgically acceptable ceramic-to-metal seals.

4. QUALITY ASSURANCE PROVISIONS

Each lot shall be inspected and tested for conformance with the requirements listed in Section 3. The supplier's test data shall accompany each shipped lot of assemblies. The specific tests and required data are listed in this section. The buyer reserves the right to test or inspect the lot for conformance with any specified requirement.

4.1 Nickel Plating

Samples of as-plated pins and form shall be mechanically deformed and examined by the supplier for plating adhesion prior to the sintering operation.

4.2 Leak Rate

Each lot shall be 100 percent leak tested according to ASTM F 134 by supplier and buyer. Leakage through contact module subassembly shall not exceed $1 \times 10^{-8} \text{ cm}^3/\text{s}$, helium reduced to STP conditions with a pressure differential of $15 \pm 2 \text{ psi}$.

4.3 Thermal Shock

Each lot shall be 100 percent thermal shocked per MIL STD 202E Method 107D, Condition C by the supplier and inspected per 4.1.

4.4 Visual Inspection

Each lot shall be 100 percent visually inspected by the supplier and buyer for quality of workmanship, i.e. defective braze joints, foreign material, handling damage, and bad plating. Headers shall be examined at 5 to 50X on a microscope.

4.5 D-Testing

Three headers from each lot or three headers from each 100 headers if the lot is more than 100, shall be destructively tested at both the supplier and buyer as follows:

4.5.1 Bend Test. Bend each pin on one header six times from perpendicular to approximately 30° off center and back. Bend in alternate directions parallel to the long axis of the form (reference Figure 1). Leak test each header per 4.2 and examine leakers per 4.5.3.

4.5.2 High Temperature Survival. Fire one header at 1000 ±50°F for 30 ±5 minutes in air. Allow a 30 minute temperature raise to 1000°F and 30 minutes to cool prior to removing assembly from oven. Leak test header per Paragraph 4.2 and examine leakers per 4.5.3.

4.5.3 Metallurgical Evaluation. Dye penetrant shall be applied to one header prior to metallurgical cross sectioning. The penetrant shall be a solution of rhodamine B, O basic violet 10 biological stain: 1 percent rhodamine, 20 percent wetting agent, 39.5 percent alcohol, and 39.5 percent water by volume. The part shall be placed in a chamber and evacuated with a vacuum roughing pump for 1 hour minimum. The penetrant should be vacuum wicked into the chamber followed by 100 psig pressurization for 30 minutes. The assembly should be rinsed to remove all surface dye penetrant and oven baked for 1 hour at 150 to 200°F. Mount the D-tested header in epoxy for a longitudinal cross section per Figure 1. The mount and epoxy should be evacuated immediately to assure complete encapsulation. Cure the epoxy at room temperature and carefully prepare metallographical mount. The sealed header shall be metallographically examined in the etched and unetched condition at 200X minimum as follows:

4.5.3.1 Pin Braze. Check each pin to ceramic seal for the following: presence of metallization on the ceramic, braze flow between pin and metallization, and adherent nickel plating to the Kovar interface.

- 4.5.3.2 Form Braze. Check each ceramic to form seal for the following: presence of metallization on the ceramic, braze flow between form and metallization, and adherent nickel plating to the Kovar interface. A filler ring of molybdenum or Kovar is acceptable in this braze joint.
- 4.5.3.3 Records. Photograph at 400X minimum any suspected problem area on each D-tested header plus a minimum of one 400X photograph of each pin-to-ceramic and form-to-ceramic interface on each D-tested header. The photographs and mount made by the buyer must be reviewed by product and materials engineering for each lot.

4.6 Mechanical Testing

Pull test three each of the pin-to-ceramic braze and push out three each of the ceramic-to-form or washer braze each week. If a molybdenum or Kovar filler ring is used in the form braze, leave it out for this test.

5. PACKAGING

Headers shall be individually packaged to avoid possible handling and shipping damage.

BDX-613-2414

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SPECIAL PROJECTS: Failure Analysis

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