Solderability and Reliability Evolution of no-Clean Solder Fluxes for Selective Soldering

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Abstract

Flux consumption for wave soldering tends to decrease, mainly due to its gradual replacement by reflow soldering methods (i.e. pin-in-paste) in many electronics applications. However, in several cases, wave soldering still remains a must, with an increasing share of "selective" soldering processes, either using wave frames with dedicated apertures or solder fountains. Such processes are more challenging for the fluxes in terms of reliability under operation, since some chemistries remaining on the printed circuit boards after soldering may promote corrosion. Thus, flux manufacturers had to adapt their formulations to minimize such issues while keeping an efficient activation level, with several types of alloys (tin-lead, tin-silver-copper and low/no-silver) and associated with the numerous types of finishes encountered.

The paper will cover the types of flux used in the electronic industry according to their chemistry and activation level (rosin-based, halides, alcohol-based or water-based flux…), and their characteristics with reference to standards. The limits of current standards will be discussed in regards to the last generation solder fluxes.

Then, the development of two low-residue new generation fluxes, an alcohol-based flux and a true VOC-free flux, will be described, according to requirements: the lab tests results (surface tension, spread tests, wettability tests…) will be presented and discussed. Reliability will be especially investigated through surface insulation resistance, electro-chemical migration test, ionic contamination as well as Bono tests to determine the candidates able to provide high processability combined with chemical inertness of residues. Finally, the performance of flux will be assessed through customer tests, involving several types of boards, finishes and different solder alloys and wave equipment.

Introduction

Flux media are described through several standards (ISO, IEC, IPC…). But the IPC J-STD-004 standard "Requirements for Soldering Fluxes" is the most commonly used. Its purpose is to classify and characterize all soldering flux materials for use in electronic assembly: liquid flux, paste flux, solder paste flux, flux-coated and flux-cored solder wire and preform. Soldering materials are classified according to their composition and activity. Four main flux categories are described: Rosin (RO), Resin (RE), Organic (OR) and Inorganic (IN). J-STD-004B standard Amendment 1 explained the difference between rosin and resin. "A Resin Flux is primarily composed of synthetic resins and/or natural resins other than rosin types. A Rosin Flux is primarily composed of natural rosin, extracted from the oleoresin of pine trees and refined. The rosins used shall have a minimum acid value of 130 as determined per ASTM D-465. A synonym for rosin is colophony."

Three flux activity levels are determined: low or no flux/flux residue activity (L), Moderate flux/flux residue activity (M) and High flux/flux residue activity (H). The activity level is assessed by the following tests: copper mirror, copper corrosion, surface insulation resistance (SIR) and electrochemical migration (ECM). The absence (0) or presence (1) of halides in the flux complements the classification, the absence of halide of 0.0% (0) being defined as below 0.05% (<0.05%). Fluxes with halide content below 0.05% may be known as halide-free. Thereby, each type of flux is identified. This identification is presented in Table 1, extracted from IPC J-STD-004B.

A few comments are necessary to fully understand the classification and its implications. The flux composition is based on the largest weight percent constituent of its non-volatile portion. Thus, an "Organic Flux" (OR) does not contain any resin or rosin as a main ingredient, but it may contain such ingredients. According to the definition of rosin, an esterified rosin, which acid index is very low, can't be classified as rosin. Rosins which have been chemically modified should not anymore be classified as rosins; nevertheless there is still some room to the interpretation of the limit between rosin and resin. It is therefore important for users not to restrict their choice to only one type of flux composition.

The halide content is determined per IPC-TM-650, test method 2.3.28.1. The concentration of chloride (Cl-), bromide (Br-), fluoride (F-) and iodide (I-) is measured by ion chromatography and is reported as the equivalent weight percentage of chloride to the non-volatile portion of the flux. Halides have been used for years as activators in flux materials, to improve the wetting properties. Sodium chloride (NaCl or Na^+Cl^- after dissolution) is the more famous example of halide. Of course it is not employed in flux as it does not help wetting at all and generates corrosion. Halides used in fluxes were mainly amine hydrochlorides or amine hydrobromides such as diethylamine hydrobromide or cyclohexylamine hydrochloride. Due to some decomposition products remaining in the residue after soldering tending to cause some electrochemical corrosion in harsh environment, their use has decreased in the electronic assembly.

The other ingredients used to enhance wettability are acids, amines, and compounds containing halogens. Halogens are a group of elements belonging to the 17^{th} column of the periodic table according to the modern IUPAC nomenclature (formerly column VIIA) including fluorine, chlorine, bromine and iodine. Halogens are highly electronegative: their valence shell contains seven electrons and easily gains one more electron to saturate to eight (octet rule), either by forming halide ions or by forming covalent bond.

Chemical compounds containing one or several halogen atoms linked by a covalent bond are commonly called halogens while the compounds containing halogen atoms in the ionic form are called halides. Thus, in the IPC J-STD-004B, halogen is the term for all chlorine (Cl) and/or bromine (Br) in compounds. The determination of halogen concentration in flux is described in EN14582. First, the combustion of the sample is done in a closed system containing oxygen (oxygen bomb test) converting the halogenated compounds to halides, which are absorbed and/or dissolved in an absorption solution. Then, the concentration of each halide is determined by ionic chromatography. As defined in the IPC J-STD-004B, low halogen materials contains ≤1000ppm (0.1%) Br, and ≤1000ppm (0.1%) Cl.

Table 1. Flux identification system as described in J-STD-004B.

Flux Composition	**Flux/Flux Residue Activity Levels**	**% Halide[1]** (by weight)	**Flux Type[2]**	**Flux Designator**
Rosin (RO)	Low	<0.05%	L0	ROL0
		<0.5%	L1	ROL1
	Moderate	<0.05%	M0	ROM0
		0.5-2.0%	M1	ROM1
	High	<0.05%	H0	ROH0
		>2.0%	H1	ROH1
Resin (RE)	Low	<0.05%	L0	REL0
		<0.5%	L1	REL1
	Moderate	<0.05%	M0	REM0
		0.5-2.0%	M1	REM1
	High	<0.05%	H0	REH0
		>2.0%	H1	REH1
Organic (OR)	Low	<0.05%	L0	ORL0
		<0.5%	L1	ORL1
	Moderate	<0.05%	M0	ORM0
		0.5-2.0%	M1	ORM1
	High	<0.05%	H0	ORH0
		>2.0%	H1	ORH1
Inorganic (IN)	Low	<0.05%	L0	INL0
		<0.5%	L1	INL1
	Moderate	<0.05%	M0	INM0
		0.5-2.0%	M1	INM1
	High	<0.05%	H0	INH0
		>2.0%	H1	INH1

In addition to the above mentioned compounds, organic weak acids are used to improve wetting performance: polycarboxylic acids and especially dicarboxylic acids with short to medium chains like succinic, adipic, sebacic acids are widely used because of their higher acid index compared to monocarboxylic acids, which provides more efficiency with the same percentage in the formulation.

Fluxes contain other ingredients. Among those, surfactants play an important role in liquid flux formulation. Their role is to lower the surface tension of the flux, which promotes spreading in the plated through holes. A considerable number of surfactants are available and many new surfactants are brought to the market every year. The solvent used in liquid flux designed for wave soldering is isopropanol (IPA) which surface tension is relatively low, around 22mN/m (or dynes/cm) at 20°C; ethanol, which surface tension is about the same, is sometimes used but in small quantity due to its tendency to easily transform acids in esters, which have no acid index and no deoxidizing ability. The effect of surfactant is even higher in water-based or VOC-free flux (VOC stands for volatile organic compound).

Deionised water surface tension is about 73mN/m, which is high; it has to be lowered by surfactants for such fluxes to allow sufficient spreading. Low VOC-free fluxes, which are often a 50/50 water/IPA mixture, have a surface tension of about 25mN/m, which is quite close to IPA alone. Moreover, surface tension decreases with temperature [1] so it is recommended to use flux after temperature conditioning. All the chemicals entering a liquid flux composition are characterized by their physical properties. Solvents are not characterized by their surface tension only, but by their viscosity, boiling point, evaporation rate, flash point, ability to dissolve or to dissociate some ingredients.

Table 2 presents a few solvents used in liquid fluxes with their boiling point, surface tension, comparative spreading rate and time to evaporation. Activators have a melting point, boiling point, sublimation or decomposition temperature, solubility, etc. When resins or rosins are involved, their color and softening temperature are key parameters for cosmetics appearance and for in-circuit-testability (ICT). As most applications are no-clean, all chemicals, including surfactants, may have an impact on post-reflow reliability, especially in case of selective soldering.
As mentioned before, the activity level of a flux is assessed by copper mirror, copper corrosion, SIR and ECM: details are given in Table 3. For liquid flux, the copper corrosion test is done on the sample “as received” with an exception for

water-based and some low-VOC fluxes (containing more than 50% water) as they may fail the test due to the presence of water. Thus, it is possible to oven dry the flux and to dissolve its non-volatile residue in the appropriate solvent before copper test. However, in this case, results from both "as-received" and reconstituted samples shall be reported. For SIR and ECM, tests coupons must be subjected to leaded and/or lead-free thermal profile, depending on the product end-use.

Table 2. Some characteristics of solvents used in liquid fluxes.

Solvent	Boiling point (°C)	Surface tension (mN/m)	Spread diameter (cm)	Evaporation
Isopropanol	82.6	22	5	<2min
Ethanol	78.4	22	5	<2min
Deionized water	100	73	2	>5 min
Solvent 1	NC	NC	5	>5min
Solvent 2	NC	NC	5	3-4min
Solvent 3	NC	NC	5	<2min

NC: non communicated.

Spread diameter measured on cleaned and degreased bare copper with 50µl flux dropped

The resistance to ECM is determined on IPC-B-25 or IPC-B-25A coupons (combs with 0.318mm lines and 0.318mm spacing). Coupons are exposed to wave soldering (pattern down) and are then exposed to temperature and humidity (65°C ± 2°C, 88.5% ± 3.5% RH condition) for 96 hours without bias, then with 10VDC during 500 hours.

For SIR, three coupons (IPC-B-24 with 0.4mm lines and 0.5mm spacing) are exposed to wave soldering pattern side down and three other coupons pattern side up. Coupons are exposed to 85°C and 85% relative humidity during 168 hours with 45-50VDC bias voltage. The minimum of 100 MΩ is required after 24 hours of exposure.

Table 3. Test Requirements for flux classification as described in J-STD-004B.

Flux Type	Copper Mirror	Corrosion	Quantitative Halide[1] (Cl^-,Br^-,F^-,I^-) (by weight)	Conditions for Passing 100 MΩ SIR Requirements[2]	Conditions for Passing ECM Requirements
L0	No evidence of mirror breakthrough	No evidence of corrosion	<0.05%[3]	No-clean state	No-clean state
L1			≥0.05 and <0.5%		
M0	Breakthrough in less than 50% of test area	Minor corrosion acceptable	<0.05%[3]	Cleaned or No-clean state[4]	Cleaned or No-clean state[4]
M1			≥0.5 and <2.0%		
H0	Breakthrough in more than 50% of test area	Major corrosion acceptable	<0.05%[3]	Cleaned	Cleaned
H1			>2.0%		

1. This method determines the amount of halide present (See Appendix B-10).
2. If a printed circuit board is assembled using a no-clean flux and it is subsequently cleaned, the user should verify the SIR and ECM values after cleaning. J-STD-001 may be used for process characterization.
3. Fluxes with halide measuring <0.05% by weight in flux solids may be known as halide-free. If the M0 or M1 flux passes SIR when cleaned, but fails when not cleaned, this flux shall always be cleaned.
4. Fluxes that are not meant to be removed require testing only in the no-clean state.

Today, most fluxes used in electronic assembly are no-clean L0 or L1 low residue. But, on one hand, lead-free SnAg3Cu0.5 (SAC305) alloy exhibits lower wetting speed than leaded SnPb traditional alloy. Low-silver and no-silver alloys wetting performances are even poorer, requiring fluxes with strong activation. On the other hand, wave soldering is turning more and more towards selective soldering: excess of non heated or partially heated no-clean flux remaining on the boards can cause concern about long term reliability in harsh environments. These contradictory requirements are the major drives to new generation no-clean flux development.

The paper will describe the procedures used to characterize fluxes, the methodology used for development. In-house tests results will be presented and a few industrial tests will be given.

Description of laboratory tests

Flux spreading

50 µl of flux (or flux solvent) is dropped on a cleaned and degreased surface. The diameter after spreading is measured. The test is preferentially performed on copper substrate but can be done on other metal finishes as electroless nickel immersion gold (ENIG), hot air levelled tin (HASL), immersion tin or on several solder masks having different surface tensions.

Residue spreading and cosmetics after soldering
50 µl of flux is dropped on a cleaned and degreased surface (alumina, solder mask, bare copper…) and heated. Several conditions may be used to check the residue appearance according to the flux location. Residues are almost non visible when the flux is in contact with wave. But, when the flux is exposed to lower temperature, which is the case for selective soldering, the residues are more or less visible depending on the formula. Conditions examples: preheat one minute at 160°C then one minute at 250°C or preheat 30sec at 100°C, 30sec at 140°C, 30sec at 160°C.

Flux wetting
A SAC305 alloy ring is placed on a test coupon and 50µL of flux is dropped in the middle of the ring. Several finishes may be used (cleaned and degreased, bare copper, copper OSP, ENIG …). The substrate is submitted to reflow after preheat. Example of test condition: 90 seconds from 20-120°C, reflow at 260°C

Solder balling
Dedicated boards with different finishes (OSP, ENIG) are dipped in flux, preheated and dipped in SAC305. Different preheat time and temperature may be used. Wave temperature is 260°C and contact time 3 seconds. Example of preheat conditions: 90 seconds from 20-120°C.

Surface Insulation Resistance Test (SIR)
Flux is sprayed on IPC-B-25 (60g/cm^2), then coupons are placed in a convection reflow at 130°C to mimic preheat conditions. Then, SIR is performed following J-STD-004B, TM 2.6.14.1.

SIR/ECM on non deactivated flux
A given flux quantity is applied on IPC-B-24 (0.2ml/comb) and the flux is dried at 40°C during 30 min. IPC-B-24 are placed in a climate chamber at 40°C/93%RH, and SIR is measured after 1h, 2h, 3h, 4h, 8h, 12h and 24h. After 24h a constant voltage of 100V is applied and SIR measurements are done at 48h, 168h, 336h and 504h. After test, coupons are visually inspected for migration and corrosion. The test is performed according to DIN EN ISO 9455-17.

Alcohol-based and water-based flux development methodology
Starting from the fact that alcohol based and water based fluxes developed years ago do not meet today's customers requirements, the same methodology is applied to their respective development. For this study, micro-balls reduction, hole filling improvement and high post-soldering residue reliability are the focussed characteristics. The adhesion of conformal coatings was considered as an additional important characteristic.

Practically, after checking performances of some existing fluxes at a laboratory level, goals to achieve were fixed. A large screening of raw materials was done. These raw materials and their combinations were submitted to spreading wettability test. The main types of ingredients were activators and surfactants. Once interesting ingredients were identified, new flux formulations were built and an optimization of the proportions was made. In parallel the ionic conductivity of the raw materials used to prepare fluxes as well as fluxes themselves was measured: in case of selective soldering or soldering using pallets, unburned fluxes remain present may generate corrosion in harsh environment. A selection of optimized versions was fully characterized at a laboratory level: flux spreading, residue spreading, residue cosmetics, wetting, solder balling, copper mirror, standardized SIR/ECM.

Finally, Bono tests [2] on flux dried at room temperature and a special SIR/ECM test on non deactivated flux (dried at low temperature) were performed to predict flux reliability. Several tests were performed in industrial conditions in wave soldering equipments to assess the real performance of new fluxes.

Results
One VOC-free flux (WFA) was taken as a reference to optimize in terms of solder balling and hole filling while keeping a similar residue reliability level. AFB and AFC were taken as initial alcohol-based fluxes: solder balling, hole filling and post-soldering reliability had to be improved. Some characteristics of these fluxes are presented in Table 4.

Table 4. Characteristics of initial fluxes.

Characteristics	Flux classification	Density	Solid Content (%)	Acid Index (mg/g)
WFA	ORL0	1,01	3.5	32
AFB	ORL0	0.80	2.0	18
AFC	ORL0	0.82	1.9	19

Acid index expressed on the total flux, not on the solid portion

The appearance of raw material solutions after preheat on copper was checked, in order to identify the materials leading to bad cosmetics and especially to identify those leading to copper discoloration (Table 5).

Table 5. Appearance of solvents and raw materials solutions after preheat at 100, 140, 160°C (3x30sec) on copper.

Ingredient	Appearance	Ingredient	Appearance
Solvent 2	Transparent	Rosin 4 in IPA	Transparent + slightly yellow
Solvent 3	Transparent	Activator 4 in DI water	White
Activator 3 in IPA	Transparent	Activator 5 in DI water	Slightly white + a few green traces on the edge
Activator 3 in DI water	Transparent	Activator 6 in IPA	Transparent + a few green traces on residue
Rosin 1 in IPA	Transparent	Activator 7 in IPA	White + a few green traces on the edge

The development of two alcohol-based fluxes (AFD and AFE) and one water-based flux (WFF) was achieved. Flux spreading was measured on several surfaces. The comparison was done with other fluxes (AFG, WFH and WFI). Although slightly below the reference fluxes, AFD and AFE spreading was quite similar whatever the surface: copper, ENIG and immersion tin. The test was performed on two types of solder masks: spreading was always higher on SM2 than on SM1 but the difference was more or less significant and was according to the flux. For VOC-free fluxes, WFF was developed: its spreading performance ranked better than WFA. Results are presented in Figure 1. Some fluxes designed for selective soldering claim a low spreading on solder mask to avoid the excess of flux in hidden areas, thus avoiding chemical reliability issues. But it is difficult to predict spreading on the numerous types of solder masks available on the market.

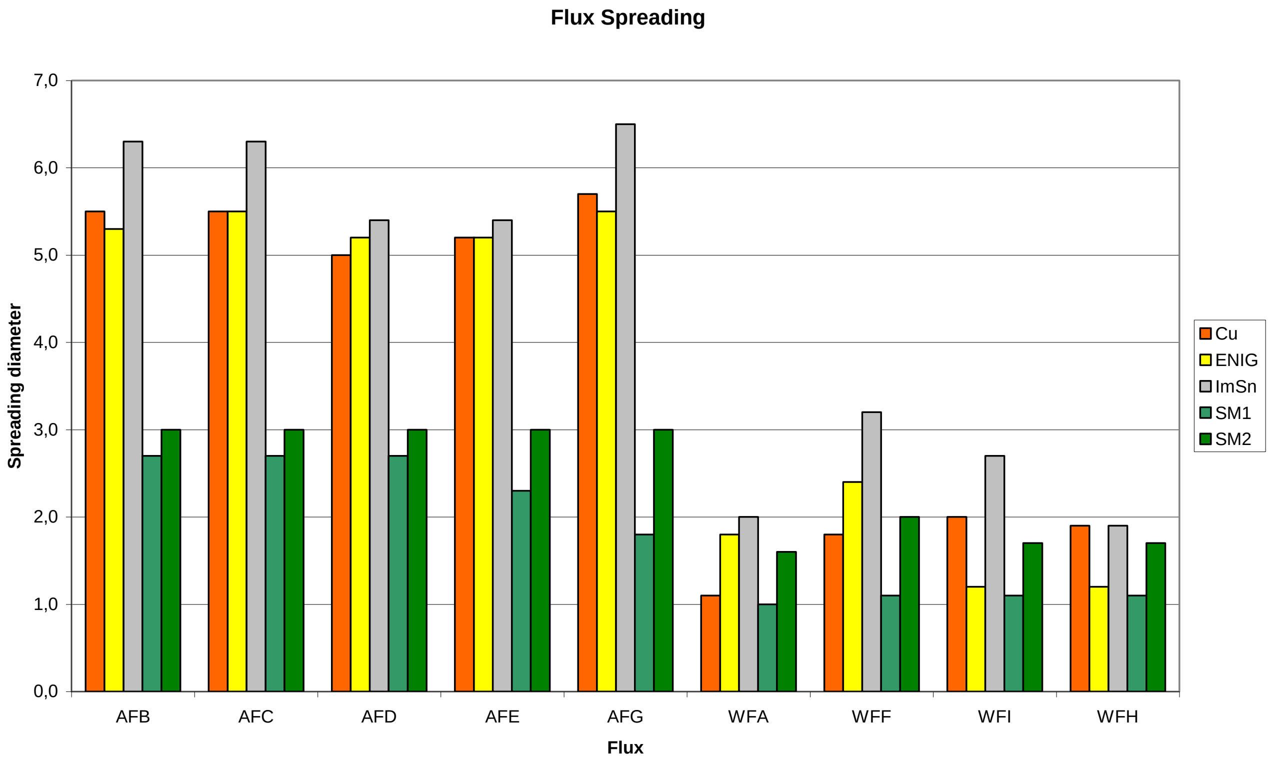


Figure 1. Flux spreading on several substrates.

The flux residue appearance after air drying was checked. AFD, AFE, WFA and WFF exhibited white residues while AFC and AFG residues were transparent. Pictures of the samples are shown in Figure 2. In this development no criterion about residue color was defined but white color residue was even preferred: ingredients leading to transparent residue may affect the chemical reliability.

AFB AFC AFD AFG

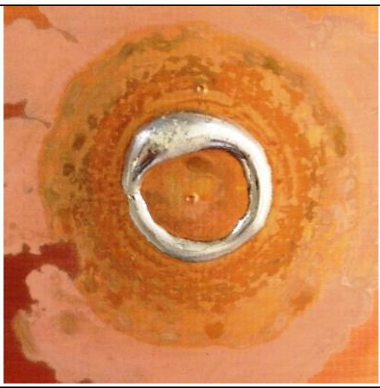
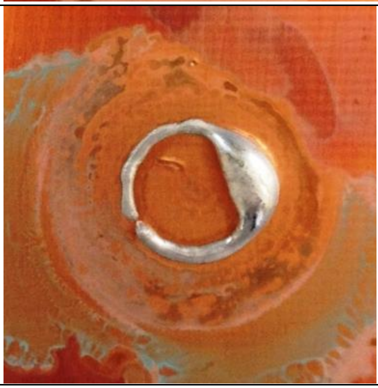
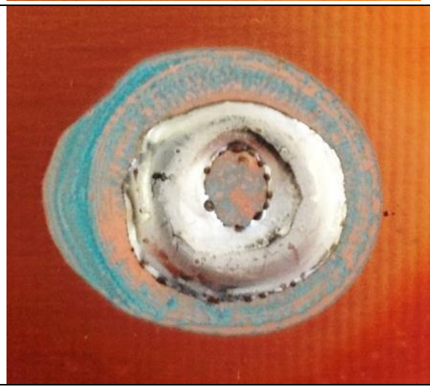
Figure 2. Residue appearance after preheat at 100, 140, 160°C (3x30sec).

Wetting performance was assessed according to Table 6. WFF performance was medium to good on copper and showed a significant improvement compared to WFA as well as WFI. pictures after test are presented in Table 7. Regarding alcohol-based fluxes, AFD wetting was significantly better than AFB but AFE performed less.

Table 6. Wetting classification.

Before test	Bad	Medium	Good
			

Table 7. Examples of water-based flux wetting performance.

Flux	WFA	WFI	WFF
Preheat 100°C 30s			
Preheat 100, 140, 160 3(x30s)			

As far as solder balling is concerned, water-based fluxes generated more micro-balls than alcohol-based fluxes. WFF performed significantly better than WFA and WFI. For alcohol-based ones, the ranking,, from best to worst, was AFD, AFB, AFE, AFG and AFC. AFC and AFD pictures are shown in Figure 3.

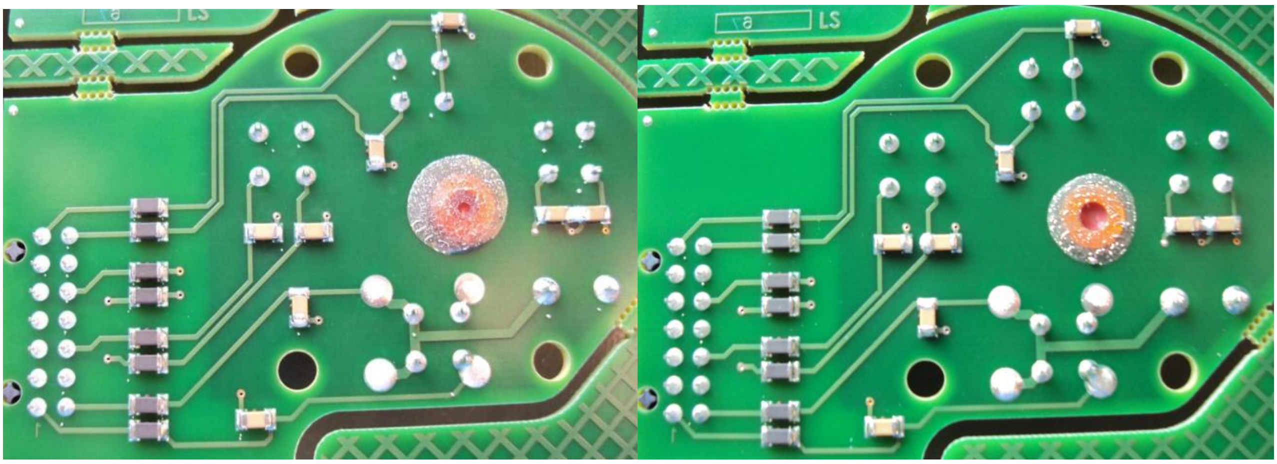


Figure 3. Solder balling test AFC (left) and AFD (right).

Coating compatibility is strongly dependant on flux residue surface tension. Following IPC-HDBK-830 (paragraph 7.3.1), the degree of risk for coating delamination during environmental stress can be evaluated by determining the surface energy of the board before coating. Table 8 (also extracted from IPC-HDBK-830) indicates the expected results according to the surface energy of the surface to coat.

Table 8: Surface energy with expected results as described in IPC-HDBK-830.

Surface Energy (dynes/cm)	**Expected Result**
Above 40	Adhesion expected to be good
35-40	Adhesion generally good, with some intermittent delamination under severe conditions
30-35	Adhesion generally poorer, with increasing incidence of delamination under severe conditions
Below 30	Coating adhesion poor in any climatic testing.

One of the goals of the study was to achieve a good compatibility with coatings. The results for acrylic coating A, which was a coating of interest, are reported. The adhesion of acrylic coating A on unprocessed board was found to be excellent. The adhesion of acrylic coating A was poor on AFG and AFC residues (30% delamination) and good on AFB and AFD residues (5% delamination) with a good correlation with flux residue surface energy (Table 9).

Table 9: Flux residue surface tension and delamination test of acrylic coating A.

board number	0	1	2	3	4	5
board and flux	unprocessed board (reference)	board by customer with AFG	AFG	AFB	AFC	AFD
flux residue after oven	x	visible residues	Visible residue	slight white residues	visible residue	slight white residues
residue surface energy (dynes/cm)	> or = 44	32	32	38	32	38
coating	Acrylic coating drying time: 30mn air + 2hrs 60°C					
adhesion test after 16h	delamination 0%	delamination 30%	delamination 30%	delamination 5%	delamination 30%	delamination 5%

As expected, WFF, AFD and AFE passed copper mirror, SIR and ECM (Figure 4) and ranked ORL0 according to IPC J-STD-004. Neither halides nor halogens were intentionally added. The Bono test was performed on WFF and AFE. Bono boards were dipped in flux and air dried at room temperature. The Bono test was performed at 85°C/85%RH, with 20VDC on test boards (9µm copper thickness / 80 microns width anode between two cathodes of 3mm width with 120 microns space between anode and cathode). Both fluxes passed with a corrosion factor (Fc) of 1% without any corrosion. Those results were similar with WFA and AFB.

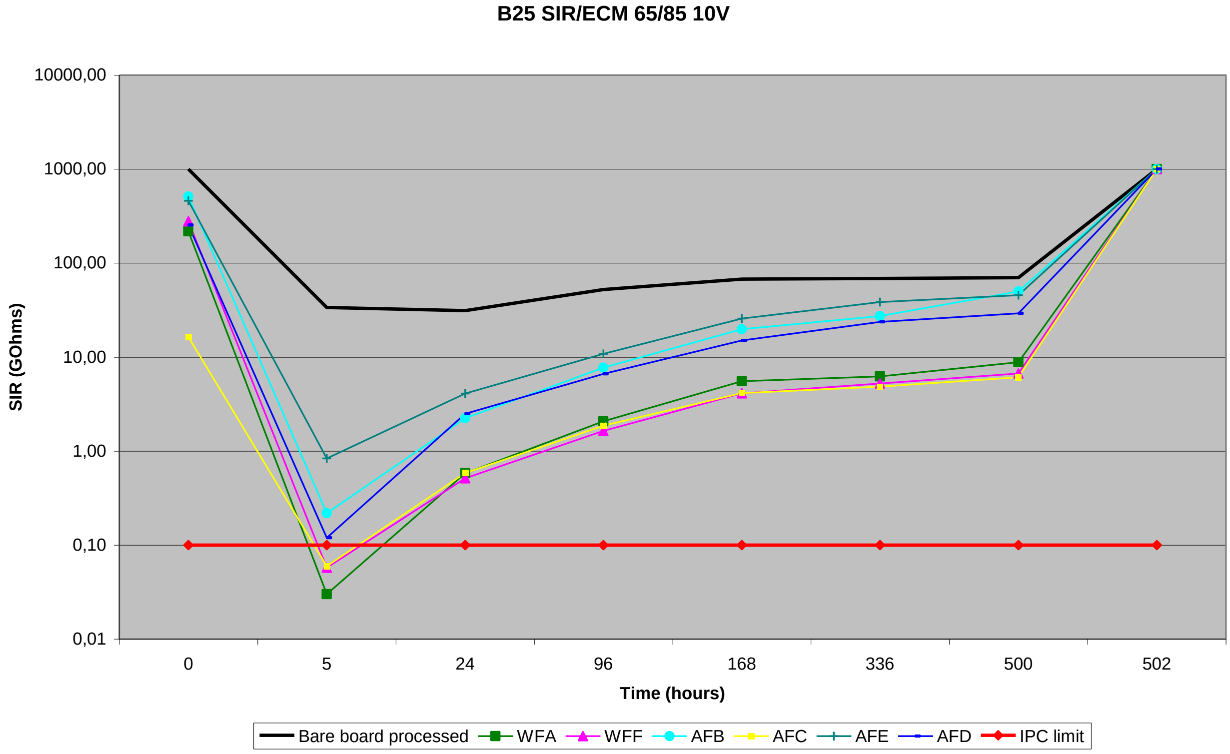


Figure 4. SIR curves of alcohol and water-based fluxes.

Alcohol-based flux AFE was submitted to the more demanding SIR/ECM test. The main difficulties of the test were the low activation/drying temperature of the flux, the low temperature (40°C) and high humidity of the test and the requirement for the SIR values to be above 500MOhms even in the 24 first hours of the test. AFE passed the test with no evidence of corrosion. Figure 5 shows the SIR results of AFE (light purple curves stand for reference boards and multi-colored curves stand for AFE).

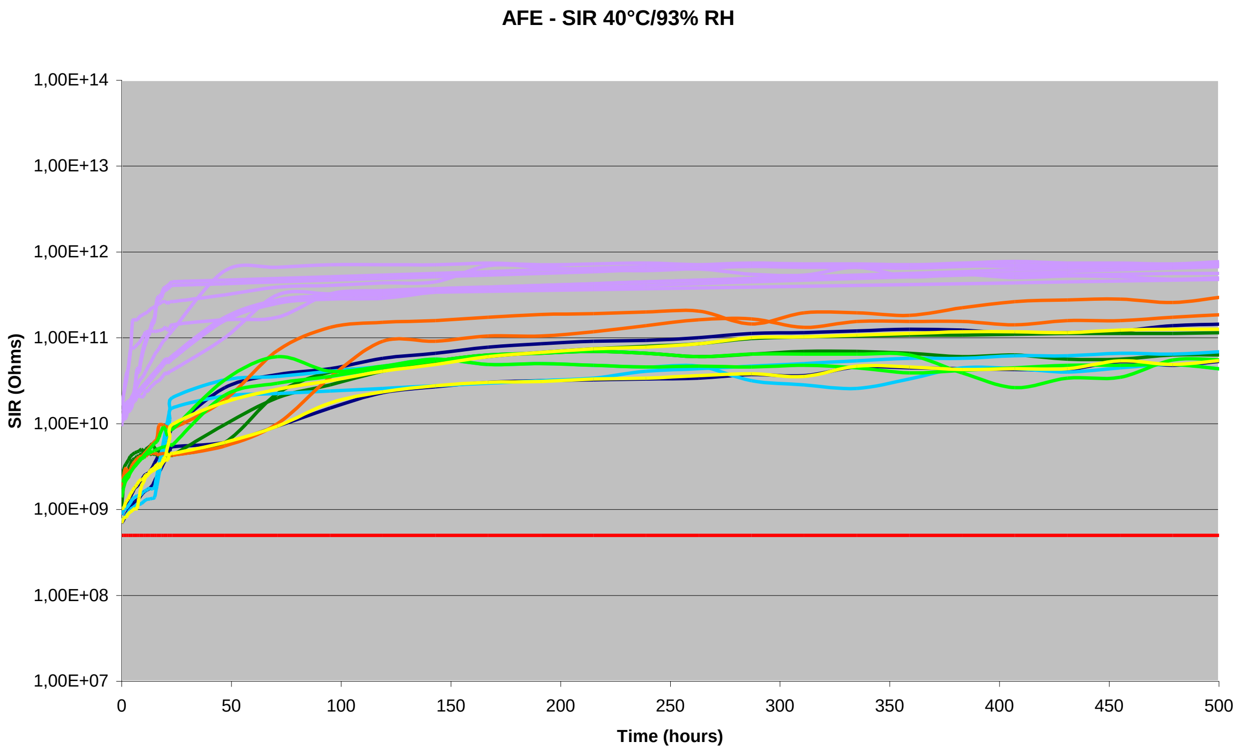


Figure 5. SIR curves of alcohol based flux AFE after low temperature activation.

Results obtained at a laboratory level in terms hole filling and solder balling were confirmed by several industrial tests. Some trials are described below.

VOC-free flux on-site trial 1: comparison of WFA and WFF

The test was conducted on production wave equipment A with 5 preheat IR (infrared) zones with set-ups from 355 to 370°C and quartz at 80%. Soldering took place on one single laminar low-silver wave (265°C) with a pump at 75%. A PCB with a 3mm diameter pins connector was soldered. No change was made in the parameters: the flux was sprayed at 20ml/min.

WFA: no solder balling issues but some insufficient hole filling, a few bridges.
WFF: no solder balling issues, good wetting and hole filling, one sporadic bridge.

VOC-free flux on-site trial 2: comparison of WF30 and WFF

The test was conducted on production wave equipment B with 3 preheat IR zones. Soldering took place on one single doped SnCu turbulent wave (265°C). Boards were manually sprayed with flux and no change was made in the parameters.

WFA: depending on the boards some insufficient hole fillings and a few solder balls
WFG: good hole filling and no solder balls, low and uniform residue, equal to WF30

VOC-free flux on-site trial 3: comparison of WFF and WFJ

WFJ flux was designed to be washed after soldering (ORM1, halide activated flux). Despite its excellent soldering performance, due to its residue quantity, a premature saturation of the cleaning bath used to occur.
The test was conducted on production wave equipment A with 5 preheat IR (infrared) zones with set-ups of 300, 320, 340, 360, 380°C and quartz at 75%. The conveyor speed was 75cm/min. Soldering took place on one single SnPb wave (250°C) with a pump at 65%. The flux was sprayed at 16ml/min (display 1ml). The test was performed on a full through hole power regulator FR4, immersion tin metalized holes, 4 boards per panel.

To achieve the same soldering quality, WFF was sprayed at 55cm/min and the conveyor speed was modified to 18ml/min. Residue were almost invisible. Further testing confirmed the good results and WFF replaced WFJ.

Alcohol-based flux on-site trial 1: AFC, AFE and AFD

The test was conducted with SnPb alloy (245°C). The conveyor speed was set at 93cm/min. Pumps for chip and laminar waves were set-up at 100% and the IR zone at 100°C (old equipment).

AFC was used for years but with a lot of residue accumulation under pallet and several solder balls on some boards.
AFE was manually sprayed with the same parameters: several solder balls were observed. Despite parameters modifications the level of solder balling did not decrease.

AFD was manually sprayed with the same parameters: one big solder ball randomly remained on the board and some residues were observed under the pallet but less than with AFC. After a few modifications, IR was fixed at 110°C and the conveyor speed at 95cm/min. Good wetting was achieved with no solder balling; some residues (less than with AFC) were still present under pallets (Figure 6).

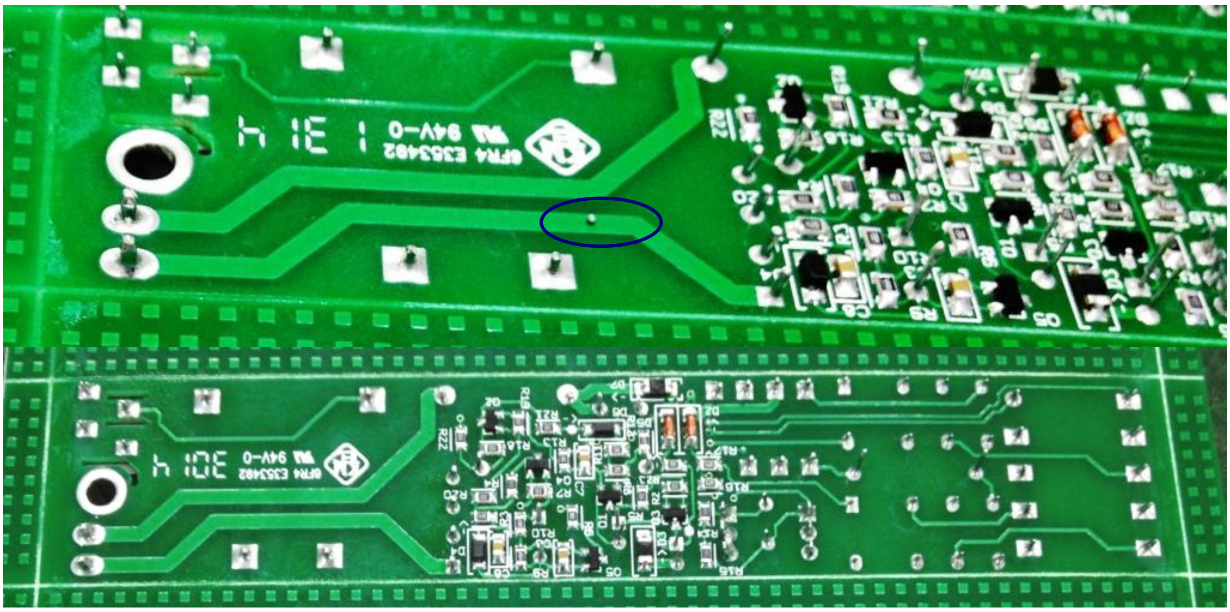


Figure 6. Board after soldering with AFD.

Alcohol-based flux on-site trial 2: AFG and AFB

The test was conducted on production wave soldering machine C with SAC305 alloy (260°C) and AFG with a conveyor speed of 90cm/min. The flux was sprayed with an ultrasonic fluxer at 3PSI. Tests were performed on FR4 single sided boards of 2mm thickness with lead-free HASL finish. Preheat temperature were respectively 300 and 270°C (maximum board temperature after preheat was defined at 95°C and dwell time 2-4 seconds). The goal was to keep the same conveyor speed and preheat parameters for AFB.

AFG: good soldering quality with low micro-ball levels.

AFB: same good soldering quality with low micro-balls levels achieved with less flux consumption (2.5PSI) with other settings changed (Figure 7).

Figure 7. Board after soldering with AFB.

Conclusions

For this study, micro-balls reduction, hole filling improvement and high post-soldering residue reliability were the focussed characteristics. The adhesion of conformal coatings was considered as an additional important characteristic.

Optimized VOC-free and alcohol-based ORL0 low-halogen fluxes were developed.

A no-clean low residue alcohol-based flux, AFD, was fully characterized. It clearly showed improved process performance in terms of soldering efficiency and micro-balls reduction with the same level of chemical reliability compared to the reference. In addition, the high surface tension of AFD residues predicted good coating compatibility in terms of adhesion, which was shown with an acrylic conformal coating. As far as selective soldering is concerned, to answer the special request of SIR/ECM on non-deactivated flux, a customized flux, AFE, was developed. The soldering performance of this flux was more or less equal to the reference fluxes.

A no-clean low residue water-based flux WFF was developed. Its residues were characterized as chemically reliable through standard SIR test and through the Bono test. Compared to the reference, WFF exhibited improved hole filling performance and significant reduction of micro-balls with several types of alloys, including low and no-silver alloys.

The improvement of the no-clean fluxes was achieved through numerous in-house experiments on fluxes combining several raw materials. Activators and especially surfactants were the key ingredients which allow the development of optimized fluxes. The characteristics of the optimized fluxes are presented in Table 10.

Table 10: Characteristics of optimized fluxes.

Characteristics	Flux classification	Density	Solid Content (%)	Acid Index (mg/g)
AFD	ORL0	0.80	4.2	35
AFE	ORL0	0.81	5.0	28.5
WFF	ORL0	1,01	3.5	32

References

[1] G. Vazquez, E. Alvarez, J. M. Navaza, Surface Tension of Alcohol Water + Water from 20 to 50 degree C, J. Chem. Eng. Data, 1995, 40 (3), pp 611–614.

[2] C. Puechagut, A.M. Laügt, E. Guéné, R. Anisko, “Solder Paste Residue Corrosivity Assessment: Bono Test”, Proceedings of IPC Apex, March 2010.

Solderability and Reliability Evolution of No-Clean Solder Fluxes For Selective Soldering

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Purpose of the study

For some high reliability markets, both alcohol-based and water-based fluxes developed years ago do not meet today's requirements

- Development of new-generation alcohol-based flux
- Development of new-generation water-based flux (real VOC-free)
- Low residue, no-clean, ORL0, low halogen, no halogen intentionally added

  - ➢Decrease micro-balling level
  - ➢Improve hole filling
  - ➢Improve post-soldering residue reliability
  - ➢Achieve good conformal coating adhesion

Flux development methodology

- Performance of existing fluxes checked at laboratory level / define improvement criteria
- Screening of raw materials: raw materials / raw materials combinations tested in terms of spreading and wettability (activators and surfactants)
- Ionic conductivity measurements of raw materials and existing fluxes: in case of selective soldering or soldering using pallets, unburned fluxes may generate corrosion in harsh environment
- Identification of raw materials with interesting performances (spreading/wetting/low contamination)
- New flux formulations and optimization of the proportions
- Selection of optimized versions fully characterized at laboratory level: flux spreading, residue spreading, residue cosmetics, wetting, solder balling, copper mirror, standardized SIR/ECM
- Bono tests on flux dried at room temperature and for some fluxes SIR/ECM test on non deactivated flux performed to predict flux reliability
- Industrial tests performed in wave soldering equipments

Laboratory Tests

- Flux spreading
- Post-soldering residue cosmetics
- Flux wetting
- Solder balling
- Surface insulation resistance (SIR) / electro-chemical migration (ECM)
- Bono test
- SIR and ECM on non deactivated flux

Characteristics of initial fluxes

Characteristics	Flux classification	Density	Solid Content (%)	Acid Index (mg/g)
WFA	ORL0	1,01	3.5	32
AFB	ORL0	0.80	2.0	18
AFC	ORL0	0.82	1.9	19

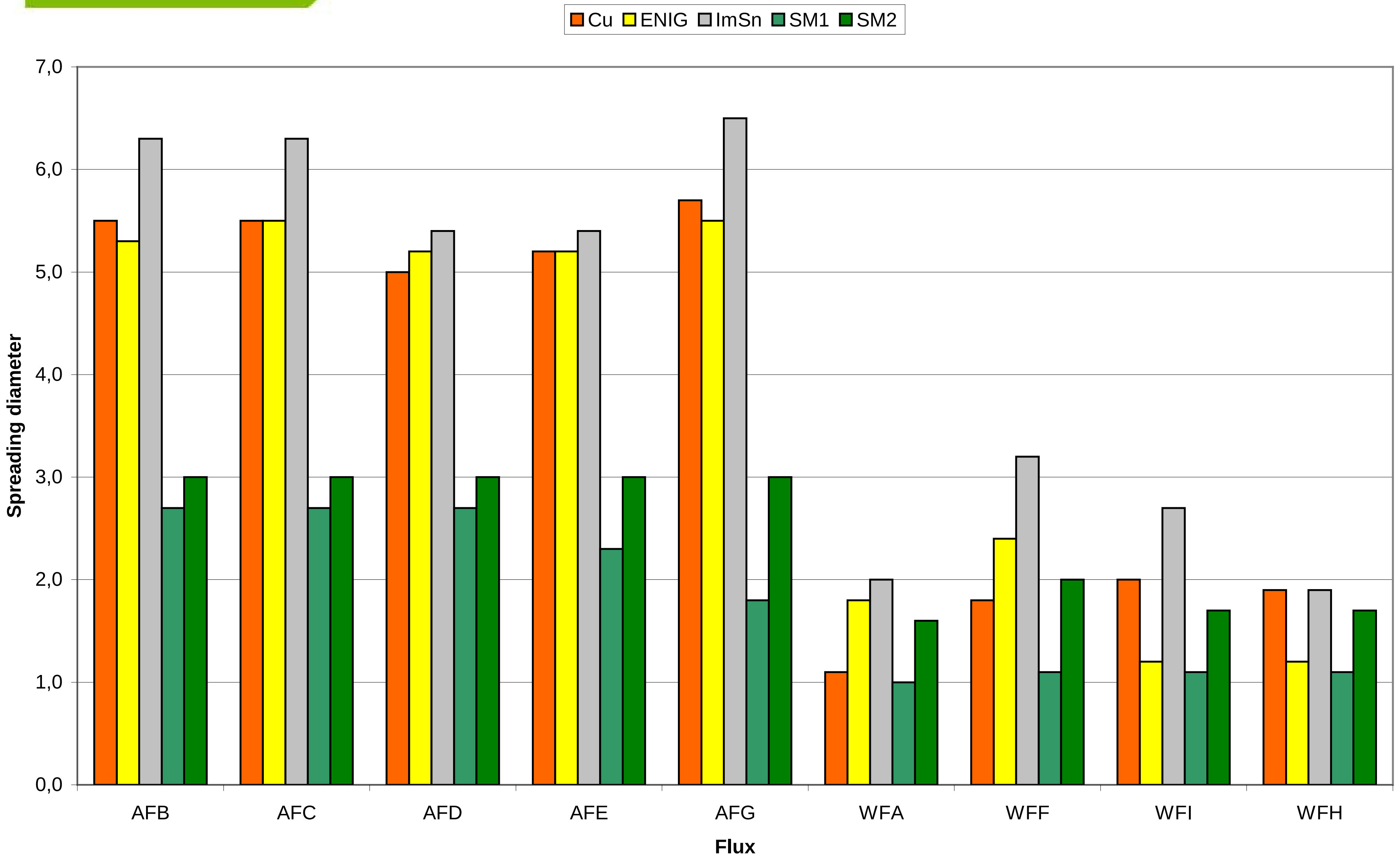
Acid index expressed on the total flux

WFA *water-based flux taken as reference*
AFB and AFC *alcohol-based fluxes taken as reference*

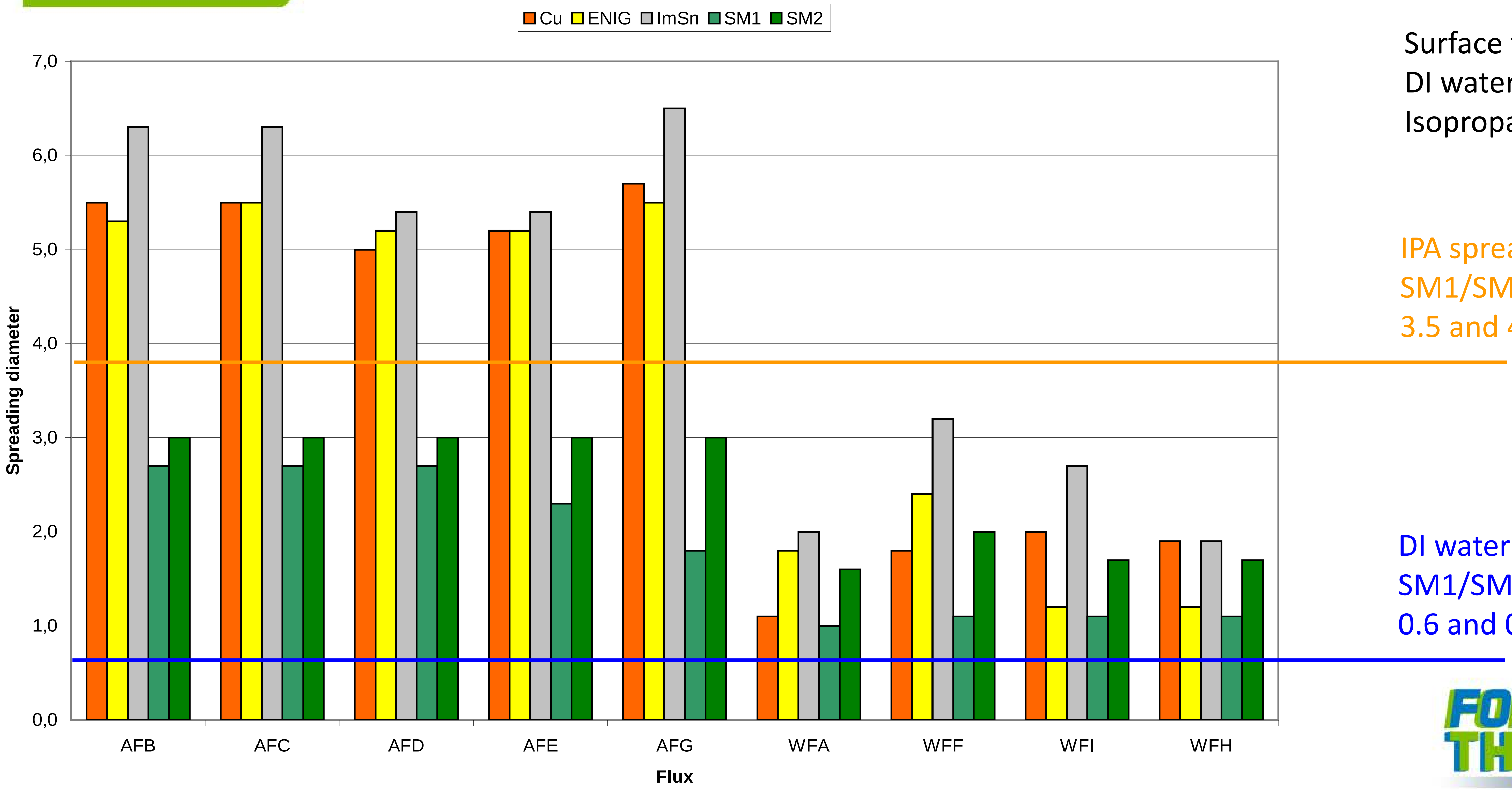
Flux spreading

- 50 µl of flux (or flux solvent) dropped on cleaned and degreased surface
- Diameter after spreading measured
- Bare copper substrate
- Boards with several metal finishes
- Several solder masks with various surface tensions

Flux spreading



Flux spreading

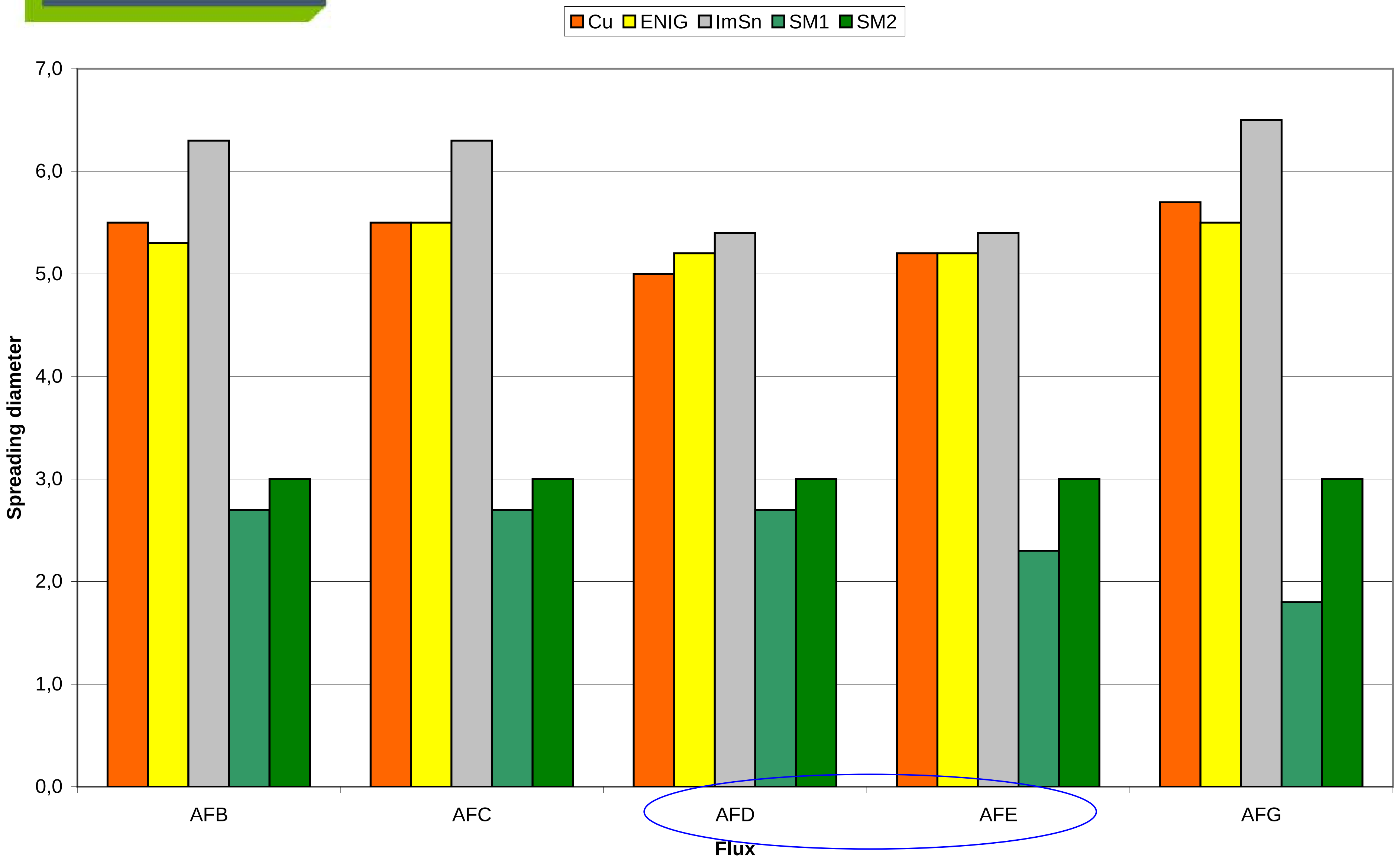


Surface tension
DI water 73mN/m
Isopropanol 22mN/m

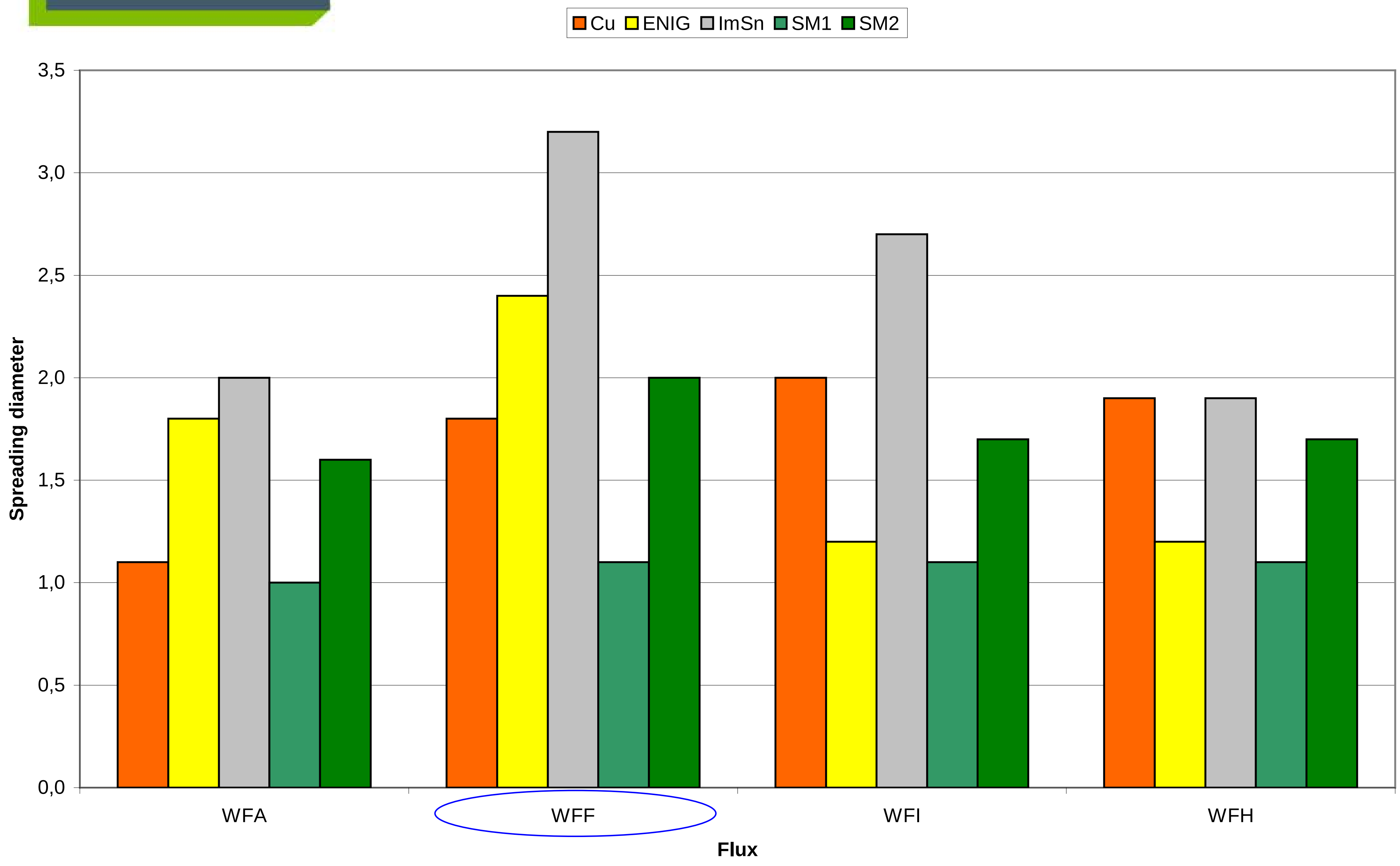
IPA spreading
SM1/SM2
3.5 and 4.0 cm

DI water spreading
SM1/SM2
0.6 and 0.7 cm

Alcohol-based flux spreading



Water-based flux spreading

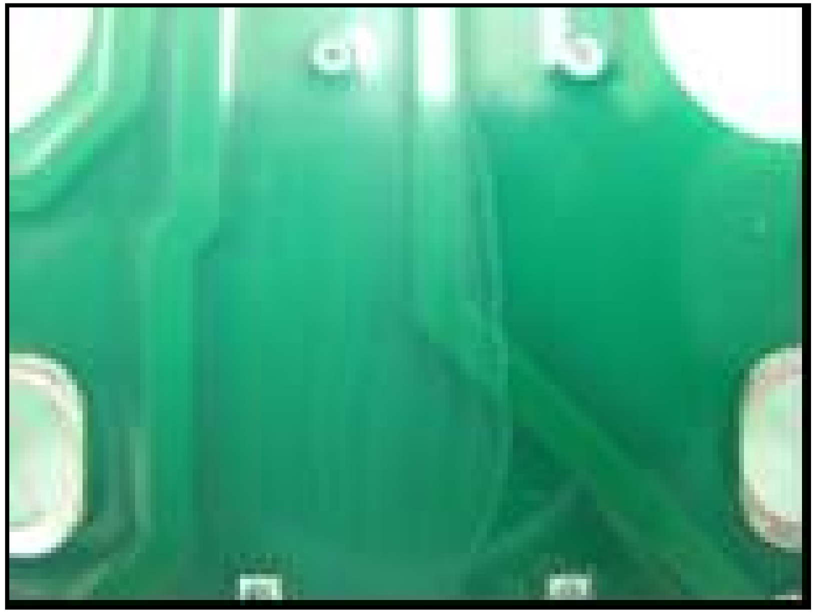


Post-soldering residue cosmetics

- 50 µl of flux dropped on a cleaned and degreased surface (alumina, solder mask, bare copper…)
- Coupons heated
- Several conditions
    - Residues almost non visible when the flux is in contact with wave
    - When flux exposed to lower temperature (selective soldering), residues more or less visible depending on the formula
    - Conditions examples:
        - preheat 1 min 160° C + 1 min 250° C
        - preheat 30sec 100° C + 30sec 140° C + 30sec 160° C

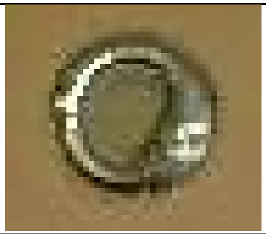
Post-soldering residue cosmetics

- AFB, AFD, AFE, WFA, WFF white residue
- AFC, AFG transparent residue

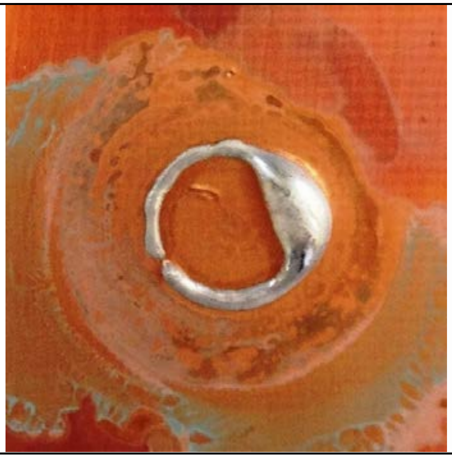
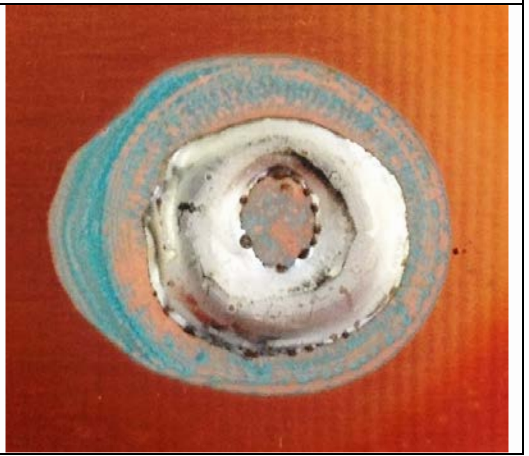


Flux wetting

- SAC305 alloy ring placed on test coupon
- 50µL of flux dropped in the middle of the ring
- Several finishes: cleaned and degreased bare copper, copper OSP, ENIG …
- Preheat and reflow 260° C

Before test	Bad	Medium	Good
			

Flux wetting

Flux	WFA	WFI	WFF
Preheat 100°C 30s			
Preheat 100, 140, 160 3(x30s)			

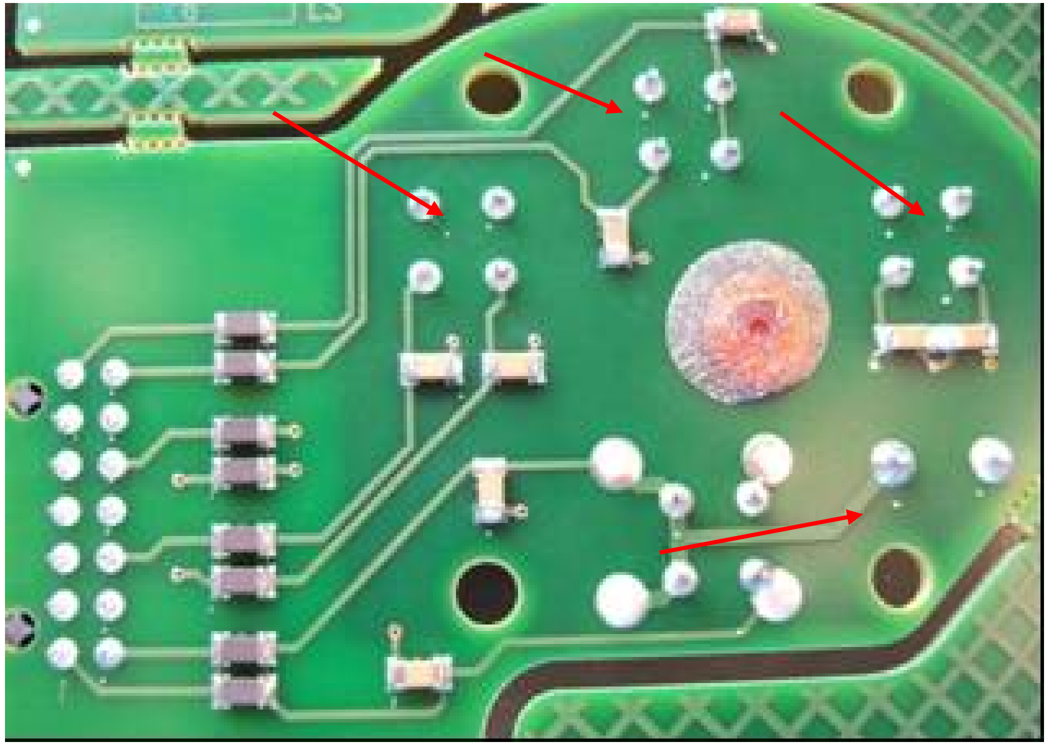
Better wetting of WFF
-compared with reference flux WFA
-compared with flux WFI

No discoloration with reference WFA
Slight green discoloration of copper
With WFF
Strong discoloration with WFI

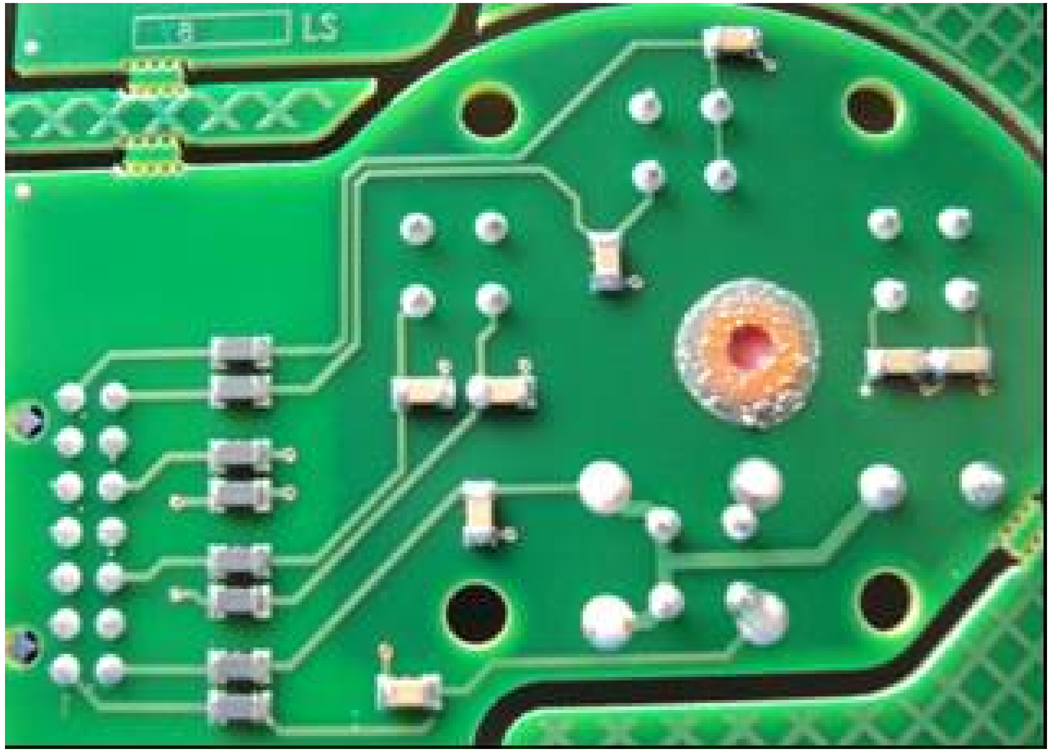
Solder balling

- Dedicated boards with different finishes (OSP, ENIG) dipped in flux, preheated and dipped in SAC305
- Different preheat time and temperature may be used
- Wave temperature 260° C
- Contact time 3 seconds
- Example of preheat conditions: 90 seconds from 20-120° C

Solder balling



AFC (one of the references)



AFD

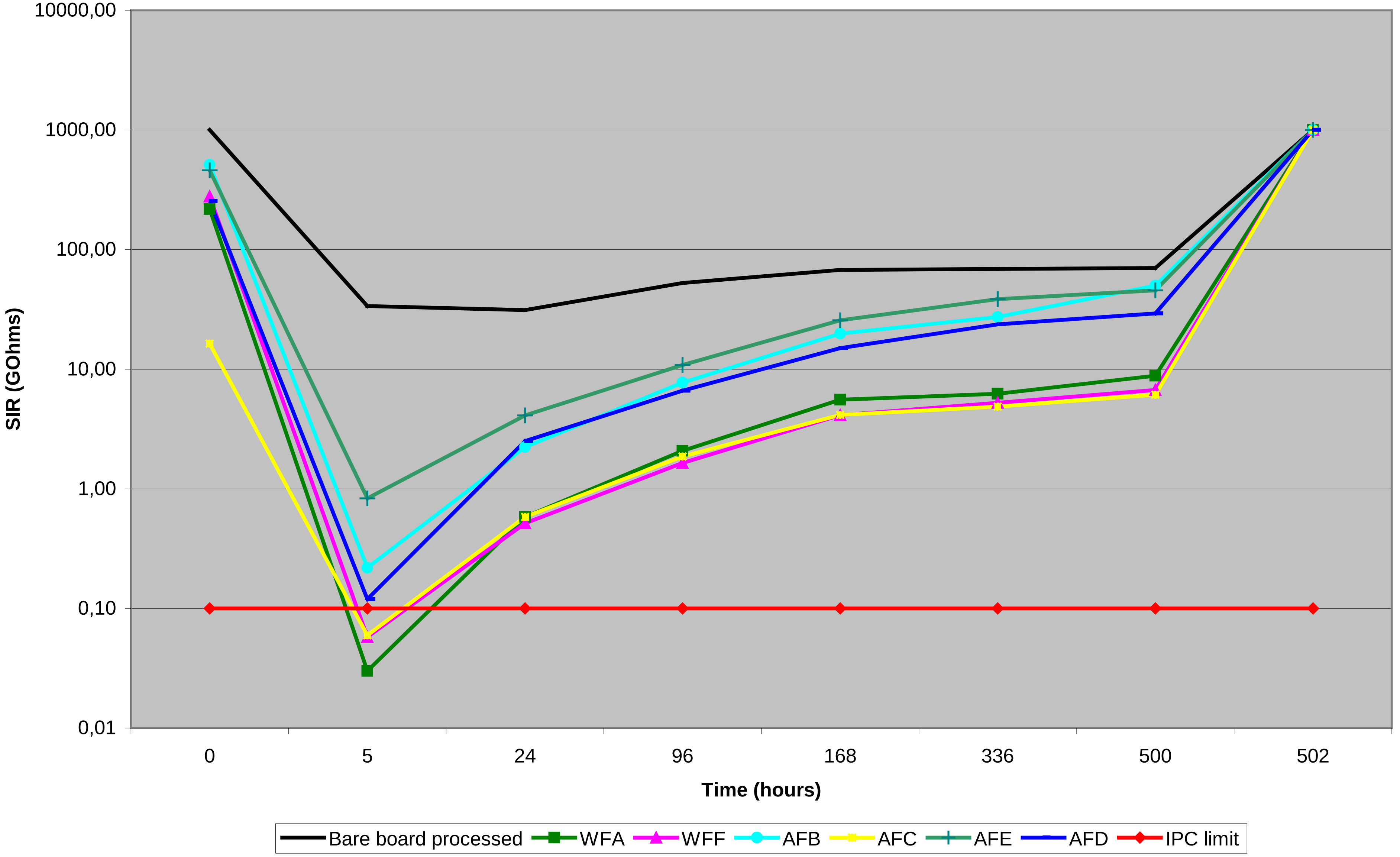
Solder balling

- More solder balls with water-based fluxes than with alcohol-based fluxes
- Significantly less solderballs for WFF compared to WFA
- Alcohol-based from best to worst AFD> AFB> AFE> AFG> AFC

Surface insulation resistance (SIR) Electro-chemical migration (ECM)

- J-STD-004B, Test method 2.6.14.1
- IPC-B-25
  - Sprayed with flux 60g/cm^2
  - Convection reflow 130°C to mimic preheat conditions (no soldering)
- Bias Voltage 10V
- 65°C / 85% RH
- 500 hours

SIR / ECM



- No dendrite growth
- No corrosion
- No SIR degradation of more than one decade between initial (96hours) and final measurements (500h)
- All fluxes passed

Bono Test

- Bono test board / FR4 epoxy single layer copper with 10 electrolytic cells
- Copper thickness 9µm
- Anode width 80µm / cathode width 3mm
- Cathode spacing 120 µm
- Bono board dipped in flux
- Bono board air dried at room temperature
- 85° C / 85% RH
- 336 hours (15 days)
- Bias 20VDC

Bono Test

- R-anode measured daily
- Fc Calculation:
  - Rd resistance value at day « d »
  - R0 resistance value at day « 0 »
  - Fc given in %

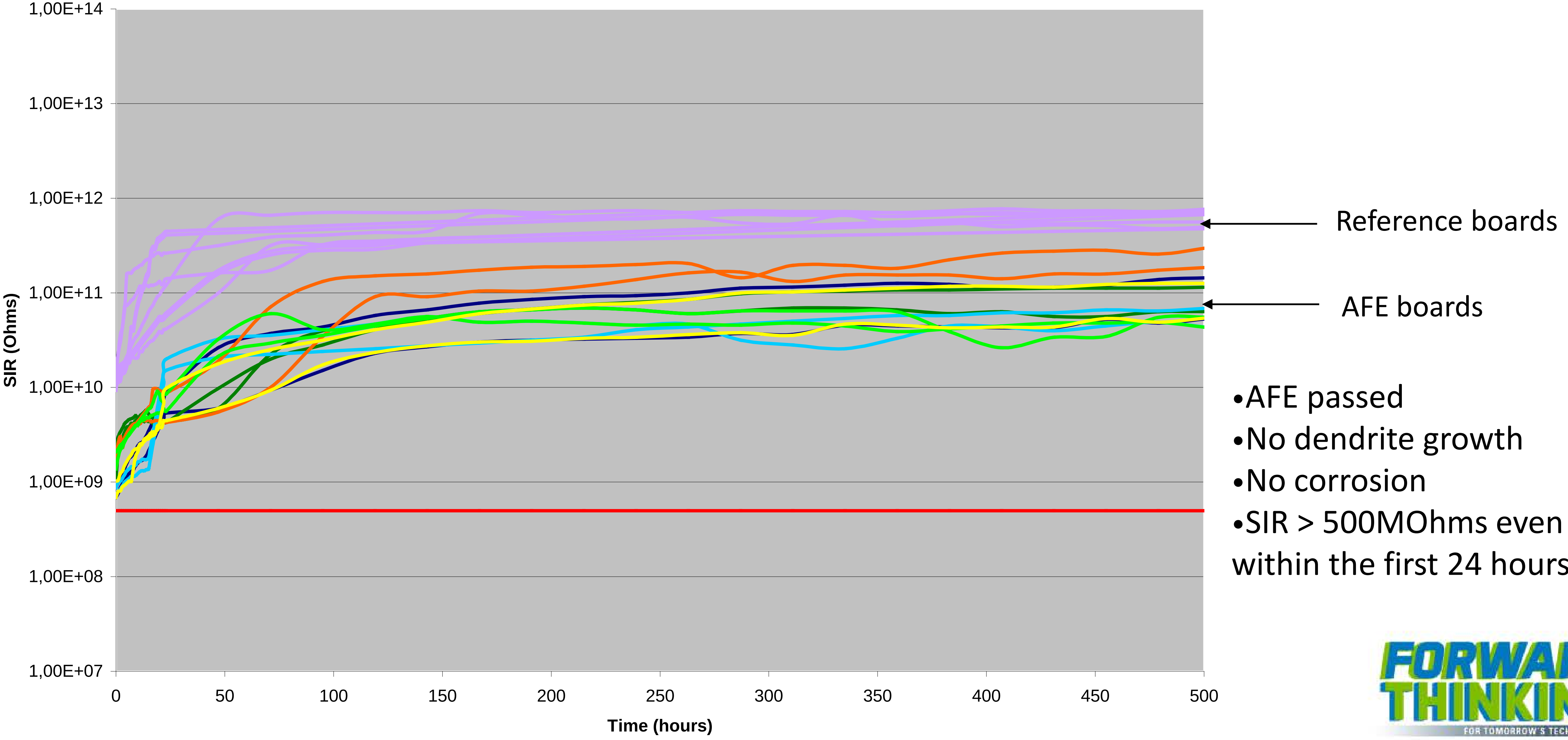
$$Fc = \frac{R_d - R_0}{R_0} \times 100$$

  - WFA, WFF passed
  - AFB, AFD, AFE passed
  - Fc 1%
  - No corrosion

SIR / ECM on non deactivated flux

- Flux applied on IPC-B-24 (calibrated quantity)
- 0.2ml flux per comb
- Flux dried at 40° C during 30 min
- Test according to DIN EN ISO 9455-17
- IPC-B-24 placed in a climate chamber at 40° C/93%RH
- SIR measured after 1h, 2h, 3h, 4h, 8h, 12h and 24h
- After 24h constant voltage of 100V applied
- SIR measurements at 48h, 168h, 336h and 504h
- After test, coupons visually inspected for migration and corrosion

SIR / ECM on non deactivated flux: AFE



- AFE passed
- No dendrite growth
- No corrosion
- SIR > 500MOhms even within the first 24 hours

Alcohol-based residue surface tension and coating adhesion

Board and flux	Unprocessed board (reference)	Board with AFG	Board with AFB	Board with AFC	Board with AFD
Residue surface energy (dynes/cm)	> or = 44	32	38	32	38
Adhesion test after 16h*	delamination 0%	delamination 30%	delamination 5%	delamination 30%	delamination 5%

* Tests with acrylic coating A / drying time 30 min air + 2 hours 60° C

According to IPC-HDBK-830
Above 40 dynes/cm: adhesion expected to be good
35-40 dynes/cm: adhesion generally good, with some intermittent delamination under severe conditions

Characteristics of final fluxes

	Flux classification	Density	Solid Content (%)	Acid Index (mg/g)
AFB	ORL0	0.80	2.0	18
AFC	ORL0	0.82	1.9	19
AFD	ORL0	0.80	4.2	35
AFE	ORL0	0.81	5.0	28.5

Performance of final fluxes

	Wetting	Solder balling	Residue cosmetics	Residue surface tension	Bono	Harsh SIR
AFB	2nd	2nd	White	Good	Yes	No
AFC	4th	3rd	Transparent	Poor	No	No
AFD	1st	1st	White	Good	Yes	No
AFE	2nd	3rd	White	Good	Yes	Yes

Conclusions alcohol-based flux

- A no-clean low residue alcohol-based flux AFD was developed
- It clearly showed better soldering efficiency and micro-balls reduction with the same level of chemical reliability compared to the reference
- The high surface tension of AFD residues predicted good coating compatibility in terms of adhesion, shown with an acrylic conformal coating
- The good soldering performance was assessed by industrial tests
- This performance was mainly achieved through the right choice and the right quantity of activators

Conclusions alcohol-based flux

- A customized no-clean low residue alcohol-based flux AFE was developed to answer the special request of SIR/ECM on non-deactivated flux for selective soldering

- The soldering performance of this flux was more or less equal to the reference fluxes despite its higher solid content and acid index

Characteristics of final VOC-free flux

Characteristics	Flux classification	Density	Solid Content (%)	Acid Index (mg/g)
WFA	ORL0	1,01	3.5	32
WFF	ORL0	1,01	3.5	32

Performance of final VOC-free flux

	Wetting	Solder balling	Residue cosmetics	Bono	Harsh SIR
WFA	2nd	2nd	White	Yes	No
WFF	1st	1st	White	Yes	No

Conclusions VOC-free flux

- A no-clean low residue water-based flux WFF was developed
- Its residue was characterized as chemically reliable through standard SIR test and through the Bono test
- Compared to the reference, WFF exhibited improved hole filling performance and significant reduction of micro-balls with several types of alloys, including low and no-silver alloys (industrial tests)
- Solid content and acid index remained similar to the initial flux

General conclusion

The improvement of the no-clean fluxes was achieved through numerous in-house experiments on fluxes combining several raw materials

Activators and especially surfactants were the key ingredients which allow the development of optimized fluxes

Questions and comments?

With special thanks to Richard Anisko and Celine Puechagut at the company

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