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LOW- AND NO-VOC CONFORMAL COATINGS OVER NO-CLEAN FLUX RESIDUES Edward A. Shearls SAIC 714 N. Senate Avenue Indianapolis, IN 46202-3112

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INTRODUCTION

The use of conformal coatings over Printed Wiring Assemblies (PWAs) today presents three manufacturing challenges. First, in response to the impending phaseout of chlorofluorocarbon-based solvents, low residue or no-clean fluxes have been developed. These fluxes are advertized as leaving no, or very little benign residue after the soldering process, but the main concern is whether these benign residues interfere with conformal coating adhesion to PWAs. Second, most conformal coatings in use release significant amounts of volatile organic compounds (VOCs) during coating application. The use of new conformal coatings with lower VOC content and less environmental impact is rapidly becoming an important issue. The Clean Air Act of 1990 drives the eventual use of no- or low-VOC conformal coatings with negligible environmental impact. The third challenge is to determine whether it is feasible and practical to apply a low-VOC coating over a no-clean flux.

This paper describes results of a three phase effort that addresses these challenges completed at the Electronics Manufacturing Productivity Facility (EMPF). Phase 1 evaluated the adhesion and performance of current (not low-VOC) acrylic, polyurethane, silicone, and parylene conformal coatings applied over test pallets manufactured with no-clean (low residue) fluxes and pastes. Phase 2 evaluated the use of currently available low-VOC conformal coatings applied over commonly used RMA and water soluble fluxes and pastes. Environmental stress screening (ESS) tests were performed in both phases to down select no-clean materials and low-VOC coatings for further testing in Phase 3, the application of low-VOC coatings over no-clean fluxes and pastes on functional boards (PWAs).

METHODOLOGY

Preliminary Compatibility Testing

Preliminary compatibility testing was performed to ensure test pallet (Phase 1 and 2) and functional test board (Phase 3) base materials (fluxes, pastes, base metals, laminate, and solder mask combinations) were compatible. Five RMA fluxes, four water-soluble fluxes, seven RMA pastes, and six water-soluble pastes comprised the matrix of representative common fluxes and pastes tested. A popular liquid photoimageable (LPI) and a popular dry film solder mask were tested.

Fluxes were applied to test pallets using individual spray bottles. Pastes were printed using an 80-mesh, 10-mil-thick screen. Pallets were wave soldered or IR reflowed according to manufacturers' technical literature. Those made with RMA materials were cleaned with 10% Armakleen E-2001 detergent. Those made with water-soluble materials were cleaned with DI water only. The flux and

paste producing the best soldered pallets were used to make Phase 2 test pallets and Phase 3 PWAs.

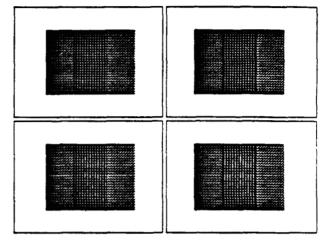
Test Pallets and Coupons

The test pallets used in the Phase 1 and Phase 2 efforts were 4.00 inches by 5.00 inches $(10.20 \times 12.75 \text{ cm})$ and had four test coupons to a pallet, with break-away tabs between the coupons (see Figure 1). Each test coupon contained a 1.00 inch by 1.50 inch $(2.54 \times 3.80 \text{ cm})$ copper rectangle with a 0.50

inch by 1.00 inch (1.27 x 2.54 cm) solder mask strip down the center. One half of the test pallets had coupons with LPI film solder mask, the other half had dry film solder mask. The majority of the coupons had bare copper on the areas adjacent to the solder mask. The remainder had hot air solder leveled (HASL) on the areas adjacent to the solder mask. The laminate was FR4 material.

The masks and the metal surfaces allowed the base metal/mask test coupon combinations:

- Copper/Photoimageable Liquid
- Copper/Dry Film
- HASL/Photoimageable Liquid
- HASL/Dry Film





Including the FR4, this arrangement allowed evaluation of conformal coating adhesion to five different substrates and interfaces after application of the various flux residues.

Phase 1. No-Clean Flux Evaluation

It was impossible to evaluate every standard (not low-VOC) conformal coating and no-clean flux. One acrylic, one polyurethane, one silicone and one parylene were randomly selected to represent their respective coating families. The coatings were applied at Specialty Coating Services of Indianapolis, Indiana. Flux and paste selection were more complicated because of the broadness of the no-clean definition. The materials available were grouped into resin, rosin, or rosin/resin free categories, and further divided by solids content, activator and carrier. Twelve pastes and eleven fluxes were chosen to represent the no-clean industry.

Liquid fluxes were applied to the test pallets with a high pressure spray system designed for noclean fluxes. Precision Dispensing Equipment of Bay Village, Ohio brought the system to the EMPF and operated it. The fluxes were processed on a nitrogen inerted wave soldering machine using a profile recommended by material vendors. No-clean solder pastes were printed to the test pallets and reflowed in a nitrogen environment using forced convective air reflow and a profile recommended by the material vendor.

In Phases 1 and 2, representative pieces of the conformal coated coupons were visually examined and tested for adhesion. Additional samples were subjected to ESS tests which included either temperature/humidity, thermal cycle, thermal shock, salt fog, a sequence of all tests, or no test. The stress tests and conditions used are listed in Table 1.

TABLE 1 ENVIRONMENTAL SCREENING TEST SUMMARY					
Stress Test	Stress Test Specification Conditions				
Temperature/Humidity	MIL-STD-810D Method 507.2	168 hours, 30 to 60° C; 85-95% relative humidity			
Thermal Cycling	IPC-TM-650 Method 2.6.6	24 hours, -55 to +125° C; 30 min per temp and 15 min dwell at 25° C between each ramp			
Thermal Shock	MIL-STD-202F Method 107D	24 hours, -55 to +125° C; 15 min per temp.			
Salt/Fog	MIL-STD-810D Method 509.2	48 hours, 35° C			

. ASTM D3359 was used to measure conformal coating adhesion, modified to eliminate as much of the subjectivity as possible. Instead of a hand-held cutting tool, an IBM robot was fitted to hold the cutting tool and the test coupon and programmed to perform the actual cutting. The coupon was cut and then rotated 90 degrees and cut again to form a lattice pattern. A specified tape was pressed over the area and then removed. Use of the robot gave uniform depth, pressure and lattice formations. The same technician performed all tape applications and ratings. Adhesion was rated for each substrate on a 5 to 0 point system, with 5 best and 0 worst as shown in Table 2.

TABLE 2	ADHESION GRADING SCHEME
GRADE	CRITERIA
5	The edges of the cuts are completely smooth; none of the squares of the lattice is detached.
4	Small flakes of the coating are detached at intersections; less than 5% of the area is affected.
3	Small flakes of the coating are detached along the edges and at intersections of cuts. The area is 5-15% of the lattice.
2	Coating has flaked along the edges and on parts of the squares. The area affected is 15- 35% of the lattice.
1	Coating has flaked along the edges of cuts in large ribbons and whole squares have detached. The area affected is 35-65% of the lattice.
्०	Flaking and detachment worse than grade 1.

Phase 2. Low-VOC Conformal Coating Evaluation

The most compatible RMA and water soluble fluxes and pastes, determined by preliminary compatibility testing, were used to make test pallets as described in that section for low-VOC conformal coating evaluation. Fourteen different conformal coatings representing the generic coating groups were applied to test pallet replicates (ten of each type shown in Table 3).

TABLE 3. TEST PALLET MATRIX					
Solder Mask	Flux		Paste		
	RMA • Water Soluble		RMA	Water Soluble	
Liquid Film	x	x	х	x	
Dry Film	x	x	х	x	

After a literature review, it was decided that LPI solder mask pallet results were the primary criteria for flux, paste, and conformal coating selections in this project, as LPI solder mask would be used on the pallets for the Phase 3 (no-clean materials with low-VOC conformal coatings) effort. LPI solder mask has been shown the most compatible with no-clean fluxes¹ and more compatible with conformal coats than other types². A solder mask comparison³ reports LPI masks reduce tombstoning and solder balls, withstand multiple reflow cycles, and are easily cleaned. Several papers describe favorable solder ball dynamics with LPI masks ^{4,5,6,7}.

The coatings were evaluated using the environmental screening tests described earlier to determine the most promising one from each generic group to use in the Phase 3 effort.

Phase 3. Low-VOC Conformal Coating Over No-Clean Flux Evaluation

Populated boards (PWAs) were manufactured for the Phase 3 evaluation using the liquid film photoimageable solder mask and the laminate from earlier testing. The no-clean fluxes and pastes that graded best in the Phase 1 effort was used for one set of these boards. The RMA fluxes and pastes and the water soluble fluxes and pastes selected in the compatibility pretesting were used to manufacture two additional sets of populated boards. A set of not-populated boards served as a final control set.

The RMA and water soluble paste and flux combinations representing current industry conditions and the no-clean fluxes and pastes used in the Phase 3 effort are shown in Table 4. These fluxes and pastes demonstrated the best adhesion with the traditional conformal coatings on the test coupons.

The low-VOC conformal coatings chosen for Phase 3 are shown in Table 5. These conformal coatings demonstrated the best adhesion when used with traditionally fluxed and cleaned test coupons.

Table 6 shows the ESS test fate for each board in a 12 replicate set. One board in each group was not ESS tested.

TABLE 4 F	LUX & PASTE SELECTION				
	PASTE	FLUX			
RMA	RMA Alpha RMA 209 Alpha 615 RMA				
ws	WS Amtech WS-465XT Lonco Organo Flux 3355 VF				
No-Clean	AIM LR5 AIM Base I	Alpha 970S Kester 970S Hi-Grade 3570-T Alpha NR200			

TABLE 5 CONFORMAL COATING SELECTED				
Conformal Coating Type Conformal Coating Selected				
Acrylic	Quick Cure 576			
Urethane	Dymax 986			
Ероху	Envibar 1244T			
Silicone	Loctite 5290			
Parylene	Parylene C			

TABLE 6 BOARD TESTING SCHEME				
BOARD NUMBER	ESS TEST			
1-3	SIR			
4-6	Sequential ESS			
7	No ESS			
8	Humidity Only			
9	Thermal Cycle Only			
10	Thermal Shock Only			
11	Salt Fog Only			
12	Spare PWA			

PWAs were visually examined and tested for adhesion in four different locations, (two on the top-side and two on the bottom-side). The slightly modified version of ASTM D3359 described earlier was used to measure conformal coating adhesion. Adhesion was rated using the grading system shown in Table 2.

RESULTS

Visual Inspection of Conformal Coatings

Visual Inspection Of Acrylic Coating

Control PWAs (non-populated PWAs) had minor dewetting on both sides and some coating discoloration. Major dewetting occurred on the component side of all processed (fluxed) assemblies, and the Plastic Leaded Chip Carriers (PLCCs) and other chips. PWAs manufactured with RMA and no-clean paste and flux also showed poor adhesion of the conformal coating on the PLCCs after being environmentally stressed. Lighter colored PLCC areas could be flaked easily using a probe or a fingernail. Acrylic coatings on the no-clean assemblies were discolored and were "bubbled" on the bottom side. PWAs made with water soluble flux and paste had good, uniform coating coverage on their bottom sides.

Visual Inspection Of Polyurethane Coating

Minor dewetting on the PLCCs and chips occurred on the boards made with RMA and no-clean paste and flux. The no-clean paste and flux displayed dewetting around the component pad areas. Control PWAs and PWAs manufactured with water soluble paste and flux displayed coatings with good, uniform coverage.

Visual Inspection Of Parylene Coating

All PWAs displayed good, uniform Parylene coatings.

Visual Inspection Of Epoxy Coating

The coating on all PWAs displayed major dewetting on the component side and the PLCCs and chips. The PLCCs exhibited adhesion problems after environmentally stressing. PWAs manufactured with RMA or water soluble paste and flux displayed good, uniform Epoxy coatings on their bottom sides. PWAs manufactured with no-clean paste had slight dewetting on their bottom sides.

Visual Inspection Of Silicone Coating

Control PWAs and all PWAs manufactured with RMA or water soluble paste and flux produced Silicone coatings with good, uniform coverage. PWAs manufactured with no-clean paste and flux displayed slight dewetting around component pad areas.

Adhesion of Conformal Coatings After Environmental Stress Testing

The top and bottom board adhesions for each PWA were rated on a 0-5 scale, then converted to a percent (100 percent maximum). Table 7 shows the overall average adhesion by coating for each ESS test. Table 8 shows the average percent adhesion for each coating as a function of the paste/flux type for each ESS test.

Adhesion of Acrylic Coating after ESS

The PWAs that went through only Humidity and only Salt Fog tests had average adhesions of 25 percent (see Table 7). No paste/flux material did well but values for boards made with no-clean materials were exceptionally poor (see Table 8). PWAs that went through all environmental stresses had an average adhesion of 55 percent, again because of poor adhesion for no-clean material boards. PWAs that saw no environmental stresses had an average adhesion of 61 percent. Thermal shock and thermal cycling tests, with resultant average adhesions of 86 and 90 percent respectively, had the least effects on PWA adhesion.

Analyzed in overall terms of paste/flux materials (see Table 8), the acrylic coated PWAs made with RMA and the water soluble paste and flux had adhesion averages of 64 and 62 percent, respectively. The control PWAs had an average adhesion of 65 percent. The no-clean paste and flux had the lowest adhesion average (37 percent).

TABLE 7 AVERAGE ADHESION BY ESS TEST vs COATING TYPE								
	ESS TEST							
COATING	HUMID	HUMID T.CYCLE T.SHOCK SALT FOG ALL NON						
ACRYLIC	25	90	86	25	55	61		
URETHANE	69	78	79	78	71	75		
PARYLENE	99	90	85	80	96	9 8		
EPOXY	91	89	86	50	80	86		
SILICONE	69	64	61	65	74	80		
average	71	82	81	61	81	80		

Adhesion of Urethane Coating after ESS

Urethane coatings produced boards with average adhesions from 69 to 79 percent (Table 7). The PWAs that went through humidity stress only had an average adhesion of 69 percent. PWAs that went through all environmental stresses had an average adhesion of 71 percent. PWAs that saw no environmental stresses had an average adhesion of 75 percent. PWAs that went through only thermal cycling, or thermal shock, or salt fog, had average adhesions of 78 or 79 percent.

In terms of paste/flux materials (Table 8), average adhesions ranged from 69 to 80 percent. The control PWAs had an average adhesion of 69 percent. The PWAs manufactured with RMA paste and flux had an average adhesion of 80 percent. The PWAs manufactured with water soluble paste and flux had an average adhesion of 77 percent. The PWAs manufactured with no-clean paste and flux had an average adhesion of 73 percent.

Adhesion of Parylene Coating after ESS

The average salt fog adhesion value of 80 percent (Table 7) for PWAs coated with Parylene is a reflection of the poor adhesion (30 percent average) on PWAs made with RMA (Table 8). Parylene coated PWAs that went through only thermal shock testing had an average adhesion of 85 percent.

PWAs that went through only thermal cycling had an average adhesion of 90 percent. PWAs that saw no environmental stresses had an average adhesion of 98 percent. PWAs that went through humidity and PWAs that saw all environmental stresses had average adhesions of 99 and 96 percent, respectively.

In terms of paste/flux materials (Table 8), the control PWAs had the best adhesion (99 percent), followed by PWAs manufactured with water soluble paste and flux (93 percent average adhesion). PWAs manufactured with RMA paste and flux had average adhesion of 87 percent. The PWAs manufactured with low-residue paste and flux had a comparable adhesion of 84 percent.

Adhesion of Epoxy Coating after ESS

The epoxy coated PWAs that went through only salt fog testing had an average adhesion of 50 percent (Table 7), again because of the poor adhesion of the boards made with no-clean materials (Table 8). PWAs that went through all environmental stresses had an average adhesion of 80 percent. The remaining ESS tests produced adhesion values that were essentially equivalent. PWAs that saw no environmental stresses and the PWAs that went through only thermal shock had average adhesions of 86 percent. PWAs that went through only thermal cycling had an average adhesion of 89 percent. Individual humidity testing produced an average adhesion of 91 percent.

In overall terms of paste/flux materials (Table 8), the control PWAs had the best average adhesion value (95 percent). The RMA paste and flux PWAs had an average adhesion of 86 percent. The water soluble paste and flux had an average adhesion of 82 percent. The no-clean paste and flux had average adhesion of 58 percent.

Adhesion of Silicone Coating after ESS

The silicone coated PWAs generally did poorly in the various individual ESS tests, with average adhesion values in the 61 to 69 percent range (Table 7). PWAs that saw all environmental stresses had an average adhesion of 74 percent. The PWAs that saw no environmental stresses had average adhesion of 80 percent.

In terms of paste/flux materials (Table 8), the PWAs manufactured with water soluble paste and flux had an average adhesion of 74 percent. Those manufactured with RMA paste and flux and the control PWAs had average adhesions of 71 and 70 percent, respectively. The PWAs manufactured with no-clean paste and flux had an average adhesion of 60 percent.

Overview Of Conformal Coating Adhesion

In terms of severity for the conformal coatings tested (Table 7), the salt fog test is most severe with an average adhesion of 61 percent. Humidity testing produces an average adhesion of 71 percent. The remaining ESS tests are equivalent in severity, with average adhesions of 81 or 82 percent. Control PWA adhesion is 80 percent.

When all environmental stresses are averaged for the various paste and flux combinations (Table 9), the PWAs coated with Parylene C had the highest average adhesion (91 percent). PWAs coated with epoxy were next with an average of 80 percent adhesion. The urethane coated boards had 75 percent average adhesion, followed by silicone-coated PWAs with 69 percent average adhesion and acrylic-coated PWAs with 57 percent average adhesion.

COATING & TEST PASTE/FLUX TYPE						
ACRYLIC	RMA	WATER SOLUBLE	NO-CLEAN	CONTROL	ESS AVE	
HUMIDITY	35	40	0	25	25	
THERMAL CYCLE	85	100	75	100	90	
THERMAL SHOCK	90	70	85	100	86	
SALT FOG	25	50	0	25	25	
ALL	72	60	15	72	55	
NONE	80	50	45	70	61	
paste/flux ave	64	62	37	65	57	
URETHANE						
HUMIDITY	80	60	75	60	69	
THERMAL CYCLE	80	80	70	80	78	
THERMAL SHOCK	80	80	75	80	79	
SALT FOG	80	80	75	80	78	
ALL	78	90	69	46	71	
NONE	80	75	75	70	75	
paste/flux ave	80	78	73	69	75	
EPOXY						
HUMIDITY	100	100	65	100	91	
THERMAL CYCLE	90	90	75	100	89	
THERMAL SHOCK	80 .	90	75	100	86	
SALT FOG	70	45	5	80	50	
ALL	89	92	40	88	77	
NONE		75	85	100	86	
paste/flux ave	86	82	58	95	80	
SILICONE	-					
HUMIDITY	70	. 80	65	60	69	
THERMAL CYCLE	60	70	65	60	64	
THERMAL SHOCK	60	60	65	60	61	
SALT FOG	75	75	30	80	65	
ALL	80	77	58	80	74	
NONE	80	80	80	80	80	
paste/flux ave	71	74	60	70	69	
PARYLENE						
HUMIDITY	100	100	95	100	99	
THERMAL CYCLE	100	80	80	100	90	
THERMAL SHOCK	95	80	65	100	85	
SALT FOG	30	100	90	100	80	
ALL	98	100	84	100	96	
NONE	100	100	95	95	98	
paste/flux ave	87	93	84	99	91	

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TABLE 9 ADHESION MATRIX AVERAGES							
COATING & TEST	PASTE/FLUX TYPE						
COATING	RMA	RMA WATER SOLUBLE NO-CLEAN CONTROL ESS AVE					
ACRYLIC	64	62	37	65	57		
URETHANE	80	78	73	69	75		
EPOXY	86	82	58	95	80		
SILICONE	71	74	60	70	69		
PARYLENE	87	93	84	99	91		

SIR Testing

SIR values are shown in Table 10 and Figures 2-6. None of the SIR value changes shown are significant value changes and none would be classed a failure. Average SIR values improved after SIR testing for the epoxy, silicone, and urethane coatings. Average SIR values decreased slightly (0.44 log ohm; less than one order of magnitude) for the acrylic coating and decreased even more (1.19 log ohm; slightly over one order of magnitude) for the Parylene coating. The average SIR value for the parylene group is almost 2 log ohms less than that of the closest group (the acrylic coating). Note that for all but the silicone group, the No-clean boards had the lowest SIR values in each group (Figures 2-6). The No-clean boards had a higher SIR value than the RMA and Control Boards in the silicone coating group.

Figure 7 shows average adhesion values for the five conformal coatings evaluated in the Phase 3 effort plotted for the RMA, Water-soluble, and No-clean fluxes and pastes used to manufacture the PWAs. The flux/paste graph lines are relatively close together over the urethane, silicone, and parylene conformal coatings, indicating all have equivalent adhesion for the materials tested.

CONCLUSIONS

A viable test vehicle (pallet) and methodology for assessing interactions between no-clean materials and low-VOC conformal coatings have been developed.

The results of this effort indicate that it is practical to use low-VOC coatings over no-clean fluxes and pastes in some circumstances. When materials are graphed against adhesion (Figure 7), it is apparent that the urethane, silicone, and parylene conformal coatings used in this study have as good adhesion over no-clean materials as over RMA and water-soluble materials.

It is important to remember these results apply only to the specific coatings tested and the specific fluxes, pastes, and solder mask over which they were applied. Coatings that did not perform well in these tests will perform very well with different PWA materials. Coating performance is related to material compatibility. It is extremely important that all materials be carefully screened for compatibility before selecting a no-clean flux/paste and low-VOC conformal coating combination. It is also important to fine tune manufacturing processes employed and then keep them constant. Small process changes can have large effects on surface conditions, which in turn effect conformal coating adhesion.

All reports and data analysis for each phase effort and the initial compatibility testing are available from the EMPF library.

TABLE 10 SIR VALUES (LOG OHMS)				
ACRYLIC COATING	INITIAL	24 HRS	96 HRS	168 HRS	FINAL
RMA	11.71	8.50	8.38	8.46	12.01
WATER SOLUBLE	11.57	8.39	8.31	8.38	11.55
NO-CLEAN	12.61	7.69	7.93	8.12	10.90
CONTROL BOARD	12.19	8.17	8.34	8.46	11.87
average	12.02				11.58
EPOXY COATING	INITIAL	24 HRS	96 HRS	168 HRS	FINAL
RMA	10.77	8.3 5	8.43	7.89	12.40
WATER SOLUBLE	10.65	**	8.33	7.81	12.23
NO-CLEAN	10.82	**	8.11	8.69	11.58
CONTROL BOARD	10.82	**	8.35	8.03	12.06
average	10.76				12.07
PARYLENE COATING	INITIAL	24 HRS	96 HRS	168 HRS	FINAL
RMA	10.80	8.56	8.19	8.11	9.76
WATER SOLUBLE	10.84	8.51	8.28	8.16	10.21
NO-CLEAN	10.41	7.83	7.66	7.59	8.88
CONTROL BOARD	11.07	8.28	8.11	8.01	9.50
average	10.78				9.59
SILICONE COATING	INITIAL	24 HRS	96 HRS	168 HRS	FINAL
RMA	11.50	8.51	8.35	. 8.26	12.03
WATER SOLUBLE	11.34	**	8.51	8.45	12.53
NO-CLEAN	11.90	**	8.42	8.32	12.27
CONTROL BOARD	11.27	**	8.23	8.07	11.86
average	11.50				12.17
URETHANE COATING	INITIAL	24 HRS	96 HRS	168 HRS	FINAL
RMA	11.04	8.00	8.14	7.82	12.34
WATER SOLUBLE	10.98	7.87	7.94	7.82	12.39
NO-CLEAN	10.84	7.72	7. 9 4	8.11	11.38
CONTROL BOARD	10.80	7.58	7.82	7.93	11.95
average	10.91				12.01

** Data lost through equipment malfunction

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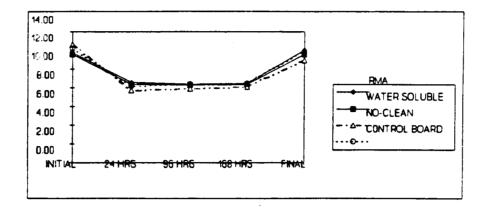


FIGURE 2 ACRYLIC COATING SIR VALUES

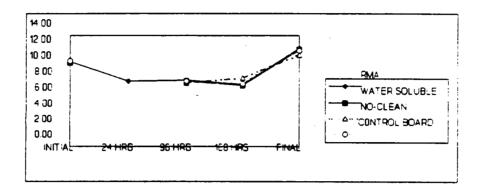


FIGURE 3 EPOXY COATING SIR VALUES

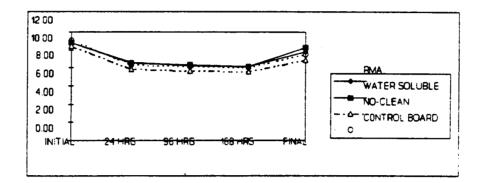
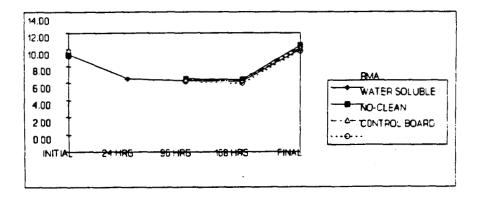


FIGURE 4 PARYLENE COATING SIR VALUES

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FIGURE 5 SILICONE COATING SIR VALUES

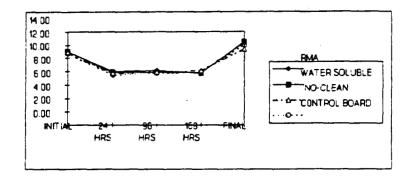


FIGURE 6 URETHANE COATING SIR VALUES

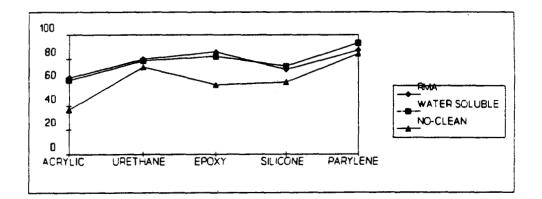


FIGURE 7 MATERIAL COMPARISON

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