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COMPARISON OF METHODS FOR CLEANING
CONTAMINATED SURFACES

PDO 6984874, Topical Report

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**Kansas City
Division**

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COMPARISON OF METHODS FOR CLEANING CONTAMINATED SURFACES

BDX-613-1048 (Rev.), UNCLASSIFIED Topical Report, Published
May 1974

Prepared by L. C. Jackson, D/814, under PDO 6984874

A study was made of alternate cleaning procedures used in the manufacture of electronic assemblies. Two entirely different means of evaluating the effectiveness of the cleaning procedures on plastics and metals were utilized: determination of strength of bonded specimens, and contaminant detection by evaporative rate analysis. Interpretive analyses are presented to explain the apparent conflict of conclusions yielded by the testing techniques. Insight was obtained into the effectiveness of cleaning procedures used to remove contaminants.

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THE BENDIX CORPORATION

KANSAS CITY DIVISION

KANSAS CITY, MISSOURI

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SUMMARY

Alternate cleaning procedures currently in use or proposed for use in manufacture of electronic assemblies were evaluated. A moldable polyester and fiberglass laminate printed circuit board was used for one comparative evaluation, while aluminum metal was used for another. Contaminants (solder flux, mold release, tape residue) were used to evaluate the effectiveness of the solvent cleaning procedures. Comparative evaluation was made by strength tests and by an evaporative rate analysis technique. Data and interpretive analyses are given. Alternate conclusions are presented.

DISCUSSION

SCOPE AND PURPOSE

The purpose of this work is to compare various cleaning procedures currently used by Bendix Kansas City Division or proposed by the Design Agency for the manufacture of certain electronic assemblies. The project encompasses a study of various contaminated surfaces, methods for removal of contaminants, and evaluation by bond strength tests and evaporative rate analysis.

ACTIVITY

Metals, plastics, and ceramics are used in the manufacture of certain assemblies. Certain areas require optimum adhesion of potting and adhesive materials. There are at least six conditions when adhesion is critical to the operation of the assembly. The three most critical areas involved the following substrates: a Celanex 3300 polyester (Celanese Corporation), 6061 aluminum alloy, and the epoxy side of a circuit board (no copper or aluminum foil present). The evaluation of cleaning procedures using these adherends would be expected to provide the necessary information that would basically relate to the other substrates.

There are two comparisons being studied. One evaluation compares the processes used for cleaning plastic surfaces; the other compares the procedures for cleaning aluminum metal. The alternate solvent cleaning procedures used for the polyester and the printed circuit board laminate are given in the appendix as Process A (currently in use) and Process D (proposed). Both processes are described in Drawing SS 297959.

The surface preparation of the aluminum alloy was a comparison of a sandblasting procedure with and without an additional solvent cleaning (that is, the nitrogen blow dry plus Process A currently in use and the proposed nitrogen blow dry only). Process A utilizes three solvents (trichlorotrifluoroethane, trichloroethylene, and isopropyl alcohol) which cover the entire solubility parameter range of most organic materials. Process D utilizes an ultrasonic technique with two solvents which cover the middle to upper position of the solubility parameter range.

The evaluation of the metal surface preparation is a comparison of a sandblasting operation involving a Number 1 flint-blasting material (Independent Gravco Company) with and without a subsequent solvent cleaning operation.

Three materials were selected to contaminate the surfaces under investigation; these were a silicone mold release (Ram Chemicals, 225), a solder flux (Alpha Metals Incorporated, Alpha 611), and a residue transfer from a pressure sensitive tape (3M Company, 470 Electroplating Tape). In addition, however, another contaminant must be considered as being present. Both the polyester and the aluminum metal were blasted with Number 1 flint. Ordinarily, the blasting of aluminum yields a light-gray colored, matte finish surface; the polyester is normally a light cream color. In both cases, the surfaces were gray to black after being blasted. The dark particles could be rubbed off using a solvent dampened cloth. Evaporative rate analysis confirmed the presence of an unknown organic contaminant.

Surface preparation and cleaning processes of the plastics and the metal coupons were performed in fireset production areas. After deliberate contamination, the coupons of polyester, printed circuit board, and aluminum were cleaned using the respective alternate procedures. Evaporative rate analysis data were obtained by a Meseran Surface Analyzer on the surface condition of the plastics and metals before and after contamination.

The production cleaned specimens were bonded for subsequent shear strength tests at room temperature at a separation rate of 0.05 inches (1.27 mm) per minute. The plastic coupons were bonded with Epon 828 (Shell Chemical Company) diethanolamine (100/12 parts by weight) and cured for 15 hours at 160°F (71°C). This condition represents the potting resin system without the glass microballoon filler material. The aluminum specimens were bonded with the adhesive Epon 828/Versamid 140 (General Mills) (2/1 parts by weight) and cured at room temperature for several days.

Contaminants on the surfaces were detected by evaporative rate analysis. Three radioactive solvents were used to assure accuracy of contaminant detection and provide other information for interpretation. Tests were made at 10 percent humidity and at 72°F (22°C).

Bond Strength

The shear strength of bonded polyester and circuit board surfaces can be used to compare the effectiveness of contaminant removal by the solvent cleaning procedures of Process A and Process D (SS 297959). The results are shown in Tables 1 and 2.

These data show that both cleaning procedures yield equivalent strength values and provide data similar to those obtained from the as-received surfaces before contamination. Tape residue on the laminate did not influence strength data, but the flux and the mold release did lower values considerably.

Table 1. Comparison of Alternate Procedures for Cleaning Contaminated Surface of Polyester Celanex 3300, Determined by Strength Data

Contaminant	Shear Strength PSI (MPa)		
	No Surface Cleaning	Process A Cleaned	Process D Cleaned
As-Received, Not Grit- Blasted	640 (4.413) 560 (3.861) 608 (4.192) 528 (3.640) 628 (4.330) 593 (4.089)**		
As-Received, Grit- Blasted	1340 (9.239) 1304 (8.991) 1216 (8.340) 1236 (8.522) 1280 (8.825) 1275 (8.797)**		
Tape Residue on Top of Grit-Blast	*	1200 (8.274) 1260 (8.687) 1192 (8.218) 1272 (8.770) 1136 (7.832) 1212 (8.356)**	1340 (9.239) 1224 (8.439) 1260 (8.687) 1212 (8.356) 1336 (9.211) 1274 (8.784)**
Mold Release Top of Grit- Blast	*	946 (6.522) 908 (6.260) 984 (6.784) 1120 (7.722) 1048 (7.226) 1001 (6.902)**	1216 (8.384) 1240 (8.549) 1184 (8.163) 1200 (8.274) 1198 (8.260) 1208 (8.329)**
Solder Flux on Top of Grit-Blast	*	1152 (7.943) 1360 (9.977) 1296 (8.966) 1164 (8.025) 1288 (8.880) 1252 (8.632)**	1360 (9.377) 1328 (9.156) 1224 (8.439) 1304 (8.991) 1376 (9.487) 1318 (9.087)**
*Insufficient number of specimens available for test. **Mean Average			

Table 2. Comparison of Alternate Procedures for Cleaning Contaminated Surfaces of Epoxy Circuit Board Laminate, Determined by Strength Data

Contaminant	Shear Strength PSI (MPa)		
	No Surface Cleaning	Process A Cleaned	Process D Cleaned
As-Received	2560 (17.651) 2320 (15.996) 1968 (13.569) 2120 (14.617) 2380 (16.410) 2270 (15.651)*		
Tape Residue	2328 (16.051) 2532 (16.768) 1940 (13.375) 2640 (18.202) 2470 (17.030) 2362 (16.285)*	2320 (15.996) 2160 (14.893) 2076 (14.314) 2088 (14.396) 2268 (15.637) 2182 (15.044)*	2460 (16.961) 2168 (14.948) 2000 (13.790) 1940 (13.376) 2408 (16.603) 2195 (15.134)*
Mold Release	308 (1.434) 200 (1.379) 360 (2.482) 344 (2.372) 386 (2.661) 300 (2.068)*	2108 (14.534) 2130 (14.686) 2160 (14.893) 1960 (13.514) 2508 (17.292) 2173 (14.982)*	1960 (13.514) 2100 (14.479) 2200 (15.168) 2256 (15.555) 2535 (17.485) 2210 (15.237)*
Solder Flux	880 (6.067) 1130 (7.791) 808 (5.570) 1160 (7.998) 600 (4.137) 916 (6.316)*	2364 (16.299) 2520 (17.375) 1764 (12.162) 2136 (14.727) 2240 (15.444) 2205 (15.202)*	2240 (15.444) 2114 (14.576) 2328 (16.051) 2460 (16.961) 2176 (15.003) 2264 (15.610)*
*Mean Average			

A comparison of the metal surfaces (Table 3), on the other hand, shows that a solvent cleaning procedure does improve bond strength. Unfortunately, the procedures used on grit-blasted surfaces also tended to lower the strength values below those obtained before the cleaning operation.

Table 3. Comparison of Alternate Procedures Used for Cleaning Contaminated 6061 Aluminum Metal Surfaces, Determined by Strength Data

Contaminant	Shear Strength, PSI (MPa)		
	No Surface Cleaning	Blow Dry Only	Blow Dry Followed by Process A Cleaning
Bare Metal As-Received	660 (4.551)	600 (4.137)	940 (6.481)
	880 (6.067)	604 (4.164)	900 (6.205)
	848 (5.847)	606 (4.178)	1064 (7.336)
	608 (4.192)	584 (4.027)	956 (6.591)
	1088 (7.501)	664 (4.578)	1044 (7.198)
	817 (5.633)*	611 (4.213)*	981 (6.764)*
Grit-Blasted Only	2108 (14.534)	1864 (12.852)	1888 (13.017)
	2468 (17.016)	1556 (10.728)	1860 (12.824)
	2108 (14.534)	2268 (15.637)	2320 (15.996)
	2168 (14.948)	2060 (14.203)	1328 (9.156)
	2288 (15.775)	1948 (13.431)	2252 (15.527)
	2228 (15.775)*	1939 (13.369)*	1930 (13.307)*
Tape Residue on Top of Grit-Blast	1540 (10.618)	1524 (10.508)	1368 (9.432)
	1708 (11.776)	1320 (9.101)	1836 (12.659)
	1720 (11.859)	1268 (8.743)	1560 (10.756)
	1760 (12.135)	1480 (10.204)	1860 (12.824)
	2180 (15.031)	1436 (9.901)	1688 (11.638)
	1782 (12.286)*	1406 (9.694)*	1662 (11.182)*
*Mean Average			

Evaporative Rate Analyses

The Meseran analysis of contaminant removal by the alternate cleaning procedures contradicts the strength data in some cases. It provides more insight into the understanding of the permanence of adhesion.

Polyester Surfaces

Part of the problem with the analysis of the Number 1 flint-blasted polyester surfaces is the presence of an unknown contaminant. Solid materials normally yield low Meseran radioactivity counts; organic residues and uncured materials, on the other hand, yield high values. Very high Meseran values were obtained with flint-blasted materials. These data are given in Table 4.

Table 4. Radioactivity Count Values of Glass Fiber Filled Polyester (Celanex 3300) Surfaces With and Without Contamination

Polyester Surface Condition	Meseran Numbers (Radioactivity Counts)								
	Surface Before Process A or D Cleaning Procedures			After Process A Cleaning			After Process D Cleaning		
	Tridecane	Tetrabromo-ethane	Diethyl-succinate	Tridecane	Tetrabromo-ethane	Diethyl-succinate	Tridecane	Tetrabromo-ethane	Diethyl-succinate
As-Received	865	129	124						
Grit-Heavy-Blast (dark colored)	1054	527	1061						
Light-Blast (light colored)	1545	333	1197						
Solder Flux Grit-Blasted (heavy)	3012	1584	1287	803	774	1033	719	689	926
Mold Release on Grit-Blast (heavy)	1625	751	1425	348	461	807	419	672	950
Masking Tape Residue on Grit-Blast (heavy)	778	825	873	312	679	631	838	889	1006

Process A appears to be more effective in removing tape residues than Process D. The advantage of A over D is somewhat less when Ram 225 is present. The difference between the two methods in removing Alpha 611 flux is not great, although Process D is slightly better. There is an indication that both cleaning processes removed some of the unknown organic residue accompanying the flint-blasting material.

Circuit Board

Process D was the most effective cleaning procedure for removing all contaminants as indicated by the very low Meseran radio-activity counts (Table 5). The data also indicate that some of the resist may have still been on the circuit board prior to the cleaning process.

6061 Aluminum

The normally expected low Meseran values for a solid material were not obtained with the flint-blasted aluminum, thereby indicating the presence of a contaminant. Neither process was found to be effective in the removal of the tape residue or the contamination from the flint-blast (Table 6).

Theoretical Considerations

Although the evaporative rate analysis data show contaminants are present on surfaces, the actual bond strength data indicate a lack of contamination. It has always been assumed that if a contaminant is present on a surface to be bonded, the subsequent strength values would be expected to be less than those of a contaminant-free surface. There is now an apparent conflict of results depending upon the method of analysis used. In effect, however, while strength tests serve to indicate that a cleaning procedure will provide an immediate acceptable bond strength, the evaporative rate analysis raises the question as to the permanence or durability of the bond over a period of time.

The explanation for the apparent disparity may be provided by the electron transfer theory. Metal is believed to be constructed as a lattice-work within which electrons move freely about. Electron freedom in plastics is severely restricted as compared to metals. The potential for electron loss is greater for metals than for plastics. Thus, the attractive force between a metal and an adhesive would be much greater than the attractive force between a plastic surface and an adhesive. It is noted that masking tape residue transfers readily to the aluminum and very poorly to the laminate circuit board. If a residue is not too many molecular layers thick, greater strength potential would be expected with metal-contaminant-adhesive bonds than with plastic-contaminant-adhesive bonds. The data in the report tend to support the theory.

Table 5. Radioactivity Count Values of Epoxy Circuit Board Laminate Surfaces With and Without Contamination

Epoxy Glass Laminate Surface Condition	Measure Numbers (Radioactivity Counts)								
	Surface Before Process A or D Cleaning Procedures			After Process A Cleaning			After Process D Cleaning		
	Tridecane	Tetrabromo-ethane	Diethyl-succinate	Tridecane	Tetrabromo-ethane	Diethyl-succinate	Tridecane	Tetrabromo-ethane	Diethyl-succinate
As-Received After Photoresist Removed by Production	130	229	777						
Solder Flux	431	608	1142	318	564	739	154	198	360
Mold Release	693	822	1416	459	508	813	131	198	324
Tape Residue	282	635	808	529	528	537	173	188	293

Table 6. Radioactivity Count Values of 6061 Aluminum Surfaces With and Without Contamination

6061 Aluminum Surface Condition	Measure Numbers (Radioactivity Counts)								
	Surface Before Blow Dry and Cleaning Procedures			Blow Dry Only			Blow Dry Followed By Process A Cleaning		
	Tridecane	Tetrabromo-ethane	Diethyl-succinate	Tridecane	Tetrabromo-ethane	Diethyl-succinate	Tridecane	Tetrabromo-ethane	Diethyl-succinate
As-Received	1560	412	804	659	311	286	287	197	273
As-Received, Trichloro-ethylene Solvent Cleaned	448	215	495						
Grit-Blast	1144	505	1239	769	660	1214	1036	808	1301
Tape Residue on Grit-Blast	1911	1008	1117	1257	1402	945	1270	755	1398

Theoretically, a molecular layer of a contaminant on a surface should have an adverse effect on adhesion. It may be ascertained that it does since, in fact, the strength data in all cases are not uniform, showing a wide variation in each set of specimens. This, in itself, is an indication of poor surface preparation.

ACCOMPLISHMENTS

Alternate cleaning procedures have been evaluated on three different substrates using strength tests and evaporative rate analysis as methods for comparative analysis.

The strength data used as a criterion for selecting Process A or D (Drawing SS 297959) for cleaning the epoxy surface of printed circuit boards or aluminum reveals both procedures are acceptable. Therefore, since Process A is already in use, Process A will continue to be used. Process D is currently available for use on the specification but will not be used at this time.

The strength data shows that the solvent procedure (Process A) should be used after surface blow dry in the cleaning of aluminum. As a result, all parts now utilize solvent cleaning; nitrogen blow dry alone is not accepted as a cleaning technique.

Neither Process A nor Process D was completely satisfactory as a cleaning procedure for the polyester when evaluated by evaporative rate analysis. Process D was more effective in contaminant removal from the epoxy board than Process A. Further studies are required relating contaminant removal, strength values, and evaporative rate analysis, and will be considered in accordance with future plans.

Verification of the effectiveness of SS 297959 cleaning and handling has been made.

Sandblasting has been identified as an operation that needs more control.

Appendix

PROCESS CLEANING PROCEDURES

PROCESS A (SS297959)

Spray clean with trichloroethylene.

Immerse in isopropyl alcohol for 15 seconds.

Spray with trichlorotrifluoroethane for 15 seconds.

Blow dry with dry nitrogen.

Vacuum bake at 160°F (71°C) for 30 minutes.

PROCESS D (SS 297959)

Immerse in trichloroethylene and then ultrasonically clean in isopropyl alcohol for 2 to 3 minutes.

Blow dry with nitrogen.

Repeat above steps.

Spray clean with trichloroethylene for 15 seconds.

Blow dry with dry nitrogen.

Vacuum bake at 160°F for 30 minutes.