CONTAMINANT IDENTIFICATION FOR AN
ELECTROMECHANICAL UNIT

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CONTAMINANT IDENTIFICATION FOR AN ELECTROMECHANICAL UNIT

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Several sources of particulate and organic contamination on piece parts of a complex electromechanical unit were identified and established as causing contact loop resistance (clr) and torque failures in some assembled units. One source, alumina particulates was reduced by developing a circuit board polishing technique. Another source, fractured glass fibers, was minimized by applying a polyurethane coating to the switch block assembly. Some organic contaminants were effectively reduced by plasma cleaning the piece parts and subassemblies. Not all sources of contamination have been identified and corrected to date.

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SUMMARY

Contamination in a complex electromechanical switch has caused contact loop resistance (clr) and torque problems; attempts were made to produce cleaner, acceptable parts. Silicon, aluminum, and iron oxide particulate and organic contamination was found on various piece parts and was established as causes for the observed clr and torque failures. A post mortem failure analysis procedure was established that minimized the amount of instrumental analysis time required for each part by using electrical test data to help locate the problem area on the circuit board for the failed rotary contact. Once the problem area was located, the following sequence was used: scanning electron microscope with energy dispersion X-ray analysis to electron microprobe analysis to Auger electron spectroscopy to gas chromatograph/mass spectrometer; this sequence provided the maximum data before the sample was destroyed.

A vibratory polishing process was developed that removed embedded aluminum oxide and glass particles from as-received circuit boards and produced surfaces with better than an 8 rms finish. Exposed glass fibers resulting from the molding operation of the switch block assembly was found to be one source of glass particulates. This source was minimized by coating the switch block with a polyurethane material that completely encapsulated all exposed glass fibers. Several piece parts showed dicotylphthalate, stearic acid, an ester wax, and a siloxane contamination. Plasma cleaning cycles were established to remove these organics. The developed plasma process produced parts cleaner than those processed by a four-stage chemical cleaning process.

Manufacturing processes have been altered to minimize identified contaminating particulate and organic sources. To further reduce other contaminants, work is continuing to analyze failed units, to evaluate processing and design changes, and to implement proven processes into manufacturing.

DISCUSSION

SCOPE AND PURPOSE

Bendix Kansas City manufactures a high-reliability switch that requires a very low contact loop resistance (clr) and a low torque input for the rotary contact to function properly. A high rejection rate of these units has been experienced. Preliminary failure analyses of some of these assemblies showed organic films on many of the individual pieces. More detailed failure analyses showed both organic films and embedded particles on the piece parts. The sources of these contaminants were not known, and it was apparent that the standard cleaning techniques being used were not removing them.

The initial purpose of this project was to identify the sources of organic contamination and to evaluate methods of producing cleaner parts. The scope was later changed to include both organic and particulate contamination identification and removal. A binocular microscope, scanning electron microscope (SEM) with energy dispersive X-ray analysis (EDAX), electron microprobe (EMPA), Auger electron spectrometer (AES), and gas chromatograph/mass spectrometer (GC/MS) were used in this effort to identify the various contaminants and to determine the effectiveness of the cleaning processes.

PRIOR WORK

Bendix has done a significant amount of previous work on cleaning and contamination control for manufacturing; however, no single assembly has been examined as extensively as this unit.

ACTIVITY

This small, complex electromechanical unit has many piece parts and subassemblies which makes failure analysis work difficult. Limitations encountered with earlier examinations resulted in establishing a post mortem procedure in which the electrical test data were used to locate the problem area on the circuit board for the failed rotary contact. The clr of the circuit boards is measured along a 360° circle. The electrical data and a map of the individual boards made it possible to look at specific paths and narrow areas for the cause of a high clr, which saved significant SEM/EMPA analysis time. Concurrent with this work, product engineering also developed a technique for opening sealed units that minimized the chances of introducing additional contamination.

An engineering team, consisting of product, process, materials, and test laboratory personnel, was formed to coordinate this activity. The team determined what type of analyses would be needed, evaluated the results obtained, and recommended and implemented process changes where applicable to minimize any identified contaminants.

Analytical Techniques

The analytical procedure involved initial examination of the failed parts in a class 1000 clean room environment under a binocular microscope, using the electrical test data and circuit board map to identify areas of concern. The part was then transferred in sealed petri dishes to the SEM and the problem areas were photographed at appropriate high magnification and EDAX data were run on any observed defects. The part was then inserted in the EMPA where elemental X-ray images were generated on the defects that were observed on the SEM/EDAX. Auger and GC/MS data were run only on parts with no inorganic defects observable with the SEM or EMPA. When organic contaminants were suspect, the GC/MS was used to identify the types of organic species. During this study, the only deviations in this procedure were caused by instrument breakdown.

The order of analyses selected provides an excellent capability for a high (> 2000X) magnification look at the surface topography of a sample and a rapid spot or area elemental analysis. The SEM/EDAX is nondestructive, and usually requires less than 15 to 20 minutes for a single analysis. However, with the available SEM/EDAX equipment, the surface of the sample must be conductive, and the maximum size of the sample is about 2.5 by 2.5 cm. Oxygen and carbon (organic) contamination cannot be detected. Although the EMPA does not have the high magnification capability of the SEM, it can detect oxygen and fairly thick (~100 nm) carbon films and can provide a quantitative elemental analysis. Thin monolayer organic films can be detected only with the AES, and the organic structure identification of these thin organic coatings requires the use of the GC/MS or other analytical equipment.

Circuit Boards

The majority of the clr problems in the failed units occurred between the rotary contacts on the switch block assembly and its two mating circuit boards. The two boards have conductive paths (Table 1) epoxy bonded to anodized aluminum. The conductor paths are flush with the epoxy. One circuit board mates to a four-finger rotary contact, and the other mates to a five-finger rotary contact. The boards are received with a sanded surface finish; sanding provides a flush surface between the conductor and epoxy.

Table 1. Composition of Contact Board and Rotary Spring Contact

Element	Composition (Weight Percent)		
	Circuit Board* (Nominal)	(Range)	Rotary Spring Contact** (Range)
Gold	71.5	70.5 to 72.5	9.5 to 10.5
Platinum	8.5	8.0 to 9.0	9.5 to 10.5
Silver	4.5	4.0 to 5.0	29.0 to 31.0
Copper	14.5	13.5 to 15.5	13.5 to 14.5
Zinc	1.0	0.7 to 1.3	9.5 to 10.5
Palladium			34.0 to 36.0
Total Base Metal Impurities		0.2 maximum	0.2 maximum
Total Platinum Group Impurities***		0.2 maximum	0.2 maximum

*Determined in accordance with ASTM B541-70 (reapproved 1975).

**Determined in accordance with ASTM B540-70 (reapproved 1975).

***Iridium, osmium, rhodium, ruthenium.

Many of the assemblies that are rejected in production fail to meet the clr requirements between these circuit boards and their mating (Table 1) contacts. Failure analyses on these boards have shown both particulate and organic contamination. Figure 1 shows a typical aluminum oxide particle that has become embedded in the contact wear track of a circuit board. Figures 2 and 3 shown embedded glass particles that were observed during SEM analyses. An embedded iron alloy particle is shown in Figure 4.

The locations of these particles correlated with observed high clr readings. Failure analyses of other units showed similar particles which also correlated with high clr readings. This established that embedded particles were the cause of many clr failures. Subsequent evaluation of piece parts prior to assembly showed aluminum oxide particles embedded in the surface of as-received circuit boards, which indicated that not all of the observed particles were being introduced during processing at Bendix. A review of vendor processing indicated that the finished circuit boards were sanded with an aluminum oxide abrasive which accounts for this source of particulate contaminant.

EDAX ANALYSIS

Figure 1. Aluminum Oxide Particle on Circuit Board

Polishing was evaluated as a method of removing these particles. Boards were vibratory polished with jewelers rouge (iron oxide), 1 μm diamond, and 6 μm diamond. The surface finish of the boards improved from a rough sanded surface to a bright, polished surface, as evident in the back-reflected SEM photomicrographs (Figure 5). SEM/EDAX evaluation showed a much cleaner surface. Very few embedded particles were observed on the polished boards, and the

EDAX ANALYSIS

Figure 2. Embedded Glass Fiber Particle
on Circuit Board

few seen were significantly smaller than those seen on unpolished boards. Best results were obtained by vibratory polishing for 20 to 30 minutes with 6 μm diamond, which produced better than an 8 rms surface finish. The rouge tended to leave an iron oxide film, and the 1 μm diamond showed no advantages over the faster polishing 6 μm diamond material.

Several of these polished boards were built into assemblies. Although the surface finish of these parts looked very good,

SEM

10 μm

EDAX ANALYSIS

Figure 3. Embedded Glass Particle on Circuit Board

they showed considerable gold alloy transfer from the board to the rotary contact during run in and testing. Figures 6 and 7 show the dimple part of the contact and the gold alloy transfer that has occurred. This gold alloy transfer caused both high torque and high clr problems.

These results indicated that a rougher surface was needed to minimize gold alloy transfer; therefore, a set of the polished boards was abrasively blasted with dolomite to a matte finish. Because dolomite is a calcium magnesium carbonate material that

Figure 4. Ferrous Alloy Contamination on Circuit Board

is soluble in dilute acids, the blasted boards were dipped in acetic acid to remove all traces of this abrasive. Units with these boards performed well during run in and functional testing;

AS-RECEIVED

100 μm

6- μm POLISHED

100 μm

Figure 5. Surface Finish of As-Received and Polished Circuit Boards

SEM: BACKSCATTER MODE

100 μ m

SEM: BACKSCATTER MODE

100 μ m

Figure 6. Gold Alloy Transfer to Contact Dimple

EMP PHOTOGRAPH

25 μm

GOLD ELEMENTAL MAP

25 μm

SILVER ELEMENTAL MAP

25 μm

OXYGEN ELEMENTAL MAP

25 μm

Figure 7. EMP Photomicrograph and Elemental X-Ray Images of Gold Alloy Transfer to Contact Dimple

therefore, the 6 μ m diamond polishing followed with dolomite blasting procedure has been incorporated into current production.

Several sources of ferrous alloy particles (Figure 4) were found. Some 304L stainless steel particles were caused by weld spatter from sealing the case. Particles of an Fe-Co-Cr-Ni alloy and a Ni-Fe-Co alloy were traced to wear debris particles generated from the spring and pins used near the circuit boards; these particles were created during functional testing of the sealed unit. Minimization of these sources is currently being evaluated.

Switch Block Assembly

The switch block assembly contains a 15-5 PH stainless steel shaft and the gold alloy contacts (Table 1) embedded in a granular glass-fiber-filled molding compound (Figure 8). SEM examination of these parts (Figures 9 and 10) showed exposed glass fibers which were suspect as the primary source of the glass particles that had been found at various locations in the assemblies (Figures 2 and 3). Note that the diameter of the glass particle in Figure 2 matches the diameter of the glass fibers shown in Figure 10. It was determined that these exposed fibers resulted from flashing during the molding operation.

Alternate molding materials and various coating materials were evaluated in an attempt to eliminate subsequent fracturing of the exposed glass fibers. SEM evaluations of these raw materials and their molded parts showed no advantages in changing to a new molding material; therefore, all efforts were diverted to evaluating coating compounds.

The basic idea for coating was to entrap all residual glass fibers or particles in a flexible film. The fibers would adhere to the switch block rather than break off and move around, causing clr problems.

The coating materials selected were polyurethane and two parylenes. Because the base of the flexible contacts would have to be coated along with the molded block to provide a good seal, strips of the contact material were coated and subjected to a mechanical flexing test to determine if there would be any lifting of the coating. Scratch tests were also used to evaluate adhesion. The polyurethane coating was selected as the best. Results indicated that it did not separate from the strip when flex tested for 3700 cycles; in addition, a coated switch block assembly (Figure 11) was built into an assembly which successfully passed 100 complete cycles at -54°C without evidence of fracture at the coating/contact/block interfaces.

Coated assemblies were tested by GC/MS to determine if any outgassing products would be detrimental during long-term storage.

1 mm

1 mm

Figure 8. Macro photograph of Switch Block Assembly

Polyurethane-coated assemblies, both with and without four-stage cleaning, were placed in clean stainless steel containers. The containers were evacuated to 26.6 μ Pa, heated to 149°C, and the outgassing components were analyzed (Figures 12 and 13). A direct probe technique was used to provide maximum sensitivity. These analyses showed that the only outgassing components detected were trichloroethylene, isopropyl alcohol, benzene, and xylene. Although more trichloroethylene was detected for the four-stage cleaned part, all these organics were present only in trace quantities and should not present any problems. Consequently, this coating process also has been incorporated into production.

Plasma Cleaning

Auger analyses were run on several piece parts after the standard four-stage cleaning process. This process is described in the Appendix. The AES spectra show a large carbon peak, indicating that organic contamination on the surfaces of these parts was not being removed during this normal cleaning. Additional parts were analyzed using the GC/MS in an attempt to determine the chemical species of the contaminant. In many cases, the GC/MS analyses were unsuccessful because of an insufficient sample size. In other cases, dioctylphthalate, stearic acid, an ester wax, and a siloxane

100 μm

50 μm

Figure 9. Exposed Glass Fibers on Switch Block Adjacent to Contact

10 μm

Figure 10. SEM Photomicrograph of Exposed Glass Fibers Along Mold Flash Area

contaminant were detected. The source of the siloxane appears to be from the RTV silicone compound used as a sealant on the ceiling tile and clean benches in the manufacturing area. The sources of the other contaminants are not known.

Plasma cleaning¹ had been used successfully to correct other organic contamination problems; therefore, it was evaluated for use on this assembly. Plasma cleaning involves exposing piece parts and subassemblies at a reduced pressure in an electric field to either an argon or oxygen atmosphere. The gas becomes ionized by the electric field, thus creating a plasma that will react with all the surfaces it contacts. Because plasma cleaning is a surface process, it does not affect the bulk properties of the material being treated. However, for electrical contacts and circuit boards, the chemical nature of the surface is critical. For instance, an oxygen atmosphere might result in a faster removal process for organics, but a higher-powered electric field is required which can cause oxidation of metal surfaces. Argon plasma cleaning will produce a surface free of both organic contamination and oxides, but requires a much longer cleaning cycle.

Figure 11. SEM Photomicrographs of Exposed Glass Fibers Encapsulated in Polyurethane Coating

100 μm

100 μm

Figure 11 Continued. SEM Photomicrographs of Exposed Glass Fibers Encapsulated in Polyurethane Coating

Figure 12. Mass Spectrum of Outgassing Products From Four-Stage Cleaned Switch Block

Figure 13. Mass Spectrum of Outgassing Products From Switch Block

Various piece parts and subassemblies were subjected to different plasma cleaning cycles and evaluated for surface contamination using AES analysis and electrical test data. The flow gas, applied wattage, vacuum level, and time of cleaning were varied to determine their effect on the finished part. The results of these tests indicated that oxygen cleaning was unacceptable for the metallic parts on this unit. Even contacts made from the precious metal alloy became oxidized. Therefore, the metallic parts had to be cleaned using an argon plasma. The ceramic parts, however, were cleaned very well using the oxygen cycle that had been developed by Sandia Laboratories, Albuquerque.²

Plasma cleaning was incorporated into production as a result of these tests. Table 2 lists the piece parts and the plasma cleaning cycles currently being used. These conditions result in parts that are electrically acceptable and cleaner than those only four-stage cleaned.

ACCOMPLISHMENTS

A failure analysis procedure has been developed to find a specific cause for clr problems. This procedure incorporates electrical test data and a map of the circuit board to assist the instrumental analyses. Also, the established sequence of SEM/EDAX to EMPA to AES or GC/MS provides the maximum amount of data before the sample is destroyed. This procedure has confirmed that embedded particles and organic contamination caused observed failures in production units.

A circuit board vibratory polishing process was developed that produced a cleaner surface on the conductor paths. Using 6- μ m diamond and 20 to 30 minutes of polishing times, a surface finish of better than 8 rms was produced. The polished boards had fewer embedded silicon and aluminum oxide particles when compared to the as-received parts from the vendor.

To reduce gold alloy transfer observed with these polished surfaces, the surfaces were given a uniform matte finish by blasting with dolomite, followed by a hot acetic acid dip, double water rinse, and four-stage cleaning. Acceptance tests of these polished and matte-finished units were favorable and resulted in incorporating these processes into the manufacturing operations.

A polyurethane coating process was developed for the switch block assembly. This coating encapsulated exposed glass fibers, preventing them from breaking off and causing clr problems in the sealed unit. This coating will also pass specified temperature cycle tests without outgassing and will withstand 100 unit test cycles at -54°C without loss of adhesion.

Table 2. Plasma Cleaning Conditions for Piece Parts and Subassemblies

Name	Conditions
Ceramic	30 min, 50 W, 53.33 Pa, 75 cm ³ /min oxygen flow
Switch Block	120 min, 20 W, 33.33 Pa, 50 cm ³ /min argon flow
Housing and Housing Plate Assembly	180 min, 20 W, 33.33 Pa, 50 cm ³ /min argon flow
Solenoid Coil	30 min, 20 W, 33.33 Pa, 50 cm ³ /min argon flow

Plasma cleaning cycles were determined for several piece parts. Both oxygen and argon plasmas were used to remove identified organic contaminants from ceramic and metallic pieces, respectively. The plasma treated parts were cleaner than those subjected to the standard four-stage cleaning process.

FUTURE WORK

Although this study has resulted in implementing many process improvements on this very complex electromechanical unit, more efforts will be required on failure analysis and additional manufacturing improvements. The task team will concentrate on continuing post mortem analyses of failed units, evaluating processing and design changes, and implementing proven processes into manufacturing operations.

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- ²J. A. Wagner, Cleaning Techniques for Al₂O₃ Ceramic Wafer. Albuquerque: Sandia Corporation Report SAND 78-0734, July, 1978.

Appendix

FOUR-STAGE CLEANING PROCESS

1. Stage I. Clean ultrasonically in trichloroethylene for 2 to 3 minutes, then blow dry with 0.3 μ m filtered dry nitrogen.
2. Stage II. Clean ultrasonically in detergent solution for 2 to 3 minutes, then perform the next operation (Stage III) immediately after removing parts from this solution. The detergent solution shall be prepared by mixing thoroughly, in the following proportions by volume.

Reference: 18.92 L Mixture

80 parts isopropyl alcohol	15.14 L
20 parts distilled or deionized water	3.78 L
0.2 parts liquid surfactant, polyoxyethylene nonionic wetting agent	38 mL
0.05 parts liquid surfactant	9.5 mL

There should be no turbidity after mixing.

CAUTION

DO NOT PERMIT THIS SOLUTION TO DRY ON PARTS.

3. Stage III. Clean ultrasonically in deionized water for 2 to 3 minutes, then blow dry with 0.3 μ m filtered dry nitrogen.
4. Stage IV. Clean ultrasonically in isopropyl alcohol for 2 to 3 minutes, then blow dry with 0.3 μ m filtered dry nitrogen.
5. Vacuum bake at 77°C for at least 60 minutes. The pressure shall be less than 25 mm of mercury.

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