The Effect of FR-4 Laminate Materials on the Surface Insulation Resistance of Wave Soldering Fluxes

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Abstract
It has been observed that, since the implementation of Pb-free processing, different flux chemistries produce different surface insulation resistance (SIR) results depending on the test coupon laminate used. The most likely reason for these results are differences in the FR-4 laminate chemistry used in the test coupons because of the higher processing temperatures used in Pb-free assembly. SIR has been measured per IPC SIR IPC-TM-650 methods 2.6.3.7 (J-STD-004B) and 2.6.3.3 (J-STD-004A) for several fluxes and types of laminates. In general, the less expensive FR-4 materials with lower glass transition temperatures (T_g) gave higher SIR values. Higher T_g materials were a little better than the lower T_g materials. Reflowed solder paste SIR was also higher on the low T_g materials than the high T_g materials but to a much lesser extent than comb down wave soldering fluxes.

Many wave soldering fluxes with rosin content, even as little as 1%, pass SIR testing per IPC-TM-650 when soldered comb-down on the high T_g FR-4 laminates. In general, the fluxes with higher rosin content have the highest SIR comb-down on these more modern laminates.

Introduction
It has been observed that, since the implementation of Pb-free processing, different flux chemistries produce different surface insulation resistance (SIR) results depending on the test coupon laminate used. Because of this issue, a detailed study of SIR testing of wave soldering fluxes was undertaken to determine the root cause of this effect on SIR values in IPC SIR testing [1,2].

Something has changed in the way fluxes are tested now compared to the past; SIR values in general are lower. Additionally, comb up (coupons passed with the comb facing up and only touching the solder wave on the back side) SIR values are now higher than comb down values (combs touching the solder wave and being soldered). In the past, comb down values were usually higher than the comb up values. This is true even with fluxes whose known raw materials haven’t changed in many years. It is unlikely that the measurement equipment has changed; temperature humidity chambers and SIR data loggers used are calibrated on a yearly basis. Coupon processing has changed to the extent that we now use lead-free solder that is processed at higher temperatures than the eutectic tin-lead solder used over a decade ago. It is also possible that the SIR test coupons have changed; the quality of the copper surface, such as roughness, may have changed or the FR-4 laminates may have changed.

Of these possible changes in testing, a change in the chemistry of the FR-4 laminate is a likely reason for the change of SIR values. FR-4 is a flame retardant brominated epoxy / woven fiberglass material, and one of the most common laminates used for electronic circuit boards [3]. The FR-4 nomenclature does not specify the type of epoxy used in the laminate, nor whether the laminate includes filler materials intended to modify the glass transition temperature, T_g, of the material. FR-4 materials used more than 15 years ago had T_g less than 130°C. Because of the increasing use of lead-free solder and the higher processing temperatures required, some FR-4 materials have T_g as high as 180°C. Many of the high T_g FR-4 laminates contain fillers, such as clay and talc, to improve the thermal properties of the material [4]. These materials may have a deleterious effect on the insulation of the material at elevated humidity; some of these fillers are hygroscopic.

We decided to test SIR per IPC-TM-650 method 2.6.3.7, 40°C, 90% R.H. and 12V bias and measure, specified by J-STD-004B [1,5], for a variety of no-clean wave soldering fluxes using different FR-4 laminates. The SIR coupons used just before the onset of this study were obtained from a PWB fabricator that used a stock FR-4 with a 180°C T_g and an inorganic filler, Laminate E. For this study, we had SIR coupons fabricated from a selection of substrates obtained from a single manufacturer of FR-4 laminate materials, A through D with T_g values from 140 – 180°C, plus laminate E. This was later expanded to include a second 140°C T_g FR-4 laminate F, from another material vendor. Some combinations of flux and laminate were also tested to the older IPC SIR method 2.6.3.3. [2], 85°C, 85% R.H., -50V bias and 100V measure. A list of properties of the various FR-4 laminate materials used in this study is given in Table 1. Figure 1 shows the coupon used for measuring volume resistivity and surface resistance, and how it is used for these two measurements [6].
Table 1. Selected physical and chemical properties of the FR-4 laminates used in this study.

<table>
<thead>
<tr>
<th>Laminate</th>
<th>Manufacturer</th>
<th>$T_g$ °C</th>
<th>$T_d$ °C</th>
<th>Moisture %</th>
<th>CTE &lt; $T_g$ PPM/°C</th>
<th>CTE &gt; $T_g$ PPM/°C</th>
<th>VR Mohm cm</th>
<th>SR Mohm</th>
<th>Chemistry</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>1</td>
<td>140</td>
<td>310</td>
<td>0.25</td>
<td>50</td>
<td>250</td>
<td>8.E+07</td>
<td>2.E+05</td>
<td>DiCY tetra-functional epoxy</td>
</tr>
<tr>
<td>B</td>
<td>2</td>
<td>140</td>
<td>320</td>
<td>0.3</td>
<td>50</td>
<td>250</td>
<td>1.E+08</td>
<td>5.E+06</td>
<td>DiCY tetra-functional epoxy</td>
</tr>
<tr>
<td>C</td>
<td>1</td>
<td>170</td>
<td>300</td>
<td>0.2</td>
<td>60</td>
<td>250</td>
<td>9.E+07</td>
<td>3.E+08</td>
<td>DiCY tetra-functional epoxy</td>
</tr>
<tr>
<td>D</td>
<td>2</td>
<td>180</td>
<td>340</td>
<td>0.15</td>
<td>45</td>
<td>230</td>
<td>3.E+08</td>
<td>3.E+06</td>
<td>Novalac</td>
</tr>
<tr>
<td>E</td>
<td></td>
<td>180</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Novalac</td>
</tr>
</tbody>
</table>

Five fluxes were used in this study, two fairly modern water-based organic acid activated fluxes, F1 and F2, two isopropanol-based organic acid activated fluxes F3 and F4 that contain no rosin, and an older water-based organic acid activated flux F5. In addition, IPC SIR per method 2.6.3.3 was measured for solder paste P1 on laminates E ($T_g$ 180°C) and F ($T_g$ 140°C) to see whether laminate made a difference for solder paste SIR measurements also. ECM at 50°C 90% RH and 5V was measured for solder paste P2 on bare copper laminates E ($T_g$ 180°C) and F ($T_g$ 140°C) because this is a difficult measurement for many soldering materials to pass.

Experimental Procedure

Coupon Preparation – Pre-Cleaning

The coupons were cleaned in an Ionograph 500M SMDII in extraction solution of 75% isopropanol (IPA) and 25% water heated to 45°C for at least one hour or until a solution resistivity greater than 300 Mohm.cm was achieved. The coupons were then baked in air at 50°C for one hour.

Coupon Preparation – Liquid Wave Soldering Fluxes

For each wave flux/laminate combination, three comb up and three comb down coupons were prepared. Flux was applied liberally using a dropper, then allowed to drain at an 80° angle for about one minute. The coupons were wave soldered using an Electrovert Vectra with a conveyor speed of 1.5 m/min, 100°C top side preheat and a SACX0807 solder pot at 265°C. Nitrogen was passed over the solder pot to reduce dross formation.

Coupon Preparation – Solder Paste

Solder paste was printed on coupons using a 6 mil stencil for IPC-B-24 coupons and for the IPC-B-25 coupon used for the 50C/90%RH ECM testing. IPC-B-24 coupons were reflowed once and IPC-B-25 coupons were printed once (3 coupons) and twice (3 coupons).

SIR Measurements

Teflon insulated wires were hand soldered to the coupons using an ROL1 core-fluxed wire. The solder paste coupons tested for 50C/90%RH/5V ECM test had the 4 segment W comb bussed together into a single comb. The coupons were mounted in an Espec ESL 2CA temperature humidity chamber and connected to a GEN3 AutoSIR datalogger. As there was room for only 32 coupons in a chamber load and because of the variability of SIR measurements between chamber loads, SIR for one wave soldering flux was measured on several laminates or SIR for several fluxes were measured on one laminate. The conditions for the various test methods used in this study are summarized in Table 2.
Table 2. Test method summary

<table>
<thead>
<tr>
<th>Test Method</th>
<th>Coupon type</th>
<th>Coupon #</th>
<th>T</th>
<th>C</th>
<th>% RH</th>
<th>Bias V</th>
<th>Measurement V</th>
<th>Measurement interval</th>
<th>Duration</th>
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<tbody>
<tr>
<td>IPC SIR 2.6.3.7</td>
<td>IPC-B-24</td>
<td>3 comb up 3 comb down</td>
<td>40</td>
<td>90</td>
<td>12</td>
<td>12</td>
<td>20 min</td>
<td>7 days</td>
<td></td>
</tr>
<tr>
<td>IPC SIR 2.6.3.3</td>
<td>IPC-B-24</td>
<td>3 comb up 3 comb down</td>
<td>85</td>
<td>85</td>
<td>-50</td>
<td>100</td>
<td>6 hours</td>
<td>7 days</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>3 reflowed solder paste</td>
<td>85</td>
<td>85</td>
<td>-50</td>
<td>100</td>
<td>1 &amp; 4 days</td>
<td>4 days</td>
<td></td>
</tr>
<tr>
<td>50C/90% RH/5V</td>
<td>IPC-B-25</td>
<td>3 reflowed solder paste</td>
<td>50</td>
<td>90</td>
<td>5</td>
<td>5</td>
<td>10 min</td>
<td>28 days</td>
<td></td>
</tr>
</tbody>
</table>

Results

Figures 2.a and 2.b show the SIR values per IPC-TM-650 method 2.6.3.7 for water-based flux F2 on laminate B, comb down and comb up respectively.

Figure 2.a. SIR per IPC TM 650 method 2.6.3.7 for flux F2 wave soldered comb down on laminate B.

Figure 2.b. SIR per IPC TM 650 method 2.6.3.7 for flux F2 wave soldered comb up on laminate B.

The SIR values for the comb up coupons are higher than for the comb down coupons. Because it is hard to compare SIR values on different laminates from separate graphs, the geometric average SIR values for all the laminates studied and the unprocessed control coupons (laminate E) were plotted together, as given in Figures 3.a and 3.b for comb down and comb up respectively.

Figure 3.a. Geometric average SIR per IPC TM 650 method 2.6.3.7 for flux F2 wave soldered comb down on five laminates.

Figure 3.b. Geometric average SIR per IPC TM 650 method 2.6.3.7 for flux F2 wave soldered comb up on five laminates.

The SIR values for the comb up coupons are higher than for the comb down coupons for all five laminates studied. There was almost an order of magnitude difference between laminate E’s SIR values and those on the other laminates. High Tg laminate D gave fairly high SIR for flux F2 in this chamber load with even for the comb down measurements; this was not the case with most of the other fluxes studied.
Figures 4.a and 4.b show the comb down and comb up average log SIR values for the laminates studied for flux F1, another low VOC flux. Here both the high Tg D and E laminates gave the lowest SIR values for the comb down measurements, not even meeting the requirements of J STD-004B of greater than 100 Mohms after one day. Laminate A, with the lowest Tg, gave the highest SIR values for comb down measurements, and met the SIR requirement for J STD 004B. Laminates B and C gave SIR values between those for laminates A and D. The comb up SIR values for flux F1 on the A, B, C and D laminates were grouped together at over 10 Gohms; laminate E’s values were an order of magnitude lower.

Figures 5.a and 5.b show the average log SIR values for the laminates studied for IPA-based flux F3; high Tg laminate E, which consistently gave the lowest comb down SIR, was replaced with low Tg laminate F prepared by a different PCB fabricator. Flux F3 is a particularly active low solids, no clean flux. The comb down SIR values for F3 show a dramatic difference of nearly two orders of magnitude between laminates F and D. Laminate A, also low Tg, gave SIR values slightly lower than laminate FNP-140, and the SIR values for laminates B and C again were between the low Tg laminates and laminate D. The comb up SIR values are grouped together, with no significant difference between any laminates. The comb down SIR values for flux F3 on laminate F were higher than the comb-up values, as they had been in the past.

Figures 6.a and 6.b show the average log SIR values for the laminates studied for flux F4, the second IPA-based active liquid flux. As with F3, the comb down SIR values for F4 show a dramatic difference of nearly two orders of magnitude between laminates F and D. Here, the laminate B SIR values are about half-way between those for laminates F and D. SIR for laminates B and C are closer to laminate D. The comb up values are very similar.

Figures 7.a and 7.b show the average log SIR values for the laminates studied for flux F5, an active, low VOC flux. For this flux also, comb down SIR values on laminate F are two orders of magnitude greater than those on laminate D. The comb down SIR values for laminates A, B and C are grouped together about half way between F and D. The comb up SIR
values are close together for all the laminates, D being slightly higher than the others.

Figure 6.a. Average log SIR per IPC TM 650 method 2.6.3.7 for flux F4 wave soldered comb down on five laminates.

Because laminate F consistently gave the highest comb down SIR values, an IPC TM 650 method 2.6.3.7 SIR chamber load of the five fluxes studied wave soldered comb down and comb up was tested on this laminate. Figure 8.a shows average comb down SIR values for the five fluxes and Figure 8.b shows the comb up SIR values. The comb down values differ by about an order of magnitude, with fluxes F4 and F5 giving the highest values and F2 giving the lowest values. For the comb up coupons, fluxes F1 and F2 gave the highest values, with F5 now giving the lowest values along with F3. Hence, some fluxes delivered better SIR on laminate F when soldered comb down than the other fluxes, but lower SIR than the other fluxes when processed comb-up. This indicates that the chemical and physical interaction between various wave soldering fluxes and FR-4 laminate materials are complex and unlikely to be well understood without an in-depth study.

While the five no-clean fluxes without rosin gave relatively low comb down SIR values on the two high Tg laminates D and E, no-clean fluxes that contain rosin gave higher SIR on laminate E. This is not surprising, it is generally known that rosin-containing soldering materials have higher SIR than those that don’t contain rosin or other resins, and a recent study confirmed this [7]. Figures 9.a and 9.b show the average log SIR values comb down and comb up respectively for four IPA-based fluxes containing different concentrations of rosin. For the comb down SIR, the values increase with increasing rosin content, especially at shorter times during the measurement. The comb up measurements are not readily attributed to any one variable.
Figures 8.b. Average log SIR per IPC TM 650 method 2.6.3.7 for laminate F wave soldered comb up with five fluxes.

Figures 9.a. Average log SIR per IPC TM 650 method 2.6.3.7 for laminate E wave soldered comb down with four no-clean fluxes containing rosin.

Figures 9.b. Average log SIR per IPC TM 650 method 2.6.3.7 for laminate E wave soldered comb up with four no-clean fluxes containing rosin.

Figures 10.a. Average log SIR per 2.6.3.3 for flux F2 wave soldered comb down on five laminates.

Figures 10.b. Average log SIR per 2.6.3.3 for flux F2 wave soldered comb up on five laminates.

Figures 11.a and 11.b show average log SIR per IPC-TM-650 method 2.6.3.3 for flux F3 for coupons soldered comb down and comb up respectively on five laminates. Here laminate F was included and laminate E was not used. The laminate F gave the highest SIR values for comb-down coupons, with the other laminates comb down average SIR values grouped together near 100Mohms. Again, there was a decrease in SIR values after an initial increase. There was little difference between the average comb up SIR values for flux F3, and again the values were relatively flat.

Figures 12.a and 12.b show average log SIR per IPC-TM-650 method 2.6.3.3 for flux F3 for coupons soldered comb down and comb up respectively for the 5 fluxes on laminate F. Flux F3 gave the lowest SIR values, while water-based F2 and IPA-based F4 gave the highest values. Fluxes F1 and F5 gave values between F3 and F4. The comb up SIR values for all the fluxes were grouped together around 10 Gohms.
Figure 11.a. Average log SIR per 2.6.3.3 for flux F3 wave soldered comb down on five laminates

Figure 11.b. Average log SIR per 2.6.3.3 for flux F3 wave soldered comb up on five laminates

Figure 12.a Average log SIR per 2.6.3.3 for five fluxes wave soldered comb down on laminate F.

Figure 12.b Average log SIR per 2.6.3.3 for five fluxes wave soldered comb up on laminate F.

Figure 13.a SIR per 2.6.3.3 for P1 SAC305 solder paste reflowed on laminates F and E at 24 hours.

Figure 13.b SIR per 2.6.3.3 for P1 SAC305 solder paste reflowed on laminates F and E at 96 hours.

Interval plots of IPC-TM-650 Method 2.6.3.3 (85°C 85% RH -48V bias 100V measure) at 1 day and 4 days at condition are given for solder paste P1 on laminates E and F in Figures 13.a and 13.b. SIR values on laminate F are a factor of two higher than those on laminate F. While this result is significant especially at 4 days, it is much less than the effect laminates have on comb down SIR values for wave soldering fluxes.

ECM at 50°C 90%RH 5V bias and measure for 28 days are given for solder paste P2 on laminates E and F with one reflow on bare copper coupons is given in Figure 14. Similar measurements for P2 reflowed twice on bare copper show similar behavior for laminates E and F. While the SIR values for all measurements start above 10Gohms, the SIR values
drop and then stabilize during the first seven days. The drop on low Tg laminate F is slightly less than an order of magnitude, while this drop is about 2 orders of magnitude for high Tg laminate E. This drop occurs for both one and two reflows. After the SIR values level out, the difference between laminates E and F are about an order of magnitude.

**Figure 14.** 50C/90%RH/5V ECM for P2 SAC305 solder paste reflowed once on laminates E and F.

**Conclusions**

In general, laminates D and E gave the lowest SIR for comb down measurements, both for method 2.6.3.7 (40C/90%RH 12V) and method 2.6.3.3 (85C/85%RH -50V bias and 100V measure). These are both Novalac high Tg materials, and contain fillers to modify the thermal properties of the materials. In general, laminate F gave the highest comb down SIR values for wave soldering fluxes. Laminates A, B and C generally gave SIR values between laminates F and D, with A being sometimes slightly higher than B and C. Comb up SIR values are less affected by laminate type, but the high Tg laminate D sometimes gave slightly higher values than the other laminates. Solder paste P1 SIR per IPC-TM-650 method 2.6.3.3 is slightly higher on laminate F than laminate E. This was also the case for the 50C/90%RH/5V ECM measurements. Since it is clear that FR4 laminate influences SIR measurements of soldering materials, we recommend the type of FR4 used should also be included in SIR test reports of soldering materials.

High comb down SIR values are achievable on high Tg laminate E by using no-clean fluxes that contain rosin. The comb-down, and to some extent the comb-up SIR values, increase as the concentration of rosin in the fluxes increase.

**References**


