

May 15, 2017

Glicoat[™]-SMD F2(LX)

Organic Solderability Preservatives / Copper Passivation

1. GENERAL INFORMATION

Most of OSPs in the market form the organic coating, not only on copper, but also on gold a little bit, in case of copper and gold mixed PWBs, for example SIMM/DIMM module PWBs. Obviously to mask gold with tape or peelable ink might solve this problem, however it always creates lots of problem to peel it off by hand. However, Glicoat-SMD F2(LX) makes no deposit on gold without any such extra process.

Glicoat-SMD F2(LX) forms uniform, very thin and transparent organic coating on copper surface like other Glicoat products, even in via-holes by chemical reaction between the active ingredient, substituted imidazole derivatives, and copper.

Glicoat-SMD F2(LX) coating has excellent heat-resistance and compatibility with non-clean soldering fluxes and solder pastes, as well as lead-free solder, so that it is suitable for PWBs with surface mount technologies as a replacement of HASL and other metal finishings.

2. FEATURES

- 1) Excellent heat-resistance to stand multiple heat cycles
- 2) No deposit on gold surface, only on copper.
- Excellent compatibility with non-clean soldering fluxes and solder pastes to achieve satisfactory solderability in plated through-holes and spreadability on SMT pads even after multiple heat cycles.
- 4) Excellent humidity resistance to prevent copper oxidation.
- 5) Non-sticky, thin and uniform coating on copper basically
- 6) Because of chemical reaction, no coating and residues (less ionic contamination) on solder mask, carbon paste and most of metal coating except gold, so that suitable for non-clean process.
- 7) Because of aqueous and weak acetic acid base solution, and no solvent contained, environmentally-friendly and less corrosive than formic acid base solution
- 8) Chemically and thermally very moderate process, so that no damage to solder mask like peeling problem by HASL and Ni/Au plating
- 9) Less solder ball problem on solder mask in comparison with HASL
- 10) Excellent solder joint strength in comparison with especially Ni/Au plating

3. PHYSICAL PROPERTIES

Appearance:	Light yellow transparent aqueous solution
pH (20°C):	3.85
Specific gravity (20°C):	1.0
Odor:	Weak acetic acid odor
Others:	Not hazardous under UN / IATA regulation

4. PROCESS OF TREATMENT

Typical process of whole Glicoat-SMD F2(LX) treatment and remarks of each process are given as follows.

The most critical processes are "3) Microetching", "7) Air knife" and "8) Glicoat-SMD F2(LX)" to control the solution and the coating consistently, and to give the best performance of Glicoat-SMD F2(LX) coating itself.

Read carefully before designing the line.

1) Degreasing

Highly recommendable especially for print-etch boards due to oil residue by punching, but also for double sided and multilayer boards due to severe copper oxidation by curing for solder mask ink.

- Water rinse
 3 cascade rinse
- 3) Microetching
 - a) Hydrogen peroxide base with no anti-tarnish ingredients contained is recommendable, because of better solder flow-up and solder paste spreadability. SPS (Sodium Persulfate) base also may be used, but it is recommendable to be followed by acid cleaning to remove away any residue.
 - b) 1.5 micron thick at least should be microetched to achieve good solderability.
- 4) Water rinse

3 cascade rinse with sufficient fresh water influent to minimize the drag-out.

5) Acid cleaning

No special chemistry is required, for example 5% sulfuric acid aqueous solution should be enough to clean away residue of SPS base microetching solution. Therefore, it might not be required after hydrogen peroxide base microetching solution.

6) Water rinse

3 cascade rinse with sufficient fresh water influent to prevent contamination into Glicoat-SMD F2(LX) solution.

It is recommendable to measure pH as a reference of drag-in of pretreatment chemistry.

7) Air knife

Blow away water residue completely, especially in through-holes, in order to prevent contamination into Glicoat-SMD F2(LX) solution. No hot air is required.

- 8) Glicoat-SMD F2(LX)
 - a) The immersion at 40°C for 60 90 sec. is typical to obtain the optimum coating thickness between 0.15 and 0.30 micron.
 - b) pH and concentration of active ingredient should be controlled between 3.80 and 4.10, 90 and 110% respectively, in accordance with the maintenance on page 6.
 - c) Add 1% of "Replenish A" to even fresh solution when making-up, and appropriate amount of "Replenish A" when sufficient coating thickness can not be obtained accidentally, in spite of all parameters like concentration, pH, temperature and immersion time within optimum range.
 - d) It is highly recommendable to immerse PWBs in the overflowed solution rather than spray, in order to make sure of Glicoat-SMD F2(LX) solution even in via-holes. To supply the solution by jet nozzle from the bottom is also helpful.
 - e) Glicoat-SMD F2(LX) solution is ready to use without dilution.

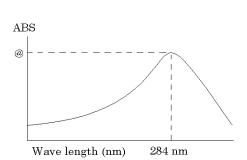
- f) Transfer roller should be designed by light weight shaft and gear type roller.
- g) No rubber materials like EPDM must be employed for any parts in Glicoat-SMD F2(LX) process. It is very difficult to clean the surface, just in case of the crystallization formed.
- 9) Squeezing rollers
 - a) It is very helpful to minimize the drag-out of Glicoat-SMD F2(LX) solution, and to form uniform coating as well rather than air knife.
 - b) Soft PP or PVAc like sponge is recommendable.
 - c) It is not recommendable to employ air knife also, since the roller is always dried so that the crystallization of the active ingredient from the drag-out solution could be easily formed, and make the roller sticky. In the worst case, the coating could be peeled off by the sticky roller, and transferred back to PWBs.
- 10) Water rinse
 - a) 3 cascade rinse with sufficient fresh water influent.
 - b) D.I. water is recommendable to decrease ionic contamination value on the solder mask.
 - c) The pressure of water spray should be moderate, since the coating is still soft.
- 11) Drying

80-90°C for 30 sec. with hot air blow to make sure of no moisture in the through-holes.

5. MEASUREMENT of COATING THICKNESS

The coating thickness can be measured by only UV spectrophotometer in accordance with the following method.

With regard the UV to specification of spectrophotometer, either double or single beam may be used. However, it is very important the band width must be 2 nm, and the absorbance of the "peak around 284 nm" must be measured. In case of bigger band width, the curve becomes broad, so that the measurement always becomes smaller. And the peak might not exactly appear at 284 nm, due to the calibration of the instrument. Therefore, UV spectrophotometer which can automatically scan the peak is highly recommendable.



Otherwise, make sure the reading is the highest around 284 nm by changing the wave length manually, for example compared with the absorbance at 283.5 and 284.5 nm.

- 1) Prepare enough "dissolving solution" by diluting 27.8 gram of 36% conc. hydrochloric acid (HCl) and 100 gram of methyl alcohol (CH_3OH) with D.I. water to 1 liter.
- 2) Scrub copper surface of sample boards (bare copper, not etched out) thoroughly by using e.g. pretreatment machine for solder mask printing, in order to remove chromate plating first and make sure of chemical reaction with the active ingredient.
- 3) Treat them with Glicoat-SMD F2(LX) and cut them into appropriate size : S cm². (e.g. 4 x 4 cm=16cm²). They should be V-cut before treatment to cut easily by hand.
- 4) Place the cut board in the beaker, pour V ml (e.g. 25 ml) of the dissolving solution, and shake the beaker for 1 minute, in order to dissolve the coating formed on the copper completely.

- 5) After taking out the board of the solution, measure *the maximum absorbance (A1) of the peak of the above solution around 284 nm*, in comparison with the dissolving solution as a reference.
 - Make sure the crystal cell is dust-free and stainless. Dust and stain would cause error in measurement.
 - Make sure no bubble in the solution.
- 6) The coating thickness (T micron) can be obtained from the following formula.

T = 0.105 x A1 x V / S

6. MEASUREMENT of ACTIVE INGREDIENT CONCENTRATION

As the same manner as the coating thickness, the concentration of the active ingredient can be measured by UV spectrophotometer in accordance with the following method.

- 1) Prepare enough "dissolving solution" by diluting 27.8 gram of 36% conc. hydrochloric acid (HCl) and 100 gram of methyl alcohol (CH₃OH) with D.I. water to 1 liter.
- 2) Dilute Glicoat-SMD F2(LX) sample solution with the dissolving solution in the exact ratio of 1:249. For example, measure precisely 0.2 ml of sample solution by using 0.2 ml hole pipette, and put it into 50 ml flask and dilute with the dissolving solution to 50 ml exactly.
- 3) Measure the maximum absorbance (A2) of the peak of the above solution around 284 nm, in comparison with the dissolving solution as a reference.
 - Make sure the crystal cell is dust-free and stainless. Dust and stain would cause error in measurement.
 - Make sure no bubble in the solution.
- 4) The concentration of the active ingredient can be obtained from the following formula.

C = 104 x A2

7. MEASUREMENT of pH

- 1) It is recommendable to use pH meter designed for Glicoat-SMD F2(LX) only. Avoid using of the pH meter for both acidic and basic chemistry.
- 2) Calibrate pH meter once a week regularly by 3-point method at pH 6.86, 4.01 and 1.68 at 20°C.
- 3) It must be measured at 20°C after cooling down sample solution, since pH of any chemical solution becomes higher as higher temperature. For example, pH of Glicoat-SMD F2(LX) at 30°C and 40°C is approx. 3.95 and 4.0 respectively.

8. MAINTENANCE

- 1) pH (20°C) : 3.80 4.10
 - a) pH is the most important factor to maintain the coating thickness consistently, so that it should be measured every day especially before production. Coating thickness becomes thinner as pH decreases, and thicker as pH increase.
 And In case of too high pH, the crystallization would appear in the solution.
 - b) Because of drag-in of rinsing water and evaporation of acetic acid, pH tends to be higher, therefore make pH lower by adding 5% acetic acid aqueous solution in case of more than pH 4.1. Do not use concentrated acetic acid.
 - c) pH of any chemical solution becomes higher at higher temperature, therefore measure pH until the solution is cooled down at 20°C. Just for reference, pH of Glicoat-SMD F2(LX) at 30°C and 40°C is approx. 3.95 and 4.00 respectively.
 - d) Use pH meter in the laboratory, and calibrate it regularly by 3-point method. Automatic pH meter equipped in the line might not be so accurate, since the electrical probe is always in the solution.
- 2) Concentration of active ingredient: 90-110%
 - a) It is recommendable to maintain the concentration of the active ingredient within the above range as much as possible, in order to keep the thickness within the optimum range consistently.
 - b) In case of too high concentration, the crystallization would appear in the solution.
- 3) Thickness of coating: 0.15 0.30 micron
 - a) Finally, the thickness of the coating has to be controlled between 0.15 0.30 micron as correct as possible.
 Less than 0.12 micron should not be thick enough to protect copper oxidation during storage and heat cycles. However, more than 0.40 micron should be too thick to be removed away by soldering flux, then could worsen the solderability.
 - b) Since the proper thickness is determined by lots of factors like storage, reflow heating, wave soldering, activation of soldering flux and design of PWB, etc., preliminary tests are recommendable.
- 4) Level of solution

Check the level of solution every day, and keep it as consistent as possible. The quantity consumed is related to drag-out with PWBs, drag-in from pretreatment process and evaporation of water and acetic acid contained in Glicoat-SMD F2(LX), etc. However, the average consumption is approx. $15-20m^2/liter (1x1m=1m^2)$, which is the size of PWB, regardless of the area of copper and solder mask.

5) Replenish A

Add 1% of "Replenish A" to even fresh solution when making-up, and appropriate amount of "Replenish A" in accordance with the following procedure, when sufficient coating thickness can not be obtained accidentally, in spite of all parameters like concentration, pH, temperature and immersion time within optimum range.

- a) Specification
 Appearance: Transparent liquid
 pH: 7-9
 Specific gravity: 1.00 +/-0.10 (20°C)
- b) Dosage
 - i) Take out 300 ml of Glicoat-SMD F2(LX) solution exactly of the production line in 300 ml beaker. The depth of the solution is approx. 7.2 cm.
 - ii) Cool down or warm up the solution at 30°C.

- iii) Put magnetic stirrer into the beaker, and add 0.5 ml each of "Replenish A" gradually until the magnetic stirrer can not be seen from the top, due to turbid solution.
- iv) Dosage of "Replenish A" can be obtained by the following formula. However, if the magnetic stirrer cannot be seen less than adding 4.5 ml, it is not necessary to add into the production line.

Dosage of "Replenish A" = $(A - 4.5) \times B / 300$

A: amount of "Replenish A" added into the beaker (ml)

B: amount of Glicoat-SMD F2(LX) solution in the production line (ml)

- c) How to add
 - i) Dilute "Replenish A" obtained by the above formula with three times of D.I. water.
 - ii) Add the diluted "Replenish A" into the bottom tank of the production line with circulating the solution, which should be kept around 40°C.
 - iii) Even if the solution becomes turbid just after dosage, keep on circulating Glicoat-SMD F2(LX) solution until it will disappear.

9. PRECAUTIONS

1) Crystallization

Especially, in case of too high pH (more than 4.1) and concentration of active ingredient (more than 120%), the crystallization tends to appear in the solution.

It is common to see a little oil-like film on the surface of Glicoat-SMD F2(LX) at the initial stage, as an indication of crystallization.

Then, some small white crystals form on the surface. As crystallization advances, the increased crystals stick on the walls of the tank, the transfer rollers, and even PWBs in the worst case.

Therefore, observe the surface regularly, and take quick action to prevent serious crystallization problem.

Trouble shooting

- a) Remove crystals on the surface with cloth like oil mat
- b) Add D.I. water to lower the concentration of active ingredient less than 10%
- c) Add 5% acetic acid solution to decrease pH between 3.80 and 4.10, heat up Glicoat-SMD F2(LX) solution in the tank to 40°C, then circulate thoroughly to dissolve the crystal.
- 2) Cleaning of squeezing rollers

The squeezing roller just after Glicoat-SMD F2(LX) treatment tends to be crystallized, especially when not operating for a long time.

Therefore, it is highly recommendable to spray the roller with small amount of water after operation to clean away Glicoat-SMD F2(LX) solution remained.

Use 5% acetic acid solution to clean the crystallization on rollers with cloth. However, in case of much crystallization accumulated and hard to remove, dip the roller into "dissolving solution, see 5. 1)" for a while, then wipe off with cloth. In addition, it is recommendable to have spare rollers, and change regularly. 3) Cleaning of wall

It is very common to have crystals on the wall of upper tank of Glicoat-SMD F2(LX), since the over-flowed solution is splashed and only the active ingredient is remained after evaporation of water and acetic acid, etc.

Therefore, clean away the crystal with brush regularly using over-flowed Glicoat-SMD F2(LX) solution.

4) Evaporation

For efficient use of Glicoat-SMD F2(LX), minimize the standby time of the process line as water and acetic acid evaporate while the system is on standby as well as in operation.

Since Glicoat-SMD F2(LX) has smell of acetic acid, ventilation is advisable. However, avoid excessive ventilation, which may cause over-evaporation and too high concentration easily.

Therefore, while the system is not in operation, minimize ventilation. Seal gaps of the compartment to decrease evaporation and smell of acetic acid.

5) Handling and packing of PWBs

Glicoat-SMD F2(LX) coating is reliable enough against moisture from a long term point of view, however could be easily decomposed by water dew and sweat.

Therefore, PWBs after Glicoat-SMD F2(LX) treatment should be handled with glove, and don't touch copper pad directly. It should be packed with plastic bag, if possible with vacuum.

10. MATERIAL for PROCESS

The following materials are compatible or incompatible with Glicoat-SMD F2(LX) solution.

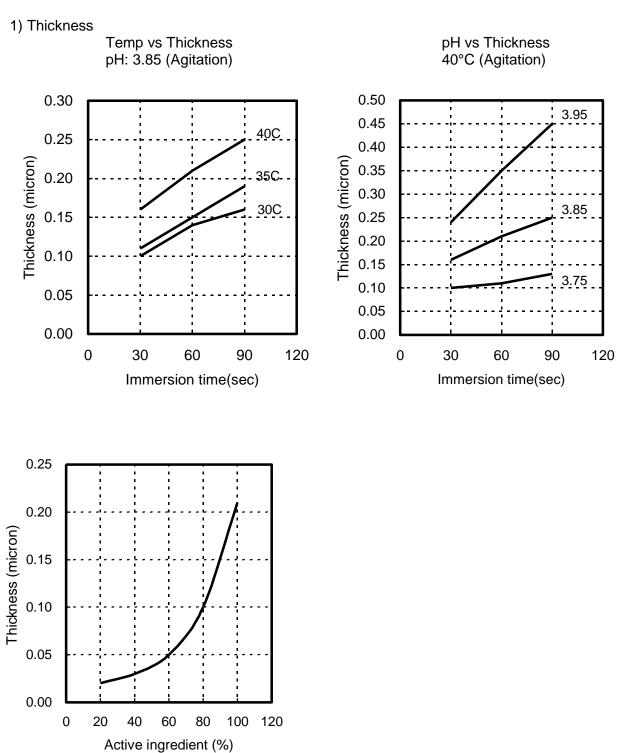
- Recommendable Hard PVC, Polyethylene, Polypropylene, Acryl resin, Poly carbonate, ABS, Teflon, Silicon rubber, SUS 304 / 316 / 316L, Titanium
- 2) Not recommendable Soft PVC, Rubber, PVA without surface treatment

11. TROUBLE SHOOTING

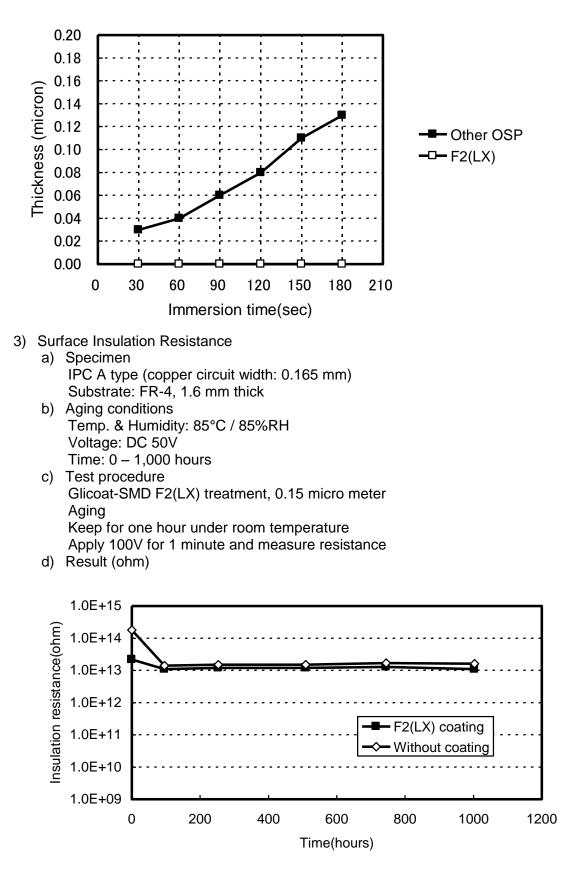
Trouble	Cause	Measure	
High pH	Excessive evaporation of acetic acid due to gaps of compartment	Seal the gaps to make the compartment airtight	
	Excessive evaporation of acetic acid due to excessive ventilation	Decrease ventilation	
	Insufficient replenishment of acetic acid	Increase replenishment of acetic acid	
	Too long standby	Operate more efficiently	
Low pH	Excessive replenishment of acetic acid	Do not add acetic acid until Ph recovers. Must use acetic acid with 5% dilution	
	Drag-in of microetching solution	Improve the efficiency of air knife. Increase the influent of rinsing water	
High concentration of active ingredient	Excessive replenishment of Glicoat-SMD F2(LX)	Decrease replenishment of Glicoat-SMD F2(LX)	
	Concentration by evaporation	Replenish deionized water and acetic acid	
Low concentration of active ingredient	Insufficient replenishment of Glicoat-SMD F2(LX)	Increase replenishment of Glicoat-SMD F2(LX)	
	Drag-in of rinsing water	Prevent drug-in of rinsing water to improve the efficiency of air knife	
Insufficient thickness	Improper operating conditions (temperature, time)	Refer to the recommended conditions	
	Low pH or low concentration of the active ingredient	Increase the pH or active ingredient	
	Excessive contents with squeezing rollers	Decrease pressure and number of contacts	
	Microetching solution with anti-tarnish ingredient contained	Change microetching solution, or consider acid rinse process	
	Anti-tarnish plating like chromate	Scrub copper surface by the pretreatment machine before Glicoat-SMD F2(LX)	
Crystallization on the surface of Glicoat-SMD F2(LX)	High pH	Replenish acetic acid and circulate in a few hours	
	High concentration of active ingredient	Dilute with deionized water to below 110%	
Foreign objects stick on PWBs	Foreign objects transfer from the rollers	Clean the roller with weak acetic acid to remove	
	Glicoat-SMD F2(LX) contains foreign objects	Filter Glicoat-SMD F2(LX) to remove foreign objects	
Insufficient heat resistance	Improper thickness of coating	Adjust thickness between 0.15 - 0.30 micron	
	Improper drying conditions, and moisture is not dried up completely	Increase the temperature and time of drying	

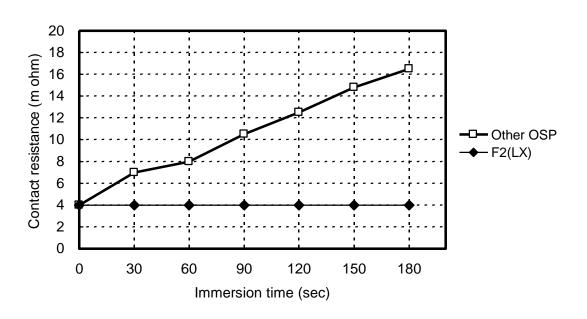
	Contamination or aging of Glicoat- SMD F2(LX)	Replace with new Glicoat-SMD F2(LX)	
Unevenness in Glicoat-SMD F2 (LX) coating	Stained surface of PWBs	Enhance microetching to remove stain	
	Insufficient thickness of microetching	Make sure of 1.5 micron at least by analyzing the conditions of microetching	
	Insufficient thickness of the coating	Adjust thickness between 0.15 - 0.30 micron	
	Excessive contacts with rollers	Use light material for shaft like carbon fiber	
	Too high air knife pressure after Glicoat-SMD F2(LX)	Decrease the pressure	
	Too high spray pressure of water rinse after Glicoat-SMD F2(LX)	Decrease the pressure	
Sudden whitening of Glicoat-SMD F2(LX)	Abnormal drug-in of microetching solution	Terminate drag-in of microetching	
Abnormal smell of acetic acid	Insufficient ventilation	Open up ventilation duct or install more ducts	
	Excessive evaporation of acetic acid due to improper sealing of the compartment	Make compartment airtight	
Excessive decrease of the level of Glicoat-SMD F2(LX)	Excessive drag-out	Prevent drag-out to improve air knife after Glicoat-SMD F2(LX)	
	Leak from the tank	Repair the tank	
	Excessive evaporation	Adjust ventilation and avoid long standby	
Excessive increase of the level of Glicoat-SMD F2(LX)	Excessive drag-in of rinsing water before Glicoat-SMD F2(LX)	Prevent drag-in of rinsing water before Glicoat-SMD F2(LX)	

12. PROPERTY



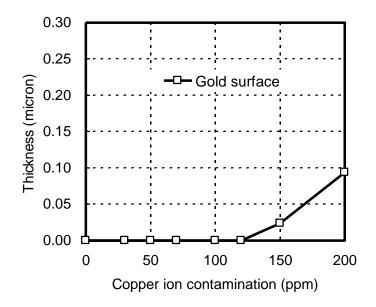
2) Film on gold





4) Contact resistance on gold surface

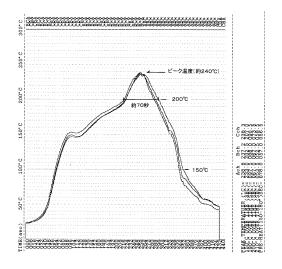
5) Influence of copper ion contamination

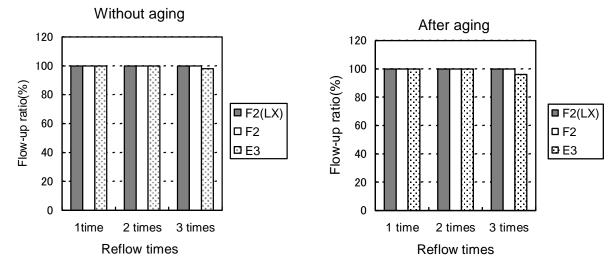


6) Solderability

6-1) Flow-up ratio

- a) Specimen Double side board, FR-4, 1.2 mm thick, Through-hole diameter: 0.6 mm, 629 holes
- b) Reflow condition
 Equipment: IR type under air
 Heat profile: As right side
 Peak temperature: approx. 240°C
 Over 200°C : approx.70 sec.
- c) Soldering condition
 Solder bath temperature: 245 255°C
 Soldering flux: JS-64MSS (Koki made)
 (Solid content: 14%, Cl content: 0.07%)
- d) Aging condition 40°C/90%RH/96hours
- e) Result





6-2) Spreadability

- a) Specimen
 FR-4, 1.6mm thick,
 Copper line: 0.8 mm width, 20 mm length, 10 lines
- b) Printing condition of solder paste1.2 mm diameter, 150 micro meter thick, by using metal mask
- c) Solder paste

Formula	Melting point (°C)	Flux content (wt%)	CI content (wt%)
Sn63/Pb37	183	9.5	0.13

- d) Reflow condition Equipment: IR type under air Heat profile: Refer to 6-1)-b) Peak temperature: approx. 240°C Over 200°C : approx.70 sec.
- e) Aging condition 40°C/90%RH/96hours
- f) Result

