

# Effect of process related residues on the performance of conformal coatings for PCBA applications

*Umadevi Rathinavelu, Morten S. Jellsen, and Rajan Ambat  
Department of Mechanical Engineering  
Technical University of Denmark  
DK 2800 Kgs, Lyngby, Denmark*

## Summary

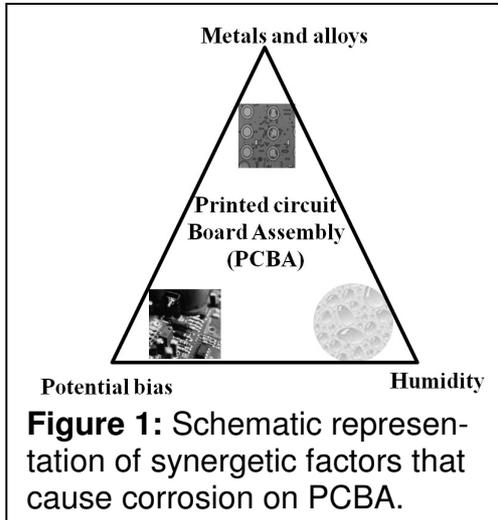
Electronic devices have become integral part of the present world and are widely used in consumer and industrial applications. Climatic reliability of the devices is becoming a prime concern these days due to various reasons such as high levels of miniaturization, new production practices using no-clean fluxes, and application of the device all climatic conditions. Heart of the electronic device is the Printed Circuit Board Assembly (PCBA). A PCBA typically consists of a number components made of various corrosion prone metallic materials, while a potential bias exists when the device is working, and interaction with humid environment brings the aqueous environment to complete the ingredients needed for corrosion. Therefore, control of corrosion on PCBA is utmost important for improving the climatic reliability of electronic devices.

Applying organic or inorganic coatings to the printed circuit boards as conformal coating is considered as one of the strategy to protect the circuits from external influence and corrosion. Conformal coatings are meant to provide a high degree of insulative protection and are usually resistant to many types of solvents and severe environments encountered in the product life cycle. Various types of conformal coatings are available for this applications namely silicone based, polyurethane or acrylic. However, in actual service conditions performance of these conformal coatings depends on various factors such as water or moisture permeability, adhesion to printed circuit board, cleanliness of the board, and permeability to other environments such as gases.

A systematic study on the performance of conformal coatings in connection with process related contaminants like activated or non activated flux residues is the focus of this paper. Different test substrates like plain laminate surface, Surface Insulation Resistance (SIR) pattern with 15 mil spacing conducting lines and a test PCBA coated with conformal coatings were investigated in this study. The coated substrates were exposed to wide range of environmental conditions ranging from full immersion in water to high humid environment at ambient temperature and their performance were evaluated. The performance of the coating was evaluated using various parameters like change in morphology of the coating on the laminate surface, decrease in surface insulation resistance across the conducting lines, and analysis of the failures like dendrites formation due to electrochemical migration (ECM) under the coating. Morphology of the coating before and after exposure was investigated using SEM, EDS, and FIB. Results show that the flux residues greatly degrade the performance of the coatings compared to the clean substrates. Type of coatings determined the permeability to environment, while contamination at the interface increased corrosion attack drastically once the coating is delaminated.

# 1 Introduction

Corrosion reliability of electronics devices are becoming a serious issue today due to a number of factors such as increased miniaturization and wide spread use together with tiny levels of contaminations resulting from manufacturing process or service conditions. Even tiny levels of contaminations can introduce reliability risk as nano-scale corrosion is enough to cause damage to the device. No-clean flux residue resulting from the soldering process is difficult to avoid, and therefore invariably found on the PCBA surface [2]. Fig. 1 shows the combination of factors causing corrosion on a PCBA when humid environments are present.



For improving the corrosion reliability of the PCBAs or to protect it from the environmental interaction, conformal coatings are used some time as the final step in the production process. Conformal coatings are synthetic resins or polymers that are applied to the PCBAs to act as a barrier to moisture, dirt, and other external contaminants [3-5]. Due to the intrinsic properties of these polymer coatings such as physical shielding (by adhering to the surface), electrical insulation, hydrophobicity, resistance to permeation of moisture, gases etc, the reliability of the PCBAs is significantly improved, however the performance the coating towards corrosion protection depends on various factors.

There are five generic types of coating used for electronic applications today namely Acrylic, Epoxy, Urethane, Silicone, and Paralyene [4]. These coatings have different thermal, chemical and electrical properties [6] depending upon the chemistry of the coating. Acrylic coating is one of the most widely used conformal coatings due to the favorable properties like high moisture resistance, dielectric stability, good reparability, and low cost [4,5]. Conformal coating based on Silicone is another type commonly used by electronic industries. Silicones are synthetic polymers based on a molecular structure of alternating silicone and oxygen atoms with organic groups attached to all or some of the silicon atoms. Due to the high average bond energy between the Si - O chemical bond, (452kJ/mol) silicones have several advantageous properties for PCBA application like high dielectric strength, high service temperatures up to 200 °C, high thermal, oxidative, and thermal shock resistance etc [2]. The flexible bond angle between Si-O-Si makes the coatings soft and flexible which also results in easy to application and repair [3][4]. However their open molecular structure makes the coating more permeable to water vapour than many other polymers.

The performance of the conformal coatings in general, depends upon the environment to which the part is exposed, cleanliness of the substrate, thickness of the coating, and importantly the chemistry of the coating. Corrosion protective nature of the conformal coatings depends on the transport of water through the coatings to the interface, which can lead to corrosion. It was stated that the amount of water, which can diffuse through the organic coatings of reasonable thickness is greater than that needed for the corrosion process [7].

Presently electronic industries use no-clean flux systems for soldering. By using no-clean flux systems, PCBAs are not cleaned after the soldering process except in high profile applications such as aerospace and military. Therefore, in most of the normal applications, conformal coatings are applied on PCBAs without prior cleaning. Presence of no-clean flux contamination under the coating enhances the corrosion failure due to its hygroscopic and corrosion property.

In this paper, the role of a low solid content no-clean wave solder flux residue on the performance of acrylic and silicone conformal coating used for PCBA applications was investigated by exposure studies, electrochemical investigations, and microscopic investigations using SEM and FIB-SEM.

## 2 Materials and methods

### 2.1 Materials

The two test substrates used in the present investigation are plain laminate and a test PCBA. The laminate is an FR (Flame Retardant)-4 type of dimension 90mm x 90mm covered with epoxy solder mask. The test PCBA shown in Figure 2 is in-house designed and produced with a series of Surface Mount (SM) components such as chip capacitors and resistors. The test PCBA was made from a FR4 epoxy laminate in accordance to IPC-4101/21, and produced according to PERFAG 2E specifications for production of rigid PC-boards [10]. The test PCBA consists of 20 circuits in total with 18 circuits of SM components and 2 circuits of SIR patterns. One SIR pattern is open, and other is covered by soldermask a reference pattern. Among the 18 SM components circuits, nine of the circuits were resistors with resistance ranging from  $68\Omega$  to  $1M\Omega$  and of sizes 0805, 0603, and 0402. Other nine of the circuits were capacitors in the range of 22pF to 100nF capacitance and housing sizes of 0805,

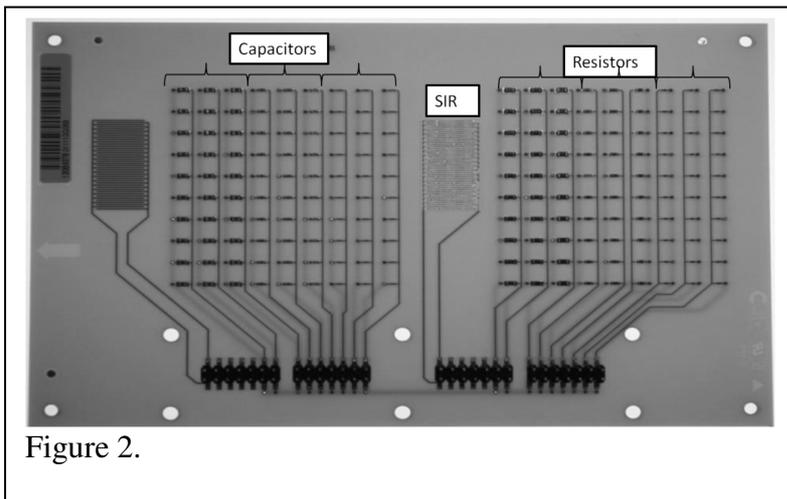


Figure 2.

0603 and 0402. Each SM circuit consists of 10 components connected in series. However, for the present investigation on the coated PCBA, only the output signal from only 5 channels were collected namely the signal from resistors  $330K\Omega$  and size 0805, capacitors 100 nF and 22 pF with housing size 0603 and 0805 and the open SIR pattern were collected.

The current signal measured on each channel is the total current across 10 identical SM components connected in series. The potential bias applied on the test PCBA is 6V across each channel using an external power supply and the electrical functionality of the components was monitored by current measurements. The current from each circuit was measured using a multiplexer circuit and software programmed using Labview to record the current-time curves for each channel.

The contaminant investigated here is a low solid content no clean wave soldering flux meant for Pb free soldering, which consists of 80-90 % of 2-propanol and 5-15 % ethyl alcohol, and about <2 % of carboxylic acid and remaining resin component. The carboxylic acids generally present in the no-clean fluxes are adipic acid, succinic acid, glutaric acid, etc. The ELPEGUARD SL 1306 N-FLZ modified acrylate resin was investigated as the conformal coating over the test substrates. The silicone conformal coating tested was Dow Corning...

## **2.2 Preparation of laminates and SIR PCBs with solder flux contamination**

Three types of laminate surfaces were prepared namely clean surface, surface contaminated with flux, surface contaminated with flux and heated to  $255\pm 5^{\circ}\text{C}$  for 45 seconds (simulation of wave soldering process temperature profile, peak temperature, and total time). The flux contamination was made by spraying with a squeeze bottle, followed by drying at room temperature for 30 minutes. The amount of flux sprayed was approximately constant on all the laminate surfaces and was verified by the weight measurement after spraying to make sure that it is within the possible error limit.

Prior to the experiment, the test PCB (Figure 2) was cleaned by immersion in 50% isopropyl alcohol using a sonicator for 30 minutes to remove any flux residues that was present. The test PCBs were then rinsed with de-ionized water and dried using hot air drier. Three types of Test PCB surfaces were prepared for the leakage current study namely clean surface, surface contaminated with the no-clean flux by spraying, and surface heated to  $255\pm 5^{\circ}\text{C}$  for 45 seconds after flux spraying.

## **2.3 Preparation of coated samples using spin coater**

Conformal coating was applied on the test substrates using the spin coater (WS-650SZ-s-6NPP-Lite Spin Processor, Laurell). For coating the plain laminate surface, conformal coating solution was placed on the laminate surface which is held by the spin coater chuck and allowed to spin. For acrylic coating, a spin speed of 120 rpm for 1 minute was used, while for silicone coating 250 rpm for 1 minute. Final acrylic coating thickness was approximately 30 to  $40\mu\text{m}$  (after curing for 24h), while the thickness of the silicone coating was 100 to  $110\mu\text{m}$  (after curing for 40 minutes at  $120^{\circ}\text{C}$ ).

The test PCBs were dip coated as the surface profile is not uniform, therefore it is difficult to get a uniform coating using spin coater. In this case, thickness of the acrylic layer on the test PCB was 10 to  $20\mu\text{m}$  after curing, while for silicone coating was 30- $40\mu\text{m}$ .

## **2.4 Exposure studies**

### *2.4.1. Acrylic coated laminates*

Acrylic coated laminates were tested by full immersion in  $60^{\circ}\text{C}$  water for 10 and 15 days test duration. After exposure, the increased weight was measured to calculate water intake followed by SEM analysis to investigate change in surface morphology.

### 2.4.2. Silicone coated laminates

Silicone coated laminates were subjected to humidity exposure at 60°C and 98%RH for 15 days and 25°C and 98% RH for 5 days. After exposure the morphology of the coated surface were analysed using light optical microscope.

### 2.5 Potentiostatic studies of coated test PCBs

A Potential bias of 6V was applied to various components of the test PCB and the leakage current was recorded using an in-house built TestPCB set up and software for every 1 second [10]. Test environment used was 25°C 98% RH for 60 hours duration.

### 2.6 Microscopic Investigation

Microstructural characterization of the laminate surface and coating was carried out using SEM (JEOL 5900 instrument). FIB-SEM was performed using a Zeiss 1540EsB cross beam for the cross sectioning of the coating in order to investigate the under film corrosion after the electrochemical experiments.

## 3 Results and Discussion

Figure 3 shows the surface of the FR4 laminate with solder mask with flux residues on it formed at room temperature and at  $255\pm 5$  °C. The laminate surface with room temperature flux contains more patches of flux residues compared to the laminate with activated flux. The typical morphology of no clean flux residue formed after the room temperature evaporation is shown in the Fig. 4. The two components seen in the morphology are acid and resin component of the flux (shown in the Figure), while most of the isopropyl alcohol component is evaporated. Residues formed at higher temperature also showed similar morphology, however the acid crystals present only to a lesser extent and broken up and distributed in the ester matrix.

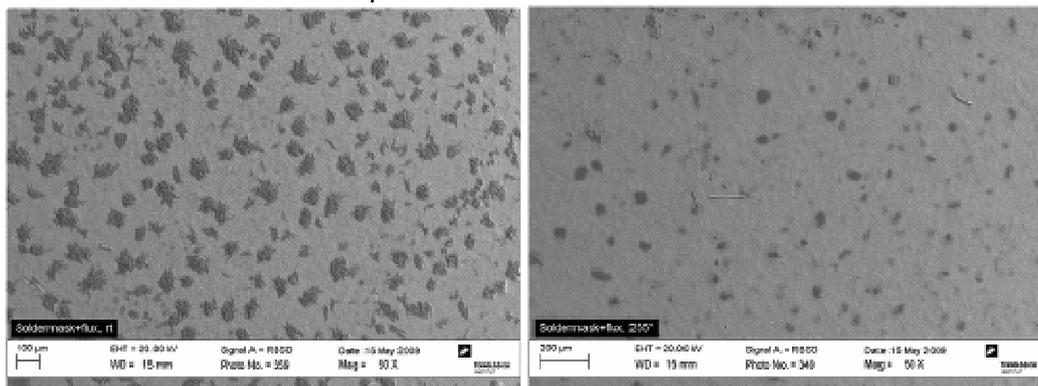
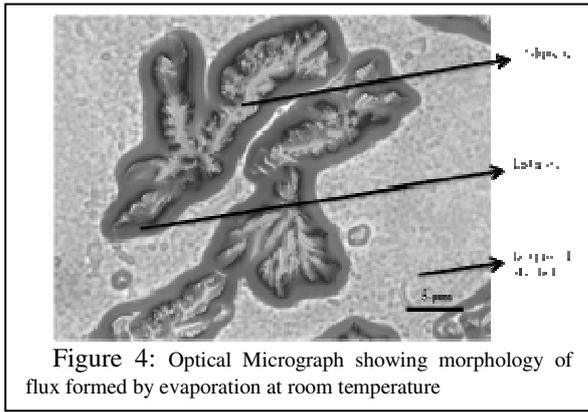


Figure 3: Typical surface morphology of flux contaminated FR4 laminate: (left) residue formed at room temperature, and (right) residue formed at  $255\pm 5$ °C.

Fig. 5 shows the wave solder temperature profile for the lead free alloy (Sn-Cu-Ag) soldering process. The overall time scale of the solder profile is about 5-6 minutes, which comprises of pre-heating, soldering, and cooling stage. However, the total time at peak soldering temperature ( $\sim 255$ °C) is about 45 seconds. Fig. 6 shows the percentage of activator components in the flux residue versus different stages of soldering process. After the pre-heating stage about 30-40 % of the initially sprayed acid has been volatilized leaving 60 % of the acid on the boards. However after the sol-



dering stage about 80-90 % of the acid component has been volatilized, therefore only about 10 % of acid component was present in the flux residue on the boards.

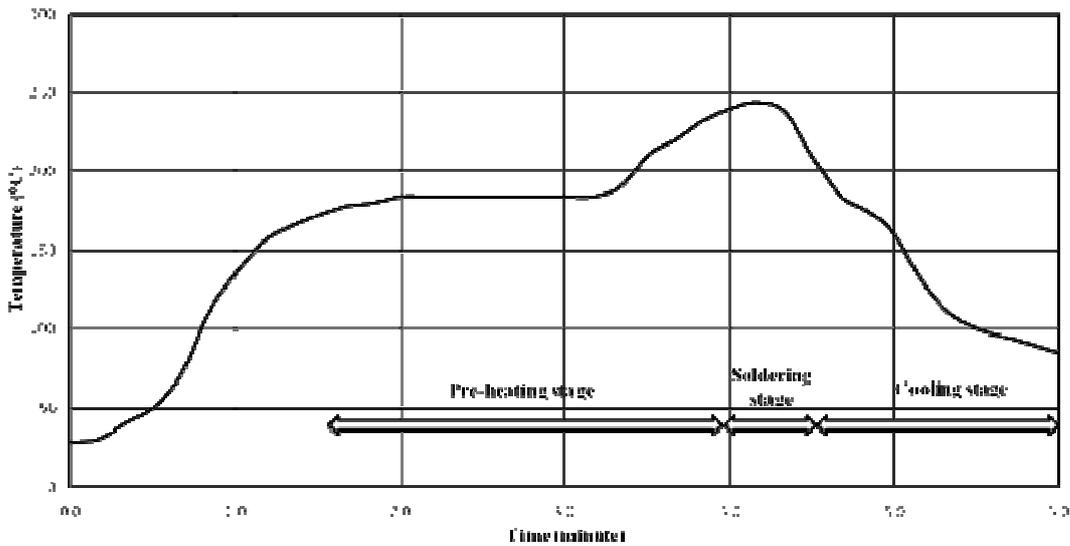


Figure 5. Thermal profile of Pb-free wave soldering processes

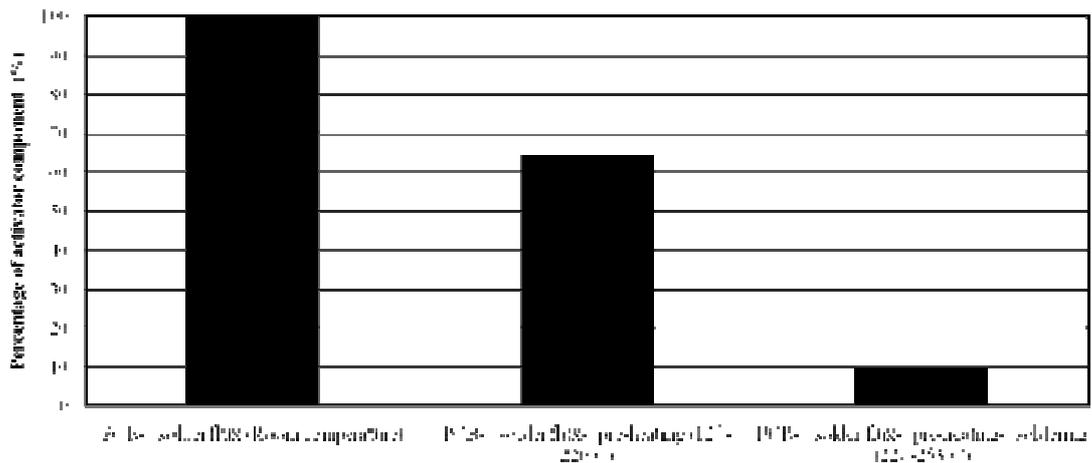


Figure 6: The percentage of activator components (carboxylic acids) left on a real PCBA from the production line removed at various stages of the soldering process.

### 3.1 Exposure studies on coated laminates

### 3.1.1 Surface morphology of acrylic coated sample after exposure to 60°C water

Fig. 7a(i) shows the optical micrograph of the surface morphology of the coating on clean laminate after total immersion in 60°C water for 10 days. It is clear that the laminate surface is not significantly affected by the exposure. The SEM pictures shown in Fig. 7a(ii) (low magnification) & (iii) (high magnification) is in agreement with this as only very few blisters or de-bonding areas are seen on the surface after exposure.

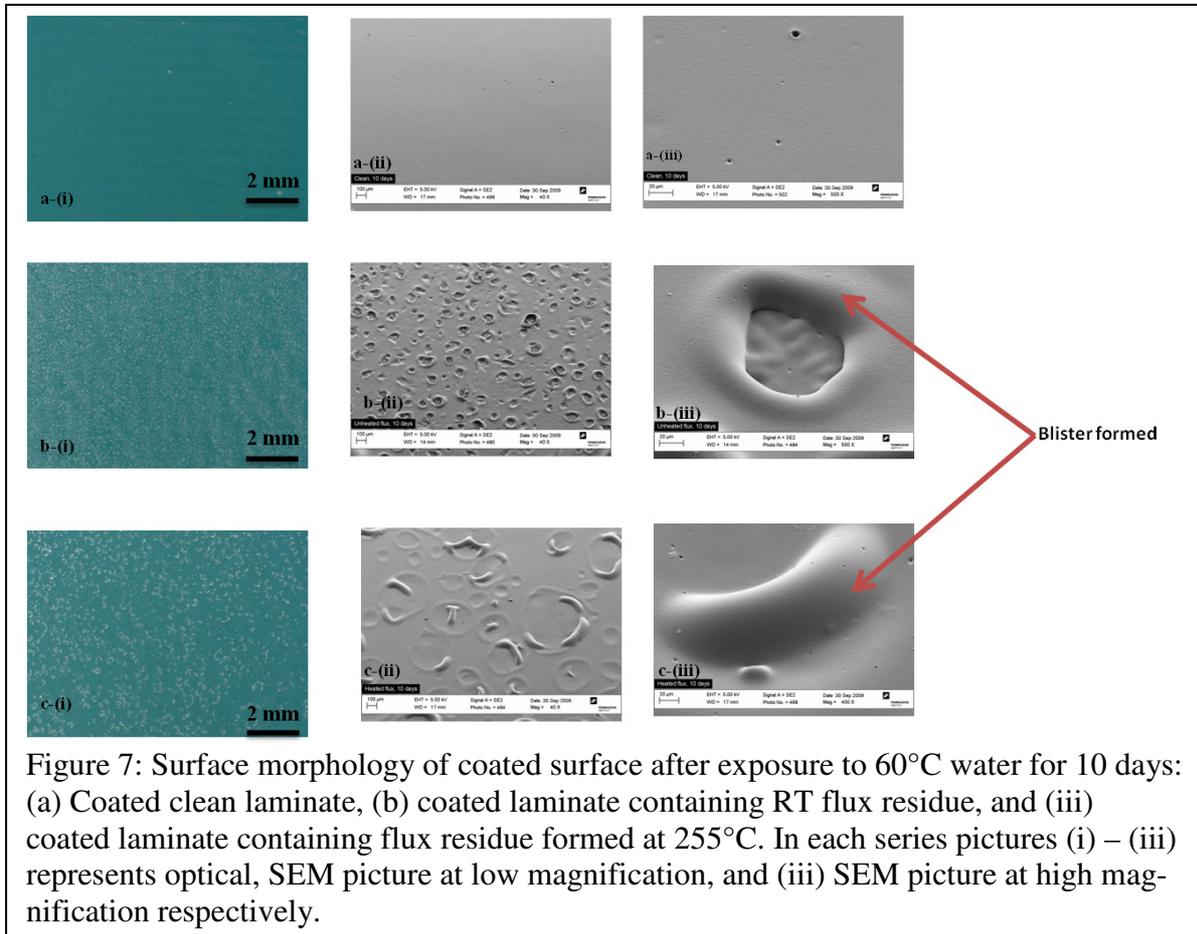


Figure 7: Surface morphology of coated surface after exposure to 60°C water for 10 days: (a) Coated clean laminate, (b) coated laminate containing RT flux residue, and (c) coated laminate containing flux residue formed at 255°C. In each series pictures (i) – (iii) represents optical, SEM picture at low magnification, and (iii) SEM picture at high magnification respectively.

Results for the laminate with room temperature flux residues and coating are shown in Fig. 7 b(i)-(iii). Clearly the surface morphology of the coated surface after exposure has changed with the appearance of numerous blisters compared to the unexposed one. The blisters were uniformly distributed on the surface with an average size ranging from 50 – 100  $\mu\text{m}$ . The high magnification picture in Fig. 7b (iii) shows that the centre part of the blister has retracted back to the surface after the specimen has removed from the solution. The laminate with flux residue formed at 255°C also showed change in morphology and formation of blisters (Fig. 7 c(i)-(iii)), but less in number and bigger size compared to the surface with room temperature flux. However, in this case the blister formation is not uniform around the edge of the circumference of the blister. For both cases, the distribution of blisters is comparable with the morphology of the surface showing flux residue distribution at room temperature and 255°C shown in Fig. 3. Similar observation was found when the laminates were exposed to slightly longer duration of 15 days except for the slightly higher number of blisters.

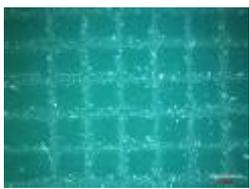
The amount of water intake per unit area of the coatings during the immersion test calculated from weight gain measurements (not shown here) showed that the contaminated surface has absorbed more water compared to the clean substrate. Also the coated substrate with heated flux has taken more water compared to the coating on substrate contaminated with room temperature flux. It correlates with the SEM micrograph that shows larger blisters on the coating over the surface contaminated with heated flux (Fig 7(c)). However, the coating over the surface contaminated with room temperature flux had more number of blister formation than on the coating over the surface contaminated with heated flux.

*3.1.1 Surface morphology and adhesion of Silicone coated laminates after exposure to humidity*

Surface morphology of silicone conformal coated laminates after exposure to 98%RH at 25oC showed visible blisters before removal from the climatic chamber, while the blisters were disappeared immediately after removal. Therefore cross-cut adhesion test was carried out to understand the state of the coating after exposure.

Table I shows the adhesion strength of the coating on the laminate after exposure to humidity using the cross-cut test. Results presented in the table shows that the adhesion strength of the coating was dependant on the cleanliness of the substrate. From the appearance after the cross cut test, the coating on clean laminate surface can be classified as class 1, while the coating on surface contaminated by non activated and activated flux was categorized as class 2 and class 4 respectively. Coating on the laminate with activated flex residue showed severe detachment after the test.

Table1: Illustration of adhesion strength of coatings on surface containing different flux contaminations

Substrate	Appearance of surface after cross cut	Adhesion class	Visual observation
Clean		1	Detachment of small flakes of coating around the edges
Non- Activated flux surface		2	The coating has flaked along the edges and at the intersection of the cuts.

			
Activated surface		4	The coating has flaked along the edges of the cut and some of the squares has been partially or fully removed

### 3.1 Electrical test on coated test PCBs

The electrical performance of a typical PCBA with non activated flux residue and conformal coated after exposure to 25 C and 98 % RH is shown in figure 8. In PCBA1 drastic increase in current was observed across the surface insulation resistance pattern and the large and medium capacitor during the exposure. The PCBA 2 showed higher leakage current in the SIR, and medium capacitor channels. The PCBA 3 showed increase in leakage current in the SIR and large capacitor channel while the medium capacitor channel did not showed any leakage current. Among the three PCBAs tested, no leakage current was seen across the SM resistor component. The time of failure (increase in leakage current) varied widely between the different channels and PCBAs and is briefly charted in fig 6???. In all the three PCBAs tested SIR showed failure in a wide range of time from 3.8 to 56.3 hours whereas the capacitors failed at approximately after 30 to 36 hours of the test duration.

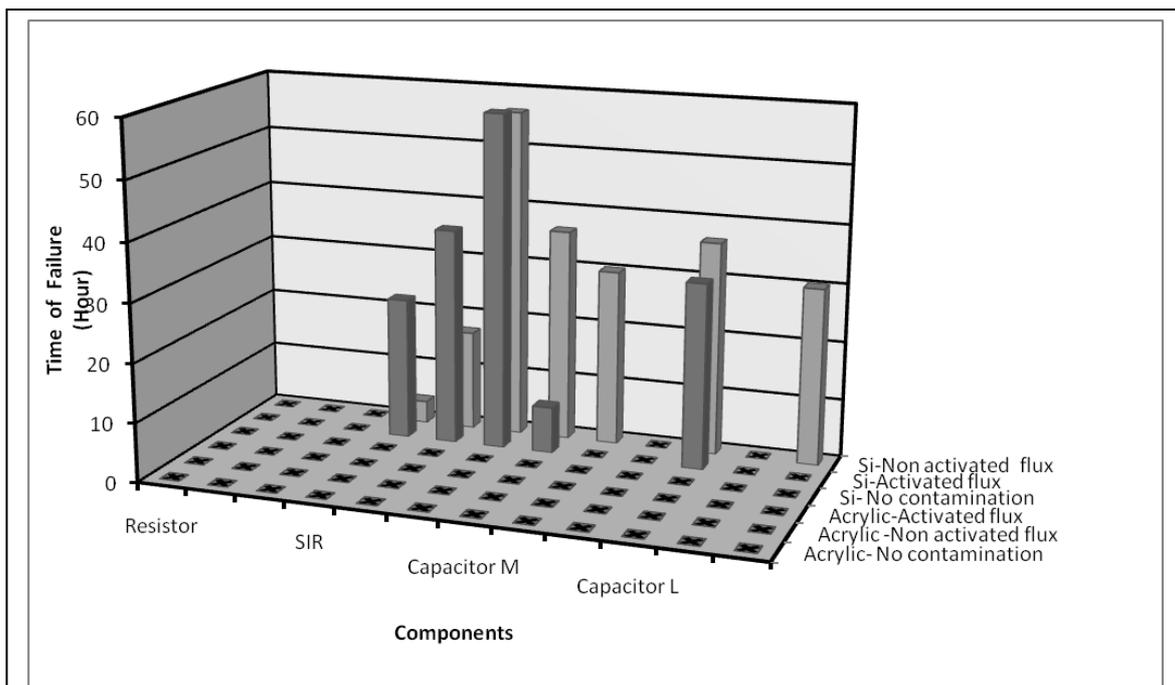


Figure 8:

The time of failure of the components containing flux residues with and without conformal coating is compared in figure 9. The time of failure of the PCBA without conformal coating was in the range of 0.2 to 1 hour while the PCBA with conformal coating was in the range of approximately 3.8 to 36.5 hours.

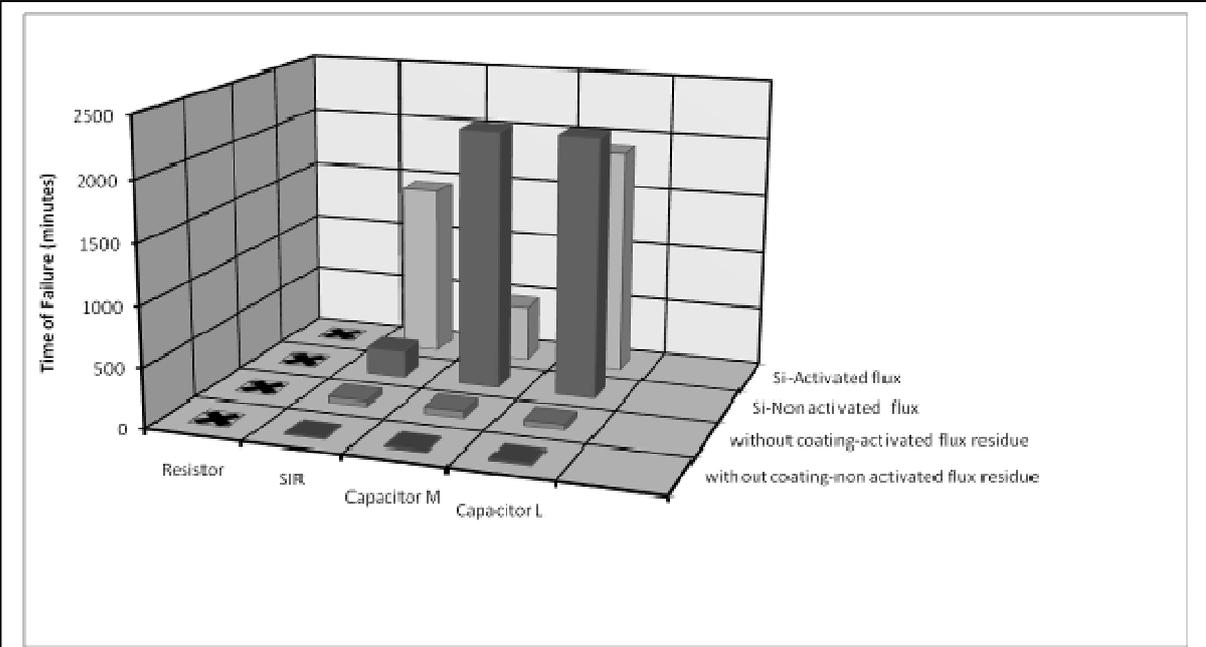


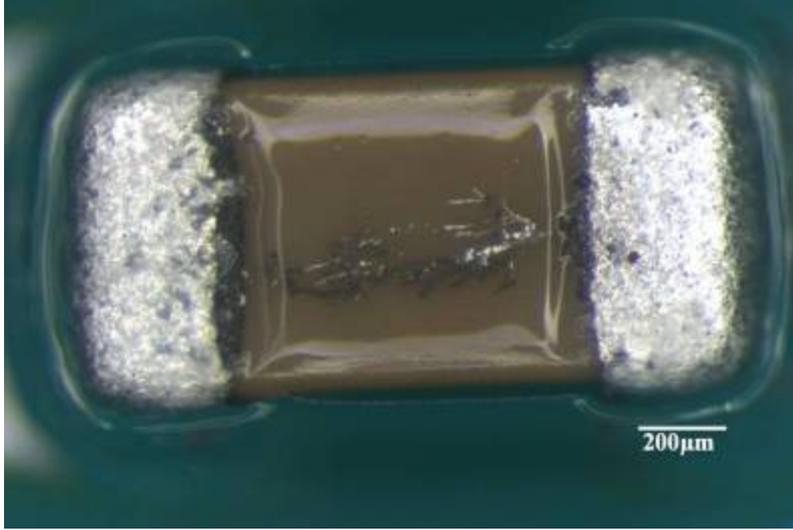
Figure 9:

### 3.2 Corrosion surface morphology of test PCB surface after exposure

The surface morphology of the conformal coated clean components observed in light optical microscope after the exposure is shown in figure 10. It can be seen that none of the components showed any signs of damage to the coating and corrosion or electrochemical migration. After removal of coating, the SEM analysis on the components (fig 10), also did not show signs of corrosion. (just explain, no need of pictures..)

Morphology of the conformal coated PCBA components which contained the non activated flux contaminant observed in the light optical microscope is shown in figure 11. Severe damage to the coating and drastic corrosion is observed on the terminals of the capacitors (fig 11 (a&b)). The SIR pattern (fig 11 (d)) showed severe corrosion and also electrochemical migration leading to formation of dendrites. Resistor did not show any deterioration of the surface. The SEM analysis of the capacitor (fig 12) also showed signs of corrosion. (show only typical ones and say that others looks similar..)

Figur 1: Surface morphology of silicone coated components that contained non activated flux residue after testing.



Figur 2: Surface morphology of silicone coated components that contained activated flux residue after testing.

Table II: showing the number of dendrites formed on the capacitors containing different contaminations (change this table to B&W)

	Clean		RT		HT	
	Medium capacitor	Large Capacitor	Medium capacitor	Large Capacitor	Medium capacitor	Large Capacitor
PCBA 1	0/10	0/10	1/10	2/10	1/10	9/10
PCBA 2	0/10	0/10	1/10	0/10	7/10	8/10
PCBA 3	0/10	0/10	0/10	1/10	5/10	4/10

### 3 References

- [1]
- [2]
- [3] etc